ORNL-2971

Vol. 6

CHEMISTRY—SEPARATION PROCESSES
FOR PLUTONIUM AND URANIUM

MARTIN MARIETTA ENERGY SYSTEMS LIBRARIES
3 4456 0361534 5

2

CENTRAL RESEARCH LIBRARY
DOCUMENT COLLECTION

CENTRAL RESEARCH LIBRARY
DOCUMENT COLLECTION
LIBRARY LOAN COPY
DO NOT TRANSFER TO ANOTHER PERSON
If you wish someone else to see this document, send in name with document and the library will arrange a loan.

BIBLIOGRAPHY ON NUCLEAR REACTOR FUEL REPROCESSING AND WASTE DISPOSAL

Volume 6

Process Chemistry and Engineering

T. F. Connolly

Oak Ridge National Laboratory
operated by
Union Carbide Corporation

United States Atomic Energy Commission
Division of Technical Information

LEGAL NOTICE

The AEC reports listed in this document are accounts of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in these reports, or that the use of any information, apparatus, method, or process disclosed in these reports may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in these reports.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, to the extent that such employee or contractor of the Commission, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.

This report has been reproduced directly from the best available copy.

Printed in USA. Price $3.50. Available from the Office of Technical Services, Department of Commerce, Washington 25, D. C.

USAEC Division of Technical Information Extension, Oak Ridge, Tennessee

ORNL-2971

Vol. 6

CHEMISTRY—SEPARATION PROCESSES
FOR PLUTONIUM AND URANIUM

BIBLIOGRAPHY ON NUCLEAR REACTOR FUEL REPROCESSING AND WASTE DISPOSAL

Volume 6

Process Chemistry and Engineering

T. F. Connolly

Oak Ridge National Laboratory
operated by
Union Carbide Corporation

United States Atomic Energy Commission
Division of Technical Information

MARTIN MARIETTA ENERGY SYSTEMS LIBRARIES

3 4456 0361534 5

PROCESSING OF NUCLEAR REACTOR FUELS – A BIBLIOGRAPHY

Introduction

This volume includes Section 6.0 of eight sections of a bibliography on nuclear reactor fuel reprocessing and waste disposal. The collection will be a unit, and cross references are made between volumes. The decimal coding of headings is designed for ease of cross referencing, filing, expansion, and for future use in an automatic microfilm viewer-printer now being developed.

Cutoff points are somewhat arbitrary: overlapping into the area of feed materials, for example, is kept to a minimum, even though fuel reprocessing and fuel refabrication can be a continuous operation. Continuous on-site reprocessing of aqueous reactor fuels is only lightly covered.

Section 1.0, Chemistry and Physics of Important Elements, contains many references to separation methods and conditions, particularly on a laboratory scale. Cross references to that section from Sections 2.0, 3.0, and 6.0 – to each of which it may be considered as peripheral – would be too numerous to be included and should be assumed for each element. Special tests or uses (e.g., as construction materials or reagents in standard processes) are treated in the appropriate sections.

The complete collection includes about 7000 abstracts, nearly all from Nuclear Science Abstracts, representing books, bibliographies, symposia, journals, and contractors' topical reports. Most of the material dates from the 1955 Geneva Conference to the present. A serious lack is material from progress reports, and a program for the detailed abstracting and indexing of progress reports is therefore under way at ORNL (with the helpful cooperation of the Cataloging Section of the AEC-DTI Extension).

TABLE OF CONTENTS

PROCESS CHEMISTRY AND ENGINEERING

	Page
ADSORPTION	6.01
CHELATION	6.02
CHLORINATION	6.04
CHROMATOGRAPHY	6.05
COMPLEX FORMATION	6.08
AMINES	6.09
GENERAL – INORGANIC	6.09
GENERAL – ORGANIC	6.10
SURVEYS	6.14
DISTILLATION AND CONDENSATION	6.15
ELECTROCHEMISTRY	6.16
FLUIDIZED BEDS	6.20
FLUORINATION	6.23
FUSED SALT AND LIQUID METAL SYSTEMS	6.25
FUSED SALTS	6.26
Chlorides	6.26
Electrochemistry	6.32
Equipment	6.36
Fluorides	6.37
Mixed Halides	6.46
Others	6.47
Surveys and Theory	6.49
LIQUID METALS	6.53
General	6.53
Equipment	6.57
Properties	6.57
Theory	6.59
HEAT TRANSFER	6.60
ION EXCHANGE	6.64
EQUIPMENT	6.64
EXCHANGERS	6.66
Attrition and Recovery	6.67
Non-Resins	6.69
Resins – Dowex	6.73
Resins – General	6.76
Resins – Phosphate and Phosphonate	6.78
Resins – Theory	6.79
PROCESSES AND METHODS	6.80
Countercurrent Methods	6.80
Elution	6.81
General	6.84
THEORY	6.87
ION TRANSFER (MEMBRANES)	6.90
PRECIPITATION	6.91
RADIATION EFFECTS	6.93
AQUEOUS SOLUTIONS	6.93
Alpha	6.93
Gamma and X	6.94
Beta (Electrons)	6.97
Theory	6.97
ON ELECTRONIC COMPONENTS	6.100
General	6.100
Semiconductors	6.103
GASES	6.105
GLASS	6.107
GRAPHITE	6.109
LUBRICANTS	6.110
METALS	6.110
Neutrons	6.110
General	6.112
Gamma	6.115
ORGANIC FLUIDS	6.116
Alcohols	6.116
Benzenes	6.117
General	6.119
Halides	6.123
Other	6.125
OTHER SOLIDS	6.128
General	6.128
Halides	6.130
Nitrates	6.130
Oxides	6.130
Others	6.131
PLASTICS AND HIGH POLYMERS	6.133

TABLE OF CONTENTS

PROCESS CHEMISTRY AND ENGINEERING

	Page
Adhesives	6.133
General	6.133
Polyethylene	6.137
Rubber	6.140
Others	6.140
SURVEYS AND THEORY	6.143
Bibliographies	6.143
Surveys	6.145
Theory	6.146
SOLVENT EXTRACTION	6.151
COSTS	6.151
EQUIPMENT	6.152
Contactors (General)	6.152
Countercurrent Extractors	6.154
Maintenance	6.155
Mixer-Settlers	6.156
Pulsed Colums	6.158
Pulsed-Column Auxiliaries	6.161
RADIATION DAMAGE TO SOLVENTS	6.163
Diluents	6.163
Ethers	6.164
TBP	6.164
Other Solvents	6.166
REAGENT ADJUSTMENT AND RECOVERY	6.168
Chlorides	6.168
Entrainment	6.168
General	6.170
HNO_3	6.170
Solvent Evaluation	6.173
TBP	6.175
SOLVENTS	6.176
Amines	6.176
Dibutyl Carbitol	6.180
Ethers	6.182
Eetones	6.183
Organophosphorous	6.185
Others	6.187
TBP – Diluents	6.189
TBP – General	6.190
TBP – Interferences	6.192
TBP – Nitrate Systems	6.193
TBP – Temperature Effects	6.195
SURVEYS AND THEORY	6.196
Flooding and Flowrate	6.196
Processes	6.200
Stage Efficiency	6.203
Interface (and Control)	6.208
ULTRASONICS, USE OF	6.210
(SPECIAL) PROCESSES	6.213
AMERICIUM RECOVERY	6.213
BISMUTH PHOSPHATE	6.213
BUTEX	6.215
DAREX	6.216
DEBOER	6.221
DIBAN ION EXCHANGE	6.221
DRUHM	6.222
EXCER	6.222
FLUOROX	6.225
FLUREX	6.228
FUSED SALT – FLUORIDE VOLATILITY	6.228
General – Flowsheets	6.229
Design	6.231
Corrosion and Maintenance	6.229
HF Recovery	6.232
Construction Materials	6.232
HCL ALCOHOL	6.233
HERMEX	6.233
METALLEX	6.235
METAL RECOVERY	6.237
NEPTEX	6.238
PUREX	6.238
General – Flowsheets	6.238

TABLE OF CONTENTS

PROCESS CHEMISTRY AND ENGINEERING

	Page
Construction Materials	6.241
Corrosion – Process	6.241
Ion Exchange	6.242
Process Control	6.242
Process Data	6.243
Reagent Adjustment and Recovery	6.245
Solvent Extraction	6.246
Waste	6.247
RALA	6.250
REDOX	6.252
General – Flowsheets	6.252
Corrosion – Process	6.253
Hazards	6.254
Process Data and Control	6.255
Solvent Extraction	6.255
Waste	6.256
SULFEX	6.257
25-TBP	6.258
General – Flowsheets	6.259
Head-End and Feed	6.260
Solvent Extraction	6.262
Waste	6.262
THOREX	6.263
General – Flowsheets	6.263
Head-End and Feed	6.264
Solvent Extraction	6.265
Ion Exchange – U^{233}	6.268
Process Data and Control	6.269
Decontamination	6.270
ThO Production	6.270
Corrosion	6.270
UAP PROCESS	6.271
ZIRCEX	6.272
ZIRFLEX	6.275
SINTERING	6.276
THERMAL DIFFUSION	6.279
ULTRASONICS	6.280
ZONE MELTING	6.284
PELLETIZING	6.285

PROCESS CHEMISTRY AND ENGINEERING

ADSORPTION 6.01

See also, under FISSION PRODUCT, POISON, AND RADIOISOTOPE REMOVAL,

ADSORPTION (2.01),

and,

under FISSIONABLE MATERIALS--RECOVERY,

RECOVERY OF IRRADIATED TH AND U--Adsorption (3.02.01),

RECOVERY OF PLUTONIUM--Adsorption (3.03.01).

716 PA-TR-2566

Picatinny Arsenal. Feltman Research and Engineering Labs., Dover, N. J.

SURFACE PHENOMENA IN SOLIDS. A Literature Review. Cecil G. C. White and Samuel Helf. May 1959. 34p. DA Project 599-04-001. (AD-208231).

The results of a literature survey on some of the fundamental principles and experimental practices involved in the study of adsorption phenomena on solids are described. One section consists of an elementary review of the theories of adsorption on solids and another is a description and discussion of the classical methods for the determination of surface area of solids. A third section is devoted to a special bibliography on previous applications of radioactive isotopes to the study of surface phenomena. (auth)

13729

THE STATE OF MICRO-QUANTITIES OF RADIOACTIVE ELEMENTS IN BOTH LIQUID AND SOLID PHASE. I. E. Starik. Zhur. Neorg. Khim. 3, 6-15(1958). (In Russian)

The experimental difficulties in the determination of the form (colloid or ion) in which microquantities of radioelements are present in solutions are discussed. An investigation was made of the adsorption and desorption of Po, using glass. The deposition of Po on copper was also studied at pH values from 1 to 13. Data on the solubility of the hydroxides of Po, Ru, Tl, Zr, Pm, and La were compared with data from the literature. The adsorptive and colloidal properties of the radioelements were discussed as a function of pH. (J.S.R.)

National Bureau of Standards, Washington, D. C.

BIBLIOGRAPHY OF SOLID ADSORBENTS—1943 TO 1953. Victor R. Deitz, comp. Mar. 1, 1956. 1528p. (NBS-C-566).

Adsorption of gases and vapors on solid adsorbents, adsorption from solutions on solid adsorbents, thermal effects in adsorption processes, theories of adsorption, refining of sugars and other applications of adsorbents, general information on adsorbents and special methods of investigation, and preparation of carbon adsorbents are the main subjects included in the bibliography. A total of 13,763 annotated references is listed. Author and subject indexes are included.

7151

IDEALIZED MODELS FOR ADSORPTION FROM SOLUTION. I. VAN DER WAALS ADSORPTION FROM REGULAR SOLUTIONS. Robert S. Hansen and Ursula H. Mai (Ames Lab., Iowa). J. Phys. Chem. 61, 573-7(1957) May.

The theory of ideal van der Waals adsorption from regular solutions of equal sized molecules is developed, and explicit limiting forms for high and low concentrations of preferentially adsorbed components and for slightly soluble systems are given. (auth)

19893

THE INFLUENCE OF A NON-AQUEOUS SOLVENT ON THE ADSORPTION OF RADIOACTIVE MICROCOMPONENTS BY A NUMBER OF ADSORBANTS. S. A. Voznesenskii, V. D. Puzako, and L. B. Levasheva. Nauch. Doklady Vysshei Shkoly Khim. i Khim. Tekhnol. No. 3, 452-4(1958).

The influence of ethyl alcohol and acetone on the adsorption of microamounts of Cs^{137} and Sr^{89} by ultramarine, iron oxide, barium sulfate, and titanium dioxide in water at room temperature was investigated. It was found that the addition of non-aqueous solvents usually increases the adsorption of the microcomponents, sometimes more than 10-fold. Only in the adsorption of cesium on barium sulfate was the adsorption decreased by the addition of ethyl alcohol or acetone to the aqueous solution. Experimental condition variations are described. (auth)

ADSORPTION 6.01

6485 A/CONF.15/P/2096
KINETICS OF ADSORPTION OF A MIXTURE OF RADIOISOTOPES ON SURFACES. Z. Dlouhý and J. Malý (Czechoslovakia). 20p.

The theoretical relations are given for the kinetics of adsorption of a radioisotope mixture from solution on a solid surface. The time dependence of the amount of isotope A adsorbed can be described by a function containing two exponentials in the form $A = K_0 - e^{-K_1 t} - e^{-K_2 t}$. The adsorption of Zn^{65}, Po^{210}, and Ca^{45} on stainless steel was studied. (W.D.M.)

CHELATION 6.02

13708 ✓ KLX-44
Kellex Corp., New York.
THE FEASIBILITY OF CONTINUOUS CHELATION. Job 11. Apr. 29, 1949. Decl. Mar. 28, 1957. 51p. Contract AT-30-1-Gen-169. $9.30(ph OTS); $3.60(mf OTS).

A survey of the feasibility of the use of chelation for continuous processing of (a) dissolver solution, (b) solutions resulting from Redox Process wastes, and (c) solutions from the treatment of wastes from the present process for plutonium recovery and decontamination, super clean-up of uranium, zirconium recovery and packaging, uranium recovery and decontamination, and fission product recovery has been made. New experimental work on batch and continuous extraction in a horizontal countercurrent flow extractor is reported and previous work is reviewed. The conclusion is drawn that continuous chelation processes are probably feasible for all of the above. Possible schemes for incorporation of chelation into process design

Crystallography and Crystal Structure

10421 ✓ UCRL-8225
California. Univ., Berkeley. Radiation Lab.
THE CRYSTAL STRUCTURE OF VANADYL BIS-ACETYLACETONATE (thesis). Richard Patrick Dodge. Mar. 31, 1958. 27p. Contract W-7405-eng-48. $0.75(OTS).

The crystal structure of the chelate vanadyl bis-acetylacetonate has been determined by x-ray diffraction. The compound is not a polymer in the solid but forms separate molecules, and there is no intermolecular bonding other than the usual Van der Waals type.

CHELATION 6.02

Ames Lab., Ames, Iowa.
SUBSTITUENT EFFECTS OF THE STABILITY OF METAL CHELATES. Wilfred G. Borduin and G. S. Hammond. Dec. 1957. 28p. Contract W-7405-eng-82. $1.00(OTS).

The stability constants of the chelate compounds of a series of substituted diaroylmethanes with several divalent metals in 75 per cent by volume dioxan-water were measured. The data were treated with the Hammett relationship, $\log k/k_0 = \rho\sigma$. The expected correlation of rho values with metal-chelate bond type was not observed. This lack of correlation is attributed to the free energy of solution of the metal chelate.

THE STRUCTURE AND CHARACTERISTICS OF THE FLUORESCENT METAL CHELATES OF o,o'-DIHYDROXY-AZO COMPOUNDS. Donald C. Freeman, Jr. and Charles E. White (Univ. of Maryland, College Park). J. Am. Chem. Soc. 78, 2678-82(1956) June 20.

A series of compounds has been synthesized and investigated in order to determine the characteristics of structure and bond type necessary for the formation of fluorescent chelates. The parent type of structure for the compounds in this series is that of o,o'-dihydroxyazobenzene. Some modifications of this basic configuration such as the bis-salicylidenediamines, salicylidene-o-aminophenols, and salicylaldehyde azines were also studied. Both non-aqueous N,N-dimethylformamide and 34% N,N-dimethylformamide in water were found to be excellent solvents for fluorescent chelation reactions. The bonds between the metal ion and the chelating agent were found to be primarily ionic in character. Transition elements do not form fluorescent chelates with this type of compound. A strong dependence of fluorescence intensity on the size of the metal ion was observed.

6527 ✓ NP-5901
Clark Univ., Worcester, Mass.
STUDY OF EQUILIBRIA BETWEEN METAL IONS AND AMINO ACID COMPLEXING AGENTS IN AQUEOUS SOLUTION. Final Report. Arthur E. Martell. Dec. 1955. 24p. Project No. NR-052-286. Contract NONR-596(00).

The thermodynamic changes associated with the formation of alkaline-earth, rare-earth, and Mn chelates of ethylenediaminetetraacetic acid (EDTA) and related ligands are tabulated. Relative thermodynamic stability constants were also calculated for EDTA chelates of transition metals and rare earths. Chelate structure and theory of chelate formation are discussed. (C.W.H.)

CHELATION 6.02

12338 ✓

THE NATURE OF URANYL 8-QUINOLINOLATE.
E. P. Bullwinkel and Paul Noble, Jr. (Arthur D. Little, Inc., San Francisco). J. Am. Chem. Soc. 80, 2955-8 (1958) June 20.

It is shown that the three moles of 8-quinolinol in the uranyl 8-quinolinolate are coördinated in an equivalent manner and that the acidic nature of this chelate is best represented by the formula $H(UO_2(C_9H_6NO)_3)$, where the anions, $UO_2(C_9H_6NO)_3^{-1}$, are linked together by hydrogen bonding of the uranyl oxygens. The uranyl tri-8-quinolinolate anion is shown to be stable in alkaline solutions and to form stable salts with cations, such as Na^+, R_4N^+, and $(C_6H_5)_4As^+$. The absorption spectra of aqueous alkaline solutions of the uranyl tri-8-quinolinolate anion and chloroform solutions of the tetraphenylarsonium salt of this anion are reported. The chemical behavior of the uranyl di-8-quinolinolate is reported and discussed with respect to its possible structure. (auth)

1119 ✓ NYO-2370
Pittsburgh. Univ.
A THERMODYNAMIC STUDY OF SOME FACTORS AFFECTING THE STABILITY AND SOLUBILITY OF METAL CHELATES. (thesis). David Fleischer. [1958] 153p. Contract AT(30-1)-860. $24.30(ph OTS); $7.50 (mf OTS).

Studies are presented on the formation and solubility of metal chelates. The reactions of 8-quinolinol and its homologs, 2-methyl- and 4-methyl-8-quinolinol, with bivalent ions of Cu, Ni, and cobalt were examined. Attention is focused on methods for the direct determination of heats of reaction. For the solubility work the compounds copper(II) and nickel(II) dimethylglyoxime were examined. Attention is directed towards separating the effects of crystal energy and solvation energy on the solubility. (W.L.H.)

6436 ISC-673
Ames Lab., Ames, Iowa.
STERIC EFFECTS ON THE FORMATION CONSTANT OF METAL CHELATES OF BETA-DIKETONES. G. A. Guter and G. S. Hammond. Aug. 1955. 62p. Contract W-7405-eng-82. $1.75(OTS).

Evidence is presented which indicates that chelating ability may be greatly altered by changing the steric requirements of the ligand. A series of formation constants was measured for three beta-diketones. Thermodynamic values could not be obtained but relative concentration constants were. The constants obtained were correlated with each other and with the basicity of the reagent toward a proton. Each correlation indicated that the formation constants for the copper chelates of diisobutyrylmethane and dipivaloylmethane were low and exhibited large separation factors. However, relatively small separation factors were found for metal chelates which do not form square planar complexes. A simple method for determining the extent of enolization of beta-diketones and the kinetics of enolization is presented. The method makes uses of spectrophotometric measurements in the ultraviolet region. Correlations were observed between the pKa of the chelating agent, the kinetics of enolization, and the kinetics of hydrolysis of corresponding esters. Variations in the pKa and enol content were explained on the basis of steric effects. A procedure for the separation of lithium from other alkali metal ions was developed, making use of the selectivity of dipivaloylmethane. The theoretical implications of this selectivity are discussed. Correlation of formation constants with a double basicity scale is reveiwed. It is suggested that when using such correlations a chelate effect be taken into account. The application of the Edward and Hammett equation to formation constants is discussed. (auth)

9667 ✓ TID-10022
Argonne National Lab., Lemont, Ill.
THE CHELATE PROCESS. II. H. W. Crandall, J. R. Thomas, and E. Zebroski. Aug. 17, 1947. Decl. Mar. 12, 1957. 19p. $0.25(OTS).

The Chelate Process for the extraction and decontamination of plutonium has been tested on a semi-works scale. Tests using C_6H_6 and $C_6H_5CH_3$ as the organic solvent gave an average of 98.7% Pu recovery for a three and a half cycle process. The average beta decontamination factors using C_6H_6 and $C_6H_5CH_3$ were $>10^7$ and 10^6 respectively. The average soft gamma decontamination factors using benzene and toluene were 10^6 and 10^4 respectively. The details of the semi-works experiments are given and the results of those experiments are critically evaluated. (T.R.H.)

SPECTROPHOTOMETRIC AND ELECTRICAL CONDUCTANCE STUDIES ON CHELATE FORMATION OF URANYL ION WITH AMMONIUM AURINTRICARBOXYLATE. Anil. K. Mukherji and Arun K. Dey (Univ. of Allahabad, India). J. Inorg. & Nuclear Chem. 6, 314-18 (1958) July.

CHELATION 6.02

The formation of a colored chelate between uranyl ion and ammonium aurintricarboxylate with λ_{max} at 540 mμ, has been reported. The chelate has a composition of 1:1. The formation constant at 25°C is $5.9 \pm 0.5 \times 10^4$, and the free energy of formation -6.51 ± 0.05 kcal at the same temperature. (auth)

8374 ✓ WADC-TR-57-764
Wright Air Development Center. Materials Lab., Wright-Patterson AFB, Ohio.
FLUORINE CONTAINING CHELATE COMPOUNDS.
Glenn R. Buell. Nov. 1957. 15p. Project title: RUBBER, PLASTIC AND COMPOSITE MATERIALS. Task title: SYNTHESIS AND EVALUATION OF NEW POLYMERS. (AD-150984).

A study was made of the feasibility of preparing liquid chelate compounds. The thermal stability of certain chelate compounds led to their study as possible liquid base stock materials. Several fluorinated diketones were chelated with metal ions and their melting points studied to determine the effect of the fluorine on the physical properties of the molecule. Only one liquid was obtained and all compounds exhibited high vapor pressures. (auth)

6475 A/CONF.15/P/1650
STUDY OF THE POSSIBILITY OF PREPARATION OF HIGH SPECIFIC ACTIVITY Co-60 THROUGH SZILARD-CHALMER'S REACTION. Amar Nath, Jagdish Shankar, and S. B. Srivastava (Atomic Energy Establishment, Trombay, India). 10p.

Several chelates of Cobalt (III) were studied. It was found that great care is necessary in the preparation and purification of the complexes so as to eliminate all traces of cobaltous impurities. The choice of the methods used for the separation of Co^{60}, along with Co(II) was guided by the ease of manipulation and the necessity to avoid decomposition of the irradiated complex during the process. The enrichment factor is profoundly influenced by the conditions of neutron irradiation, such as γ flux and temperature. It also depends on the time which elapses between irradiation and the chemical separation and the temperature at which the sample is stored. (auth)

COMPLEXONES: A BIBLIOGRAPHY OF ANALYTICAL, BIOLOGICAL, AND INDUSTRIAL APPLICATIONS. M. R. Verma and K. J. Therattil (National Physical Lab. of India, New Delhi). J. Sci. Ind. Research (India) 15A, Suppl., 1-41 (1956) Sept.

The name complexones is applied to a group of compounds which are essentially derivatives of iminodiacetic acid. They have the remarkable property of chelating with a large number of inorganic ions, and the conditions under which chelation occurs differ considerably for different ions. References are presented to some of the important papers on the applications of complexones, including their preparation, purification, and estimation. The references are arranged under suitable heads to facilitate easy reference. (572 references.) (C.H.)

7476
CONTINUOUS CHELATION-EXTRACTION PROCESS FOR THE SEPARATION AND PURIFICATION OF METALS.
J. R. Thomas, T. E. Hicks, B. Rubin, and H. W. Crandall (to U. S. Atomic Energy Commission). U. S. Patent 2,916,349. Dec. 8, 1959.

A continuous process is presented for separating metal values and groups of metal values from each other. A complex mixture, e.g., neutron-irradiated uranium, can be resolved into component parts. In the present process the values are dissolved in an acidic solution and adjusted to the proper oxidation state. Thenceforth the solution is contacted with an extractant phase comprising a fluorinated β-diketone in an organic solvent under certain pH conditions whereupon plutonium and zirconium are extracted. Plutonium is extracted from the foregoing extract with reducing aqueous solutions or under specified acidic conditions and can be recovered from the aqueous solution. Zirconium is then removed with an oxalic acid aqueous phase. The uranium is recovered from the residual original solution using hexone and hexone-diketone extractants leaving residual fission products in the original solution. The uranium is extracted from the hexone solution with dilute nitric acid. Improved separations and purifications are achieved using recycled scrub solutions and the "self-salting" effect of uranyl ions.

CHLORINATION 6.03

See also, this section,

FUSED SALTS--Chlorides (6.10.01.01),
SPECIAL PROCESSES--Zircex (6.17.24).

CHLORINATION 6.03

3056

APPARATUS FOR REACTING A DENSE CHLORINATING VAPOR WITH A SOLID. M. J. Polissar (to U. S. Atomic Energy Commission). U. S. Patent 2,725,284. November 29, 1955.

An apparatus is described for conducting a reaction between a vaporizable and a solid material comprising two communicating chambers of an hour glass configuration disposed along an inclined axis, the upper chamber forming a reservoir of vaporizable material, and the lower chamber being adopted for the solid material, a jacketed condenser extending in the upper chamber, a tube from the upper to the lower chamber to supply inert gas, a controlled discharge outlet into the condenser admitting the vaporizable material, a condenser vent, and a means of heating each chamber. (auth)

CHROMATOGRAPHY 6.04

See also, this section,

PROCESSES AND METHODS--Elution (6.12.03.

16547

THEORY OF CHROMATOGRAPHY. PART II. ENRICHMENT OF ISOTOPES BY CHROMATOGRAPHY. E. Glueckauf (Atomic Energy Research Establishment, Harwell, Berks, Eng.). Trans. Faraday Soc. 54, 1203-5(1958) Aug.

It is shown that the isotope separation data obtained by elution chromatography can be represented by a linear plot of the log of the local separation coefficient as ordinate against the fraction of the eluted mixture on a probability scale abscissa. The gradient of this plot has the value $\delta\sqrt{N}$, where N is the number of theoretical plates in the column (obtainable from the shape of the elution curve) and δ is the separation factor minus one. This technique of evaluation has been applied to the $Na^{24}-Na^{22}$ separations by Betts, Harris, and Stevenson. It gives values of δ of 1.78×10^{-4}, 1.38×10^{-4}, and $\sim 0.7 \times 10^{-4}$ at 5, 25, and 55°C, respectively, indicating a heat of isotopic exchange of 0.4 cal/mole. (auth)

4717

APPARATUS FOR CONTINUOUS ELECTROCHROMATOGRAPHY. Harold H. Strain (Argonne National Lab., Lemont, Ill.). Anal. Chem. 30, 228-31(1958) Feb.

CHROMATOGRAPHY 6.04

A simple, efficient, adaptable, and economical apparatus has been tested for continuous electrochromatographic separations. The stabilization medium for the flow of the background electrolytic solution and concomitant transverse electrical migration is a tapered sheet of soft, thick industrial filter paper. The support for the paper is a block of solidified polystyrene foam. Evaporation of the background electrolytic solution is prevented by the polystyrene foam support and by a loose curtain of polyethylene sheeting. This apparatus has been utilized for the continuous separation of various inorganic ions. (auth)

PAPER CHROMATROGRAPHY OF RADIOACTIVE SUBSTANCES. (RADIOCHEMICAL STUDIES ON "BIKINI ASHES" (MARCH 1, 1954), PART III). (STUDIES OF THE ANALYTICAL CHEMISTRY OF FILTER PAPER XVI). Shoichi Nakano. Bull. Chem. Soc. Japan 29, 219-24(1956) Mar.

6221

THE SEPARATION AND QUANTITATIVE DETERMINATION OF PLATINUM, PALLADIUM, RHODIUM, AND IRIDIUM ON PAPER STRIPS. N. F. Kember and R. A. Wells (Chemical Research Lab., Teddington, Middlesex, England). Analyst 80, 735-51(1955) Oct.

Chromatographic methods are described for the separation of Pt, Pd, Rh, and Ir in microgram amounts on paper strips. After separation, the metals are extracted from the sections of the strips, and the Pt, Pd, and Rh are determined by colorimetric methods and the Ir by microtitration. Analyses have been carried out by this technique on mixtures of all four metals containing large amounts of base metals. (auth)

11137

ION DIMENSIONS AND R_F VALUES IN RADIAL PAPER CHROMATOGRAPHY. C. Bergamini and A. Rovai (Univ. of Florence, Italy). Anal. Chim. Acta 15, 43-5(1956) July.

The radial paper chromatography of some metals: Zn, Cd, Hg; Li, Na, K, Cs; Be, Mg, Ca, Sr, Ba, carried out using as mobile phase the upper layer of n-butanol–water–nitric acid mixtures of increasing acidity, shows that the phenomenon of the deepening of the color towards the external edge of the chromatogram is not to be attributed to the presence and the nature of the complex-forming agent, and that the ionic mobility does not always result in a linear function of the ionic radius. This behavior results from the interaction of various factors capable of provoking a variation of the dimensions of the solvated ion and consequently of its mobility. (auth)

CHROMATOGRAPHY 6.04

1756

THEORY OF CHROMATOGRAPHY. PART 10. FORMULAE FOR DIFFUSION INTO SPHERES AND THEIR APPLICATION TO CHROMATOGRAPHY. E. Glueckauf (Atomic Energy Research Establishment, Harwell, Berks, England). Trans. Faraday Soc. 51, 1540-51(1955) Nov.

A mathematical analysis of the diffusion process and the practical application of several diffusion equations to ion exchange and chromatography are discussed. (C.W.H.)

7558 ORNL-1690

Oak Ridge National Lab., Tenn.

SEPARATION OF URANIUM(VI) FROM OTHER METALS BY FILTER PAPER PARTITION CHROMATOGRAPHY WITH 2-METHYLTETRAHYDROFURAN AND ITS QUANTITATIVE DETERMINATION. I. SEPARATION STUDIES BY USE OF RADIOISOTOPES. H. P. Raaen and P. F. Thomason. Aug. 4, 1954. Decl. Mar. 2, 1957. 48p. Contract W-7405-eng-26. $0.45(OTS).

Radioisotopes were used in a study of the microseparation of U^{6+} from other metals by filter-paper partition chromatography with 2-methyltetrahydrofuran. Autoradiography of the chromatograms established the efficiency of the separations. The sorption gradient of the separated U^{233} was determined by α-count measurements of the chromatograms; the quantitative removal of U^{233} from the paper strip was demonstrated by the same technique. Maximum condensation of the U^{233} band was effected by a 1.75-hr

4771

A MATHEMATICAL THEORY OF CHROMATOGRAPHY AND MODIFICATION OF THE DIFFUSION METHOD OF ISOTOPE SEPARATION. Prem Behari Mathur (Univ. of Allahabad, India). J. Sci. Ind. Research (India) 14B, 667-9 (1955) Dec.

A mathematical analysis of isotopic separation by the chromatographic process is presented. The separation occurs, it is theorized, as a result of different electrical potentials developed during diffusion. The nature of the medium, concentration and mass effects, and process variables are also discussed. (C.W.H.)

13835

ON SOME CHARACTERISTIC FEATURES OF THE CHROMATOGRAPHIC SEPARATION OF MIXTURES OF RADIOACTIVE SUBSTANCES. M. M. Senyavin, G. M. Kolosova, and V. A. Nikashina. Zhur. Neorg. Khim. 3, 104-8(1958). (In Russian)

The general problems and characteristics of the chromatographic separation of mixtures of radioactive substances were investigated. The static and kinetic properties of the process are discussed. (J.S.R.)

13783

A PRACTICAL DEVICE FOR CHROMATOGRAPHIC SEPARATIONS. B. K. Preobrazhenskii. Zhur. Neorg. Khim. 3, 119-20(1958). (In Russian)

A description is given of a practical device for chromatographic separation. It completely prevents formation of bubbles in the columns, and complete reproducibility of results is obtained. The air pressure after preliminary degassification of the solution is maintained by the use of a hollow glass float closely fitting the walls of the tube. (J.S.R.)

5841 USNRDL-TR-131

Naval Radiological Defense Lab., San Francisco.

CONSTANT FLOW-RATE DEVICE FOR ELECTROLYTE ELUANTS IN COLUMN CHROMATOGRAPHY. R. K. Main, L. J. Cole, L. M. Bryant, and S. K. Morris. Jan. 15, 1957. 11p. Project NM 006-015.

A device is reported which was designed for the accurate control of the flow of an electrolyte eluant through a chromatographic column. This device, operating on the principle of volume delivery, offers certain advantages over those control devices based on the principle of capillary throttle. It has been utilized successfully in laboratory studies on fractionation of nucleic acids by column chromatography. (auth)

13249 AEC-tr-3665

CHROMATOGRAPHIC SEPARATION OF MIXTURES OF NITRO SUBSTANCES. Viktor Ettel, Jan Pospisil, and Zdenek Deyl. Translated for Los Alamos Scientific Lab. from Chem. listy 52, 623-30(1958). 14p. $3.30(ph), $2.40(mf) JCL.

Conditions were ascertained for the chromatographic separation of aromatic nitro compounds distinguished by the presence of further functional groups. Paper impregnated with formamide was used, and the individual parts of the mixtures were washed out progressively with solvents of increasing polarity. The method described was used in the separation of several mixtures obtained as products or by-products of the nitration of toluene. (auth)

CHROMATOGRAPHY 6.04

13231 NP-7590
Rio de Janeiro. Centro Brasileiro de Pesquisas Físicas.
A STOCHASTIC THEORY OF CHROMATOGRAPHY. H. Macedo, A. L. Zamith, and J. Danon. 1958. 17p. (Notas de Física Vol. IV, No. 18).

A microscopic theory is presented in which the stochastic variable is the displacement of the particles along the chromatographic system. This treatment allows an easy derivation of the main results of the chromatographic theories based on the theoretical plate concept. (auth)

13221 IGO-TM/CA-06 (Suppl. 6)
United Kingdom Atomic Energy Authority. Industrial Group. Capenhurst Works, Capenhurst, Ches., England.
VAPOUR PHASE CHROMATOGRAPHY. A Bibliography. C. E. H. Knapman. [Nov. 5, 1958]. 17p.

A bibliography of 114 references from report and published literature on vapor phase chromatography available up to Oct. 1958 is presented. (J.E.D.)

12492
SEPARATION OF FISSION PRODUCTS AND CORROSION ELEMENTS ON ALUMINUM OXIDE. Dorde S. Nemoda. Bull. Inst. Nuclear Sci. "Boris Kidrich" (Belgrade) 9, 95-103(1959).

Use of Al_2O_3 for the chromatographic separation of long-lived radioactive isotopes and corrosion elements from a mixture of fission products was examined. By proper choice of eluents, the mixture was separated into fractions containing one or more elements. Experiments were carried out with artificial mixtures containing the following elements: Cs^{137}, $Sr^{90}-Y^{90}$, $Ba^{140}-La^{140}$, $Ce^{144}-Pr^{144}$, $Zr^{95}-Nb^{95}$, $Ru^{103}-Rh^{103}$, Te, Fe, Co, Ni, Mo, Cr, Al, Pb, and U. (auth)

CHROMATOGRAPHY ON PAPER IMPREGNATED WITH ION EXCHANGE RESINS. IV. THE ADSORPTION OF METAL IONS ON DOWEX-50 FROM DILUTE HF SOLUTION. Michael Lederer (Institut du Radium, Paris). J. Chromatog. 2, 209-10(1959) Mar.

A simple method is described which employs ion exchange papers for determining whether a metal ion in a given solution adsorbs on a cation exchanger. Applications are described in which Dowex-50 or Dowex-50 papers were employed for separating a number of divalent transition elements from Pa(V) or any other of the fast-moving metal ions. Zinc and Cd which complex readily in HCl were strongly adsorbed from HF solution. (C.H.)

20917
ION EXCHANGE CHROMATOGRAPHY. M. J. Cabell (Atomic Energy Research Establishment, Harwell, Berks, Eng.). Chem. in Can. 11, No. 4, 25-30(1959) Apr.

A description is presented of the two main methods used in ion exchange chromatography, break-through and elusion. Accounts of how these separations are controlled by the solution chemistry of the particular process chosen and the way various factors influence the course of a column separation are given. Two examples of ion exchange separation are described, the first being an application to a research problem in which it was necessary to separate sub-microgram amounts of materials and the second being a description of a process which is used on a large scale as an industrial operation. (auth)

20978
THEORY OF CHROMATOGRAPHY. PART XII.
CHROMATOGRAPHY OF HIGHLY RADIOACTIVE GASES. E. Glueckauf (Atomic Energy Research Establishment, Harwell, Berks, Eng.). p.69-89 of "Gas Chromatography, 1958." C. H. Desty, ed. New York, Academic Press Inc., 1958. 391p.

As a result of the temperature rise occurring in the adsorbent of the column during the passage of a highly radioactive band, the rear end of the band tends to move faster than its front. This results in a marked contraction of the bands, which finally reach a width which is independent of the original width of the feed band and of the length of the column. Both these effects are opposite to the behavior of the inactive gases in a column, and they are quantitatively discussed. One of the most useful aspects of these effects is that they afford a simple means of concentrating dilute radioactive gases into small volumes, without the need for refrigeration, provided that the total radioactive power is in the watt range. Explicit expressions are derived for the breakthrough volume, the shape of the elution curve, the peak maximum and the mean band width under steady-state conditions, as well as for the volume of

column required to attain the steady state. Finally the effect is discussed of radioactive heating on the separation of the fission gases krypton and xenon. It is shown that as long as the radioactive power of the adsorbed gases is moderate, separation is slightly more efficient, due to band contraction, though the separation of the peak maxima is less. But for higher radioactive power the latter effect gains in importance, and when the ratio of power to gas flow rate approaches a value of 14 J/ml of gas, separation is no longer possible for these gases, except under specified conditions. (auth)

7453 CEA-tr-R-768
MÉTHODE CHROMATOGRAPHIQUE DE SEPARATION DES MÉTAUX PAR FORMATION DES COMPLEXES D'ADSORPTION. (Chromatographic Method for the Separation of Metals by the Formation of Adsorption Complexes). A. M. Gurvich (Gourvich) and T. B. Gapon. Translated into French by B. De Trezvinsky from Zavodskaya Lab. 23, 1037-42(1957). 16p.

A chromatographic technique is described which involves adsorption of cation complexes. A complexant such as dimethylglyoxime, α-nitroso-β-naphthol, or o-oxyquinoline which is strongly adsorbed by activated carbon is used to complex cations as solution flows through the activated carbon column. The cation is then eluted leaving the complexant on the carbon. (T.R.H.)

COMPLEX FORMATION 6.05

See also, under CHEMISTRY AND PHYSICS OF IMPORTANT ELEMENTS,

RARE EARTHS--Complex Formation (1.32.(

and,

under FISSIONABLE MATERIAL--RECOVERY,

RECOVERY OF PLUTONIUM--Complex Format: (3.03.04).

AMINES 6.05.01

2734 NYO-6591
Pennsylvania State Univ., University Park. Coll. of Chemistry and Physics.
A THERMODYNAMIC STUDY OF SOME COMPLEXES OF METAL IONS WITH POLYAMINES. Charles R. Bertsch, W. Conard Fernelius, and B. P. Block. Apr. 30, 1956. 20p. Contract AT(30-1)-907. $3.30 (ph OTS); $2.40(mf OTS).

The dissociation constants of the following amines have been determined at 10, 20, 30, and 40°C: N,N,N',N'-tetramethylmethanediamine (I), 1,3-propanediamine (II), 1,4-butanediamine (III), cis- and trans-1,2-cyclohexanediamines (IV and V, resp.), 2,2',2"-triaminotriethylamine (VI), and 1,3-diamino-2-propanol (VII), and at 10°C for trans(?)-1,2-cycloheptanediamine (VIII). Formation constants and enthalpy and entropy changes have been determined for the following: II with Ag^+, Cu^{2+}, and Ni^{2+}; III with Ag^+; IV and V with Cu^{2+}, Ni^{2+}, Zn^{2+}, and Cd^{2+}; VI with Cu^{2+}; and VII with Ag^+ and Ni^{2+} at the four temperatures. Formation constants have been determined for VIII with Cu^{2+}, Ni^{2+}, Zn^{2+}, and Cd^{2+}

9989
INFRARED ABSORPTION SPECTRA OF INORGANIC COÖRDINATION COMPLEXES. XII. THE CHARACTERISTIC NH_3 DEFORMATION VIBRATIONS OF SOLID INORGANIC COMPLEXES. G. F. Svatos, D. M. Sweeny, San-ichiro Mizushima, Columba Curran, and J. V. Quagliano (Univ. of Notre Dame, Ind.). J. Am. Chem. Soc. 79, 3313-15(1957) July 5.

The infrared spectra of a number of solid metal amine complexes have been investigated. It has been possible to determine the three deformation vibrations of the ligated molecules in these complexes. The NH_3 degenerate deformation is observed in the region of 1650 to 1560 cm.$^{-1}$; the NH_3 symmetric deformation in the region of 1350 to 1150 cm.$^{-1}$; and the NH_3 rocking frequency in the region of 950 to 650 cm.$^{-1}$. Each of these absorption bands has a characteristic absorption profile. A theoretical explanation for the range of these deformation frequencies is presented. (auth)

9135
MECHANISM OF SUBSTITUTION REACTIONS OF COMPLEX IONS. VIII. Hydrolysis of cis- and trans-CHLOROHYDROXO-BIS-(ETHYLENEDIAMINE)-COBALT(III) COM-

COMPLEX FORMATION 6.05

AMINES 6.05.01

PLEXES. Ralph G. Pearson, Robert E. Meeker, and Fred Basolo (Northwestern Univ., Evanston, Ill.). J. Am. Chem. Soc. 78, 2673-6(1956) June 20.

The rates of reaction of cis- and trans-chlorohydroxo-bis-(ethylenediamine)-cobalt(III) complexes with H_2O reported. They are considerably faster than those of the corresponding dichloro complexes. A discussion is given of the products of reaction of one mole of hydroxide ion with one mole of cis- or trans-$[Coen_2Cl_2]^+$. (auth)

SYNTHESIS AND PROPERTIES OF SOME FLUORO-BIS (ETHYLENEDIAMINE)-COBALT(III) COMPLEXES. William R. Matoush and Fred Basolo (Northwestern Univ., Evanston, Ill.). J. Am. Chem. Soc. 78, 3972-6(1956) Aug. 20.

A somewhat different approach to the synthesis of fluoroamminecobalt(III) salts is presented. Preparation of five new complexes is described and the isolation of optically active compounds containing a metal-fluoride bond is reported for the first time. Isolation of such optically active forms provides a proof of structure for three different pairs of geometric isomers. Reactions of dextro-$[Coen_2F_2]^+$ give no evidence for a Walden inversion type reaction as is known for levo-$[Coen_2Cl_2]^+$. (auth)

4455 CEA-tr-A246
LES COMPLEXES DE L'ACIDE ÉTHYLÈNEDIAMINE-TETRACÉTIQUE AVEC LE VANADIUM ($-H_4Y$). (Complexes of (Ethylenediamine)Tetra- Acetic Acid with Vanadium ($-H_4Y$).) G. Schwarzenbach and J. Sandera. Translated by B. Moreau from Helv. Chim. Acta 36, 1089-1100(1953). 16p.

The formation of ethylenediaminetetraacetic acid complexes of the type VY^{2-} with divalent vanadium was studied by measurements of the pH. The stability constant of the complex VY^- in which the metal had the III valence was obtained from the variation of the oxidation-reduction potential V(III)/V(II) produced during the addition of complexon. Tetravalent vanadium forms the complex VOY^{2-}, which was studied polarographically. Pentavalent vanadium does not form complexes with EDTA. Salts having the yellow color of the anion VY^- and the blue color of VOY^{2-} were prepared. (J.S.R.)

6387 ✓ A/CONF.15/P/1600
CHEMICAL SPECIFICITY OF CATION EXCHANGE. I. CHARGE AND RADIUS EFFECTS WITH POLY-AMINO COBALTIC COMPLEXES. Bahattin Baysal (Univ. of Ankara). 13p.

Data are presented for the cation exchange equilibria in replacing La^{3+} from the cation resin Dowex-50 for five cobalt(III) complexes, three of tripositive charge (the trisethylenediamine complex, the hexamino, and the cisbisaquotetramino), on dipositive (the pentamino-chloro) and one unipositive (the tetraminoxalate). The studies extended over a range of mole fraction of cobalt complex from 0.05 to 0.85. (W.D.M.)

GENERAL - INORGANIC 6.05.02

956 AEC-tr-2658
INORGANIC COMPLEXES. A. E. van Arkel. Translated from Chem. Weekblad 52, 193-7(1956). 20p.

A theory of the formation and structure of inorganic complexes is presented. The correlations between the different groups of compounds which are formed by variation of the original complex are discussed in detail. (W.L.H.)

16926 ISC-943
Ames Lab., Ames, Iowa.
COMPLEX METAL HALIDES IN FUSED ALKALI NITRATES. Marlowe L. Iverson and F. R. Duke. Dec. 1957. 79p. Contract W-7405-eng-82. $2.25(OTS).

Solubility relationships and reaction kinetics using a mixture of alkali nitrates as solvent for inorganic salts were investigated. Association effects observed show that complex ions are formed between metal ions and halide ions. The rate studies suggest a mechanism for a dichromate-nitrate reaction. Solubility dependence on halide concentration is interpreted in terms of complex ion formation. (T.R.H.)

9192
COMPLEX FLUORIDES. PART V. COMPLEXES FORMED BY MOLYBDENUM HEXAFLUORIDE, TUNGSTEN HEXA-FLUORIDE, AND RHODIUM TETRAFLUORIDE. B. Cox, D. W. A. Sharp, and A. G. Sharpe (University Chemical Lab., Cambridge, England). J. Chem. Soc., 1242-4(1956) May.

The hexafluorides of molybdenum and tungsten combine with potassium, rubidium, and cesium fluorides to form complexes of general formulae M_2MoF_8 and M_2WF_8. The

COMPLEX FORMATION 6.05

GENERAL - INORGANIC 6.05.02

complex fluoride which is obtained by interaction of bromine trifluoride and sodium hexachlororhodate(III) is sodium hexafluororhodate(IV), Na_3RhF_6. (auth)

ON THE RELATIVE STABILITY OF ALUMINUM, TITANIUM, VANADIUM, IRON, AND COPPER TARTRATE COMPLEXES IN ALKALI MEDIA. I. V. Pyatnitskii and A. P. Kostyshina. Zhur. Neorg. Khim. 4, 1341-6(1959) June. (In Russian)

The stability of aluminum, copper, iron, titanium, and vanadium tartrate complexes was determined using bond magnitudes as criteria (the ratio between the concentrations of complexed and free ions at a certain standard acid condition). A method is suggested for determining the ratio of the bonds combining the complexes of two metals. The partition constants of aluminum, copper, iron(III), and vanadium hydroxyquinolinates between the aqueous solution and chloroform were 2.6×10^{-33}, 7.3×10^{-23}, 1.5×10^{-37}, and 4.2×10^{-23}, respectively. The relative stability of copper and iron tartrate complexes in alkali solution (pH 13) and aluminum, iron(III), titanium, and vanadium(IV) tartrate complexes in ammonium solution (pH 9.5) was determined. (R.V.J.)

20932

THE FLUORIDE COMPLEXING OF SCANDIUM(III) IN AQUEOUS SOLUTION: FREE ENERGIES, HEATS AND ENTROPIES. John W. Kury, Armine D. Paul, Loren G. Hepler, and Robert E. Connick (Univ. of California, Berkeley). J. Am. Chem. Soc. 81, 4185-9(1959) Aug. 20.

The complexing of Sc(III) in perchlorate solutions at an ionic strength of 0.5M was determined by studying the potential of the ferrous-ferric electrode as a function of the HF, H^+, and Sc(III) concentrations. The data can be interpreted by assuming the presence of the species ScF^{2+}, ScF_2^+, $ScF_3(aq)$ and ScF_4^-. Equilibrium constants for the formation of these complexes at 15, 25, and 35° were evaluated and from them the heat and entropy changes of the reactions calculated. The scandium fluoride complexes are more stable than those of other trivalent ions of comparable radius. The extra stability of the complex scandium fluorides arises from an irregularity in the heat term, rather than in the entropy term. (auth)

ON THE COMPLEX FORMATION IN THE SYSTEM $UO_2(NO_3)_2-KHCO_3-H_2O_2-H_2O$. E. V. Komarov. Zhur. Neorg. Khim. 4, 1313-23(1959) June. (In Russian)

GENERAL - ORGANIC 6.05.03

6466 A/CONF.15/P/1439

RADIOCHEMICAL SEPARATIONS ON ANION EXCHANGE COLUMNS WITH EMPLOYMENT OF STRONG COMPLEXING AGENTS. T. Schonfeld, W. Wald, and Maria Bruno (Vienna Univ.). 19p.

Only a few investigations have been concerned with the use of strong complexing agents. Various radiochemical separations using anion-exchange resins and strong complexing agents are being studied. Results on several systems are reported, among them separations of $Pb^{210}-Bi^{210}$ and $Sr^{90}-Y^{90}$ using strongly basic anion exchangers and EDTA or other complexing agents. The special features of the application of strong complexing agents to radiochemical separations on anion exchange columns are discussed. (auth)

6602

STUDIES OF THE EXTRACTION OF METAL COMPLEXES. XXXI. INVESTIGATIONS WITH SOME 5,7-DIHALOGEN DERIVATIVES OF 8-QUINOLINOL. David Dyrssen, Margareta Dyrssen, and Ebbe Johansson (Royal Inst. of Technology, Stockholm, Sweden). Acta Chem. Scand. 10, No. 3, 341-52(1956).

Equilibrium constants describing the chloroform extraction of La(III), Th(IV), and U(VI) complexes with the 5,7-dichloro, 5,7-dibromo, 5,7-diiodo and 5-chloro-7-iodo derivatives of 8-quinolinol (oxine) were investigated at 25°C. The ionic strength in the aqueous phase was kept constant at 0.1M. The overall equilibrium constant K for the extraction of metal complexes is increased by halogen substitution of the reagent. This effect, which is very pronounced for Th, is discussed in terms of the separate dissociation and distribution equilibria. The dihalogen substituted oxines are

SPECTRAL INVESTIGATIONS OF METAL COMPLEXES OF β-DIKETONES. I. NUCLEAR MAGNETIC RESONANCE AND ULTRAVIOLET SPECTRA OF ACETYLACETONATES. R. H. Holm and F. A. Cotton (Massachusetts Inst. of Tech., Cambridge). J. Am. Chem. Soc. 80, 5658-63(1958) Nov. 5.

COMPLEX FORMATION 6.05

GENERAL – ORGANIC 6.05.03

A report of proton resonance and ultraviolet spectra of a broad range of metal ion complexes of acetylacetone is presented. The n.m.r. spectra of diamagnetic complexes provide no support for the postulate of benzenoid resonance in the chelate rings, as has been suggested by Calvin nor are any great differences in electron density at the unique carbon atom in the ring indicated. Definitive ultraviolet spectra of thirty acetylacetone complexes are reported. All of these data in general and particularly in certain comparisons indicate that the electrostatic model of Belford, et al., cannot account for the variation in energy of the presumed $\pi-\pi^*$ transition. The energy of this transition cannot be correlated with any simple parameter of the complexed

10356 ✓ ISC-921
Ames Lab., Ames, Iowa.
ANNOTATED BIBLIOGRAPHY OF ALPHA, BETA, GAMMA, DELTA-TETRAPHENYLPORPHINE.
Charles V. Banks and Samuel Anderson. Oct. 1957. 17p. Contract W-7405-eng-82. $0.50(OTS).

The references in this bibliography deal with the methods of preparation and purification of alpha, beta, gamma, delta-tetraphenylporphine, the properties of some of its metal complex salts, and its analytical applications. The references are listed chronologically. (auth)

13718
COMPLEXES OF VANADIUM (IV). X. THE VANADIUM (IV)–MALONATE SYSTEM. R. Trujillo and F. Torres (Uviversidad de La Laguna). Anales real soc. espan. fis. y quim. (Madrid) Ser. B. 54, 377-82 (1958) May. (In Spanish)

The complex formation of V^{4+} with malonic acid was studied using a solution containing $VO(ClO_4)_2$, the metallic Vion, and malonic acid. The absorption spectrum was obtained. The formula for the complex, obtained by the method of Job and from molar relations, was determined to be $[VO-(Mal)-VO]^{2+}$. (J.S.R.)

ION EXCHANGE SEPARATION OF METAL IONS.
James S. Fritz and Gerald R. Umbreit (Iowa State Coll., Ames). Anal. Chim. Acta 19, 509-16(1958) Dec.

An ion exchange method that depends on the selective complexation of metal ions by EDTA (ethylenediaminetetraacetic acid) is discussed. Selectivity is achieved by proper choice and control of pH. The uncomplexed metal ions are retained by a short cation exchange column, while the complex metals pass quantitatively through the column. Quantitative ion exchange separations are attained quickly because a short (4-cm) column is used. Excellent pH control is effected by incorporating a buffer in the sample to the separated and by treating the resin column with the same buffer solution. The ion exchange retention of several metal cations in EDTA solution was studied as a function of pH. From the behavior of individual ions, predictions can be made as to the mixtures that can be separated by this method. Several actual separations are reported. (J.H.M.)

✓ **17033**
THE DIPHENATES OF THORIUM, CERIUM, ZIRCONIUM, AND URANIUM. COMPOSITION, STRUCTURE, AND PROPERTIES. HETEROMETRIC STUDY. M. Bobtelsky and A. H. I. Ben-Bassat (Univ. Hebraique, Jerusalem). Bull. soc. chim. France No. 8-9, 1138-44(1958) Aug.-Sept. (In French)

Thorium nitrate forms with diphenic acid (D) at pH ~ 4 the soluble complex $(Th_2D)^6$ which is quantitatively changed into insoluble ThD. Cerium(III) nitrate forms, in a solution containing 50% ethyl alcohol at a pH 4 to 5, the final insoluble compound $Ce(III)D_3$ and the intermediate compounds Ce(III)D and $Ce(III)D_2$. Zirconium nitrate forms the final insoluble compound ZrD (ZrOD). The intermediate compounds have the composition Zr_4D (soluble), Zr_3D, and Zr_2D. Uranium nitrate forms with diphenic acid only solutions. In the presence of thorium nitrate it forms quantitatively an insoluble compound (or a co-precipitate) having the composition $ThUD_4$.

13721 ✓
CONTRIBUTION TO THE STUDY OF THE COMPLEXES OF THE CITRATES AND TARTRATES. IV. THE COMPLEX OF VANADIUM WITH CITRATE. Clara Heitner-Wirguin and Débora Friedman (L'Université Hébraïque, Jérusalem). Bull. soc. chim. France No. 7, 970-3(1958) July. (In French)

The existence of a stable complex $(VOCl)^{2-}$ was shown by means of pH and spectrophotometric measurements. The possible existence of a 2VO/Cl complex is discussed. (tr-auth)

COMPLEX FORMATION 6.05

GENERAL – ORGANIC 6.05.03

MANGANESE-54, URANIUM-233 AND COBALT-60 COMPLEXES OF SOME ORGANIC ACIDS. Norman C. Li, William M. Westfall, Arthur Lindenbaum, James M. White, and Jack Schubert (Argonne National Lab., Lemont, Ill. and Duquesne Univ., Pittsburgh). J. Am. Chem. Soc. 79, 5864-70(1957) Nov. 20.

By using Mn-54 with ion-exchange technique, we have determined the formation constants of manganous complexes of 20 organic acids at pH 7.2 to 7.3, u = 0.16. The effect of –OH, double bonds, configuration, and chelate ring size on complex stability is brought out by comparison of the formation constants of the 20 acids. Uranium-233 was used with ion-exchange and solvent-extraction methods for the study of uranyl complexes of bioxalate, glycolate, and serine. Cobalt-60 complexes of four organic acids were studied by ion-exchange and solvent-extraction techniques. Several of the complexes also have been studied by pH method. An important assumption in the use of ion-exchange method for complex study is that the complex itself is not taken up by the cation exchanger. This has been demonstrated experimentally for the first time for some metal complexes by means of acetate labeled with tritium and with C^{14}. (auth)

8990 AEC-tr-3212

STUDY OF THE COMPLEX COBALT OXALATES BY MEANS OF ANION EXCHANGE. THE THEORETICAL BASIS OF THE METHOD. V. V. Fomin and V. V. Sinkovskii (Sinkovsky). Translated by Lydia Venters from Zhur. Neorg. Khim. 1, 2316-26(1956). 19p.

A method for determination of the stability constant of the complex ions in solution by means of anion exchange was worked out. The method has been used for the determination of the stability constant of the complex cobalt oxalates. It has been found that cobalt ions Co^{2+}, $CoC_2O_4^0$, $Co(C_2O_4)_2^{2-}$, and $Co(C_2O_4)_3^{4-}$ exist in the solution at the ammonium oxalate concentration from 1×10^{-4} to 3×10^{-1} mole/l which indicates amounts of cobalt. By experiment relationships between the stability constants (expressed in concentrations) to the second and third ion forms of the stability constant relative to the first one have been found; they equal relatively $K_1 = 1.5 \times 10^2$ and $K_2 = 4.5 \times 10^3$. Using the previously determined stability constants of the first complex $[Co(C_2O_4)]$, the stability constants of all the ions have been calculated, which equal accordingly $\beta_1 = 3.1 \times 10^4$, $\beta_2 = 4.6 \times 10^6$, and $\beta_3 = 1.35 \times 10^8$. (auth)

13322

PREPARATION OF OXALATES OF METALS OF ATOMIC NUMBER GREATER THAN 88. R. B. Duffield (to U. S. Atomic Energy Commission). U. S. Patent 2,872,467. Feb. 3, 1959.

A method is presented for the preparation of oxalates of metals of atomic number greater than 88. A solid peroxide of the heavy metal is contacted with an aqueous oxalic acid solution at a temperature of about 50°C for a period of time sufficient to form the insoluble metal oxalate which is subsequently recovered as a pure, crystalline compound.

8300

STUDIES ON THE EXTRACTION OF METAL COMPLEXES. XXII. THE COMPLEX FORMATION OF THORIUM WITH 1-NITROSO-2-NAPHTHOL AND 2-NITROSO-1-NAPHTHOL. David Dyrssen, Margareta Dyrssen, and Ebbe Johansson (Research Inst. of National Defence, Sundbybery, Sweden and Royal Inst. of Tech., Stockholm, Sweden). Acta Chem. Scand. 10, No. 1, 106-12(1956).

The extraction of Th with chloroform or hexone using 1-nitroso-2-naphthol and 2-nitroso-1-naphthol as complexing agents has been studied at 25°C. The perchlorate ion concentration in the aqueous phase was kept constant at 0.1 M. The mean complexity constants are found to be $10^{8.50}$ for 1-nitroso-2-naphthol and $10^{8.30}$ for 2-nitroso-1-naphthol. These values are lower than for oxine, but higher than for acetylacetone. (auth)

11372

ON FOCUSING ION EXCHANGE. V. SEPARATION OF METAL IONS, ESPECIALLY RADIOACTIVE INDICATORS BY EXCHANGE WITH PROTONS. E. Schumacher and H. J. Streiff (Univ. of Zurich). Helv. Chim. Acta 41, 824-43(1958). (In German)

Focusing ion exchange (FI) has extensively been studied using a pH gradient for producing a gradient in chelating tendency. NTA, EDTA, Chel CD, Chel ME, and Chel DE are complexing agents, HCl or $HClO_4$ in suitable concentrations are complex-destroying agents. Apparatus for FI-separation on paper-strips is described. A simple radiograph for evaluation of separated activity-distributions has been in use for over a year. pH plotted against length of strip shows two distinct steps, one in the strong acid region between pH 0 and 1, one between pH 2 and 6, $0.8 < pH < 2$ is a region of smoothly varying pH, where separations preferentially

COMPLEX FORMATION 6.05

GENERAL - ORGANIC 6.05.03

take place. The value of the electrical field along the strip is discussed. The influence of process-parameters on separation has been evaluated with Co, Cu and Ba, and Sr rare earths as test-mixtures. Optimal conditions are found. Some applications are mentioned, such as separation of Cs^{137} from alcaline earths, rapid qualitative analyses of some alloys, rapid analysis of a fission mixture of activities, qualitative analysis of group II-ions, and analysis of rare earths-mixtures. Some interesting observations in regard to complex-chemistry are made: all complexing agents used are forming ammonium-ions in pH 0 to 1 region. This has several implications on the separation process and on the magnitude of measured stability constants, NTA complexes of rare earths show anionic behavior probably by taking up Cl^- or F^- ion. (auth)

FOCUSING ION EXCHANGE. VII. QUANTITATIVE MICROANALYSIS OF HEAVY METALS AND RARE EARTHS. Ernst Schumacher and Hans Jakob Streiff (Univ. of Zürich). Helv. Chim. Acta 41, 1771-83(1958). (In German)

Focusing ion exchange as a method of separation of metal-ions, taking place in homogeneous solution in the presence of a complex-formation and an electrical-potential gradient, is compared with heterogeneous ion exchange and ionophoresis, and the distinctions are stressed. The phenomenon of "double-focusing", meaning the stationary existence of two lines of the same metal in the presence of two complexing agents (one of which is an amino-poly-acetic acid), offers a possibility for the estimation of μg- and ng amounts of various metals, including Fe^{3+}, Cu^{2+}, Pb^{2+}, Co^{2+}, Y^{3+}, and heavy rare earths. This can not be realized with ordinary ion exchange nor electrophoresis. One of the lines of a double-focus, being located on the acid side of a pH-gradient, changes its intensity proportionally to the added amount of a chelating agent (NTA, EDTA,

10101

THE APPLICATION OF SOLVENT EXTRACTION TO THE STUDY OF CHELATE COMPLEXES OF THORIUM. Margareta Dyrssen (Royal Inst. of Tech., Stockholm, Sweden). Rec. trav. chim. 75, 748-52(1956) June.

The extraction method is very useful for studies of the complex formation of metal ions M^{N+} with chelating agents. These reagents are very often organic acids HA which form uncharged complexes MA_N of very low solubility in water but of appreciable solubility in organic solvents. The distribution ratio, q, of the metal M between the two liquid phases is measured as a function of the concentration $[A^-]$ of free ligand. Mathemathical treatment of the function log q (log $[A^-]$) very often enables the calculation of at least two parameters a and b for the complex formation. (auth)

6603

STUDIES ON THE EXTRACTION OF METAL COMPLEXES. XXXII. N-PHENYLBENZOHYDROXAMIC ACID. David Dyrssen (Research Inst. of National Defence, Sundbyberg, Sweden). Acta Chem. Scand. 10, No. 3, 353-9(1956).

N-Phenylbenzohydroxamic acid (PBHA) was investigated at 25°C as a solvent extraction agent for La(III), Th(IV), and U(VI). Chloroform was used as the organic solvent and the ionic strength in the aqueous phase was kept constant at 0.1M by addition of $HClO_4$ and $NaClO_4$. The values of 1/N log K (where K is overall constant, and N = ionic valence) for La, Th, and U are −4.80, −0.17, and −1.57, respectively. It is shown that Th and U may easily be separated from La by batch extraction, while a good separation of Th and U would require a simple extraction column. The stoichiometric dissociation constant k_a of PBHA is $10^{-8.15 \pm 0.01}$ and its distribution constant k_d between chloroform and water is $10^{2.33 \pm 0.01}$. The mean complexity constant 10^{β} for the formation of Th tetra-N-phenylbenzohydroxamate is $10^{9.45}$ and the distribution constant λ_4 of this complex between chloroform and water is $10^{3.45 \pm 0.03}$. (auth)

22030

THE EXTRACTION OF CITRATE AND TARTRATE COMPLEXES OF THE TRANSITION METALS. Max Ziegler (Universität, Göttingen, Ger.). Naturwissenschaften 46, 492(1959). (In German)

The citrate and tartrate complexes of the transition metals palladium, vanadium, uranium, iron, bismuth, gold, and cerium can be separated as substituted ammonium salts by liquid-liquid extraction from aqueous solutions with organic solvents. The citrate complex of rhodium and the tartrate complex of zirconium can be extracted in a similar manner. The organic solvent and the solution acidity for the best results are tabulated for each element. (J.S.R.)

COMPLEX FORMATION 6.05

SURVEYS 6.05.04

6466 ✓ A/CONF.15/P/1439
RADIOCHEMICAL SEPARATIONS ON ANION EXCHANGE COLUMNS WITH EMPLOYMENT OF STRONG COMPLEXING AGENTS. T. Schonfeld, W. Wald, and Maria Bruno (Vienna Univ.). 19p.

Only a few investigations have been concerned with the use of strong complexing agents. Various radiochemical separations using anion-exchange resins and strong complexing agents are being studied. Results on several systems are reported, among them separations of $Pb^{210}-Bi^{210}$ and $Sr^{90}-Y^{90}$ using strongly basic anion exchangers and EDTA or other complexing agents. The special features of the application of strong complexing agents to radiochemical separations on anion exchange columns are discussed. (auth)

6493 ✓ A/CONF.15/P/2204
APPLICATION OF ION EXCHANGE FOR INVESTIGATION OF THE STATE OF RADIOACTIVE SUBSTANCES IN SOLUTIONS. B. Nikolskii (Nikolsky) and V. Paramonova (U.S.S.R.). 22p.

Reaction mechanisms in ion-exchange reactions in solutions are reviewed. Applications are discussed in quantitative determinations of ions of elements in solutions and in studies of the state of radioactive elements in solutions, especially in extremely dilute solutions. As examples the complex-forming processes are considered in systems of zirconium–nitric acid, uranyl–acetic acid, zirconium-95–perchloric acid–sodium perchlorate, and zirconium with nitrate ion and in mixtures of nitric acid and perchloric acid, in which zirconium-95 was present in microconcentration. Data are presented from studies on the interaction of radioyttrium ions with lactic acid complex ions and complex formation of uranyl ions in water solutions of sodium nitrate and sodium acetate and in acetate–nitrate solutions. (C.H.)

103 ✓ AEC-tr-3854
SOME PHYSICOCHEMICAL CONSIDERATIONS CONCERNING THE ELIMINATION OF RADIOACTIVE METALS BY MEANS OF COMPLEX PRODUCERS. Hans Jörg Heller and Alexander Catsch. Translated by Lydia Venters (Argonne National Lab.) from Strahlentherapie 109, 464-82(1959). 31p. JCL.

This paper was previously abstracted from the original language and appears in NSA, Vol. 13, as abstract No. 17748.

4199 AECD-3938
[Knolls Atomic Power Lab., Schenectady, N. Y.] RADIOCHEMISTRY III. Nuclear Engineering Course, 1947–1948. E. Zebroski. Apr. 15, 1948. Decl. with deletions Jan. 9, 1956. 16p. Contract [W-31-109-Eng-52]. $3.30(ph OTS); $2.40(mf OTS).

The radiochemistry, including complex ion formation, and reactor applications of the actinide elements are discussed. (C.W.H.)

8201
SOME RELATIONSHIPS AMOUNG THE STABILITIES OF METAL COMPLEXES. Harry Irving and Hazel Rossotti (Inorganic Chemistry Lab., Oxford, England and Royal Inst. of Tech., Stockholm, Sweden). Acta Chem. Scand. 10, No. 1, 72-93(1956).

DISTILLATION AND CONDENSATION 6.06

See also, this section,

SPECIAL PROCESSES--Fused Salt-Fluoride Volatility (6.17.11),

and,

under FISSION PRODUCT, POISON, AND RADIOISOTOPE REMOVAL,

DISTILLATION (2.15),

and,

under FISSIONABLE MATERIAL--RECOVERY,

VOLATILITY METHODS--Fluoride Volatility (3.02.18.01),

General (3.02.18.02).

SEPARATION OF FLUID MIXTURES. R. Lipscomb, A. Craig, S. Labrow, and J. F. Dunn (to U. S. Atomic Energy Commission). U. S. Patent 2,857,745. Oct. 28, 1958.

An apparatus is presented for separating gaseous mixtures by selectively freezing a constituent of the mixture and subsequently separating the frozen gas. The gas mixture is passed through a cylinder fitted with a cooling jacket, causing one gas to freeze on the walls of the cylinder. A set of scraper blades are provided in the interior of the cylinder, and as the blades oscillate, the frozen gas is scraped to the bottom of the cylinder. Means are provided for the frozen material to pass into a heating chamber where it is vaporized and the product gas collected.

HOW TO USE MASS TRANSFER COEFFICIENTS. James O. Osburn (State Univ. of Iowa, Iowa City). Chem. Eng. 65, No. 10, 169-72(1958) May 19.

Steady-state mass-transfer information needed in the design of gas absorption, extraction, drying, crystallization, and distillation equipment may be obtained through the use of mass-transfer coefficients. The proper application of these coefficients and the calculational methods employed are presented and discussed. (D.E.B.)

7486 KLX-10076

Vitro Labs., West Orange, N. J.
ELECTROKINETIC PROCESSES–NUCLEAR ASPECTS SUMMARY REPORT [FOR PERIOD] NOVEMBER 1, 1953–NOVEMBER 30, 1956. A. C. Werner and H. Katz. July 12, 1957. Decl. May 7, 1958. 13p. Contract AT(30-1)-850. $3.30(ph), $2.40(mf) OTS.

Progress in electrophoretic deposition is summarized, and references are made to previous reports in which the information is reported in detail. (W.L.H.)

A DISCUSSION OF THE CONTROLLING RATES OF VACUUM DISTILLATION OF METALS. J. E. Vivian. Sept. 24, 1953. Decl. Mar. 29, 1957. 6p. (LWS-24923; CRD-R-92)

The relative rates of evaporation from liquid metal surfaces and diffusion to the surface in the liquid phase are compared and their effect on the separation of metal mixtures by vacuum distillation is discussed. It is concluded that under some conditions liquid phase diffusion may be significant, and that the relative rates will vary with temperature.

5755 AD-105431

Institute of Tech., Wright-Patterson AFB, Ohio.
A STUDY OF THE AUTOMATIC CONTROL OF A CONTINUOUS DISTILLATION COLUMN (thesis). Edwin Goodell Park, Jr., and Robert Arthur Vince. Aug. 1956. 58p. (GAO-56).

The application of automatic control to chemical processing and operation has previously been done largely by a trial and error procedure. The purpose of this procedure was to determine modes of control and the suitable constants of these modes which gave the most stable response. Williams and co-workers have developed a theoretical method of accomplishing this evaluation for a continuous distillation column through computations made on a large-scale analog computer. The goal of this thesis has been to check experimentally their results so that the validity of their method could be determined. A small six-plate distillation column was operated to determine the stability

DISTILLATION AND CONDENSATION 6.06

afforded by various combinations of control modes and constants. Automatic control for this column was provided by a Foxboro Hyper-Reset Controller and a Foxboro Stabilflo Valve. The results of the experiments indicate that within the limits of the apparatus used, a verification of the work of Williams and do-workers has been obtained. (auth)

19833 UCRL-8787
California. Univ., Berkeley. Lawrence Radiation Lab. SOLUTION OF MULTISTAGE SEPARATION PROBLEMS BY USING DIGITAL COMPUTERS (thesis). John H. Duffin. Aug. 1959. 266p. Contract W-7405-eng-48. $4.00(OTS).

Methods of solving various problems in multistage separation operations are presented. These methods are illustrated by solving specific problems using both medium (IBM 650) and large (IBM 701) digital computers. Separations by distillation normally encountered in the petroleum and chemical industries are covered in detail from the initial considerations of variable analysis to the final convergent solution of a given problem. Various methods of attacking a problem are presented and combinations of these methods that produce convergent solutions are suggested and illustrated. By means of appropriate equations, an operating unit is simulated on a computer and its behavior is studied as a steady-state or convergent solution is approached. Problems in distillation occur where an infinite rather than a discrete mixture is used, and methods of attack and illustrations are given for problems of this type. By using these methods it is possible to simulate such complex industrial units as crude-oil distillation columns. Nonideal equilibrium problems are also covered; methods of attack are presented as well as illustrations for a specific problem. Since methods developed for distillation operations should apply, with appropriate modifications, to any multistage process, liquid-liquid extraction is considered finally. A method of attack for problems of this type is developed and solutions of problems are given. (auth)

ELECTROCHEMISTRY 6.07

See also, this section

Excer Process

And

Molten Salts, Fused Salts, And

Liquids.

See also, this section,

FUSED SALTS--Chlorides (6.10.01.01),

SPECIAL PROCESSES--Flurex (6.17.10),

and,

under FISSIONABLE MATERIAL--RECOVERY,

ELECTROCHEMICAL (3.03.05).

4314
ELECTRODEPOSITION OF URANIUM AT THE MICROGRAM LEVEL. Charles L. Rulfs, Anil K. De, and Philip J. Elving (Univ. of Michigan, Ann Arbor). J. Electrochem. Soc. 104, 80-3(1957) Feb.

The recovery of microgram and submicrogram quantities of U by electroplating has been studied, using radioactive U^{233} as a tracer with and without the presence of microgram quantities of natural U as carrier. Optimum results were obtained in an ammonium oxalate medium with electrolysis at 80 to 85°C being performed first in acidic solution and then in alkaline solution. A small Pt disk which fitted into a flow counter was used as cathode, and a Pt spiral served as anode. With 20 μg of U carrier present and a volume of 25 ml, an average recovery, based on alpha counting, of 94 ± 3% was obtained for amounts of U^{233} ranging from 0.03 to 0.13 μg. (auth)

ELECTROCHEMISTRY 6.07

10472 KAPL-1692
Knolls Atomic Power Lab., Schenectady, N. Y.
THE BEHAVIOR OF REPRESENTATIVE FISSION PRODUCTS AND PLUTONIUM IN THE KAPL ELECTROREFINING PROCESS AND A REVIEW OF THE STATUS OF THE PROCESS. L. W. Niedrach, A. C. Glamm, and G. R. Fountain. Mar. 27, 1957. 36p. Contract W-31-109-Eng-52. $0.30(OTS).

New decontamination data are presented from multi-throughput runs with the KAPL electrorefining process. It is shown that decontamination from both noble metals and active metals is satisfactory even after as many as 15-bath throughputs. A method is described for calculating the decontamination obtainable by electrorefining from measurements of the distribution coefficient for a fission product, plutonium or other material between the metal and the salt phases. The current status of electrorefining as a method for processing reactor fuels is reviewed. An indication is also given of the types of fuel for which an electrorefining process would be most adaptable. (auth)

6663 ✓ AERE-C/R-779
Gt. Brit. Atomic Energy Research Establishment, Harwell, Berks, England.
A USEFUL ELECTROLYTIC CELL. F. Hudswell, K. L. Wilkinson, and J. C. Waldron. Sept. 10, 1951. Decl. Mar. 4, 1957. 12p.

The electrolytic cell described is constructed from perspex, and can be used for obtaining electrodeposits on defined areas of one or both sides of a metal sheet or foil. Stirring is uniform on both sides of the foil, since the stirrers (usually the anodes) are geared together. The foil is held in a vertical plane to minimize the collection of loose particles. Provision is made for the removal of the electrolyte into an auxiliary chamber before interrupting the current. The cell is especially useful for the electrodeposition of radioactive materials from small volumes of solution. (auth)

ELECTROLYTIC CELLS. R. Q. Boyer (to U. S. Atomic Energy Commission). U. S. Patent 2,733,202, January 31, 1956.

An improved electrolytic reduction cell has been developed comprising a casing provided with a catholyte passage of maximum length within the casing, and contacting electrode structure arranged to afford a large uniform reducing action upon the metallic ions contained in the catholyte. The arrangement provides for a pool of liquid metal in the lower portion of the cell, with the electrolyte in the upper portion floating thereon. The cathode structure mounted on a removable frame is rotatable and is utilized to propel the electrolyte through the elongated passage forward in the casing. A gastight cover supporting the electrode structure depending therefrom is constructed in part of transparent material to facilitate observation of the interior of the cell, and a syphon mechanism is provided for charging and discharging electrolyte. (auth)

388
THE INFLUENCE OF TEMPERATURE ON ISOTOPIC SEPARATION UNDER THE EFFECT OF A CONTINUOUS CURRENT IN MOLTEN GALLIUM. Maurice Goldman, Guy Nief, and Étienne Roth. Compt. rend. 243, 1414-16 (1956) Nov. 5. (In French)

2620 ✓
ELECTROCONDUCTIVITY OF CONCENTRATED AQUEOUS SOLUTIONS OF LiCl, NaCl, AND KCl AT HIGH TEMPERATURES. I. M. Rodnyanskii and I. S. Galinker. (Khar'kov V. V. Dokuchaev Agri. Inst.) Doklady Akad. Nauk 105, 115-18(1955) Nov. 1. (In Russian)

Investigations of electroconductivity of 1 to 3N salt solutions of LiCl, NaCl, and KCl up to 340°C are discussed, and tabulations are given. (R.V.J.)

5039 ✓
ELECTROLYTIC CELLS. R. Q. Boyer (to U. S. Atomic Energy Commission). U. S. Patent 2,733,202, January 31, 1956.

An improved electrolytic reduction cell has been developed comprising a casing provided with a catholyte passage of maximum length within the casing, and contacting electrode structure arranged to afford a large uniform reducing action upon the metallic ions contained in the catholyte. The arrangement provides for a pool of liquid metal in the lower portion of the cell, with the electrolyte in the upper portion floating thereon. The cathode structure mounted on a removable frame is rotatable and is utilized to propel the electrolyte through the elongated passage forward in the casing. A gastight cover supporting the electrode structure depending therefrom is constructed in part of transparent material to facilitate observation of the interior of the cell, and a syphon mechanism is provided for charging and discharging electrolyte. (auth)

ELECTROCHEMISTRY 6.07

8490
ELECTRODEPOSITION OF ACTINIDE ELEMENTS AT TRACER CONCENTRATIONS. Russell F. Mitchell (Tracerlab, Inc., Richmond, Calif.). Anal. Chem. 32, 326-8 (1960) Mar.

The electrodeposition of the actinides from an electrolyte of ammonium chloride–hydrochloric acid is a rapid method for recovering trace concentrations of these elements in high yield. The plating procedure produces deposits which are adherent and uniformly distributed over the area of the plate. The effects of such parameters as current density, pH of electrolyte, salt concentration, volume of electrolyte, and the time of plating on the electrodeposition are discussed. (auth)

146 KAPL-1762
Knolls Atomic Power Lab., Schenectady, N. Y.
BUBBLE STIRRING IN A LOW TEMPERATURE MODEL OF A FUSED ELECTROLYSIS CELL. R. E. Coffman and L. W. Niedrach. June 28, 1957. 22p. Contract W-31-109-Eng-52. $0.75(OTS).

The rapid settling of the oxide feed in the KAPL process for electrowinning U from its oxides causes operating problems. The possibility of making use of the anodically generated gases to stir the bath is considered as a means of overcoming the settling problem. Effects of variables on the efficiency of such stirring are described. (auth)

147 NAA-SR-1997
Atomics International Div., North American Aviation, Inc., Canoga Park, Calif.
SEPARATIONS CHEMISTRY QUARTERLY PROGRESS REPORT [FOR] JANUARY–MARCH 1957. G. E. Brand, A. G. Buyers, W. J. Gardner, and E. E. Motta. Sept. 30, 1957. 25p. Contract AT-11-1-GEN-8. $0.75(OTS).

Two multikilogram scale remotely operated experiments using irradiated and natural U were conducted to study fission product removal by oxide drossing. Analytical results are not yet available. The diffusion coefficient of Ce in molten U was found to be 1×10^{-4} cm^2/sec at 1200°C. A series of nonconsumable electrode arc melting experiments using Th–U alloys has been completed showing that Pa and U can be evaporated at temperatures over 2000°C. Thorium was electrolyzed from molten salt baths by using an Fe cathode and a molten Zn anode. A theoretical equation was derived relating the decontamination factor to the standard electrode potentials of Th and other elements. (auth)

5764 NP-6239
Rio de Janeiro. Centro Brasileiro de Pesquisas Fisicas and Brasil. Escola Nacional de Química, Rio de Janeiro.
ISOTOPIC EXCHANGE AND EVOLUTION OF METAL SURFACES IN ELECTROLITIC SOLUTIONS. U. Camerini, J. Danon, and M. Malagolowkin. Dec. 26, 1956. 19p. (Notas de Física Vol. III, No. 8).

Studies of isotopic exchange between several metals and electrolytic solutions are reported using Bi^{212} as tracer. (T.R.H.)

16374
STUDIES OF THE ANODIC BEHAVIOR OF ALUMINUM. II. COULOMETRY OF BARRIER LAYER PRODUCTION. Robert C. Plumb (Cambridge Univ., Eng.). J. Electrochem. Soc. 105, 498-502(1958) Sept.

In order to avoid solvent action by the electrolyte during anodic oxidation of aluminum, the electrolyte must have a high buffering capacity. With such an electyte the conversion of aluminum to aluminum oxide is essentially quantitative. The film thickness is proportional to the voltage drop across the film, and the proportionality constant does not depend on the length of time which the voltage is left applied. The film contains, as an essential part of its structure, a quantity of the anion from the electrolyte. The film appears to be nonstoichiometric, having excess aluminum during formation, the amount of excess decreasing with increased length of anodizing time. The coulombic transfer during film formation is quantitatively accounted for during formation by oxidation of aluminum to the trivalent state and liberation of oxygen gas. After the film growth has ceased, the current can be only partially accounted for by oxygen evolution, but it is thought that this is because of difficulty in nucleating oxygen bubbles. (auth)

2094
THEORETICAL ELECTROMOTIVE FORCES FOR CELLS CONTAINING A SINGLE SOLID OR MOLTEN CHLORIDE ELECTROLYTE. Walter J. Hamer, Marjorie S. Malmberg, and Bernard Rubin (National Bureau of Standards, Washington, D. C.). J. Electrochem. Soc. 103, 8-16(1956) Jan.

From thermodynamic data, electromotive forces have been calculated for reversible galvanic cells of the type $M/MCl_n/Cl_2(z)$ where M is a metallic element in the solid, liquid, or gaseous state and MCl_n is the correspond-

ing chloride in the solid or liquid state. Results are given for temperatures from 25 to 1500°C. The chlorides are listed in a series and compared with the electromotive force series of the elements in aqueous solutions. Comparisons are given with results obtained from galvanic cells with fused chlorides or from measurements of decomposition voltages. (auth)

82 AEC-tr-2640
AN INTRODUCTION INTO METHODS OF INVESTIGATION OF THE KINETICS OF ELECTRODE PROCESSES. Heinz Gerischer. Translated from Z. Elektrochem. 59, 604-12(1955). 18p.

Methods for the measurement of the speed of partial processes of an electrode reaction are reported. Topics such as the stationary current voltage curve, the unstationary switch-on processes with constant current, and constant potential are discussed. Examples are given to show how to determine the speed parameters of the essential phase boundary reaction. (J.H.M.)

7468 AECU-3962(Pt. III)
Michigan. Univ., Ann Arbor.
THE FARADAIC ADMITTANCE OF ELECTROCHEMICAL PROCESSES. III. EXPERIMENTAL TEST OF THE THEORETICAL EQUATIONS. Report No. 44. Henry H. Bauer, David L. Smith, and Philip J. Elving. Aug. 14, 1958. 26p. Contract AT(11-1)-70, Project 8. $4.80(ph), $2.70(mf) OTS.

An evaluation of the two sets of equations obtained to describe the behavior of a simple redox reaction under the influence of a small superposed alternating potential is presented. The cadmium system was investigated under a variety of conditions. While either set of equations may be applicable to results for a particular system involving fixed concentration of depolarizer and background medium, neither set is of general applicability, e.g., neither describes satisfactorily the variation of the phase angle with frequency and that of the heterogeneous rate constant with frequency and depo-

ON THE THEORY OF OXIDATION-REDUCTION REACTIONS INVOLVING ELECTRON TRANSFER. II. APPLICATIONS TO DATA ON THE RATES OF ISOTOPIC EXCHANGE REACTIONS. R. A. Marcus (Polytechnic Inst. of Brooklyn, N. Y.). J. Chem. Phys. 26, 867-71(1957) Apr.

The rates of some homogeneous isotopic exchange reactions in solution are considered in the light of a recently developed quantitative theory of redox processes. The relative importance of several factors influencing the rates of these reactions is discussed. These factors include the Coulombic repulsion between the ionic reactants and the extent of solvation of the ions. Free energies and entropies of activation of various reactions are calculated from the theory without the use of any adjustable parameters. The agreement with the experimental data is considered to be satisfactory. On the basis of the theory and of earlier experiments in heavy water an experimental method is tentatively suggested for distinguishing electron and atom transfer mechanisms. This method applies to halide-catalyzed exchange reactions of metal aquo-ions, and to other anion-catalyzed reactions of this type not involving breakable OH bonds. (auth)

RECENT DEVELOPMENTS IN FLUORIDE VOLATILITY PROCESSING OF REACTOR FUELS. R. K. Steunenberg, J. Fischer, S. Vogler, M. J. Steindler, M. Adams, G. Goring, R. C. Vogel, W. A. Rodger, W. J. Mecham, and W. B. Seefeldt. 32p. (A/CONF.15/P/539)

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

Development work on the processing of fuel alloys containing enriched U and Zr is reported. The alloy is dissolved at a temperature of 600°C in an approximately equimolar melt of NaF and ZrF_4, which is sparged with HF vapor. The resulting tetrafluorides of Zr and U dissolve in the melt as they are formed. The U is volatilized as the hexafluoride by sparging the melt with F_2 or BrF_5 vapor. Data on the dissolution rates and the U volatilization have been obtained in both laboratory and semi-works investigations. Equipment problems are discussed and a design as well as initial operating experience is presented of a semi-works scale dissolver-hydrofluorinator of all graphite construction. Corrosion data on metal and graphite under dissolution conditions are reported. Included are recent studies relating to fission product behavior in fluoride systems, the corrosion rates of certain metals in fluorine at elevated temperatures, and the handling of PuF_6 in the vapor phase.

20199 AAEC/E-27
WORK WITH ALKALI METALS. P. P. Pugachevich. Translated by H. J. de Bruin from Zhur. Fiz. Khim. 31, 2140-2(1957). 6p.

ELECTROCHEMISTRY 6.07

Methods are described for purifying alkali metals in a vacuum and production techniques for alkali metal amalgams. The glass devices used for these purposes have no vacuum taps, ground in stoppers or rubber tubes; thus the purity of the metals and amalgams is not affected by the vapor of the vacuum lubricant, water or otherwise. The alkali metal amalgams produced remained unaltered in quality through years of storage. (auth)

22104
RADIATION ELECTROCHEMICAL PROCESSES IN AQUEOUS URANIUM SALT SOLUTIONS. N. B. Miller, Ts. I. Zalkind, and V. I. Veselovskii (L. Ia. Karpov Physicochemical Inst.). p.117-22 of "Radiation Electrochemical Processes."

A study was made of the connection between radiation chemical conversions occurring in irradiated solutions and the electrochemical parameters of the system. The electrochemical processes arising during the action of radiation from radon and cobalt-60 on solutions of uranium salts in sulfuric and perchloric acids were investigated. The action of optical radiation on the same system was investigated in parallel. Due to differences in the electrochemical behavior of gold and platinum electrodes it was possible to obtain a stationary potential difference of ~0.8 to 0.85 v by the action of gamma radiation on uranyl salt solution. (C.H.)

544 KAPL-M-GFM-8
Knolls Atomic Power Lab., Schenectady, N. Y.
REPORT ON THE EVALUATION OF BP-85 INCONEL-TYPE COVERED ELECTRODE. G. F. McKittrick and W. A. Owczarski. June 26, 1959. 18p. OTS.

The development and production of a new Inconel-type covered electrode capable of meeting higher quality standards required for radioactive system service were undertaken. This electrode is intended for welding Inconel to itself, Inconel to dissimilar metal, and carbon steel overlaying. Results from an evaluation of the BP-85 are summarized. (W.D.M.)

FLUIDIZED BEDS 6.08

See also, this section,

SPECIAL PROCESSES--Fluorox (6.17.09),

and,

under FISSIONABLE MATERIAL--RECOVERY,

FLUIDIZED BEDS (3.02.09).

15
FLUIDIZATION TECHNIQUES AS APPLIED TO REACTOR FUEL PROCESSING. S. Lawroski, A. A. Jonke, N. Levitz, and E. J. Petkus. 14p. (A/CONF.15/P/542)

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

Developments in fluidized-bed technology as applied to the processing of nuclear fuel materials, both irradiated and non-irradiated are discussed. Continuous conversion of recovered and decontaminated $UO_2(NO_3)_2$ solution to UO_3 in a fluidized-bed calciner was demonstrated in pilot scale equipment. Concentrated $UO_2(NO_3)_2$ solution is continuously atomized and fed to the hot agitated UO_3 bed, resulting in thermal decomposition of the $UO_2(NO_3)_2$. Recent studies show that from this UO_3 it is possible to produce UO_2 of high chemical reactivity by H reduction in a fluidized-bed reactor. The improved reactivity results from the ability to control and maintain uniform reaction temperatures in the fluidized-bed reactor. Multistage, fluidized-bed equipment for pilot-scale production of UO_2 and UF_4 has been designed and successfully operated. Use of such equipment permits countercurrent contact between reactant gases and solids, and allows temperature gradients to be used. The development of separation processes consisting of dissolution in aqueous media, evaporation in a fluidized-bed calciner, and fluorination in a fluidized-bed reactor are described. Separated UF_6 can then be decontaminated by proved methods employing fractional distillation. Work on application of this process to Pu-bearing fuels has also been initiated.

562 Y-1233
Union Carbide Nuclear Co. Y-12 Plant, Oak Ridge, Tenn.
DEVELOPMENT OF A CONTINUOUS COUNTER-CURRENT HIGH EFFICIENCY GAS-SOLIDS CONTACTOR. R. P. Levey, Jr., A. de la Garza, S. C.

FLUIDIZED BEDS 6.08

Jacobs, H. M. Heidt, and P. E. Trent. Sept. 3, 1958. 80p. Contract W-7405-eng-26. $2.25(OTS).

Experimental conversions of UO_3 to UO_2 and UO_2 to UF_4 in a batch fluidized bed pilot-scale contactor have demonstrated that conversion rates approaching those predicted from basic kinetic data are attainable. Further studies with fluidized beds in columns incorporating a very slight taper show that solids mixing with attendant product contamination is reduced by a factor of ten as a direct result of the taper. Experiments with tapered fluidized beds made the design of continuous (as opposed to the step enrichment of a multistage contactor) countercurrent contactors practical. Design procedures, in generalized form, are presented. The procedures take into consideration heat transfer, mixing losses, reaction kinetics, and chemical equilibria. (auth)

Fluidized Bed Reactor Studies, W. G. May, Chem. Eng. Prog. 55, 12, p. 49, Dec. 1959.

Scaling up from pilot plant units is discussed.

General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
FLUIDIZATION STUDIES WITH STEEL BALLS AND WATER. V. P. Kelly. Apr. 29, 1958. 54p. Contract [W-31-109-Eng-52]. $9.30(ph), $3.60(mf) OTS.

Fluidization studies were made of steel ball in water to determine the parameters which influence solids dispersion and to correlate fluidization data pertinent to fluidized-bed reactor design. Beds of $^3/_8$- and $^3/_4$-in. balls were fluidized in 6- and 9-in. pipes with water from 13 to 52°C. Density fluctuations were measured by a gamma absorption technique. Five conclusions were made from the results: (1) Beds of solids can be fluidized without channeling or slugging, although dispersion may not be absolutely uniform. (2) Optimum uniformity is obtained with approximately equal bed-depth and bed-diameter and with conical flow distributors. (3) Fluid viscosity change has little or no effect on fluidization quality and fluid velocity-bed expansion relationships. (4) Knowledge of physical laws governing fluid beds is inadequate for the selection of optimum parameters for a fluid-bed reactor. (5) Gamma absorptiometry is practical for the study of density fluctuations. (D.E.B.)

6396 AERE-R-2996
United Kingdom Atomic Energy Authority. Research Group. Atomic Energy Research Establishment, Harwell, Berks, England.
HEAT TRANSFER IN BEDS OF FLUIDISED PARTICLES: A LITERATURE SURVEY. H. Bannister. Oct. 1959. 64p. BIS.

A review is given of the literature of heat transfer in fluid beds with particular emphasis on gas supported systems since these are of greater industrial importance at present. Some mention is, however, made of liquid fluidization. The period covered is approximately 1940–59. (auth)

11366 UCRL-8602
California. Univ., Berkeley. Lawrence Radiation Lab.
A NEW CORRELATION FOR MASS TRANSFER IN THE FLOW OF GASES THROUGH PACKED BEDS AND FOR THE PSYCHROMETRIC RATIO. Edward J. Lynch and Charles R. Wilke. Feb. 10, 1959. 31p. Contract W-7405-eng-48. $1.00(OTS).

A new method is proposed for the correlation of mass transfer in the flow of gases through packed beds. This method is based on an empirical extension of the equations of Lin, Moulton, and Putnam and von Karman. The proposed equation agrees well with data on the vaporization from solids over a range of Schmidt numbers from 0.24 to 3.15. Modifications of the proposed equation appear to be applicable to mass transfer in packed columns with liquid flow and to psychrometric work. More extensive data will be necessary to confirm the suitability of these applications. (auth)

63 NP-6977
California Inst. of Tech., Pasadena. Guggenheim Jet Propulsion Center.
THE APPARENT ORDER OF HETEROGENEOUS SPRAY REACTIONS. Report No. 27. F. A. Williams. Sept. 1958. 8p. Contract DA-04-495-Ord-446.

The order and rate of a heterogeneous reaction involving liquid or solid particles in a gaseous medium are sometimes measured in devices which produce steady flow of the heterogeneous mixture. The objective of this study is to show that the apparent order of the heterogeneous reaction (with respect to the material composing the particles) obtained in this manner is primarily a function of the particle size distribution and is largely independent of the single-particle reaction order. (W.L.H.)

FLUIDIZED BEDS 6.08

1779
HEAT TRANSFER IN PACKED BEDS. ANALYTICAL SOLUTION OF TEMPERATURE PROFILES IN FIXED- AND MOVING-BED REACTORS AND HEAT EXCHANGERS. Andrew Pusheng Ting (Catalytic Construction Co., Philadelphia). Ind. Eng. Chem. 47, 2293-9(1955) Nov.

A mathematical study was made of temperature profiles in a gas-solids bed in exchanger or reactor tubes being heated or cooled externally. The gas streams may flow in either fixed or moving beds of solids. Analytic equations were developed for predicting temperature profiles in beds under the following conditions of heating or cooling on the shell side: con- and countercurrent shell fluid, shell fluid at constant temperature, and constant heat flux along tube height. Assumptions are rodlike gas flow, uniform gas velocity, constant effective thermal conductivity, existence of film resistance next to tube wall, and negligible thermal conduction in directions of material flows. Equations provide accurate solutions for uniform gas velocity; for nonuniform velocity average effective thermal conductivities should be used. (auth)

10058 IGRL-IB/CA-23
Gt. Brit. Capenhurst Works. Capenhurst, Ches., England.
HEAT TRANSFER IN STATIC GRANULAR BEDS. Information Bibliography. E. McCoy and A. J. Townend. May 1957. 6p.

6317
A FLUIDIZED BED PROCESS FOR THE PRODUCTION OF URANIUM TETRAFLUORIDE. N. M. Levitz, E. J. Petkus, H. M. Katz, and A. A. Jonke (Argonne National Lab., Lemont, Ill.). Chem. Eng. Progr. 53, 199-202(1957) Apr.

Advantages of the use of single and multistage fluidized beds over methods now in use are illustrated. Development of a process for improving the reduction and hydrofluorination operations in the production of UF_4 is presented. (W.L.H.)

11669
IMPROVEMENTS IN OR RELATING TO APPARATUS FOR THE TRANSFER OF GRANULAR OR OTHER FINELY DIVIDED SOLID MATERIAL. Robert Lipscomb (to United Kingdom Atomic Energy Authority). British Patent 812,189. Apr. 22, 1959.

A valve is described which will permit transfer of powders or grains from one area to another without the two areas being interconnected. Two conical plates are arranged on a movable axis so that in the "up" position one plate engages a knife edge and in the "down" position a knife edge of the lower plate engages a seal surface. Thus, one area is connected with the valve's intermediate chamber at a time. (T.R.H.)

11565 ANL-SL-SL-1039
Argonne National Lab., Lemont, Ill.
COMMENTS ON "APPLICATION OF FLUIDIZATION TO NUCLEAR TECHNOLOGY." Ju Chin Chu. Apr. 4, 1956. Decl. Mar. 16, 1957. 52p. Contract [W-31-109-eng-38]. $9.30(ph OTS); $3.60(mf OTS).

The preparation of UO_3 with high reactivity towards hydrofluorination is described. The reaction rate is correlated with process conditions and physical characteristics of the system for conversion of UO_3 to UF_6. Design data are developed for multiple stage fluidization, and the volume of fluidized bed is determined. Heat transfer coefficients of submerged surface in a fluidized bed are determined. Uniform distribution of fluidizing gas is predicted by pressure drop and solid-flow gradient calculation. The calcination of radioactive waste by fluidization with steam is reported. A process recommendation is made for production of UF_4 by fluidizing UO_3 with gaseous NH_3 and HF. The energy balance in a fluidized reactor is derived. (L.T.W.)

10697 ✓
LIQUIDLIKE PROPERTIES OF FLUIDIZED SYSTEMS. Junji Furukawa and Tsutomu Ohmae (Kyoto Univ.). Ind. Eng. Chem. 50, 821-8(1958) May.

Although many studies of fluidization are available for use in design or operational procedures, the chemical engineering approach does not lead to a true understanding of a fluidized particle system. In this investigation, the nature and properties of a liquid system are used as a basis for deriving equations applicable to a fluidized system. Correlations based on experimental data were found for expansion, viscosity, surface tension, and miscibility. (auth)

FLUIDIZED BEDS 6.08

11296 KY-240
Union Carbide Nuclear Co. Paducah Plant, Ky.
FLUIDIZATION IN TAPERED BEDS. W. R. Rossmassler and R. L. Harris. Feb. 14, 1958. 12p. Contract W-7405-eng-26. $0.50(OTS).

The concept of a tapered fluidized bed is explored briefly. Data are given showing that in bench-scale apparatus a tapered bed gives better fluidization with some materials that are difficult to fluidize in a conventional cylindrical bed. (auth)

9223 ANL-5725
Argonne National Lab., Lemont, Ill.
STUDIES OF PARTICLE SIZE DISTRIBUTION IN FLUIDIZED BEDS. H. M. Katz. May 1957. 20p. Contract W-31-109-eng-38 $0.20(OTS).

A technique for the study of distribution of particle sizes in fluidized beds consisting of two particle sizes has been devised, and data have been obtained for a number of such systems. It has been shown that both static electricity in the bed and surface adsorption of moisture affect the particle size distribution. Experimental data indicate that the effects of these variables can be eliminated by use of a conducting column of conducting particles and the use of dry air for fluidization. In addition, a qualitative relationship between minimum fluidizing velocity for mixed particle sizes and pressure drop across the bed has been demonstrated. (auth)

8897
CALCULATE FLUIDIZED HEAT TRANSFER BY NOMOGRAPH. Chin-Yung Wen and Liang-Tseng Fan (West Virginia Univ., Morgantown). Chem. Eng. 64, No. 7, 254-7 (1957) June.

Methods are given for calculating heat transfer in fluidized beds using nomographs. (W.L.H.)

12790 AEC-tr-3648
FLOW PATTERN AND HEAT CONDUCTION IN FLUIDIZED BEDS. F. Fetting and E. Wicke. Translated from Dechema Monograph 24, 146-69(1955). 28p. $4.80(ph), $2.70(mf) JCL or LC.

The measured heat coefficient a as a function of the rate of flow u has been shown for glass beads in air as well as for activated charcoal in air and carbon dioxide. (W.L.H.)

8973 USNRDL-TR-134
Naval Radiological Defense Lab., San Francisco.
A FEASIBILITY STUDY FOR THE DESIGN OF AUTOMATIC COUNTING AND SIZING INSTRUMENTATION. L. D. Miller. Nov. 16, 1956. 43p. Projects NS 081-001 and NE 051-500.

A feasibility study of automatic counting and sizing instruments was made, by making a literature survey of methods as well as an evaluation of instruments in production. An instrument using an intercept length comparison method appears to be the most advantageous one at the present time. It allows fast counting and sizing of irregularly shaped particles in the size range of ~0.5 to 3000 microns projected diameter. (auth)

FLUORINATION 6.09

See also, this section,
FUSED SALTS--Fluorides (6.10.01.04),
and,
under HAZARDS AND PROTECTION,
FLUORINE (4.01.05),
and,
under FISSION PRODUCT, POISON, AND RADIOISOTOPE REMOVAL,
FLUORINATION AND HYDROFLORINATION (2.19).

FLUORINATION 6.09

14234 NP-7516
TEPLOTY SUBLIMATSII TETRAFTORIDOV URANA, NEPTUNIYA, PLUTONIYA I AMERITSIYA. (Heat of Sublimation of Uranium, Neptunium, Plutonium, and Americium Tetrafluorides.) E. G. Chudinov and D. Ya. Choporov. [nd]. 3p.

The vapor pressure and heat of sublimation of active element fluorides were studied in relation to the pyrometallurgical treatment of nuclear fuels. A modified Knudsen method was used for measuring the tetrafluoride vapors. Radiometric measurements of the quantity of evaporated materials were made starting with the specific activity of the investigated materials. Measurements were made on UF_4 (using the isotopes of U^{233} and mixtures of U^{233} and U^{238}), NpF_4 and PuF_4 (using isotopes of Np^{237} and Pu^{239}), and AmF_4 (using Am^{241}). The tables of sublimation heat and entropy calculated from the obtained vapor pressure values are presented, including data on ThF_4. (R.V.J.)

13774 NACA-RM-E58D11
Lewis Flight Propulsion Lab., Cleveland.
COMPATIBILITY OF METALS WITH LIQUID FLUORINE AT HIGH PRESSURES AND FLOW VELOCITIES. Harold W. Schmidt. July 15, 1958. 16p.

Nickel, stainless steel, brass, and aluminum were evaluated for compatibility with liquid fluorine at flow velocities up to approximately 400 ft/sec and pressures up to 1500 lb/sq in. gage. Configurations were varied to produce severe turbulence and impact effects. Two rotating-vane flowmeters were also tested. None of the metals exhibited any measurable physical or chemical changes. In a run made with Teflon, fluorine reacted violently. (auth)

112 NACA-RM-E56K21
Lewis Flight Propulsion Lab., Cleveland.
MATERIAL COMPATIBILITY WITH GASEOUS FLUORINE. Harold G. Price, Jr., and Howard W. Douglass. Nov. 19, 1956. 5p.

Static tests on the compatibility of fluorine with nonmetals at atmospheric temperature eliminated many materials from further consideration for use in fluorine systems. Several materials were found compatible at atmospheric pressure. Only Teflon and ruby (aluminum oxide) were compatible at 1500 psig. (auth)

2610
CATALYSTS FOR FLUORINATION. (to United Kingdom Atomic Energy Authority)(U.S.A.). British Patent 794,022. Nuclear Eng. 3, 510(1958) Nov.

When fluorine reacts with hydrocarbon the heat produced tends to establish large temperature differences between different parts of the catalyst mass which slows up the reaction. By plating a metal (copper) inert in fluorine with silver, cobalt, manganese, or cerium a good catalyst material is produced whose high thermal conductivity reduces the temperature differences.

11730 K-532
Carbide and Carbon Chemicals Corp. K-25 Plant, Oak Ridge, Tenn.
STABILITY OF FREON-113 WITH FLUORINE. A. V. Faloon and J. D. Gibson. Dec. 16, 1949. 5p. Contract [W-7405-eng-26]. $0.10(OTS).

The stability of Freon-113 (1,1,2-trichloro-1,2,2-trifluoroethane) with fluorine was studied for the purpose of determining the feasibility of using it as a solvent in the direct fluorination of various compounds. Freon-113 was treated with a fluorine–nitrogen mixture at a temperature of 80°C, at atmospheric pressure, and a contact time of 3 minutes. Runs were made using 20, 40, and 60% fluorine. A copper cylinder filled with copper shot to give a free space of 55.0% was used as the reactor. Under the conditions investigated Freon-113 was found to be stable and can be used as a solvent for compounds undergoing treatment with elemental fluorine. (auth)

FORCE CONSTANTS OF METAL HEXAFLUORIDES. Howard H. Claassen (Argonne National Lab., Lemont, Ill.). J. Chem. Phys. 30, 968-72(1959) Apr.

Equations are given relating fundamental frequencies and force constants for a general quadratic potential function for XY_6 molecules of symmetry O_h. Tables of force constants are given for hexafluorides of Mo, W, Re, Os, Ir, Pt, U, Np, and Pu. (auth)

7388
RECOVERY OF URANIUM VALUES FROM WASTE MATERIAL. (to U. K. Atomic Energy Authority (U.S.A.)). British Patent 768,831. Nuclear Eng. 2, 224(1957) May.

FLUORINATION 6.09

The material is hydrofluorinated to convert metal values to non-volatile fluorides. The residue is reacted with fluorine gas (at 400 to 500°C) forming uranium hexafluoride vapors which may be separately condensed. The process can be used for recovering uranium from sweepings, metal pieces (bolts, nuts) copper or other metallic parts of the isotope separating apparatus, etc. Fluorination is carried out on trays in a chamber constructed of fluorine resistant material (nickel, Monel metal) in the form of a muffle furnace into which hydrofluoric acid, fluorine and an inert gas (nitrogen, helium) are introduced as required. The inert gas is used for flushing between the treatment with acid and the treatment with fluorine. (auth)

10359 NLCO-729
National Lead Co. of Ohio, Cincinnati.
A LITERATURE SURVEY ON THE PHYSICAL CHEMISTRY, ANALYSIS AND RECOVERY AND/OR DISPOSAL OF HYDROFLUORIC ACID. Earl W. Mautz. Apr. 4, 1958. 25p. Contract AT(30-1)-1156. $0.75(OTS).

A listing of literature references relating to the physical chemistry, analysis, and recovery and/or disposal of anhydrous and aqueous hydrofluoric acid is reported. (auth)

10830 NYO-2048(Del.)
New Brunswick Lab., AEC, N. J.
SULFURIC ACID EXTRACTIVE DISTILLATION PROCESS FOR THE RECOVERY OF ANHYDROUS HYDROFLUORIC ACID FROM BY-PRODUCT AQUEOUS HYDROFLUORIC ACID. Preliminary Process Design and Economic Study. R. W. Legassie and J. P. Termini. Feb. 1954. Decl. with deletions Feb. 27, 1957. 29p. $4.80(ph OTS); $2.70 (mf OTS).

Laboratory and bench-scale developmental work at the New Brunswick Laboratory has established the technical feasibility of recovering anhydrous HF by the extractive distillation of aqueous 70 wt. % HF with H_2SO_4 in a packed tower. Anhydrous HF is removed as an overhead product while the bottoms, consisting of approximately 50 wt. % H_2SO_4 and less than 1 wt. % HF, is fed to a concentrator to produce a 50 wt. % H_2SO_4 for recycling. A preliminary process design and an estimate of the investment and operating costs are presented. The cost for concentrating aqueous 70 wt. % HF in this plant is estimated to range from $0.031 to $0.035 per pound of acid without amortization and from $0.047 to $0.053 per pound including a five year amortization. (auth)

1794 ORNL-2038
Oak Ridge National Lab., Tenn.
A COMBINED DISTILLATION-ELECTROCHEMICAL

METHOD FOR RECOVERY OF HYDROFLUORIC ACID. J. A. Marinsky and A. J. Giuffrida. May 25, 1956. Decl. Sept. 6, 1956. Contract W-7405-ENG-26. $0.30(OTS).

Electrodialysis in an ion-exchange membrane cell was shown to be technically feasible for the concentration of an azeotropic mixture of HF and water. A flowsheet is presented for recovery of anhydrous HF by distillation and electrodialysis of the azeotropic residue. In the electrodialysis step, 2.6 kwhr of energy per pound of anhydrous product was consumed. With electricity at 1¢ per kilowatt-hour, the total operating cost of the electrodialysis equipment alone, including amortization, would be 6¢ per pound of HF. (auth)

FUSED SALT AND LIQUID METAL SYSTEMS 6.10

See also, under FISSIONABLE MATERIAL--RECOVERY

RECOVERY OF IRRADIATED TH AND U--Fused Salts and Liquid Metals (3.02.11),

RECOVERY OF PU--Fused Salts and Liquid Metals (3.03.07),

and, — Fluorination (3.03.00)

under PLANTS AND EQUIPMENT,

HIGH TEMPERATURE SYSTEMS (5.10).

FUSED SALT AND LIQUID METAL SYSTEMS 6.10

FUSED SALTS 6.10.01

CHLORIDES 6.10.01.01

9991
PREPARATION OF PURE FUSED LITHIUM CHLORIDE–POTASSIUM CHLORIDE EUTECTIC SOLVENT. H. A. Laitinen (Univ. of Illinois, Urbana), W. S. Ferguson (The Ohio Oil Co., Littleton, Colo.) and R. A. Osteryoung (Rensselaer Polytechnic Inst., Troy, N. Y.). J. Electrochem. Soc. 104, 516-20(1957) Aug.

Hydrolytic decomposition occurs during the fusion of a eutectic mixture of lithium chloride-potassium chloride containing traces of moisture if the fusion conditions are not controlled. The resultant contamination by hydroxyl ion greatly lowers the utility of this mixture as a fused salt solvent. The effectiveness of various procedures used for preparation of the fused salt solvent was followed by observation of the characteristic polarographic residual current using a platinum microelectrode. A preparative method is described which involves drying the mixture under moderate vacuum, fusion under anhydrous hydrogen chloride, and removal of the hydrogen chloride from the melt. (auth)

5060 AECU-4544
Carnegie Inst. of Tech., Pittsburgh. Metals Research Lab.
STUDY OF EQUILIBRIUM BETWEEN METALS AND THEIR POLYVALENT CHLORIDES IN LiCl–KCl EUTECTIC MELT. Ling Yang, Robert G. Hudson, and Chien-yeh Chien. [1959]. 27p. OTS.

Dilute solutions of uranium, thorium, zirconium, and columbium chlorides in LiCl–KCl eutectic melts were made by anodic dissolution of these metals. After equilibrium was reached between the solution and the metal, the electrode potential was measured relative to a AgCl reference electrode. The nature of the metal ions in the equilibrated melt was determined either from the slope of the isothermal plots of potential versus the logarithm of concentration, or by using Faraday's law. It was found that in the temperature range of 760 to 830°K, U^{3+}, Th^{4+}, Zr^{4+}, and Nb^{3+} are the predominant species existing in the uranium chloride (0.26 to 2.08% U), thorium chloride (0.19 to 1.47% Th), zirconium chloride (0.07 to 0.86% Zr) and niobium chloride (0.13 to 0.43% Nb) melts, respectively. For niobium chloride melts of higher concentrations than that shown above, other species of niobium ions, probably Nb^{4+}, coexists with Nb^{3+} in appreciable amounts. The results are explained on the basis of the magnitude of the free energy of formation of the chlorides of these metal ions of different valence states and the tendency for these metal ions to form complex ions with the chloride ions in the melt. (auth)

7299 AECU-4551
Massachusetts Inst. of Tech., Cambridge. Lab. for Nuclear Science.
A NEW METHOD FOR PREPARING HYDROXIDE-FREE ALKALI CHLORIDE MELTS. Donald L. Maricle and David N. Hume. [1959]. 8p. Contract AT(30-1)-905. OTS.

Molten salts have been used extensively as solvents for electrochemical processes. The difficulty of removing the last traces of residual moisture from the salts has often necessitated rather arduous drying procedures. If the moisture is not removed, hydrolysis occurs upon fusion. The hydroxide ion thus produced precipitates metal ions, attacks glass, and is reduced more easily than the alkali metals, thus diminishing the usable potential range of the solvent. It was found that if rigorously dried hydrogen chloride is bubbled through the melt for twenty minutes before the argon flush, the hydroxide ion impurity is removed. (W.L.H.)

3786 NAA-SR-202
North American Aviation, Inc., Downey, Calif.
AN EXPLANATION OF THE EFFECT OF ADDED METALS ON THE DISTRIBUTION OF RARE EARTHS BETWEEN LIQUID BISMUTH AND KCl–LiCl. D. Cubicciotti. Jan. 8, 1953. Decl. Nov. 9, 1955. 10p. Contract AT-11-GEN-8. $0.15(OTS).

An explanation is proposed of experimental results obtained on the distribution of rare earths between liquid Bi and molten LiCl–KCl. It is shown that the mass-action law predictions of the distribution agree with experimental observations, if it is assumed that the equilibrium governing the distribution is the partial reduction of the LiCl by the rare earth metal. The distribution of the rare earth is affected by added metals inasmuch as they affect the equilibrium. (auth)

FUSED SALT AND LIQUID METAL SYSTEMS 6.10

FUSED SALTS 6.10.01

CHLORIDES 6.10.01.01

2929
PHASE DIAGRAM AND VAPOR PRESSURE IN THE SYSTEMS $NaCl-ZrCl_4$, $KCl-ZrCl_4$, AND NaCl-KCl (1:1 MOLAR)-$ZrCl_4$. L. J. Howell and H. H. Kellogg (Columbia Univ., New York) and R. C. Sommer (American Smelting and Refining Co., South Plainfield, N. J.). J. Metals 9, 193-200(1957) Jan.

Portions of the phase diagrams of the systems $NaCl-ZrCl_4$, $KCl-ZrCl_4$, and NaCl-KCl (1:1 molar)-$ZrCl_4$ have been determined. Particular attention was given to locating compositions which might be useful for electrodeposition of Zr metal. The vapor pressure of $ZrCl_4$ in equilibrium with the condensed phases at various temperatures has been measured by a dew-point technique. Several melt regions which show promise as potential electrolytes for deposition of Zr are described. (auth)

10004
THE REDUCTION OF Th^{4+} IONS IN CHLORIDE MELTS BY METALLIC THORIUM. M. V. Smirnov and L. E. Ivanovskiĭ (Sverdlovsk Acad. of Sciences). Zhur. Fiz. Khim. 31, 801-7(1957) Apr.

Studies of thorium reactions with chloride melts containing 6 to 11% by weight $ThCl_4$ showed that at 500 to 900°C the metal dissolves in the melts reducing Th^{4+} to Th^{2+}. The thorium potential and the potential of an indifferent molybdenum electrode were measured against a chlorine electrode. Calculations were made of the temperature dependence of constants in thermodynamic equations for the state of equilibrium potentials. (R.V.J.)

3324 AD-98464
Columbia Univ., New York. School of Mines.
PHYSICAL CHEMISTRY OF NaCl-KCl MELTS CONTAINING DISSOLVED TITANIUM CHLORIDES. Final Report [for] February 1, 1953 to April 30, 1956. Warren C. Kreye. June 1956. 39p. Contract Nonr-266 (24).

11295 ISC-929
Ames Lab., Ames, Iowa.
SOLUBILITY OF WATER IN MOLTEN ALKALI CHLORIDES. William J. Burkhard and J. D. Corbett. July 1957. 50p. Contract W-7405-eng-82. $1.50(OTS).

The solubility of water in molten mixtures of lithium chloride and potassium chloride containing 50.0, 53.0, 60.0, and 68.6 mole % lithium chloride was determined at 480 and 390°C in the pressure range zero to 26 mm. Melts were dried by alternate hydrogen chloride flushing and evacuation, and the solubility was determined by measuring the quantity of water vapor absorbed by the melt. The solubility versus pressure curves follow Henry's law from zero to 18 mm pressure at 480° and from zero to 10 mm at 390°. The reciprocal Henry's law constants, K, are 14, 11.8, 11.3, and 10.8 micromoles water per mole lithium chloride per mm for the

10007
SOLUBILITY OF IRON IN FUSED MIXTURES OF LiCl-KCl and NaCl-KCl. V. P. Kochergin and G. I. Stolyarov. Zhur. Priklad. Khim. 29, 730-3(1956) May. (In Russian)

Investigations were made to reveal the mechanisms of iron solubility in melted electrolytes and the rate of iron solubility in fused salts of LiCl-KCl and NaCl-KCl. Results show that in transition from salt eutectics of LiCl-KCl to the easily melting mixture of NaCl-KCl, the rate of iron solubility is reduced. An increased rate of iron solubility with the increase of temperature in fused chlorides of alkali metals has been observed. An analytical method of determining iron solubility in a fused eutectic containing hydrogen is discussed. (R.V.J.)

57 CCC-1024-TR-139
Richmond, Va. Univ.
STUDY OF THE SYSTEM $NaCl-KCl-LiCl-AlCl_3$ IN THE REGION OF MOLE RATION 1:1:1:(3-6). W. E. Trout, Jr. and W. J. Triner, Jr. Oct. 3, 1955. 29p.

Preliminary studies have been made of the phase diagram of the system $NaCl-KCl-LiCl-AlCl_3$ in the region of mole ratios 1:1:1:(3-6), i.e., with variation only in the ratio of $AlCl_3$ to an equimolar mixture of the other three salts. Techniques were developed for loading fusion cells, and cooling curves of mixtures were obtained. In each case the temperature at which solid first appeared was noted. A low-melting region was found to lie between 1:1:1:4.1 and 1:1:1:4.5. The lowest point observed was at 62°C for the mixture 1:1:1:4.24. (auth)

9994
THERMODYNAMICS OF MOLTEN MIXTURES OF ALKALINE METAL CHLORIDES AND THORIUM TETRACHLO-

FUSED SALT AND LIQUID METAL SYSTEMS 6.10

FUSED SALTS 6.10.01

CHLORIDES 6.10.01.01

RIDE. W. V. Smirnov and L. E. Ivanovskiĭ (Sverdlovsk Electrochemical Lab. Acad. of Science). Zhur. Fiz. Khim. 31, 640-7(1957) Mar. (In Russian)

The equilibrium potentials of Th–Zn alloy electrode against a chlorine electrode were measured in molten eutectic mixtures of Li and K chlorides containing 2.98 to 40 × 16% by weight $ThCl_4$ and in pure molten $ThCl_4$ at 840°C. It was observed that melts with less than 25% by weight of $ThCl_4$ behave as ideal solutions. Equations were developed for the temperature and concentration dependence of the electrode potential Th/Th^{+4} in molten mixtures of alkaline metal chlorides of less than 25% by weight $ThCl_4$ with respect to the chlorine electrode. (R.V.J.)

3477 NAA-SR-3940

Atomics International Div., North American Aviation, Inc., Canoga Park, Calif.

THE SOLUBILITY OF SALTS IN METALS. S. J. Yosim and E. B. Luchsinger. Aug. 1, 1959. 15p. Contract AT-11-1-GEN-8. OTS.

A series of experiments to determine the solubilities of metal chlorides in their metals was carried out using a visual technique. The systems studied included Bi, Hg, Pb, Cd, Zn, Sn, In, Ag, and Cu with their chlorides. The results show that $BiCl_3$ and Hg_2Cl_2 are most soluble, the former being completely miscible with Bi at 778°C, while the latter is soluble to the extent of 7 mole % at 600°C. $PbCl_2$ is slightly soluble (1 mole % at 1,000°C), while the remaining salts are considered insoluble in their metals. There does not appear to be any single criterion for predicting the solubility of salts in their metals. Experiments were also carried out to see if nonspecificity of solubility exists at high temperatures–i.e., to determine if metals dissolve salts other than their own halides. It was found that solubility does occur. It was also found that the less stable the salt, the greater is the solubility of the salt in a given metal, until the solubility approaches that of the salt of the metal. Thermodynamic calculations indicate that the solubility of a foreign salt in a metal can be explained in terms of oxidation-reduction reactions to form the halide of the metal, which in turn dissolves in the metal. These calculations suggest that $BiCl_3$ dissolves in bismuth metal as BiCl. (auth)

13723

EQUILIBRIUM POTENTIALS OF ZIRCONIUM IN CHLORIDE MELTS. M. V. Smirnov and V. A. Chemezov (Electrochemical Lab., Urals Branch of the Academy of Sciences, USSR). Doklady Akad. Nauk S.S.S.R. 120, 122-5(1958) May 1. (In Russian)

The dependence of the equilibrium potential of zirconium on temperature and ion concentration in chloride melts was investigated. (R.V.J.)

EQUILIBRIUM POTENTIALS OF TITANIUM IN CHLORIDE MELTS. M. V. Smirnov, L. E. Ivanovskii, and N. A. Loginov (Electrochemical Lab., Urals Branch of Academy of Sciences, USSR). Doklady Akad. Nauk S.S.S.R. 121, 685-8(1958) Aug. 1. (In Russian)

The electromotive force of $Ti/TiCl_2$, LiCl, KCl(melt)/LiCl, KCl(melt)/8.05% $PbCl_2$, LiCl, KCl(melt)/Pb was measured at 410 to 710° with titanium concentrations of 0.2, 0.32, 0.49, 0.94, 5.3, and 5.7 wt. % (which correspond to concentrations in mole units of 4.66×10^{-4}, 7.45×10^{-4}, 1.14×10^{-3}, 2.19×10^{-3}, 1.25×10^{-2}, and 1.35×10^{-2}). A diagram of electromotive force between titanium and chloride was plotted with empirical equations for 700, 800, 900, and 1000°K. (R.V.J.)

4477

EQUILIBRIUM BETWEEN TITANIUM METAL, TITANIUM DICHLORIDE, AND TITANIUM TRICHLORIDE IN MOLTEN SODIUM CHLORIDE-STRONTIUM CHLORIDE MELTS. S. Mellgren and W. Opie (National Lead Co., South Amboy, N. J.). J. of Metals 9, 266-9(1957) Feb.

It has been demonstrated that an equilibrium exists between divalent, trivalent, and metallic titanium in a fused chloride system using sodium and strontium chlorides as solvents. This equilibrium is unaffected by temperature change between 650 and 800°C (within analytical error range of 5%) or by varying the total titanium concentration between 1.7 and 5.0% total titanium in solution. It is markedly affected by varying the $SrCl_2$ to NaCl mole ratio of the solvent, the divalent titanium at equilibrium decreasing with increasing NaCl content. The solutions studied are not ideal. (auth)

4274

SOME PHYSICAL PROPERTIES OF MOLTEN AND SUPERCOOLED GALLIUM TRICHLORIDE. N. N. Greenwood and

FUSED SALT AND LIQUID METAL SYSTEMS 6.10

FUSED SALTS 6.10.01

CHLORIDES 6.10.01.01

K. Wade (The Univ., Nottingham, Eng.). J. Inorg. and Nuclear Chem. 3, 349-56(1957) Jan.

The viscosity of molten gallium trichloride has been measured both above and below the melting-point, and a small discontinuity in slope of the log $\eta - 1/T$ curve has been found near the melting-point despite the symmetrical and nonpolar nature of the gallium-trichloride dimer. The density and surface tension have also been determined over a range of temperature and the results used to calculate the parachor and related properties of the compound. (auth)

1862 BNL-345
Brookhaven National Lab., Upton, N. Y.
LIQUID METAL FUEL REACTOR FUEL PROCESSING STUDIES. C. Raseman, R. Bauman, and J. Weisman, comps. Jan. 1955. Decl. Mar. 18, 1957. 22p. (LMFR-9). $0.75(OTS).

Small scale studies and loop tests were carried out on Bi – Ce – U/LiCl – KCl systems to estimate the effect of U and Ce concentration on the respective distribution coefficients, the effect of metal-metal ion additives, the effect of redox additives, and the distribution coefficients of the nonvolatile fission products obtained by pile irradiation. The corrosion of stainless steel by liquid Bi – U alloys was investigated. (C.W.H.)

12246 ✓
STUDY OF FUSED SALTS AT ELEVATED TEMPERATURES BY X-RAY DIFFRACTION. II. STRUCTURE OF THE LIQUID STATE OF THE CHLORIDES LiCl, NaCl, KCl, $BaCl_2$ AND OF THE FLUORIDE CaF_2. GENERAL CONSIDERATIONS ON THE STRUCTURE OF FUSED HALOGENIDES. Jerzy Zarzycki (Centre de Recherches des Glaceries de Saint-Gobain, France). J. phys. radium 19, Suppl. No. 4, 13A-19A(1958) Apr. (In French)

The analysis of the radial distribution curves deduced by Fourier transform from the x-ray diffraction spectra of molten salts confirms the results already obtained for alkali fluorides. In the liquid state the low-range order of the crystal is conserved but the first coordination number is much lower. This proves the lacunar structure of molten halides. It is shown that the holes are actually "fluctuating cracks" between ionic clusters grouping 1-2 ionic shells around a central ion. (auth)

7933 ✓ ORNL-2323
Oak Ridge National Lab., Tenn.
ACTIVITIES IN THE KCl – $FeCl_2$ AND LiCl – $FeCl_2$ SYSTEMS. C. Beusman. May 15, 1957. 106p. Contract W-7405-eng-26. $0.55(OTS).

Phase equilibria in the $FeCl_2$ – KCl and the $FeCl_2$ – LiCl systems were studied by means of thermal analysis and vapor pressure measurements. The solid-liquid equilibria were studied by simultaneous conventional cooling curves and differential cooling curves. Petrographic and x-ray analysis helped identify two compounds in the $FeCl_2$ – KCl system: K_2FeCl_4 and $KFeCl_3$. The $FeCl_2$ – LiCl system showed no compounds, and consisted of continuous solid solution formation. By combining total vapor pressure measurements with transportation measurements, average molecular weight values were calculated for the pure salt vapors studied, as well as for vapors from binary mixtures From these data, vapor phase association was found to exist not only in pure salt vapor, but also in the vapor mix-

10384 ✓
STUDY OF INTERACTION OF CHLORIDES OF ALKALI EARTH METALS IN MELTS. III. STUDY OF INTERACTION OF SODIUM, POTASSIUM, CALCIUM, CESIUM, RUBIDIUM, AND LITHIUM CHLORIDES IN MELTS. V. Ye. Plyuchchev, L. N. Komissarova, L. V. Meshchaninova, and L. M. Akulkina. Zhur. Neorg. Khim. 1, No. 4, 820-33 (corrections in No. 12, 2874) (1956). (Translated from Referat. Zhur. Khim. No. 8, 1957, Abstract No. 26145.)

7158 ✓
URANIUM DIOXIDE ANODES IN MELTED CHLORIDE ELECTROLYTES. M. V. Smirnov and L. E. Ivanovskiĭ. (Ural Div., Academy of Sciences, U.S.S.S.R.). Zhur. Obshcheĭ Khim. 27, 295-9(1957) Feb. (In Russian)

Electrodes prepared from UO_2 were used as anodes in the electrolysis of fused molten chlorides at current densities up to 3 A/cm^3. Investigations were made of the behavior of UO_2 anodes in molten K and Li chlorides, and polarization of the anodes at 550° was measured. A suggestion has been offered that UO_2 electrodes behave in melts identical to metallic anodes. Their potential can be determined by the UO_2^{++} mole-mg concentration in electrolyte. (R.V.J.)

FUSED SALT AND LIQUID METAL SYSTEMS 6.10

FUSED SALTS 6.10.01

CHLORIDES 6.10.01.01

12672 AERE-Lib/Trans-760
REACTION OF THORIUM OXIDE WITH ITS CHLORIDE IN MOLTEN SALT SOLUTIONS. M. V. Smirnov and L. E. Ivanovskii (Ivanowskii). Translated by S. Botcharsky from Zhur. Neorg. Khim. 1, 1843-9(1956). 12p.

The reaction between ThO_2 and its chloride was studied at 840°C temperature. The formation of $ThOCl_2$ was investigated by heating the oxide in a molten eutectic mixture of Li and K chlorides with varying content of ThCl. It has been found that $ThOCl_2$ is practically insoluble in molten chloride or fluoro-chloride solutions and that in molten chloride solutions in contact with $ThOCl_2$ the thoryl ions (ThO^{2+}) are not present. (R.V.J.)

7253
CONVERSION MEASUREMENT FOR THE DETERMINATION OF THE MOBILITY OF Li^6, Li^7, AND Pb IN MOLTEN $LiCl-PbCl_2$ MIXTURES. A. Klemm and E. U. Monse. Z. Naturforsch. 12a, 319-34(1957) Apr. (In German)

14227 ISC-1071
Ames Lab., Ames, Iowa.
TRANSPORT NUMBERS IN FUSED SILVER CHLORIDE–ALKALI METAL CHLORIDE SYSTEMS. Allen Lee Bowman and F. R. Duke. Aug. 1958. 41p. Contract W-7405-eng-82. $7.80(ph), $3.30(mf) OTS.

The transport number of the Cl^- ion was determined directly by following the migration of Cl^{36} in the alkali metal chlorides, AgCl, and mixtures of AgCl with LiCl, NaCl, and KCl. The transport numbers of the cations were determined by measurement of Φ or by following the migration of Ag^{110m}. Also a set of equations is proposed with adjustable parameters to describe the conductivity, fluidity, self-diffusion, and transport numbers in a pure fused salt. (J.R.D.)

16836
TRANSPORT NUMBERS IN PURE FUSED SALTS. THE ALKALI METAL CHLORIDES. Frederick R. Duke and Allen L. Bowman (Inst. for Atomic Research, Iowa State College, Ames). J. Electrochem. Soc. 106, 626-7 (1959) July.

The transport number of the chloride ion has been determined in fused alkali chloride salts by a radiochlorine tracer technique, using a membrane cell. Values obtained are compared with theoretical predictions of Mulcahy and Heymann and of Sundheim. (auth)

SOME EXPERIMENTS ON THE FUSED SALT PROCESSING OF LIQUID METAL FUELS. J. K. Higgins. June 1958. 9p. (AERE-M/M-194)

The reaction between a fused NaCl, KCl, $MgCl_2$ eutectic and a liquid Bi solution containing small amounts of a rare earth Ce (~100 ppm by wt) and U (~1000 ppm by wt) is considered. Experimental results are given for the transfer of Ce and U from the metal to the salt phase in a silica container. It is concluded that contamination from this container presents serious difficulties to an electrolytic method for the processing of liquid metal fuels.

THERMODYNAMIC PROPERTIES OF SOME FUSED CHLORIDES. B. F. Markov, J. K Delimarsky, and I. D. Panchenko (Inst. of General and Inorganic Chemistry, Academy of Sciences of the Ukrainian S.S.R., Kiev). J. Polymer Sci. 31, 263-8(1958) Sept.

The stability of complexes and associates formed in compound molten electrolytes can be quantitatively estimated from the excess thermodynamic functions (free enthalpy, entropy, enthalpy). These functions can be most conveniently determined by measuring the electromotive force of chemical cells of the type $Me_I[Me_IX_m, Me_{II}X_n]\ X_2$, where X is halogen. In this way the following systems were experimentally investigated: $BeCl_2-NaCl$; $MgCl_2-LiCl$; $MgCl_2-NaCl$; $MgCl_2-KCl$; $MgCl_2-RbCl$; $PbCl_2-LiCl$; $PbCl_2-NaCl$; $PbCl_2-KCl$; $PbCl_2-RbCl$. The dependence of the excess partial free enthalpy on the mole fraction can be expressed by the equation $Z_1^E = \alpha(1 - X_1)^\beta$, where α and β are constants

3207 BNL-403
Brookhaven National Lab., Upton, N. Y.
LIQUID METAL FUEL REACTOR IN-PILE FUEL PROCESSING LOOP (LOOP B): CONSTRUCTION, OPERATION, EXPERIMENTAL RESULTS. C. J. Raseman, H. Susskind, and C. H. Waide. Jan. 1957. 44p. (LMFR-13). $1.50(OTS).

FUSED SALT AND LIQUID METAL SYSTEMS 6.10

FUSED SALTS 6.10.01

CHLORIDES 6.10.01.01

A molten solution of natural U in Bi at 500°C was circulated through the Brookhaven reactor in a type 347 stainless steel system. The U–Bi stream leaving the reactor was equilibrated with He gas and fused salts to determine distribution coefficients for certain fission products between metal and salt and metal and gas phases. The solubility of Xe at 500°C and 1 atmosphere (Xe) was found to be 0.4 ppm and to provide a value of 1.3×10^6 for the Henry's law constant k in the expression $p = kx$. The removal of the Xe by reduction of its partial pressure as a result of the removal of blanket gas at reduced pressure was shown to be within LMFR design requirements. More than half the I (the Xe precursor) in the loop was found to concentrate on the vessel walls. Other fission products were also studied to determine the degree of deposition on the walls. Salt contacting experiments were conducted with a LiCl-KCl eutectic and a $MgCl_2$-NaCl-KCl eutectic, and the results agreed favorably with previous small-scale tracer experiments. Indicative of these results are the ranges of the partition coefficients (ppm in salt/ppm in metal) for the following fission products and the $MgCl_2$-KCl-NaCl salt: Cs, 400 to 1020; Ba, 342 to 490; Sr, 125; Ce, 3.17 to 3.82; TRE, 4.07 to 7.55; Zr, 0.03 to 0.14; Ru, 0.9; I, 48 to 235; and Po, 0.73 to 0.76. These are compared to U, for separation purposes, with coefficients of 0.03 to 0.06. The loop was operated continuously in the reactor for 6446 hours, at the end of which shutdown was forced by a heater failure Leakage was discovered at several points in the system, and it was decided to remove the loop from the reactor. (auth)

21001 ✓

PRODUCTION OF OXYGEN-FREE AND ANHYDROUS FUSED SALT ELECTROLYTE FROM OXYGEN CONTAINING COMPOUNDS OF URANIUM. Milton J. Arnoff (to Horizons Titanium Corp.). U. S. Patent 2,887,356. May 19, 1959.

Fused U salt baths may be prepared oxygen-free from uranium compounds containing oxygen. Fused alkali metal chlorides, with or without alkaline earth chlorides are prepared and the U compound and a chlorinating agent added. The U is converted to chloride and the oxygen leaves as water or carbon dioxide. In one example a KCl–NaCl eutectic was heated to 700°C and $UO_2(NO_3)_2 \cdot 6H_2O$ was added. Water vapor and N oxides were released violently. Then HCl was added, and the mixture cooled and analyzed. Substantially all the U was converted to UCl_4. (T.R.H.)

21957 OOR-1486.13

Illinois. Univ., Urbana.

ELECTRODE REACTION KINETICS IN FUSED POTASSIUM CHLORIDE–LITHIUM CHLORIDE (thesis). Technical Report No. 6. D. K. Roe. July 15, 1959. 47p. Project No. 1486. Contract DA-11-022-ORD-1987.

The kinetic parameters of four electrode couples in fused KCl–LiCl eutectic at 450°C were experimentally measured using the voltage step method. A theoretical discussion of electrode kinetics is given along with the modifications from which the kinetic parameters are calculated by this method. Differences between the theoretical and experimental current-time curves, during the initial period of kinetic control ($< 2 \times 10^{-5}$ sec), are discussed with respect to the validity of the theoretical assumptions. A method of correcting for such differences was devised. The corrections proved to be significant for the slower electrode reactions which were investigated. Considerable attention was given to preparation and purification of the eutectic melt. The method adopted involved displacement of heavy metal ion impurities by metallic magnesium and filtration of the molten eutectic through sintered quartz. The experimental results show that some charge transfer reactions are very rapid, as might be expected in fused salts, but the rate of the bismuth couple was surprisingly slow. The kinetic parameters of molar exchange current density, transfer coefficient, and heterogeneous rate constant for each electrode couple are, respectively: Cd(II)/Cd(liq), 210 amps. $cm.^{-2}$, 0.13, 0.4 cm. $sec.^{-1}$; Zn(II)/Zn(liq), 150 amps. $cm.^{-2}$, 0.10, 0.4 cm. $sec.^{-1}$; Bi(III)/Bi(liq), 8 amps. $cm.^{-2}$, 0.48, 9×10^{-4} cm. $sec.^{-1}$; and V(III)/V(II), 30 amps. $cm.^{-2}$, 0.67, 0.3 cm. $sec.^{-1}$. The equipment used in the kinetic studies was also used to measure the differential double layer capacity of a platinum surface in contact with the melt. The variation of double layer capacity with potential indicates that chloride ions are specifically adsorbed over a span of about 0.6 volts. (auth)

FUSED SALT AND LIQUID METAL SYSTEMS 6.10

FUSED SALTS 6.10.01

CHLORIDES 6.10.01.01

AEC-tr-3382

ON SOLUTIONS OF METALLIC CADMIUM IN MOLTEN CHLORIDES. S. Karpachev and A. Stromberg. Translated for Atomics International from Zhur. Fiz. Khim. 13, 397-405 (1939). 12p. (Figures omitted).

In melted mixtures containing chlorides of potassium, sodium, and cadmium, the dependence of the potential of the carbon electrode on the concentration of the metallic cadmium dissolved in molten salts was investigated. It was found that depending on the concentration of the dissolved metal, the potential of the carbon electrode varies according to the usual thermodynamic equation. The dependence of the solubility of the metallic cadmium in molten mixtures of chlorides of potassium and sodium with cadmium chloride on the content of the latter was studied. (auth)

ELECTROCHEMISTRY 6.10.01.02

2988 / AECD-3700
Carnegie Inst. of Tech., Pittsburgh. Metals Research Lab.
ELECTROCHEMICAL SEPARATIONS IN NON-AQUEOUS SOLUTIONS. Occasional Report No. 3 for the Period June 1, 1953 to June 1, 1954. G. M. Pound, Gerhard Derge, June Fullmer, Earl Roland, and Joan Pacacha. June 1, 1954. Decl. with deletions Oct. 31, 1955. 44p. Contract AT(30-1)-1432. $0.30(OTS).

The electrolytic decomposition potentials of pure fission product metal chlorides in molten LiCl–KCl eutectic were measured directly by the current-voltage method as a function of temperature. Certain improvements in the technique relating to dehydration and oxygen removal permitted measurements for even the most electropositive metals. In most cases, the measured values are in reasonable agreement with the thermodynamically calculated values. This means that, in general, there were no large (>0.2 volt) effects due to over-voltage or non-ideality of

83 AEC-tr-2642
ON THE EFFECT OF THE ANION ON ELECTRODE POTENTIALS OF METALS IN FUSED SALTS. Yu. K. Delimarskii. Translated from Ukrain. Khim. Zhur. 21, 449-56(1955). 14p.

A scale of electrode potentials has been constructed in fused halides with the aid of the zero-electrode method. Electrode potentials of heavy metals during transition from chlorides to bromide to iodides to fluorides became more negative. The influence of anions on electrode potentials of heavy metals can be explained by the regular change in degree of electrolytic dissociation during transfer from chlorides to iodides, and also by the deformation of ions which occur as a consequence of one-sided or mutual polarization. (W.L.H.)

6722 ✓
ELECTROKINETIC EFFECTS IN LIQUID METALS.
G. E. Pikus and V. B. Fiks (Semiconductor Inst., Academy of Sciences, Leningrad). Soviet Phys.–Solid State 1, 972-80(1960) Jan.

The theory of electrokinetic effects in liquid metals is developed. These effects are caused by inelastic scattering of current carriers at the metal boundaries and lead to the creation of a transfer flow when an electric current is passed through liquid metal contained in a thin capillary, and to the creation of a transfer current when the liquid flows through the capillary. Using Onsager's relationships, formulas were obtained for the transfer current and transfer potential. (auth)

12665 ✓ IGRL-IB/R-13
United Kingdom Atomic Energy Authority. Industrial Group H. Q., Risley, Lancs, England.
ELECTROLYSIS OF FUSED SALTS. Information Bibliography. B. Yates, comp. Apr. 1956. 16p.

5750
THE ISOTOPE EFFECT OF A DIRECT ELECTRIC CURRENT THROUGH LIQUID AND SOLID METALS.
E. Haeffner, Th. Sjöborg, and S. Lindhe (AB Atomenergi, Stockholm, Sweden). Z. Naturforsch. 11a, 71-5(1956) Jan. (In English).

The isotope separation effect of a direct electric current in a liquid metal is demonstrated by passing a current through mercury, which is enclosed in a capillary tube. The second part of the paper deals with an attempt of establishing an isotope effect when a direct current is passed through an uranium wire. (auth)

FUSED SALT AND LIQUID METAL SYSTEMS 6.10

FUSED SALTS 6.10.01

ELECTROCHEMISTRY 6.10.01.02

10434 NBS-5205
National Bureau of Standards, Washington, D. C.
REFERENCE ELECTRODES FOR MOLTEN SALTS.
Progress Report [for] January 1 to March 31, 1957.
Vernon A. Lamb and Roger J. Labrie. Apr. 2, 1957. 9p.
NBS Project 0506-20-3280. $1.80(ph OTS); $1.80(mf OTS).

The properties of the electrode system Ag/AgCl–NaCl/porcelain, in which the porcelain is in the form of a sodium-ion-conductive capsule were studied. Experiments were carried out in which porcelain electrodes were intercompared and also measured against some of the previously developed electrodes in order to obtain data on the reproducibility, stability, range of usefulness, and durability of porcelain electrodes. (For preceding period see NBS-5100.) (W.L.H.)

6581 ✓
ELECTROMIGRATION OF RADIOACTIVE IONS IN FUSED SALTS. APPLICATION TO THE SEPARATION OF ISOTOPES. Harirao Jeevan Arniker (Université, Paris).
Ann. phys. (13) 4, 1291-1339(1959) Nov.-Dec. (In French)

The use of asbestos paper to support a liquid phase for chromatography and electrophoresis of fused salts is described. Ionic mobility studies using radioactive tracer studies are possible by this technique. Several ionic mobility studies are described. The technique was also applied to $Na^{24}-Na^{22}$, $Li^{6}-Li^{7}$, $K^{39}-K^{41}$, $Rb^{85}-Rb^{87}$, and $Cs^{137}-Cs^{131}$ separations. (T.R.H.)

United Kingdom Atomic Energy Authority. Industrial Group H.Q., Risley, Lancs, England.
ELECTROLYSIS OF FUSED SALTS. Information Bibliography. B. Yates, comp. Apr. 1956. 16p. (IGRL-IB/R-13).

This bibliography is a selection of references to reports, journal articles, and patents. They are taken from *Engineering Index* (1934 -), *Chemical Abstracts* (1907 -), *Metallurgical Abstracts* (1934 -), *Industrial Arts Index* (1955 -), and the index to the "Information Bulletin." 153 references.

7357
APPARATUS FOR THE DETERMINATION OF ELECTRIC CONDUCTIVITY OF FUSED SALTS. V. N. Tsyss, V. F. Drobnin, A. I. Nikitin, and A. I. Savchenko (Kazan' Mining-Metallurgical Inst.). Sbornik Nauch. Trudov Kazan. Gorno-Met. 11, 166-70(1956). (Translated from Referat. Zhur. Khim. No. 2, 1957, abstract No. 4942)

A device for the determination of electric conductivity of fused salts is described in which the earphones usually utilized as the zero instrument of the Kohlrausch bridge have been replaced by an electronic system which has made it possible to increase considerably the accuracy of determination. Detailed instructions are provided for operating the device and a wiring diagram of the zero instrument is included. (tr-auth)

TRANSFERENCE IN BINARY MOLTEN SALT SOLUTIONS. Benson Ross Sundheim (New York Univ., New York). J. Phys. Chem. 61, 485-6(1957) Apr.

The coupling between diffusion and electrical conduction is treated for a solution of two molten salts having an ion in common. The quantities of transport are related to transference numbers, diffusion potential and the steady state in electrolysis. (auth)

16929 NYO-7444 ✓
New York Univ., New York.
JUNCTION POTENTIALS IN MOLTEN SALTS.
Technical Progress Report. Benson R. Sundheim.
Feb. 17, 1958. 5p. Contract AT(30-1)-1938. $1.80 (ph OTS); $1.80(mf OTS).

Two discussions of junction potentials in solutions of molten salts having an ion in common are presented. (W.L.H.)

7133 NP-tr-49
THE ELECTROCHEMISTRY OF FUSED SALTS. P. Drossbach. Translated by M. H. Rand from Excerpt (p.14-100) of book published by Springer-Verlag, Berlin, 1938. 57p.

The electromotive force of a galvanic cell containing a mixed electrolyte is discussed, pointing out that all processes at the electrodes take place at a fixed rate in a definite manner. The polarization voltage at high current densities is greater than the equilibrium value which fluctuates. A number of important rules concerning conductivities and densities of fused salts and conditions governing current efficiency and energy consumption are discussed, along with polarization voltages and sources of error. The only thorough investigation of a metal's sol-

FUSED SALT AND LIQUID METAL SYSTEMS 6.10

FUSED SALTS 6.10.01

ELECTROCHEMISTRY 6.10.01.02

ubility in its fused salt is the work on the $Bi/BiCl_3$ system. The construction of electrolysis apparatus and some modifications are described along with methods of operation. Mathematical development and experimental proof are presented throughout to support information given. (J.R.D.)

5633 ✓
TEMPERATURE BEHAVIOR OF THE ISOTOPE EFFECT IN CURRENT PASSAGE THROUGH LIQUID ALKALI METALS. A. Lodding (Chalmers Technische Hochschule, Göteborg). Z. Naturforsch. 14a, 934-7(1959) Nov. (In German)

The temperature dependence of the Haeffner effect was studied in K between 79 and 335°C. There was an increase in the mass effect (μ) up to ~250°C; above 300°C μ decreased however. These findings agree with earlier studies and through new measurements confirmed the behavior of Rb. (tr-auth)

533 CF-54-8-64
Oak Ridge National Lab., Tenn.
MEASUREMENTS OF THE ELECTRICAL CONDUCTIVITY OF MOLTEN FLUORIDES. N. D. Greene. Aug. 16, 1954. Decl. Sept. 26, 1958. 22p. Contract [W-7405-eng-26]. $4.80(ph OTS); $2.70(mf OTS).

Measurements of the electric conductivity of molten fluorides from 1000 to 1700°F, together with a brief description of the measuring apparatus, are given. Redeterminations of the electrical conductivity of KNO_3, NaOH, and $LiNO_3$ melts were found to be within ± 10% of values reported. (J.E.D.)

83 AEC-tr-2642
ON THE EFFECT OF THE ANION ON ELECTRODE POTENTIALS OF METALS IN FUSED SALTS. Yu. K. Delimarskii. Translated from Ukrain. Khim. Zhur. 21, 449-56(1955). 14p.

A scale of electrode potentials has been constructed in fused halides with the aid of the zero-electrode method. Electrode potentials of heavy metals during transition from chlorides to bromide to iodides to fluorides became more negative. The influence of anions on electrode potentials of heavy metals can be explained by the regular change in degree of electrolytic dissociation during transfer from chlorides to iodides, and also by the deformation of ions which occur as a consequence of one-sided or mutual polarization. (W.L.H.)

APPARATUS FOR THE DETERMINATION OF ELECTRIC CONDUCTIVITY OF FUSED SALTS. V. N. Tsyss, V. F. Drobnin, A. I. Nikitin, and A. I. Savchenko (Kazan' Mining-Metallurgical Inst.). Sbornik Nauch. Trudov Kazan. Gorno-Met. 11, 166-70(1956). (Translated from Referat. Zhur. Khim. No. 2, 1957, abstract No. 4942)

A device for the determination of electric conductivity of fused salts is described in which the earphones usually utilized as the zero instrument of the Kohlrausch bridge have been replaced by an electronic system which has made it possible to increase considerably the accuracy of determination. Detailed instructions are provided for operating the device and a wiring diagram of the zero instrument is included. (tr-auth)

13037 ✓ AERE-M/M-194
United Kingdom Atomic Energy Authority. Research Group. Atomic Energy Research Establishment, Harwell, Berks, England.
SOME EXPERIMENTS ON THE FUSED SALT PROCESSING OF LIQUID METAL FUELS. J. K. Higgins. June 1958. 9p.

The reaction between a fused sodium, potassium, magnesium chloride eutectic and a liquid bismuth solution containing small amounts of a rare earth cerium (~100 ppm by wt) and uranium (~1000 ppm by wt) is considered. Experimental results are given for the transfer of cerium and uranium from the metal to the salt phase in a silica container. It is concluded that contamination from this container presents serious difficulties to an electrolytic method for the processing of liquid metal fuels. (auth)

1485
ELECTROMIGRATION OF RADIOACTIVE IONS IN FUSED SALTS. APPLICATION TO THE SEPARATION OF ISOTOPES. Presented at the International Conference on Radioisotopes in Scientific Research, Sept. 9-20, 1957 at Paris. No. UNESCO/NS/RIC/214. H. J. Arnikar and M. Chemla. London, Pergamon Press, Ltd., 1957. 9p. (In French)

FUSED SALT AND LIQUID METAL SYSTEMS 6.10

FUSED SALTS 6.10.01

ELECTROCHEMISTRY 6.10.01.02

6764 V AERE-Lib/Trans-702
INFORMATION ON THE ELECTROLYSIS OF FUSED SALTS. P. Drossbach. Translated by F. Hudswell from Z. Elektrochem. 59, 512-25(1955). 31p.

Investigations are communicated on methods of measuring the polarization voltage. The current efficiencies and polarization voltages are given for the electrolysis of $TiCl_4$ in KCl + LiCl, of $CrCl_2$ and of $CrCl_3$ in KCl + NaCl, and of TiCl in KCl + NaCl. A survey is presented on the measurements of single potentials and overvoltages in fused salts. (auth)

6597 AERE-Lib/Trans-699
THE SEPARATION OF TANTALUM, TITANIUM AND SODIUM AS SODIUM-LEAD ALLOYS FROM FUSED SALT MIXTURES. P. Drossbach. Translated by F. Hudswell Z. Elektrochem. 57, 548-58(1953). 24p.

A thyratron device is described with which it is possible to measure polarization potentials and single potentials down to 0.8 μ sec after switching off the current. Measurements of current yields of polarization potentials for the electrolysis of Na halides in mixtures with Na cyanide with the formation of a Na-Pb alloy and in the electrolysis of K_2TaF_7 and K_2TiF_6 in molten salt mixtures. (auth)

V 5810 NP-5992
Goteborg. Chalmers Tekniska Högskola. Institutionen för Fysik.
ISOTOPE ENRICHMENT BY COUNTERCURRENT ELECTROMIGRATION IN MOLTEN SALTS (thesis). Arnold Lundén. 1956. 114p.

Up to the present date isotopes of Li, Cl, K, Cu, Zn, Br, Ag, Cd, Tl, and Pb have been enriched by countercurrent electromigration in molten chlorides, bromides and nitrates. The theory of the experiments is reviewed briefly and systematical relations between the observed mass effects are discussed. Different properties of the separation columns are studied. Glass packed columns have 20 to 50 theoretical plates per cm length while quartz packed columns have 5 to 10. Attempts by various methods to enrich isotopes of Li, Mg, Cl, K, Ca, Cu, Zn, Ga, Br, Rb, Sr, Ag, Cd, In, Sn, rare earths, Tl, and Pb are reviewed. (auth)

8021
RECOVERY POTENTIALS FOR MOLTEN SYSTEMS OF LiCl-KCl, AND LiCl-$BaCl_2$. A. A. Kolotiĭ and Yu. K. Delimarskiĭ. Zhur. Priklad. Khim. 30, 406-41(1957) Mar. (In Russian)

Recovery potentials from eutectic and equimolar complexes of LiCl-KCl and LiCl-$BaCl_2$ at various temperatures were studied. It was found that eutectic compounds are more amenable to electrolytic recoveries of Li. The difference of LiCl-$BaCl_2$ recovery potentials at temperature intervals of 550 to 700° was about 0.21 to 0.27 v, while the difference for LiCl and KCl in the same temperature interval was only 0.11 to 0.17 v. The disadvantage of the LiCl-$BaCl_2$ system is its high melting point. Experiments showed that metallic Li recoveries from chlorides should be carried out at the lowest possible temperatures. (R.V.J.)

9672
THE ELECTRICAL CONDUCTIVITY OF SOLUTIONS OF ALKALI METALS IN THEIR MOLTEN HALIDES. H. R. Bronstein and M. A. Bredig (Oak Ridge National Lab., Tenn.). J. Am. Chem. Soc. 80, 2077-81(1958) May 5.

The electrical conductivity of alkali metal solutions in their molten halides has been measured by means of a synthetic sapphire conductance cell. The specific conductance increases with increasing metal concentration. The equivalent conductance of K, Λ_K, in both KCl and KBr also increases, namely, from 2800 ohm^{-1} cm^2 (K-KCl, 820°), 6100 (K-KBr, 760°), and 6500 (K-KBr, 870°) at infinite dilution, to 38,000, 83,000, and 71,000 ohm^{-1} cm^2 at 19, 23, and 20 mole % K, respectively, the maximum concentrations measured. However, Λ_{Na}, for Na in NaBr at 895° decreases from 12,500 to a minimum of 7300 ohm^{-1} cm^2 at ca. 9 mole % metal. At 805° Λ_{Na} in NaBr decreases from ca. 12,000 at infinite dilution, i.e., approximately the same value as at 895°, to less than 5000, and in NaCl at both 845 and 890° from ca. 6000 to less than 3000 ohm^{-1} cm^2. These values are

ISOTOPE EFFECT BY PASSAGE OF CURRENT IN MOLTEN LITHIUM METAL. A. Lundén, A. Lodding, and W. Fischer. Z. Naturforsch. 12a, 268-9(1957) Mar. (In German)

FUSED SALT AND LIQUID METAL SYSTEMS 6.10

FUSED SALTS 6.10.01

ELECTROCHEMISTRY 6.10.01.02

6750 AEC-tr-2899
FERROELECTRICS. Herbert Sachse. Excerpts translated from Ferroelektrika; Technische Physik in Einzeldarstellungen. Bd. XI. Springer-Verlag (Berlin). 1955. 99p.
The ferroelectric properties of titanates, zirconates, stannates, niobates, and tantalates are treated. General theory, pressure dependence, field dependence, and applications are discussed. (T.R.H.)

THE INFLUENCE OF TEMPERATURE ON ISOTOPIC SEPARATION UNDER THE EFFECT OF A CONTINUOUS CURRENT IN MOLTEN GALLIUM. Maurice Goldman, Guy Nief, and Étienne Roth. Compt. rend. 243, 1414-16 (1956) Nov. 5. (In French)

11623 AEC-tr-3658
THE MECHANISM OF THE CATHODE PROCESS IN THE ELECTROLYSIS OF A FUSED $NaCl-CaCl_2$ MIXTURE ON AN IRON CATHODE. L. Suskii. Translated by A. L. Monks (Oak Ridge National Lab.) from Zhur. Fiz. Khim. 32, 1785-95(1958). 30p. (Includes original, 11p.) $4.80(ph), $2.70(mf) JCL or LC.
The cathode process in the electrolysis of a fused $NaCl-CaCl_2$ mixture on an iron cathode is examined. Factors given special attention were the sequence of electro-chemical processes for the discharge of individual ions at the cathode, and the cathode potential as a function of time. Additional measurements were carried out to confirm the validity of the hypothesis according to which the anodic potential was assumed to be constant. Results indicate that calcium is separated at the cathode and sodium is precipitated out. (J.R.D.)

14288 AEC-tr-3667
STUDIES OF THE CATHODIC PROCESS DURING ELECTROLYSIS OF THE PURE MOLTEN CHLORIDES OF CALCIUM, SODIUM AND POTASSIUM. L. Suskii. Translated by A. L. Monks (Oak Ridge National Lab.) from Zhur. Fiz. Khim. 32, 1393-1403(1958). 28p. (Includes original, 11p.) $4.80(ph), $2.70(mf) JCL or LC.
This paper was previously abstracted from the original language and appears in NSA, Vol. 12, as abstract No. 16212.

IMPROVEMENTS IN OR RELATING TO REFINING METAL BY ELECTRODEPOSITION. Allan Robert Gibson (to United Kingdom Atomic Energy Authority). British Patent 810,091. Mar. 11, 1959.
A process for purifying metals by electrodeposition from a molten salt bath is described. The metal to be purified is placed on the graphite anode plate in the molten salt. The cathode is made of the pure metal which is being refined. An alumina cup or other inert-material cup is provided at the anode to catch electrodeposited metal which does not adhere. An inert gas atmosphere is provided. (T.R.H.)

409
United Kingdom Atomic Energy Authority. Industrial Group H.Q., Risley, Lancs, England.
ELECTROLYSIS OF FUSED SALTS. Information Bibliography. B. Yates, comp. Apr. 1956. 16p. (IGRL-IB/R-13).
This bibliography is a selection of references to reports, journal articles, and patents. They are taken from *Engineering Index* (1934 -), *Chemical Abstracts* (1907 -), *Metallurgical Abstracts* (1934 -), *Industrial Arts Index* (1955 -), and the index to the "Information Bulletin." 153 references.

EQUIPMENT 6.10.01.03

See also, under Plants and Equipment, Pumps — For Fused Salts and Liquid Metals (5.6.03.04).

15055 ORNL-2337
Oak Ridge National Lab., Tenn.
INTERIM REPORT ON CORROSION BY ALKALI-METAL FLUORIDES: WORK TO MAY 1, 1953. G. M. Adamson, R. S. Crouse, and W. D. Manly. June 5, 1959. 36p. Contract W-7405-eng-26. $1.25(OTS).
In connection with the search for container materials suitable for use in high-temperature molten-fluoride reactor systems, the corrosion of several metals by various fused fluoride mixtures was studied in thermal-

FUSED SALTS 6.10.01

Equipment 6.10.01.03

convection loops made from the metal being tested. In these tests the temperature drop was fairly large, but the velocity of the liquid was low and not subject to control. The fluoride mixture used in most of the study contained 43.5 mole % KF, 10.9 mole % NaF, 44.5 mole % LiF, and 1.1 mole % UF_4, and is referred to as "fluoride 14." In a series of screening tests Inconel, or similar high-nickel alloys, was shown to be the most promising container material. The data reported are from loops that were operated for only relatively short times, 500 hr, and with impure fluoride mixtures. The lack of purity of the mixtures was an important consideration in the tests reported. With fluoride 14 an average figure for the maximum microscopically visible attack on Inconel was 0.012 to 0.015 in. after 500 hr of circulation. The attack was in the form of small voids which did not connect with the surface or with each other. It is shown that for the times used here the attack was caused primarily by the reduction of fuel impurities by chromium metal from the container and by the leaching of sufficient chromium to reach equilibrium concentrations in reactions with the fluorides, primarily with UF_4. In contrast to the effects of impurities, the depth and type of attack varied only slightly when changes were made in the major components of the various fluoride mixtures studied. The only effect definitely established was that, in every case, mixtures containing uranium fluoride caused more attack than did similar mixtures containing no uranium. When the loop was operated at a fluid temperature of 1650°F, the attack was slightly deeper than it was at 1300°F. The addition of zirconium hydride to the fuel almost completely eliminated the attack. Alkali metals also acted as inhibitors. It was found that type 316 stainless steel might be considered for a container for molten fluoride mixtures that do not include potassium fluoride. With such stainless steels and zirconium fluoride-base mixtures, the maximum depth of attack was about the same as that found with Inconel; however, more material was mass-transferred and cold-leg deposits were found. When alkali-metal-base mixtures were circulated in type 316 stainless steel, plugging occurred in a relatively short time; the plugging was probably caused by the formation of a compound similar to K_2NaCrF_6. (auth)

17119 CF-58-8-87
Oak Ridge National Lab., Tenn.
REFLECTIONS ON THE HOT-ZONE ATTACK OF INCONEL BY FUSED FLUORIDE. J. L. Scott. Aug. 22, 1958. 18p. Contract [W-7405-eng-26]. $3.30(ph OTS); $2.40(mf OTS).

Evidence is presented that the hot-zone attack of nickel-base alloys in contact with fused fluorides occurs by a grain-boundary diffusion process. Predictions based on the grain-boundary diffusion model are shown to compare favorably with existing data. Certain anomalies are pointed out with regard to the corrosion of Inconel by Fuel No. 30 ($NaF-UF_4-ZrF_4$) and tests which would lead to a better understanding of fused-salt corrosion are outlined. (auth)

FLUORIDES 6.10.01.04

12536 CF-59-2-61
Oak Ridge National Lab., Tenn.
PROCESSING OF MOLTEN SALT POWER REACTOR FUEL. D. O. Campbell and G. I. Cathers. Apr. 1, 1959. 18p. Contract [W-7405-eng-26]. $3.30(ph), $2.40(mf) OTS.

Fuel reprocessing methods are being investigated for molten salt nuclear reactors which use $LiF-BeF_2$ salt as a solvent for UF_4 and ThF_4. A liquid HF dissolution procedure coupled with fluorination has been developed for recovery of the uranium and $LiF-BeF_2$ solvent salt which is highly enriched in Li^7. The recovered salt is decontaminated in the process from the major reactor poisons; namely, rare earths and neptunium. A brief investigation of alternate methods, including oxide precipitation, partial freezing, and metal reduction, indicated that such methods may give some separation of the solvent salt from reactor poisons, but they do not appear to be sufficiently quantitative for a simple processing operation. Solubilities of LiF and BeF_2 in aqueous 70 to 100% HF are presented. The BeF_2 solubility is appreciably increased in the presence of water and large amounts of LiF. Salt solubilities of 150 g/liter are attainable. Tracer experiments indicate that rare earth solubilities, relative to $LiF-BeF_2$ solvent salt solubility, increase from about 10^{-4} mole % in 98% HF to 0.003 mole % in 80% HF. Fluorination of uranium from $LiF-BeF_2$ salt was demonstrated. This appears feasible also for the recovery of the relatively small concentration of uranium produced in the $LiF-BeF_2-ThF_4$ blanket. A proposed chemical flowsheet is pre-

FUSED SALTS 6.10.01

FLUORIDES 6.10.01.04

sented on the basis of this exploratory work as applied to the semicontinuous processing of a 600 Mw power reactor. (auth)

15152 ✓
HIGH-TEMPERATURE PROCESSING OF MOLTEN FLUORIDE NUCLEAR REACTOR FUELS. Warren R. Grimes, James H. Shaffer, Newman V. Smith, Richard A. Strehlow, Wilfred T. Ward, and George M. Watson (Oak Ridge National Lab., Tenn.). Chem. Eng. Progr. 55, Symposium Ser. No. 27, 65-70(1959).

The removal of certain undesirable fission-product fluorides from reactor fuels consisting of molten mixtures of uranium tetrafluoride with other fluorides has been successfully accomplished in small-scale experiments by precipitation techniques which may be adaptable to large-scale application. Data relating to the solubility of xenon and other rare gases are presented and are pertinent to the ease of removal of such gaseous fission-product poisons. The rare earth trifluorides crystallize from $NaF-ZrF_4-UF_4$ or $LiF-BeF_2-UF_4$ fuel mixtures as the simple, uncomplexed trifluoride. Solutions containing more than one rare earth fluoride yield as the primary phase a solid solution of the trifluorides. Absolute solubilities in the range of 450° to 550°C are relatively low. Accordingly by use of equilibria such as $SmF_{3(d)} + CeF_{3(s)} \rightleftharpoons CeF_3 \cdot SmF_{3(ss)} + CeF_{3(d)}$ (where d, s, and ss refer to dissolved, solid, and solid solution, respectively) it is possible to deplete the liquid of nuclearly harmful rare earth species. The fuel mixture is, in the process, essentially saturated with cerium trifluoride at the temperature of separation. Selective precipitation of polyvalent cations from fluoride melts by addition of oxides has been successful in laboratory-scale experiments. Chromatographic separation of cations of various valences by precipitation on oxide beds has shown promise in preliminary experiments. (auth)

11920 ✓
AN INVESTIGATION OF THE PHASE DIAGRAM OF THE TERNARY SYSTEM SODIUM FLUORIDE–ALUMINUM FLUORIDE–MAGNESIUM FLUORIDE. A. A. Kostyukov and A. B. Karpov. Trudy Leningrad. Politekh. Inst. No. 188, 58-66(1957). (Translated from Referat. Zhur. Met. No. 6, 1958, p.35)

The methods of thermal analysis and microstructural analysis are used to study the following binary systems: (1) $NaF-MgF_2$; (2) MgF_2-AlF_3; (3) $Na_3AlF_6-MgF_6$; (4) $Na_3AlF_6-NaMgF_3$; and (5) $NaMgF_3-AlF_3$. Systems (1) and (2) are binary accessory systems, while (3), (4), and (5) are sections of the ternary system $NaF-AlF_3-MgF_2$, a study of which is necessary to clarify the effectiveness of addition of MgF_2 to the electrolyte during Al refining to reduce the m.p. of the electrolyte, to develop crystal-optical methods of monitoring the composition of the electrolyte in Al baths when MgF_2 is used as an addition, and to clarify the chemical reaction among the starting components. It is shown that (1) $Na_3AlF_6-MgF_2$ and $NaMgF_3-AlF_3$ are not binary systems. The phase diagrams of

9693 MLM-1086
Mound Lab., Miamisburg, Ohio.
DENSITY AND VISCOSITY OF FUSED MIXTURES OF LITHIUM, BERYLLIUM, AND URANIUM FLUORIDES. B. C. Blanke, E. N. Bousquet, M. L. Curtis, and E. L. Murphy. Dec. 1956. 88p. Contract AT-33-1-GEN-53. $13.80(ph), $4.80(mf) OTS.

The density of the ternary system lithium fluoride–beryllium fluoride–uranium fluoride was investigated by determining the loss in weight of a solid of known volume immersed in the fused solution over the temperature range of the liquidus to 900°C. The viscosity of the same system over this temperature range was measured by use of a concentric cylinder viscometer. The results are assembled in tabular and graphic form.

1251
THE SYSTEMS CaF_2-LiF AND $CaF_2-LiF-MgF_2$. W. E. Roake (General Electric Co., Richland, Wash.). J. Electrochem. Soc. 104, 661-2(1957) Nov.

Temperature-composition relationships have been measured in the CaF_2-LiF and the $CaF_2-LiF-MgF_2$ systems in the areas of liquidus temperatures below 1000°C. A binary eutectic composition of 80.5 mole % LiF and 19.5 mole % CaF_2 was observed to melt at 769°C. A ternary eutectic composition of 59.0 mole % LiF, 2.9 mole % MgF_2, and 13.1 mole % CaF_2 was observed to melt at 672°C. (auth)

FUSED SALT AND LIQUID METAL SYSTEMS 6.10

FUSED SALTS 6.10.01

FLUORIDES 6.10.01.04

4311
MELT DIAGRAMS FOR LiF–NaF–CaF SYSTEMS. G. A. Bukhalova, K. Sulaimankulov, and A. K. Bostandzhiyan (Rostov-on-Don Construction Inst.). Zhur. Neorg. Khim. 4, 1138-40(1959) May. (In Russian)

The surface crystallization and melt diagrams of LiF–NaF–CaF systems were plotted and analyzed. The combined component crystallization lines converge at the eutectic point of 35.5% Na_2F_2, 46.5% Li_2F_2, and 20% CaF_2 at the melting point 607°C. (R.V.J.)

3584 ✓ ORNL-2396
Oak Ridge National Lab., Tenn.
GUIDE TO THE PHASE DIAGRAMS OF THE FLUORIDE SYSTEMS. J. E. Ricci. Dec. 3, 1958. 117p. Contract W-7405-eng-26. $2.75(OTS).

The phase equilibria occurring in the fused fluoride systems investigated by the Fused Salt Chemistry Section of Oak Ridge National Laboratory are discussed in detail. (T.R.H.)

PHASE EQUILIBRIA IN THE ALKALI FLUORIDE–URANIUM TETRAFLUORIDE FUSED SALT SYSTEMS. II. THE SYSTEMS KF–UF_4 AND RbF–UF_4. R. E. Thoma, Herbert Insley, B. S. Landau, H. A. Friedman, and W. R. Grimes (Oak Ridge National Lab., Tenn.). J. Am. Ceram. Soc. 41, 538-44(1958) Dec.

Detailed phase diagrams are presented for the binary systems KF–UF_4 and RbF–UF_4. Data for the determination of phase boundaries were obtained in large part by quenching after equilibration. Thermal analysis and visual observation were used as supplementary methods. Phase identifications were made by x-ray-powder diffraction and by optical microscopy. In the system KF–UF_4 four compounds have been identified. Two of these, $3KF \cdot UF_4$ and $7KF \cdot 6UF_4$, melt congruently at 957°C. and at 789°C., respectively. The compound $2KF \cdot UF_4$ melts incongruently to $3KF \cdot UF_4$ and liquid at 755°C., and the compound $KF \cdot 2UF_4$ melts incongruently to UF_4 and liquid at 765°C. There are three eutectic points in the system: 15.0 mole % UF_4, 735°C.; 38.5 mole % UF_4, 740°C.; and 54.5 mole % UF_4, 735°C. The compound $2KF \cdot UF_4$ decomposes during cooling to $3KF \cdot UF_4$ and $7KF \cdot 6UF_4$ at 608°C. Two other compounds, $KF \cdot 3UF_4$ and $KF \cdot 6UF_4$, have been described by another investigator. One of these, $KF \cdot 3UF_4$, was not obtained at all in the present investigation. The other, $KF \cdot 6UF_4$, was not obtained under equilibrium conditions and was found only in KF-UF_4 mixtures that had been exposed to the atmosphere while molten. Seven solid compounds are present in the system RbF–UF_4. All of these melt incongruently with the exception of $3RbF \cdot UF_4$ and $RbF \cdot UF_4$, which melt at 995°C. and at 735°C., respectively. The incongruently melting compounds and their melting points, peritectic compositions, and melting products are: $2RbF \cdot UF_4$, 818°C., 38 mole % UF_4, $3RbF \cdot UF_4$ + liquid; $7RbF \cdot 6UF_4$, 693°C., 44 mole % UF_4, $RbF \cdot UF_4$ + liquid; $2RbF \cdot 3UF_4$, 722°C., 56.5 mole % UF_4, $RbF \cdot 3UF_4$ + liquid; $RbF \cdot 3UF_4$, 730°C., 57 mole % UF_4, $RbF \cdot 6UF_4$ + liquid; and $RbF \cdot 6UF_4$, 832°C., 70.5 mole % UF_4, UF_4 + liquid. The three eutectic temperatures and compositions in the system are: 10.0 mole % UF_4, 710°C.; 43.5 mole % UF_4, 675°C.; and 55.0 mole % UF_4, 714°C. (auth)

12357✓ ANL-WAR-SL-1140
Argonne National Lab., Lemont, Ill.
SELECTION OF OPTIMUM FLUORINATING AGENT FOR FUSED-SALT PROCESS. G. E. Goring. Dec. 26, 1956. Decl. Mar. 11, 1957. 21p. Contract [W-31-109-eng-38]. $4.80(ph OTS); $2.70(mf OTS).

Qualitative consideration of flowsheets using BrF_3, BrF_3–BrF_5, BrF_5, and ClF_3 showed that BrF_3–BrF_5 would afford the most realistic comparison with elemental F_2. Then, using a plant capacity of 100 STR cores per year as a basis, an economic comparison between the F_2 and BrF_3–BrF_5 systems was made. It was shown that the F_2 system could effect a saving on the order of $90,000 per year in such a plant, due mainly to lower manpower requirements. Differences in capital charges between the two alternates would be relatively minor, and differences in chemicals and utilities costs would be negligible. (auth)

11052 ✓ CF-54-7-59
Oak Ridge National Lab., Tenn.
DISSOLUTION OF METALS IN FUSED FLUORIDE BATHS. R. E. Leuze and C. E. Shilling. July 8, 1954. Decl. Mar. 8, 1957. 5p. Contract [W-7405-eng-26]. $1.80(ph OTS); $1.80(mf OTS).

FUSED SALT AND LIQUID METAL SYSTEMS 6.10

FUSED SALTS 6.10.01

FLUORIDES 6.10.01.04

A method is presented for processing enriched reactor fuel elements. The method calls for contacting the metal with HF in the presence of a molten fluoride bath so that the metal fluoride is dissolved. This exposes fresh metal surface for further reaction. A summary of the results obtained for various metals and various dissolving conditions is given. (W.L.H.)

9240 BMI-1186
Battelle Memorial Inst., Columbus, Ohio.
VAPOR PRESSURE AND EQUILIBRIUM STUDIES OF THE SODIUM FLUORIDE–BERYLLIUM FLUORIDE SYSTEM. Karl A. Sense, Richard W. Stone, and R. B. Filbert, Jr. May 27, 1957. 32p. Contract W-7405-eng-92. $6.30 (ph OTS); $3.00(mf OTS).

A new study made on the vapor pressure of BeF_2 over the temperature interval 802 to 1025°C showed good agreement with previous data taken over the 802 to 968°C range. Excessive scatter of the vapor-pressure data in the lower temperature region prevented a melting-point determination of BeF_2 by the transpiration method alone. Thermal analysis showed the melting point to be about 545°C. Vapor pressures of the $NaF-BeF_2$ system were measured over the range 509 to 1061°C. On the basis of previously developed theory it was concluded that the complex $NaBeF_3$ exists in the vapor phase in addition to NaF and BeF_2.

8852
AN INVESTIGATION OF FUSED-SALT SYSTEMS BASED ON THORIUM FLUORIDE. II. V. S. Emelyanov and A. I. Evstyukhin. J. Nuclear Energy 5, No. 1, 108-14(1957).

On the basis of thermal, x ray, and other analyses, phase diagrams have been constructed of the system $NaF-ThF_4$, which includes four chemical compounds (Na_4ThF_8, α-Na_2ThF_6, β-Na_2ThF_6, $NaThF_5$, $NaTh_2F_9$), and of the system $KF-ThF_4$, which comprises six chemical compounds (K_5ThF_9, K_3ThF_7, $K_3Th_2F_{11}$, $KThF_5$, KTh_2F_9, KTh_6F_{25}). In an x ray examination of melts of the system $NaF-KF-ThF_4$, the compound $NaK(ThF_6)$ was found, and its structure determined. A triangular portion of the $NaF-Na_2ThF_6-KThF_5-KF$ constitution diagram has been plotted, and the polythermal section $NaF-KThF_5$ constructed. (auth)

7483
DECONTAMINATION OF NEUTRON-IRRADIATED REACTOR FUEL. A. G. Buyers, F. D. Rosen, and E. E. Motta (to U. S. Atomic Energy Commission). U. S. Patent 2,918,366. Dec. 22, 1959.

A pyrometallurgical method of decontaminating neutron-irradiated reactor fuel is presented. In accordance with the invention, neutron-irradiated reactor fuel may be decontaminated by countercurrently contacting the fuel with a bed of alkali and alkaline fluorides under an inert gas atmosphere and inductively melting the fuel and tracking the resulting descending molten fuel with induction heating as it passes through the bed. By this method, a large, continually fresh surface of salt is exposed to the descending molten fuel which enhances the efficiency of the scrubbing operation.

8239 AEC-tr-2538
DETERMINATION OF DECOMPOSITION POTENTIALS OF MOLTEN FLUORIDES OF ALKALINE AND ALKALINE EARTH METALS. Yu. K. Delimarskii and F. F. Grigorenko. Translated from Ukrain. Khim. Zhur. 21, 561-8(1955). 17p.

An electrochemical series of metals in molten fluorides was established at 1000°C and at temperature of fusion. Methods for determining the decomposition potentials of molten salts were presented. It was hypothesized that decomposition potentials depend on the magnitude of the ion radii. (W.L.H.)

12340
PHASE EQUILIBRIA IN THE SYSTEMS $NaF-ZrF_4$, UF_4-ZrF_4 AND $NaF-ZrF_4-UF_4$. C. J. Barton, W. R. Grimes, H. Insley, R. E. Moore, and R. E. Thoma (Oak Ridge National Lab., Tenn.). J. Phys. Chem. 62, 665-76(1958) June.

Equilibrium diagrams for the binary systems UF_4-ZrF_4 and $NaF-ZrF_4$ and the ternary system $NaF-ZrF_4-UF_4$ described in this paper are based on data obtained by thermal analysis, differential thermal analysis, equilibration of small samples followed by quenching, visual observations of phase changes and filtration experiments. Phase boundaries were established for the temperature interval between liquidus temperatures and about 300°. UF_4 and ZrF_4 form a complete series of solid solutions with the minimum melting temperature at 765° for the solution containing 77 mole % ZrF_4. The $NaF-ZrF_4$ system contains the compounds $3NaF \cdot ZrF_4$ and $7NaF \cdot 6ZrF_4$ which melt

FUSED SALT AND LIQUID METAL SYSTEMS 6.10

FUSED SALTS 6.10.01

FLUORIDES 6.10.01.04

13727

SOLUBILITY OF NOBLE GASES IN MOLTEN FLUORIDES. I. IN MIXTURES OF $NaF-ZrF_4$ (53–47 MOLE %) AND $NaF-ZrF_4-UF_4$ (50–46–4 MOLE %). V. R. Grimes, N. V. Smith, and G. M. Watson (Oak Ridge National Lab., Tenn.). J. Phys. Chem. 62, 362-6(1958) July.

Solubilities of He, Ne, Ar, and Xe were determined at pressures from 0.5 to 2 atmospheres in $NaF-ZrF_4$ (53–47 mole %) at 600, 700, and 800°. The solubilities increase linearly with gas pressure, decrease with increasing atomic weight of the solute and increase with increasing temperature. Henry's law constants in moles of solute/(cm^3 solution)(atmosphere) at 600° are $21.6 \pm 1 \times 10^{-6}$, $11.3 \pm 0.3 \times 10^{-6}$, $5.06 \pm 0.15 \times 10^{-6}$, and $1.94 \pm 0.2 \times 10^{-6}$ while heats of solution are 6.2, 7.8, 8.2, and 11.1 kcal/mole for He, Ne, Ar, and Xe, respectively. Solubilities and heats of solution for He and Xe in $NaF-ZrF_4-UF_4$ (50–46–4 mole %) are very similar to the values obtained with these gases in the $NaF-ZrF_4$ mixture. (auth)

1251

THE SYSTEMS CaF_2-LiF AND $CaF_2-LiF-MgF_2$. W. E. Roake (General Electric Co., Richland, Wash.). J. Electrochem. Soc. 104, 661-2(1957) Nov.

Temperature-composition relationships have been measured in the CaF_2-LiF and the $CaF_2-LiF-MgF_2$ systems in the areas of liquidus temperatures below 1000°C. A binary eutectic composition of 80.5 mole % LiF and 19.5 mole % CaF_2 was observed to melt at 769°C. A ternary eutectic composition of 59.0 mole % LiF, 2.9 mole % MgF_2, and 13.1 mole % CaF_2 was observed to melt at 672°C. (auth)

16269

THERMODYNAMIC STUDIES AT LOW TEMPERATURES. V. N. Kostryukov, O. P. Samorukov, and P. G. Strelkov (Inst. of Problems in Physics, Moscow). Zhur. Fiz. Khim. 32, 1354-61(1958). (In Russian)

The heat capacities of BF_3, CF_4, and SiF_4 were investigated between 12 to 13°K and the melting points. In the solid state BF_3 reveals no phase transitions. Tetrafluoromethane has a second order phase transition with a jump in the heat capacity at 76.09 ± 0.012°K. The heat effect of the transition is 414.5 cal/mole and the entropy 5.11. Tetrafluorosilane does not reveal any phase transitions in the solid state. The triple points of BF_3, CF_4, and SiF_4 were determined, and corrected for the experimentally determined value of impurity depression. (tr-auth)

7101

THE BARIUM FLUORIDE–URANIUM TRIFLUORIDE SYSTEM. R. W. M. D'Eye and F. S. Martin (Atomic Energy Research Establishment, Harwell, nr. Didcot, Berks). J. Chem. Soc. 1847-51(1957) Apr.

The system UF_3-BaF_2 has been examined by x-ray and density measurements and by thermal analysis. Uranium trifluoride dissolves in solid BaF_2 to the extent of about 50 mole %, the cell constant a decreasing from 6.189 to 6.034 kX; barium fluoride dissolves in UF_3 to the extent of about 20 mole %. These two conjugate solid solutions exist between definite concentration limits. It is also shown that UF_3 may form a solid solution in SrF_2. The melting point of UF_3 is found to be about 1495°; its addition to BaF_2 increases the melting point of the latter, and it is suggested that the system has a peritectic point at about 1400°. (auth)

2735 ORNL-1956(Del.)

Oak Ridge National Lab., Tenn.

ENTHALPIES AND HEAT CAPACITIES OF SOLID AND MOLTEN FLUORIDE MIXTURES. W. D. Powers and G. C. Blalock. Feb. 1, 1956. Decl. with deletions Nov. 13, 1958. 31p. Contract W-7405-eng-26. $6.30 (ph OTS); $3.00(mf OTS).

The enthalpies and heat capacities of seventeen fluoride mixtures in the liquid state have been determined using Bunsen ice calorimeters and copper block calorimeters. The fluoride mixtures were composed of the fluorides of two or more of the following metals: lithium, sodium, potassium, beryllium, zirconium, and uranium. The enthalpies and heat capacities of most of these mixtures were studied in the solid state also. Estimates of the heat of fusion have been made. General empirical equations have been developed which represent the enthalpies and heat capacities of the fluoride mixtures in the liquid and in the solid state. (auth)

FUSED SALT AND LIQUID METAL SYSTEMS 6.10

FUSED SALTS 6.10.01

FLUORIDES 6.10.01.04

1951 ✓ CF-58-11-40
Oak Ridge National Lab., Tenn.
RESULTS OF EXAMINATIONS OF FUSED SALT MIXTURES BY OPTICAL AND X-RAY DIFFRACTION METHODS. R. E. Thoma. Nov. 14, 1958. 54p. Contract [W-7405-eng-26]. $9.30(ph OTS); $3.60(mf OTS).

A summary of results of examinations of fused salt mixtures is presented. Phase analyses are derived from the two complementing techniques: measurements of optical properties using the polarizing light (petrographic) microscope, and solid phase identification using x-ray-diffraction methods. Investigations of most of the samples on which data are reported were made as a part of a program of phase equilibrium studies of fluoride fused salt mixtures. (auth)

5230 ✓
VAPOR PRESSURE AND DERIVED INFORMATION OF THE SODIUM FLUORIDE–ZIRCONIUM FLUORIDE SYSTEM. DESCRIPTION OF A METHOD FOR THE DETERMINATION OF MOLECULAR COMPLEXES PRESENT IN THE VAPOR PHASE. Karl A. Sense, C. A. Alexander, R. E. Bowman, and R. B. Filbert, Jr. (Battelle Memorial Inst., Columbus, Ohio). J. Phys. Chem. 61, 337-44(1957) March.

By use of the transpiration method, the vapor pressures of the $NaF-ZrF_4$ system were measured over the range 599 to 1075°C. A partial phase diagram of the $NaF-ZrF_4$ system derived from the vapor pressure data shows a constant boiling point to exist at 25 mole % ZrF_4. Plots showing vapor–liquid–solid equilibria are presented. A plot is given showing the change of the total vapor pressure of the $NaF-ZrF_4$ system with composition for various temperatures. (auth)

2741 ORNL-1877(Rev.)
Oak Ridge National Lab., Tenn.
DISSOLUTION OF METALS IN FUSED FLUORIDES. R. E. Leuze, G. I. Cathers, and C. E. Schilling. Oct. 12, 1955. Decl. Mar. 6, 1957. 15p. Contract W-7405-eng-26. $0.30(OTS).

In scouting tests, a number of metals used in nuclear reactor fuel elements were dissolved by $ZrF_4-KF-NaF$ fused salt at 675°C through which HF was being passed. These included type 304 stainless steel at 4 mils/hr; type 347Nb stainless steel at 7 mils/hr; Th at 14 mils/hr; nonirradiated U at 17 mils/hr; Zr at 22 to 35 mils/hr; Ti at 31 mils/hr; and Zircaloy-2 at 22 to 46 mils/hr. Only small amounts of volatile fission products formed when irradiated U was dissolved. Variables that appear to affect the dissolution rate are the composition of the fused fluoride, the fused fluoride temperature, the HF flow rate, the metallurgical characteristics of the material being dissolved, and the presence of other metals. The low dissolution rate of 0.001 mil/hr observed for nickel suggests that it may be suitable as a material of construction for reaction vessels. (auth)

1851 CF-55-5-59
Oak Ridge National Lab., Tenn.
MEASUREMENT OF THE VISCOSITY OF COMPOSITION 78. S. I. Cohen and T. N. Jones. May 16, 1955. Decl. Aug. 14, 1957. 3p. Contract W-7405-eng-26. $1.80 (ph OTS); $1.80(mf OTS).

Viscosity measurements were made on Composition 78 ($BeF_2-LiF-NaF$; 28–16–56 mole %), and the viscosity was found to vary from about 7 centipoises at 575°C to about 2.9 centipoises at 800°C. The viscosity may be represented throughout this temperature range by $\mu = 0.105 \exp(3560/T)$, where T is in °K. (L.T.W.)

2736 ✓ ORNL-2278(Del.)
Oak Ridge National Lab., Tenn.
VISCOSITY MEASUREMENTS ON MOLTEN FLUORIDE MIXTURES. S. I. Cohen and T. N. Jones. July 12, 1957. Decl. with deletions Nov. 13, 1958. 50p. Contract W-7405-eng-26. $7.80(ph OTS); $3.30(mf OTS).

A summary is presented of the experimental viscosity program on fused fluoride mixtures which has been carried out at the Oak Ridge National Laboratory. The experimental techniques which have been developed are described, data on the viscosity of 36 mixtures are tabulated, and several correlations involving these data are discussed. (auth)

1852 CF-56-5-33(Del.)
Oak Ridge National Lab., Tenn.
MEASUREMENT OF THE VISCOSITY OF COMPOSITIONS 12, 14, AND 107. S. I. Cohen and T. N. Jones. May 9, 1956. Decl. with deletions Mar. 15, 1957. 7p. Contract [W-7405-eng-26]. $1.80(ph OTS); $1.80(mf OTS).

FUSED SALT AND LIQUID METAL SYSTEMS 6.10

FUSED SALTS 6.10.01

FLUORIDES 6.10.01.04

The viscosity of Composition 12 or Flinak (NaF–LiF–KF; 11.5–46.5–42 mole %) varied from 9.1 centipoises at about 500°C to 1.95 centipoises at about 800°C and may be represented throughout this range by $\mu = 0.0391 \exp (4219/T)$, where T is in °K. The viscosity of Composition 14 or Fulinak ($NaF-LiF-KF-UF_4$; 10.9–44.5–43.5–1.1 mole %) varied from 8.8 centipoises at about 500°F to 1.8 centipoises at about 800°C and may be represented throughout this range by $\mu = 0.0348 \exp (4265/T)$. The viscosity of Composition 107 ($NaF-LiF-KF-UF_4$; 11.2–45.3–41–2.5 mole %) varied from 8.2 centipoises at about 525°C to 3.0 centipoises at about 700°C and may be represented throughout this range by $\mu = 0.0292 \exp (4507/T)$. (L.T.W.)

2601 NLCO-747
National Lead Co. of Ohio, Cincinnati.
THE RECOVERY OF MAGNESIUM AND HF FROM MAGNESIUM FLUORIDE: A LITERATURE SEARCH. Earl W. Mautz. May 26, 1958. 26p. Contract AT(30-1)-1156. $1.00(OTS).

Literature references relating to the recovery of Mg and Hf from MgF_2 are presented. Reports on the manufacture of HF are comprehensively included. (auth)

7512 WADC-TR-56-4
Stanford Research Inst., Menlo Park, Calif.
THE THERMODYNAMIC PROPERTIES OF MOLTEN SALTS: MOLYBDENUM HEXAFLUORIDE. [Period Covered] June 1954 to October 1955. A. P. Brady, J. K. Clauss, and O. E. Myers. Nov. 1955. 55p. Project No. 1252. Contract AF33(616)-2558.

Assembling and testing of apparatus for measuring heat capacities from about 50°K to room temperature has been completed, including calibration of thermocouples by comparison with a Pt resistance thermometer and with a Cu one. This apparatus has been used to determine the absolute entropy of MoF_6 vapor at 298.16°K. The result, 79.7 ± 0.6 cal/mole·deg, agrees well with that calculated from its spectrum, 80.05 cal/mole·deg. A previously

5232
THE VAPOR PRESSURE OF SODIUM FLUORIDE. Karl A. Sense, C. A. Alexander, R. E. Bowman, R. W. Stone, and R. B. Filbert, Jr. (Battelle Memorial Inst., Columbus, Ohio). J. Phys. Chem. 61, 384(1957) March.

8854
PARAMAGNETIC RESONANCE OF IMPURITIES IN NaF. B. Bleaney and W. Hayes (Clarendon Lab., Oxford). Proc. Phys. Soc. (London) B70, 626-8(1957) June.

The paramagnetic resonance spectrum of a single crystal of NaF has been observed before and after irradiation with gamma rays, electrons, and neutrons, and also after annealing. Results are summarized and analyzed. (M.H.R.)

1ZZZ6
THERMODYNAMIC PROPERTIES OF LITHIUM FLUORIDE. N. K. Voskresenskaya, V. A. Sokolov, Ye. I. Banashek, and N. Ye. Shmidt. Izvest. Sektora Fiz. Khim. Anal. Inst. Obshchei Neorg. Khim. Akad. Nauk S.S.S.R. 233-8(1956). (Translated from Referat. Zhur. Fiz. No. 4, 1957, Abstract No. 8993.)

The specific heat C_p of crystalline LiF is determined in the temperature range from 317 to 658°K using a method previously described (V. A. Sokolov, Zhur. Tekh. Fiz. 18, 813(1948)). The data obtained fit, within an average error of 0.25%, the relation C_p (cal/° mol) = $10.32 + 3.90 \times 10^{-3}\,T - 1.36 \times 10^{+5} \times T^{-2}$. A measurement was made of the enthalpy of LiF in the interval 673 to 1410°K. The results are expressed by the equations: $H_T - H_{293.16}$(cal/mol) = $10.00\,T + 2.217 \times 10^{-3}\,T^2 + 122176\,T - 3539$ (solid phase) and $H_T - H_{293.16} = 32 + 15.175\,T$ (1128 to 1410°K; liquid phase). $\Delta H_m = 6477$, $\Delta S_m = 5.78$ entropy units. The standard values are: $H_{298.16} = 1548$ cal/mol and $S_{298.16} = 8.53$ entropy units. The values of C_p, H, S, and Z are calculated in the range 50 to 1400°K and tabulated.

72 AEC-tr-3399
INVESTIGATION OF THE SYSTEM $HF-ZrF_4-H_2O$ BY THE ISOTHERMIC SOLUBILITY METHOD (ISOTERM 0.5°). I. V. Tananayev, N. S. Nikolayev, and Iu. A. Buslayev. Translated by Lydia Venters (Argonne National Lab.) from Zhur. Neorg. Khim. 1, 274-81(1956). 13p.

FUSED SALT AND LIQUID METAL SYSTEMS 6.10

FUSED SALTS 6.10.01

FLUORIDES 6.10.01.04

The solubility of zirconium fluorides in hydrofluoric acid from 0 to 100% of HF at 0.5°C and the solid phases of the composition $ZrOF_2 \cdot 2H_2O$, $ZrF_4 \cdot 3H_2O$, $H_2ZrF_6 \cdot H_2O$, and ZrF_4 were determined. The procedure for the determination of fluorine and zirconium in combination and the reactions of thermal decomposition of $ZrOF_2$, $ZrOF_2 \cdot 2H_2O$, $ZrF_4 \cdot 3H_2O$, and $H_2ZrF_6 \cdot H_2O$ was studied. (auth)

1717 ✓

AN INVESTIGATION OF THE PHASE DIAGRAM OF THE QUATERNARY SYSTEM CRYOLITE–ALUMINUM FLUORIDE–CALCIUM FLUORIDE–ALUMINA. G. A. Abramov, A. A. Kostyukov, and L. B. Kulakov. Trudy Leningrad. Politekh. Inst. No. 188, 45-57(1957). (Translated from Referat. Zhur. Met. No. 6, 1958, p.35.)

Methods of thermal and optical analysis are used to study the phase diagram of the $5NaF \cdot 3AlF_3-CaF_2-Al_2O_3$ section of the quaternary system $Na_3AlF_6-AlF_3-CaF_2-Al_2O_3$ for the purpose of arriving at a better substantiated approach to the selection of the optimum composition of the electrolyte of Al baths, and in order to develop a crystal optical method of determining the molar ratio $NaF:AlF_3$ in baths containing CaF_2 and a crystal optical method of determining the CaF_2 content of Al bath electrolytes. The following is established: (1) the surface of the liquidus

HIGH-TEMPERATURE CENTRIFUGE FOR FUSED SALT STUDIES: THE INVARIANT POINT BETWEEN LiF AND Li_2BeF_4. Layton J. Wittenberg (Mound Lab., Miamisburg, Ohio). J. Am. Ceram. Soc. 42, 209-11 (1959) May.

A high-temperature centrifugal-filtration apparatus is described for separating the liquid phase from the solid phase in a fused salt system. The filtrate can be observed as it is obtained while the sample is maintained in a controlled atmosphere. The application of this apparatus to a phase rule study is demonstrated by locating the invariant point between the compounds LiF and Li_2BeF_4 in the binary system $LiF-BeF_2$ at 66.7 ± 0.8 mole % lithium fluoride and 456° ± 2°C. (auth)

14234 ✓ NP-7516

TEPLOTY SUBLIMATSII TETRAFTORIDOV URANA, NEPTUNIYA, PLUTONIYA I AMERITSIYA. (Heat of Sublimation of Uranium, Neptunium, Plutonium, and Americium Tetrafluorides.) E. G. Chudinov and D. Ya. Choporov. [nd]. 3p.

The vapor pressure and heat of sublimation of active element fluorides were studied in relation to the pyrometallurgical treatment of nuclear fuels. A modified Knudsen method was used for measuring the tetrafluoride vapors. Radiometric measurements of the quantity of evaporated materials were made starting with the specific activity of the investigated materials. Measurements were made on UF_4 (using the isotopes of U^{233} and mixtures of U^{233} and U^{238}), NpF_4 and PuF_4 (using isotopes of Np^{237} and Pu^{239}), and AmF_4 (using Am^{241}). The tables of sublimation heat and entropy calculated from the obtained vapor pressure values are presented, including data on ThF_4. (R.V.J.)

16535 ✓ CF-58-1-36

Oak Ridge National Lab., Tenn.

SOME ASPECTS OF THE BEHAVIOR OF FISSION PRODUCTS IN MOLTEN FLUORIDE REACTOR FUELS. Mark T. Robinson, William A. Brooksbank, Jr., Samuel A. Reynolds, Henry W. Wright, and Thomas H. Handley. Jan. 17, 1958. 19p. Contract [W-7405-eng-26]. $3.30(ph), $2.40(mf) OTS.

Observations are reported on the behavior of several fission product elements in molten $NaF-ZrF_4-UF_4$ fuels, irradiated in capsule experiments, forced-convection in-pile loop experiments, and in the Aircraft Reactor Experiment (ARE). The rare gases have been observed to escape readily from the fuels in dynamic tests, although in static tests the rate of escape is very low. Ruthenium and niobium deposit on the Inconel walls of the fuel container, probably as metals. Other fission products studied (Sr, Zr, La, Ce) appear to remain in the fuel. The results obtained are entirely consistent with theoretical predictions. It is suggested that the observed noble metal deposit may serve to reduce corrosion of metals by molten fluoride fuels. The unsatisfactory nature of Cs^{137} as a fission monitor in such fuels is reported, and the use of Zr^{95} as a substitute is discussed. (auth)

FUSED SALT AND LIQUID METAL SYSTEMS 6.10

FUSED SALTS 6.10.01

FLUORIDES 6.10.01.04

DISSOLUTION OF METALS IN FUSED FLUORIDE BATHS. R. E. Leuze and C. E. Shilling. July 8, 1954. Decl. Mar. 8, 1957. 5p. (CF-54-7-59)

A method is presented for processing enriched reactor fuel elements. The method calls for contacting the metal with HF in the presence of a molten fluoride bath so that the metal fluoride is dissolved. This exposes fresh metal surface for further reaction. A summary of the results obtained for various metals and various dissolving conditions is given.

PHASE EQUILIBRIA IN THE FUSED SALT SYSTEMS $LiF-ThF_4$ AND $NaF-ThF_4$. R. E. Thoma, H. Insley, B. S. Landau, H. A. Friedman, and W. R. Grimes (Oak Ridge National Lab., Tenn.). J. Phys. Chem. 63, 1266-74(1959) Aug.

Detailed phase equilibrium diagrams are presented for the binary systems $LiF-ThF_4$ and $NaF-ThF_4$. Data for the determination of phase boundaries and solid state transitions were obtained from thermal analysis of heating and cooling curves and by quenching after equilibration. Phases were identified with the use of the x-ray diffractometer and the petrographic microscope. Four compounds, $3LiF{\cdot}ThF_4$, $7LiF{\cdot}6ThF_4$, $LiF{\cdot}2ThF_4$, and $LiF{\cdot}4ThF_4$, have been identified in the system $LiF-ThF_4$. These melt incongruently with the exception of $3LiF{\cdot}ThF_4$. Five compounds, $4NaF{\cdot}ThF_4$, $2NaF{\cdot}ThF_4$, $3NaF{\cdot}2ThF_4$, $NaF{\cdot}ThF_4$, and $NaF{\cdot}2ThF_4$ have been identified in the system $NaF-ThF_4$. These melt incongruently with the exception of $2NaF{\cdot}ThF_4$ and $3NaF{\cdot}2ThF_4$ which melt congruently. A metastable solid phase having the composition $7NaF{\cdot}6ThF_4$ has been identified. The compounds $4NaF{\cdot}ThF_4$ and $3NaF{\cdot}2ThF_4$ do not exist as equilibrium phases at low temperatures. The compound $4NaF{\cdot}ThF_4$ decomposes on cooling into NaF and $2NaF{\cdot}ThF_4$ at 565°; the compound $3NaF{\cdot}2ThF_4$ decomposes on cooling into $2NaF{\cdot}ThF_4$ and $NaF{\cdot}ThF_4$ at 693°. Optical measurements and x-ray diffraction data have been obtained for each of the solid phases observed in the systems $LiF-ThF_4$ and $NaF-ThF_4$. (auth)

21958 ORNL-2421
Oak Ridge National Lab., Tenn.
SOLUBILITY RELATIONS AMONG SOME FISSION PRODUCT FLUORIDES IN $NaF-ZrF_4-UF_4$ (50-46-4 MOLE %). W. T. Ward, R. A. Strehlow, W. R. Grimes, and G. M. Watson. Jan. 30, 1958. 34p. Contract W-7405-eng-26. $1.25(OTS).

A study of the solubility relations among some of the fission products in a fuel mixture was carried out. Fluorides such as those of Ce^{141}, La^{140}, Sm^{153}, Y^{91}, and Ba^{133} were used with $NaF-ZrF_4-UF_4$ fuel solution. Experimental procedure is described and data are tabulated. The data reveal that fission product precipitation in $NaF-ZrF_4-UF_4$ is not a reactor problem. Reasonable estimates of the solubility of other rare earths can be made from a consideration of the atomic radii of those investigated. The chemical feasibility of solid-solvent extraction of rare-earth poisons from the molten fluoride fuel was also established. (J.R.D.)

22006 NP-tr-295
EQUILIBRIUM IN THE SYSTEM $NaF-Na_2BeF_4-H_2O$. A. V. Novoselova. Translated by F. Hilton (U.K.A.E.A. Atomic Energy Research Establishment) from Zhur. Obshcheĭ Khim. 4, 1206-10(1934). (Handwritten MS. copy). 10p. $1.80(ph), $1.80(mf) JCL or LC.

Lixiviation of the melt resulting from reaction of sodium silicofluoride with beryl was investigated for the purpose of optimizing extraction conditions for Be in the form of double salt. Data on the solubility of NaF at 0, 20, 40, 60, 80, and 94°C are tabulated along with the solubility of Na_2BeF_4 at the same temperatures and the joint solubility of $NaF-Na_2BeF_4$. From these data an equilibrium diagram was prepared. (J.R.D.)

21946 CF-59-10-18
Oak Ridge National Lab., Tenn.
RESULTS OF EXAMINATIONS OF FUSED SALT MIXTURES BY OPTICAL AND X-RAY DIFFRACTION METHODS. R. E. Thoma. Oct. 7, 1959. 72p. $12.30 (ph), $4.50(mf) OTS.

A summary of results of examinations of fused salt mixtures is presented. Phase analyses are derived from the two complementing techniques: measurements of optical properties using the polarizing light (petrographic) microscope, and solid phase identification using x-ray-diffraction methods. Investigations of most of the samples on which data are reported were made as a part of a program of phase equilibrium studies of fluoride fused salt mixtures. (auth)

FUSED SALT AND LIQUID METAL SYSTEMS 6.10

FUSED SALTS 6.10.01

MIXED HALIDES 6.10.01.05

5194 AEC-tr-2863
ELECTROMOTIVE FORCES OF THERMOCHAINS OF FUSED SALTS. B. F. Markov. Translated by J. M. Crabtree from Doklady Akad. Nauk S.S.S.R. 108, 115-17 (1956). 5p.

The electromotive forces of thermochains of fused Ag halides in the pure state and in mixtures with the alkali metal halides were measured. (W.L.H.)

9681
COMPLEX IONS IN FUSED SALTS. F. R. Duke and M. L. Iverson (Iowa State Coll., Ames). J. Phys. Chem. 62, 417-19(1958) Apr.

The complex formation constants for a series of bivalent metal halides were determined using fused KNO_3-NaNO_3 eutectic as solvent. The method involved measuring the increase in solubility of the slightly soluble metal chromate as halide ion was added. (auth)

9217
THE SOLUBILITY OF THE POST-TRANSITION METALS IN THEIR MOLTEN HALIDES. John D. Corbett, Samuel von Winbush, and Frank C. Albers (Iowa State Coll., Ames). J. Am. Chem. Soc. 79, 3020-4(1957) June 20.

The solubilities of the respective metals in molten PbI_2, $SbCl_3$, SbI_3, $ZnCl_2$, ZnI_2, CdI_2 and $GaBr_2$ are reported. An interpretation of the solution of the post-transition representative metals in their molten halides is presented in terms of the formation of slightly stable subhalides. The apparent stabilities of the lower halides increase both with increasing atomic weight of the metal within a group, and with increasing atomic weight of the halide with a given metal, except for cadmium. The halide effect is attributed to a corresponding decrease in the extent to which the higher oxidation state of the metal is stabilized by complex formation with halide. The relative

ELECTRICAL CONDUCTANCE OF THE LiCl–KCl EUTECTIC MELT CONTAINING HALIDES AND ALKALI TITANIUM FLUORIDES AS SOLUTES. George J. Janz, C. T. Brown, H. J. Gardner, and C. Solomons (Rensselaer Polytechnic Inst., Troy, N. Y.). J. Phys. Chem. 62, 1479-82(1958) Dec.

The electrical conductances over the temperature range 350 to 400° of the LiCl–KCl eutectic melt and solutions of NaCl, LiF, NaF, KF, Li_2TiF_6, Na_2TiF_6, and K_2TiF_6 in this melt are reported for the region of dilute concentrations of the solutes. A reduced total specific conductance is found on the addition of fluorides to the chloride eutectic. The decrease in total conductance on the addition of the alkali titanium fluorides to the chloride melt is interpreted in light of the previous cryoscopic results as due to a primary dissociation with the formation of alkali fluoride and TiF_4, the latter most probably existing as $(TiF_4Cl_2)^{-2}$ or some similar complex ion. (auth)

13720
ELECTROLYSIS OF THE OXIDES OF NICKEL, COBALT, AND IRON IN A FUSED MIXTURE WITH A BASE OF POTASSIUM FLUOBORATE. S. Aléonard. Bull. soc. chim. France No. 6, 827-9(1958) June. (In French)

If the oxides NiO, CoO, and Fe_2O_3 are dissolved in a fused mixture of potassium fluoride and potassium fluoborate, all the metal deposited in a primary phase in the form of crystalline fines is borided in proportion to the subsequent deposition of boron. The borides of all three metals can be obtained in this manner. (tr-auth)

13725
CRYOSCOPY OF THE LiCl–KCl EUTECTIC MELT CONTAINING ALKALI HALIDES AND ALKALI TITANIUM FLUORIDES AS SOLUTES. G. J. Janz, C. Solomons, H. J. Gardner, J. Goodkin, and C. T. Brown (Rensselaer Polytechnic Inst., Troy, N. Y.). J. Phys. Chem. 62, 823-6(1958) July.

The cryoscopic behavior of NaCl, LiF, NaF, KF, Li_2TiF_6, Na_2TiF_6, and K_2TiF_6 in the LiCl–KCl eutectic melt is reported. In the regions of dilute concentrations, the behavior of the alkali halides is in accord with complete dissociation, the deviations from thermodynamic ideality being attributed to solid solution formation, and for LiF, in addition, an activity coefficient of 2. The cryoscopy of the alkali titanium fluorides similarly is in accord with a primary dissociation to form the alkali fluoride and titanium tetrafluoride in the melt. The results are discussed in the light of the various theories and models of ionic mixtures. (auth)

FUSED SALT AND LIQUID METAL SYSTEMS 6.10

FUSED SALTS 6.10.01

MIXED HALIDES 6.10.01.05

4447 NYO-7741
New York Univ., New York.
TECHNICAL PROGRESS REPORT. Benson R. Sundheim. Oct. 9, 1958. 12p. Contract AT(30-1)-1938. $3.30(ph OTS); $2.40(mf OTS).
The absorption spectra obtained when eutectic mixtures of lithium and potassium chlorides or bromides serve as solvents and alkali halides or halogens serve as solutes are discussed. (W.L.H.)

OTHERS 6.10.01.06

6248
TRANSPORT NUMBERS AND STRUCTURES IN FUSED $AgNO_3$-$NaNO_3$ MIXTURES. Frederick R. Duke, R. W. Laity, and Boone Owens (Iowa State Coll., Ames). J. Electrochem. Soc. 104, 299-300(1957) May.
The transport numbers of the ions in $NaNO_3$-$AgNO_3$ mixtures are very closely proportional to the mole fractions. The transport number of the nitrate ion remains essentially constant. It is thus concluded that there are no complex species in $NaNO_3$ or $AgNO_3$ and that all cations present are structurally in identical positions. This argues strongly against any concept except complete dissociation in this nitrate system. (auth)

8299 ISC-923
Ames Lab., Ames, Iowa.
SURFACE TENSIONS OF SOME BINARY FUSED SALT SYSTEMS. June L. Dahl and F. R. Duke. June 1957. 82p. Contract W-7405-eng-82. $2.25(OTS).
The surface tensions of eight pure fused salts: $NaNO_3$, KNO_3, LiCl, NaCl, $PbCl_2$, KCl, and $ZnCl_2$ and nine binary mixtures: $NaNO_3-KNO_3$, $AgNO_3-NaNO_3$, $AgNO_3-KNO_3$, NaCl−KCl, $PbCl_2$−LiCl, $PbCl_2$−NaCl, $PbCl_2$−KCl, $PbCl_2$−RbCl, and $PbCl_2$−CsCl were measured by the maximum bubble pressure method. Surface tension showed essentially linear variation with temperature in all cases. (W.L.H.)

10367 UCRL-5227
California. Univ., Livermore. Radiation Lab.
FUSED LITHIUM SALTS: A BIBLIOGRAPHY COVERING 1950-57. Dorothy Baughman and G. R. Maynard. May 19, 1958. 13p. Contract W-7405-eng-48. $3.30 (ph OTS); $2.40(mf OTS).

7467 NP-6034
Pennsylvania State Univ., University Park. Coll. of Mineral Industries.
ON THE PROPERTIES OF SOME MIXTURES OF FUSED SALTS. Technical Report No. 69. Tormod Forland. June 1956. 117p. Contract N6-onr-269, Task Order 8.
Properties and particularly the thermodynamic properties, of mixtures of fused salts on the basis of various structural models for the fused mixtures are described. Four idealized models were considered, and thermodynamic functions were derived on this basis. For mixtures of fused salts containing monovalent and divalent cations the available experimental material indicates at least in some cases, that the fused systems may be described as a random mixture of the two kinds of cations, the charge being insignificant for the entropy of mixing. Further, the available experimental material indicates that

3568 CF-58-6-58
Oak Ridge National Lab., Tenn.
MOLTEN SALT COMPOSITIONS. J. P. Blakely. June 12, 1958. 12p. Contract [W-7405-eng-26]. $3.30 (ph OTS); $2.40(mf OTS).
A list of fused-salt mixtures which are of importance in molten salt research is presented. The list is a revision and extension of the list published in ORNL CF-57-6-81. The liquidus temperatures given are believed to be within 10°C of the correct values. (auth)

COMPLEX IONS IN MOLTEN SALTS. IONIC ASSOCIATION AND COMMON ION EFFECT. Ervin R. Van Artsdalen (Oak Ridge National Lab., Tenn.). J. Phys. Chem. 60, 172-7 (1956) Feb.
The concepts of complex ion and ideality are considered in the case of molten salt solutions. Freezing point depression measurements in molten $NaNO_3$ as solvent are used to determine the nature and number of ions into which several solutes dissociate. It is demonstrated that a number of salts of relatively simple oxy-acids as well as many other simple salts are stable and remarkably ideal in solution in molten sodium nitrate. Partial dissociation and com-

FUSED SALT AND LIQUID METAL SYSTEMS 6.10

FUSED SALTS 6.10.01

OTHERS 6.10.01.06

plex ion formation are shown to exist at high dilution in the case of $PbCl_2$, $CuCl_2$, $CdCl_2$, $ZnCl_2$, and $CdBr_2$. The important species in the solutions appear to be those with zero, two and four chlorines per +2 metal ion. The occurrence of the common ion effect is amply substantiated with these compounds and chloro anions containing the +2 metal exist even at low concentrations. Reactions are proposed which account for the results and equilibrium constants are calculated for these reactions. (auth)

16208
TRANSPORT NUMBERS OF THE PURE FUSED SALTS, $LiNO_3$, $NaNO_3$, KNO_3, AND $AgNO_3$. Frederick R. Duke and Boone Owens (Iowa State Coll., Ames). J. Electrochem Soc. 105, 548-9(1958) Sept.

The transport experiments were done in a cell in which the electrodes were of solid Ag surrounded by molten $AgNO_3$. When studying salts other than $AgNO_3$, cells were used which contained a capillary constriction between the alkali nitrate which filled the upper portion of the cell and the $AgNO_3$ at the bottom in contact with the Ag. Transport numbers and equivalent conductances of the ions and salts are tabulated, in addition to experimental and calculated transport numbers. (J.R.D.)

FUSION OF LITHIUM TITANATES WITH OTHER SALTS. I. N. Belyaev and N. P. Sigida (Rostov-on Don State Univ.). Zhur. Obshcheĭ Khim. 26, 1553-8(1956) June. (In Russian)

Investigations were made of lithium titanate fusion with fluorides, sulfates, silicates, pyrophosphates, and other salts of Li, Na, and K. Results showed that at temperatures up to 1100° lithium titanates do not diffuse into salts of Li_2SO_4, Li_2MoO_4, Li_2WO_4, $LiPO_3$, LiCl, and LiF, and in temperatures up to 1100° they do not melt in Li_2SiO_3, Li_3PO_4, Li_3VO_4 (the mixtures with up to 15% Li_2TiO). A certain similarity has been established between lithium titanates and barium titanates by comparing the diffusion of Li_2TiO_3 in the above salts and by establishing the temperatures of methanite melting points. The studies also established that Li_2TiO_3-2NaF, Li_2TiO_3-2KF, Li_2TiO_3-2RbF, and $Li_2TiO_3-Na_2SiO_3$ have stable diagonal cross sections of corresponding triple intersystems. (R.V.J.)

11059 NYO-8021
Tufts Univ., Medford, Mass.
SOLID-LIQUID EQUILIBRIUM IN THE LITHIUM–LITHIUM HYDRIDE SYSTEM. III. Period Covered: June 1, 1956 to June 1, 1957. Charles E. Messer, Regina A. Seales, and John Mellor. July 26, 1957. 12p. Contract AT(30-1)-1410. $3.30(ph OTS); $2.40(mf OTS).

The freezing points of mixtures of Li and LiH as a function of composition have been redetermined on a purer sample than that previously employed. Four individual samples, two in which the composition was varied by removal of hydrogen from LiH and two in which H_2 was added to Li, gave substantial agreement. The freezing point of "stoichiometric" LiH, 99.6 to 99.8% of theoretical H_2, was found to be 688°C ± 1°. Removal of H_2 lowered the freezing point to a monotectic of 685°C ± 1°. The freezing point was essentially constant at this value at compositions

[illegible] CEA-tr-R-[illegible]
CONDUCTIBILITÉ ÉLECTRIQUE DES SULFURES D'ÉTAIN, D'ANTIMOINE, DE BISMUTH ET DE NICKEL À L'ÉTAT FONDU. (Electric Conductivity of the Sulfides of Tin, Antimony, Bismuth, and Nickel in the Fused State.) Yu. K. Delimarskii (Delimarsky) and A. A. Velikanov. Translated into French from Zhur. Neorg. Khim. 3, 1075-8(1958). 12p.

The electric conductivity of Bi_2S_3 and NiS in the fused state exceeds that of fused salts and approaches the conductivity of some metals. The conductivity of Sb_2S_3 has a value of the same order as that of fused salts. Positive temperature coefficients were found for Sb_2S_3 and SnS, but the electric conductivity of Bi_2S_3 and NiS is characterized by negative temperature coefficients. The variation of the specific electric conductivity of the fused sulfides is described by the Frenkel equation. It is hypothesized that the tin and antimony sulfides have a conductivity of electrolytic character, whereas that of bismuth and nickel sulfides is an electron conductivity. The character of the electric conductivity varies in the same order as the chemical properties of the corresponding oxides.

20181 ORNL-2762
Oak Ridge National Lab., Tenn.
DENSITIES OF SOME MOLTEN SALT MIXTURES CONTAINING NITRATES. G. Pedro Smith, Guy F. Petersen, and William M. Ewing. Aug. 21, 1959. 8p. Contract W-7405-eng-26. $1.80(ph), $1.80(mf) OTS.

FUSED SALT AND LIQUID METAL SYSTEMS 6.10

FUSED SALTS 6.10.01

OTHERS 6.10.01.06

Values of the densities of molten salt mixtures have been determined as needed for the computation of molar extinction coefficients from absorption spectra. Constants in the density function measured during the past eighteen months are reported. (auth)

6223

ACID-BASE REACTIONS IN FUSED SALTS. II. THE ABSOLUTE CONCENTRATION OF NO_2^+ ION IN FUSED NITRATE. F. R. Duke and S. Yamamoto (Iowa State Coll., Ames). J. Am. Chem. Soc. 81, 6378-9(1959) Dec. 20.

The reactions $S_2O_7^= + NO_3^- \rightarrow NO_2^+ + 2SO_4^=$ and its subsequent reaction $NO_2^+ + NO_3^- \xrightarrow{slow!} 2NO_2 + O$ were studied kinetically at 235 and 275°C. The equilibrium in the first reaction was found to lie sufficiently far to the right so that its equilibrium constant could be determined. It was possible to establish a scale of acidity in fused nitrates based on the absolute concentration of NO_2^+. (auth)

18893 ISC-992

Ames Lab., Ames, Iowa.

IONIC MOBILITIES IN SELECTED FUSED SALT SYSTEMS. Boone B. Owens and F. R. Duke. Aug. 1957. 60p. $1.75(OTS).

Transport numbers were determined in the pure fused salts $LiNO_3$, $NaNO_3$, KNO_3, and $AgNO_3$ by measuring the volume changes of the material in the electrode compartments which took place during electrolysis of the melt. Transport numbers in the two binary fused salt systems $AgNO_3-NaNO_3$ and $AgNO_3-KNO_3$ were determined by measuring the volume changes accompanying electrolysis and the concentration changes accompanying electrolysis. The transport numbers of the pure fused salts were found to agree with a radius fraction expression which implied that the mobility of an ion moving through a melt is inversely proportional to its size. It is pointed out that this equation failed for other fused salts, so, factors other than the size must also affect the mobility of the ions in a fused salt. The variation with composition of the cation mobilities in the binary systems studied is discussed in terms of ordered and disordered regions in the melt. (auth)

SURVEYS AND THEORY 6.10.01.07

OXIDATION STATES AND COMPLEX IONS OF URANIUM IN FUSED CHLORIDES AND NITRATES. D. M. Gruen and R. L. McBeth (Argonne National Lab., Lemont, Ill.). J. Inorg. & Nuclear Chem. 9, 290-301 (1959) Mar.

Oxidation states of uranium in fused LiCl–KCl eutectic, pyridinium chloride, and $LiNO_3-KNO_3$ eutectic were characterized spectrophotometrically. The (III), (IV), and (VI) oxidation states of uranium are stable in LiCl–KCl eutectic. The (IV) and (VI) oxidation states have been observed in pyridinium chloride. In $LiNO_3-KNO_3$ eutectic the (III) and (IV) states are oxidized to the (VI) oxidation state. It was found that Al as well as U metal reduces U(IV) to U(III) while Mg metal reduces U(III) to U(O) in LiCl–KCl eutectic. On the basis of these experiments the U(III)–U(O) standard potential versus a 1 M Pt reference electrode can be predicted to lie between −1.77 and −2.58 v. The U(IV)–U(III) potential versus the same electrode can be expected to be more positive than −1.77 v. Spectrophotometric evidence indicates that U(IV) changes its co-ordination number from six to eight in going from fused pyridinium chloride to LiCl–KCl eutectic. Changes were observed in the UO_2(II) spectrum on addition of chloride ion to UO_2(II) solutions in $LiNO_3-KNO_3$ eutectic which were correlated with the formation of chlorocomplexes of uranyl ion. (auth)

THE THERMODYNAMICS OF DILUTE SOLUTIONS OF $AgNO_3$ AND KCl IN MOLTEN KNO_3, FROM ELECTROMOTIVE FORCE MEASUREMENTS. I. EXPERIMENTAL. Milton Blander, F. F. Blankenship, and R. F. Newton (Oak Ridge National Lab., Tenn.). J. Phys. Chem. 63, 1259-61(1959) Aug.

Measurements of the activities of $AgNO_3$ were made in a fused salt concentration cell at 370 and 436° in dilute solutions of Ag^+ and Cl^- ions. The activities of $AgNO_3$ obeyed the Nernst law at the low concentrations of $AgNO_3$ measured when no Cl^- ions were present. The dissolution of KCl in a $AgNO_3-KNO_3$ mixture lowered the activity of $AgNO_3$. The lowering was larger the larger the amount of KCl added and for a given concentration of KCl was also larger the smaller the initial concentration of $AgNO_3$. This shows that the Nernst law is not obeyed for $AgNO_3$ in the presence of Cl^- ion. (auth)

FUSED SALT AND LIQUID METAL SYSTEMS 6.10

FUSED SALTS 6.10.01

SURVEYS AND THEORY 6.10.01.07

THE THERMODYNAMICS OF DILUTE SOLUTIONS OF $AgNO_3$ AND KCl IN MOLTEN KNO_3 FROM ELECTROMOTIVE FORCE MEASUREMENTS. II. A QUASI-LATTICE MODEL. Milton Blander (Oak Ridge National Lab., Tenn.). J. Phys. Chem. 63, 1262-5(1959) Aug.

A new theory based on a quasi-lattice model of the molten reciprocal salt system A^+, B^+, C^- and D^- dilute in A^+ ions in which the A^+ and C^- ions have an extra-coulombic interaction yields the result for the activity coefficient of the component

AD: $\gamma_{AD} = \left[(1-X)^Z\left(1+\frac{X}{\beta(1-X)}\right)^{Z-1}\right]N_{D^-}$, where Z is the quasi-lattice coördination number, $\beta = e^{-\Delta E/RT}$, ΔE is the energy of formation of a mole of the ion pairs A^+-C^- and X, which can be calculated from the theory, is the fraction of positions adjacent to an A^+ ion occupied by C^- ions. $(1-X)^Z$ is the fraction of A^+ ions that have no C^- ions adjacent to them. In the "complex" ion terminology these are "free" or "uncomplexed" A^+ ions and $(1-X)^Z$ alone, in that terminology, should be equal to γ_{AD}. The other terms are significant for low values of β and high values of N_{C^-}. A comparison of the theory and measurements of the Ag^+, K^+, Cl^-, NO_3^- system at 370 and 436° indicates that the theory is valid at low values of X (< 0.15) and of N_{Ag^+} ($\leq 0.720 \times 10^{-3}$). A value of $\Delta E = -5.6_6$ kcal./mole and -5.6_4 kcal./mole for the energy of formation of the ion pair Ag^+-Cl^- is derived from the comparison for Z = 6 at 370 and 436°, respectively. While the value of ΔE is slightly sensitive to the arbitrary choice of Z the value of $Z\beta$ is relatively insensitive to Z. At higher concentrations of Ag^+ ions and higher values of X, the error due to the approximations made in the derivation of the theory becomes significant. (auth)

14707 ✓ A/CONF.15/P/1795
Argonne National Lab., Lemont, Ill.
DEVELOPMENTS IN PYROMETALLURGICAL PROCESSING. J. H. Schraidt and M. Levenson. 13p. $0.50 (OTS).

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

The results of recent work on pyrometallurgical methods and on the problems and status of application to actual reactors as a basic part of the reactor fuel cycle are presented. Information on melt refining includes the progress made on oxide drossing of short-cooled uranium and the melt refining of plutonium-rich alloys. This information also includes the fates of short-lived fission products, which have only recently been determined. The status of extraction processes is presented for blanket processing by liquid magnesium extraction of plutonium and for uranium–bismuth fuel purification by fused salt extraction. Considerable new information has been ob-

14613 A/CONF.15/P/940
Argonne National Lab., Lemont, Ill.
THE CHEMISTRY OF FUSED SALTS. Dieter M. Gruen, Sherman Fried, Peter Graf, and Robert L. McBeth. 26p. $0.50(OTS).

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

Recent work done on the chemistry of fused salts is reviewed, and the directions in which further information is being developed is indicated. Oxidation states of many actinide and rare earth ions dissolved in fused alkali and alkaline earth nitrates and chlorides have been determined for the first time by means of high-temperature spectrophotometry. Information concerning coordination numbers in fused salts has been obtained by comparing absorption spectra of transition metal ions in crystals and in melts. An interesting technique for separating metal ions dissolved in fused

15037 ✓ AFOSR-TN-58-773
Rensselaer Polytechnic Inst., Troy, N. Y.
BIBLIOGRAPHY ON MOLTEN SALTS. Technical Note No. 9. Charles T. Brown, Henry J. Gardner, Gerome Goodkin, George J. Janz, Max R. Lorenz, James D. McIntyre, and Cyril Solomons, comps. Sept. 1958. 81p. Project No. 9972–7772. ARDC Contract No. AF49(638)-50. (AD-201776).

Approximately 1000 references covering contributions in the technical press from the earliest date to 1958 are presented. Each reference is listed by author, journal, and title of the article. The subject classification, arbitrarily selected, reflects the particular interests of physical chemistry as it pervades the more specialized

FUSED SALT AND LIQUID METAL SYSTEMS 6.10

FUSED SALTS 6.10.01

SURVEYS AND THEORY 6.10.01.07

fields of electrochemistry, extractive metallurgy, ceramics, and various aspects of high temperature technology. (W.D.M.)

5575 NYO-4805
Massachusetts Inst. of Tech., Cambridge. Dept. of Metallurgy.
KINETICS OF REACTIONS BETWEEN LIQUID METALS AND LIQUID SALTS. Progress Report Covering Period March 1, 1958 to December 15, 1958. Thomas B. King. 24p. Contract AT(30-1)-1985. $4.80(ph OTS); $2.70(mf OTS).
The kinetics of reactions involving both transport control and phase boundary reaction control are being studied by direct rate measurements and by electrochemical polarization techniques. Measurements of diffusion coefficients are proceeding as part of the program to aid in the interpretation of the kinetic results. Transport control is confirmed for some systems, but side reactions have complicated the study of phase-boundary control. (auth)

16927 NAA-SR-Memo-1285
North American Aviation, Inc., [Downey, Calif.]
A PROPOSED PROGRAM FOR THE STUDY OF THE CHEMISTRY OF METAL-SALT SYSTEMS AT HIGH TEMPERATURE. Daniel Cubicciotti, Archie G. Buyers, Francis J. Keneshea, Jr., and Samuel J. Yosim. Feb. 23, 1955. Changed from OFFICIAL USE ONLY Sept. 9, 1958. 10p. $1.80(ph OTS); $1.80(mf OTS).
A program is outlined for a study of metal-salt solutions. Included are investigations of the equilibrium solubilities of certain metals in their salts, some of the properties of the solutions, and the mechanisms of exchange between a liquid metal phase and a fused salt phase. (auth)

20875 AFOSR-TN-58-17
Rensselaer Polytechnic Inst., Troy, N. Y.
PHYSICAL PROPERTIES AND CONSTITUTION OF MOLTEN SALTS—ELECTRICAL CONDUCTANCE, TRANSPORT, AND CRYOSCOPY. Technical Note No. 6. Cyril Solomons and George J. Janz. Dec. 1957. 98p. Project Chem 40-45. Contract AF49(638)-50. (AD-148056).
A review was made of the literature on theories and experimental techniques for electrical conductance, electrical transport, and cryoscopy of molten salt systems. Structural and constitutional aspects are emphasized. 278 references. (auth)

5278
A PRELIMINARY SPECTROPHOTOMETRIC STUDY OF COMPLEX IONS IN FUSED SALTS. D. M. Gruen (Argonne National Lab., Lemont, Ill.). J. Inorg. and Nuclear Chem. 4, 74-6(1957) Feb.
A spectrophotometric technique for the investigation of complex ions of transition metals in fused salts is presented. (W.L.H.)

5191 ✓ AEC-tr-2834
COMPLEX FORMATION IN BINARY FUSED SALTS. L. N. Antipin. Translated from Uspekhi Khim. 25, 632-42 (1956). 17p.
An abstract of this paper appears in Nuclear Science Abstracts as NSA 11-96. (T.R.H.)

1098 ✓ CF-56-8-61
Oak Ridge National Lab., Tenn.
PREDICTION OF THE THERMAL CONDUCTIVITY OF FUSED SALTS. W. R. Gambill. Aug. 10, 1956. Decl. Sept. 26, 1958. 10p. Contract [W-7405-eng-26]. $1.80(ph OTS); $1.80(mf OTS).
A method for predicting thermal conductivities of fused salts and salt mixtures at their melting points was developed. It allows for the atomic or lattice portion arising from the short-range order present in liquids, and the ionic portion arising from a drift of ions and subsequent energy transfer between the atoms.

4377
CHROMATOGRAPHY AND SPECTROPHOTOMETRY OF FUSED SALTS. Dieter M. Gruen (Argonne National Lab., Lemont, Ill.). Nature 178, 1181-2(1956) Nov. 24.
Chromatographic adsorption on alumina of transition metal cations, Fe^{3+}, Co^{2+}, Ni^{2+}, Cu^{2+}, and UO_2^{2+} in $LiNO_3$ - KNO_3 eutectic mixtures are discussed. (W.L.H.)

FUSED SALT AND LIQUID METAL SYSTEMS 6.10

FUSED SALTS 6.10.01

SURVEYS AND THEORY 6.10.01.07

8237 NP-5942
Stanford Research Inst., Menlo Park, Calif.
THE THERMODYNAMIC PROPERTIES OF MOLTEN SALTS. Report No. 7. Bimonthly Progress Report for February–March 1956. Orlo E. Myers. Apr. 5, 1956. 8p. SRI Project No. CU-1102. Task No. 73017. Contract AF 33(616)-2558.

The objective of this study is to determine the absolute entropies, heats of formation, and free energies of formation of selected metallic fluorides in the temperature range of 800 to 1900°F. An electroformed nickel capsule to contain corrosive and hygroscopic fluorides for high-temperature calorimetry was constructed. Low-temperature heat capacity data for NbF_5 were prepared for machine computation. A drop-calorimeter installation is described for high-temperature heat capacity determinations. This apparatus lacks only the precision electrical connections and final adjustment for its completion. (auth)

6754 NRL-Memo-603
Naval Research Lab., Washington, D. C.
THERMAL AND RELATED PHYSICAL PROPERTIES OF MOLTEN MATERIALS. Progress Report No. 17 [for] February 1–April 30, 1956. Chemistry Div. B. E. Walker, C. T. Ewing, and R. R. Miller. June 7, 1956. 6p.

The final determinations of heat content and specific heat of salt "D" have been completed over the range 30 to 900°C. Salt "F" is being measured to further evaluate the hypothesis on the prediction of specific heat on salt mixtures. For the thermal conductivity apparatus, Hastelloy B, Inconel, and vacuum poured Monel are being procured for use with the salt mixtures. (auth)

4555
PYROPROCESSING FOR NUCLEAR FUELS. Robert C. Reid and J. Edward Vivian (Massachusetts Inst. of Tech., Cambridge) and Dick Duffey (Univ. of Maryland, College Park). Nucleonics 14, No. 2, 22-5(1956) Feb.

Pyroprocessing of nuclear fuels by molten metal or fused-salt extraction and slagging, is offered as a more convenient and less expensive method than the currently used aqueous process. The basic components of a pyroprocessing system are outlined. (D.E.B.)

PYROPROCESSING METHODS FOR NUCLEAR REACTOR FUELS. J. R. Foltz, F. D. Rosen, and W. J. Gardner (North American Aviation, Inc., Canoga Park, Calif.). Chem. Eng. Progr. 52, Symposium Ser. No. 19, 117-22 (1956).

Some of the advantages of pyroprocessing fused salt and metallic spent fuels are reviewed, and the basic chemistry of several proposed processes is briefly discussed. Small-scale operations are described as an approach to approximating full-scale operations. Some of the types of problems encountered during small-scale operations and their solutions are developed. (auth)

8186 AEC-tr-2543
THE RELATIONSHIP BETWEEN CONCENTRATION AND ACTIVITY IN FUSED SALT MIXTURES. H. Flood, O. Fykse, and S. Urnes. Translated from Z. Elektrochem. 59, 364-70(1955). 20p.

The activities of the salt components of the four binary mixtures NaF–KCl, NaCl–KF, LiF–KCl, and LiCl–KF are determined from the liquid curves of the equilibrium diagrams. The results are compared with the values calculated with the help of a general formula for the activities in salt mixtures. It becomes evident that in all the mixtures the deviations from the ideal agree with Temkin's structural model of the fusions. With higher lattice energies (NaF, LiF), a simplified theoretical formula clearly randers values which are slightly too high for the activity coefficients of their components. For components with lower lattice energies the agreement is quite satisfactory; because of solidification of the solutions, however, the components NaCl and KCl cannot be more thoroughly examined. (auth)

635 NP-5809
Stanford Research Inst., Menlo Park, Calif.
THE THERMODYNAMIC PROPERTIES OF MOLTEN SALTS. Report No. 4. Bimonthly Progress Report for August–September 1955. A. P. Brady. Oct. 10, 1955. 7p. SRI Project No. CU-1102. Contract AF 33(616)-2558.

The absolute entropies, heats of formation, and free energies of formation of selected salts in the temperature range of 800 to 1900°F are determined. Assembling and testing of apparatus for measuring heat capacities from about 50°K to room temperature has been completed, including calibration of thermocouples by comparison with a platinum and with a copper resistance thermometer.

FUSED SALT AND LIQUID METAL SYSTEMS 6.10

FUSED SALTS 6.10.01

Surveys and Theory 6.10.01.07

This apparatus has been used to determine the absolute entropy of molybdenum hexafluoride vapor at 298.16°K. The result, 79.7 ± 0.6 cal/mole °C, agrees well with that calculated from its spectrum, 80.05 cal/mole °C. A solution calorimeter has also been assembled and tested. By comparing the heat of solution in aqueous sodium hydroxide of molybdenum hexafluoride with that of the oxide, the ΔH of formation of the fluoride was found to be −392.2 cal/mole at 298.16°K. These data, together with other data extant in the literature, allow calculation of the desired thermodynamic functions over the temperature range of interest. (For preceding period see NP-5685.) (auth)

LIQUID METALS 6.10.02

See also, under PLANTS AND EQUIPMENT,

HIGH TEMPERATURE SYSTEMS--Vessels (5.10.04).

GENERAL 6.10.02.01

8679 TID-3544
Technical Information Service Extension, AEC.
LIQUID METAL TECHNOLOGY. A Literature Search. James M. Jacobs, comp. Jan. 1960. 27p. OTS.

Two hundred seventeen references to unclassified reports and published literature on liquid metal technology are presented. (W.L.H.)

PYROMETALLURGICAL PROCESSING. Premo Chiotti and Adolf F. Voigt. 17p. (ISC-1018). A/CONF.15/P/517

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

A brief review of the general methods used in the pyrometallurgical purification of liquid metals is given with special emphasis on recent work on the pyrometallurgical separation of fission products and Pu from U and the separation of U from Th. The use of thermodynamic data in estimating the effectiveness of various pyrometallurgical separations is considered. Those fission products which extract into Ag, including the alkaline earths, rare earths, and others, can readily be removed from the silver layer by melting the latter with NaCl containing a small amount of AgCl. The Ag is thus recovered in good condition for reuse and the extracted material is concentrated. Extraction studies were made with Ag on a U-0.2% Pu alloy to determine the behavior of Pu in the system at this concentration. Other extractants, rare earths, and alkaline earths were also studied in extractions with this alloy. Relative to the problem of separating small amounts of U from Th the phases which exist in Mg-rich Mg-Th-U alloys were determined. The solubility of U in Mg, Mg-16 wt. % Th, Mg-35 wt. % Th and Mg-45 wt. % Th liquid solutions was measured. Various possible purification procedures for the removal of fission products from the Th-Mg solution or from the massive uranium obtained are considered. Th can be separated from Mg by distillation of the Mg. An alternate procedure wherein the Th is precipitated as ThH_2 was investigated. The major part of the Th can be precipitated at 675°C and one atmosphere H pressure. Under these conditions the residual Th content in the Mg was found to be 8.1 wt. %. More complete separation can be obtained by lowering the temperature and/or increasing the H pressure.

PURIFICATION OF REACTOR FUELS AND BLANKETS BY CRYSTALLIZATION FROM LIQUID METAL SOLVENTS. Harold M. Feder and Robert J. Teitel. 37p. (A/CONF.15/P/540)

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

A review of investigations of reactor fuels and blanket reprocessing methods which have common features is presented. These methods include dissolution of the irradiated material in a low-melting liquid metal, with separation by selective oxidation and reduction, and cooling to form intermetallic compounds of Th, U, or Pu with the solvent metal. During crystallization, separations may take place because of favorable solubility relations or coprecipitation, with final separation by filtration, centrifugation, or decantation, and subsequent volatilization or leaching to recover the desired material. The specific recovery processes are discussed, and possible areas of

FUSED SALT AND LIQUID METAL SYSTEMS 6.10

LIQUID METALS 6.10.02

GENERAL 6.10.02.01

future applications are pointed out, along with a discussion of unsolved problems. 76 references.

16605
FUEL AND BLANKET PROCESS DEVELOPMENT.
L. Burris, Jr. (Argonne National Lab., Lemont, Ill.).
p.263-71 of PROCEEDINGS OF THE 1957 FAST REACTOR INFORMATION MEETING HELD AT CHICAGO, Ill., NOVEMBER 20-21, 1957.

With the emphasis on the importance of the over-all fuel cycle in fast reactors, attention is now being focused on one of the steps within the fuel cycle—fuel reprocessing. Major types of fuel processing are discussed, with emphasis being given to the more recent development of pyrometallurgical processes which have many potential advantages in processing fast reactor fuels. Types discussed include solvent extraction, fluoride volatility processes, pyrometallurgical process development, fractional distillation or sublimation, fractional crystallization, liquid-liquid extraction, cyclic oxidation and reduction, and electrochemical. (W.D.M.)

20001
REVIEW OF DEVELOPMENTS IN REPROCESSING OF IRRADIATED NUCLEAR FUELS BY PYROMETALLURGICAL METHODS. Presented at Nuclear Engineering and Science Conference, April 6-9, 1959, Public Auditorium, Cleveland, Ohio. Preprint V-50. L. F. Coleman, J. H. Schraidt, and G. J. Bernstein (Argonne National Lab., Lemont, Ill.). New York, Engineers Joint Council, 1959. 29p. $0.50.

Results of experimental programs indicate the problems and promises of pyrometallurgical methods for reprocessing irradiated nuclear fuels. The processes of melt refining, solvent extraction by fused salts and liquid metals, and fractional crystallization from metal solvents, being developed are discussed. (auth)

14812 A/CONF.15/P/538
Argonne National Lab., Lemont, Ill.
DEVELOPMENTS IN MELT REFINING OF REACTOR FUELS. L. Burris, N. R. Chellew, S. Lawroski, G. A. Bennett, A. A. Chilenskas, M. Ader, H. M. Feder, J. B. Knighton, I. O. Winsch, J. Wolkoff, and W. A. Rodger. 12p. $0.50(OTS).

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

After investigation of various oxides, zirconia were selected as the crucible material. Data are presented on cerium removal rates from up to 5 kg of material, determined as a function of temperature. Studies were also made on the possibility of effecting separation of zirconium as a carbide or nitride. Separation of purified metal is effected by top pouring. On a 2 to 5 kg scale purified ingot yields were slightly greater than 95% using charges of small reactor fuel pins. Methods are discussed for handling fission product gases and volatilized metals including sodium which may accompany the fuel pin charges and the volatile fission product metals such as cesium. A brief description is given of the auxiliary process under development for processing crucible residues and material deliberately removed to provide the necessary periodic removal of noble elements. A description of the equipment and tests of various materials of fabrication are discussed. The results of preliminary experiments to ascertain the behavior of plutonium are also described. (auth)

PYROMETALLURGICAL TREATMENTS OF NUCLEAR FUELS. Jean Petit (Commissariat a l'Energie Atomique, Saclay, France). pp. 363-79 in "Atti del Congresso Scientifico. Volume I. Sezione Nucleare, Giugno 1957." (In Italian)

The pyrometallurgical processing of irradiated nuclear fuel is reviewed. The processes used in this treatment include distillation, fractional crystallization liquid-liquid extraction with molten metals, formation of oxides, formation of halides, electrolysis, and the iodide of the Van Arkel process, and each is briefly discussed. The potential value of pyrometallurgical procedures is considered with respect to the decontamination efficiency, the products obtained, and the installations needed. A brief description is given of pilot plants in the United States which process irradiated fuels by pyrometallurgical techniques. (J.S.R.)

THE CHEMISTRY OF PYROMETALLURGICAL PROCESSES: A REVIEW. Harold M. Feder. May 1, 1957. p.667-718 of SYMPOSIUM ON THE REPROCESSING OF

FUSED SALT AND LIQUID METAL SYSTEMS 6.10

LIQUID METALS 6.10.02

GENERAL 6.10.02.01

IRRADIATED FUELS HELD AT BRUSSELS, BELGIUM, MAY 20-5, 1957. 1165p. (TID-7534(Bks. 1-3))

The scope of this review is judged to be the separation of U, Pu, Th, fission elements or other unwanted elements from each other in whatever combination they may occur and to whatever extent is required in connection with neutron reaction technology. However, useful examples which are only indirectly connected with such technology will be cited occasionally. 84 references.

PYROMETALLURGICAL PROCESSING OF NUCLEAR MATERIALS. H. M. Feder. August 1955. 5p. (UN-544)

Prepared for the First U. N. International Conference on the Peaceful Uses of Atomic Energy, 1955.

A detailed examination of oxidative slagging is presented. This process is designed to remove from irradiated U or U-Pu alloy a significant fraction of the fission product elements in order to permit the reuse of the metal as pile fuel. The success of the purification will be judged by the mechanical strength, thermal stability, radiation damage stability and nuclear properties of the purified material. 7 references.

3727 NAA-SR-3703

Atomics International Div., North American Aviation, Inc., Canoga Park, Calif.

PROCESSING REFABRICATION EXPERIMENT (PRE) PROGRAM BIBLIOGRAPHY. G. L. Schalin. Nov. 15, 1959. 13p. Contract AT(11-1)-GEN-8. OTS.

A listing is presented of all documents concerning the Pyroprocessing Refabrication Experiment (PRE), later renamed Processing Refabrication Experiment, from its inception as a project under AEC Contract AT(11-1)-GEN-8, General Order 7501, in June 1956, until its phase-out in July 1959. Also listed are documents released prior to the establishment of the Pyroprocessing Refabrication Experiment program at Atomics International that contain essential technical information upon which the PRE was based. (auth)

5392 NP-tr-36

METALLOTHERMIC METHODS IN CHEMISTRY AND METALLURGY. G. V. Sansonov and Yu. D. Chistyakov. Translated by J. M. Crabtree from Uspekhi Khim. 25, 1223-48(1956). 24p.

Theoretical, experimental, and practical data on metallothermic methods for the extraction, purification, and production of metals and alloys are reviewed. The mechanism of metallothermic reduction is examined in a general manner. Three fundamental processes of metallothermic reduction are reviewed: metallothermic process carried out in vacuo, the process conducted at atmospheric pressure without a protective atmosphere, and the process carried out in an atmosphere of an inert gas. The in vacuo technique is used for the production of Ba, Sr, Li, and Be. Cr is produced at atmospheric pressure without a protective atmosphere. Ti, Zr, and Ta are produced in an atmosphere of an inert gas. The production of alloys and preparation of refractory materials by this method are also reviewed. 45 references. (J.S.R.)

PYROMETALLURGICAL PROCESSING: ECONOMICS AND PROPOSED ENGINEERING APPLICATIONS. J. H. Schraidt, W. A. Rodger, M. Levenson, S. Lawroski, D. C. Hampson, J. Graae, L. F. Coleman, L. Burris, Jr., G. J. Bernstein, and G. A. Bennett. p.748-804 of SYMPOSIUM ON THE REPROCESSING OF IRRADIATED FUELS HELD AT BRUSSELS, BELGIUM, MAY 20-5, 1957. 1165p. (TID-7534(Bks. 1-3))

Pyrometallurgical methods for the reprocessing of irradiated fuels from three demonstration reactors are described. The reactors involved are the Experimental Breeder Reactor No. II, Sodium Reactor Experiment, and Liquid Metal Fueled Reactor. Economics of the processes are discussed and flow-sheets are given.

10510 LM/TAB-4

Mine Safety Appliances Corp., Callery, Penna.

LIQUID METALS TECHNOLOGY ABSTRACT BULLETIN FOR THE PERIOD JANUARY 1 TO May 31, 1957. June 18, 1957. 28p.

2338 LM/TAB-5

MSA Research Corp., Callery, Penna.

LIQUID METALS TECHNOLOGY ABSTRACT BULLETIN FOR THE PERIOD JUNE 1 TO OCTOBER 31, 1957. Nov. 11, 1957. 37p.

FUSED SALT AND LIQUID METAL SYSTEMS 6.10

LIQUID METALS 6.10.02

GENERAL 6.10.02.01

HIGH TEMPERATURE FUEL PROCESSING CONFERENCE HELD AT AMES LABORATORY, NOVEMBER 10-11, 1953. Oct. 6, 1954. Decl. Mar. 4, 1957. 44p. (TID-5189)

A survey on high-temperature fuel processing was presented by representatives of laboratories actively engaged in research on this program. Studies reported include volatilization of Pu and fission products, scavenging, liquid salt extraction, electrolytic refining, C slagging coupled with UCl_3 extraction, fluorine extraction, and liquid metal extraction.

VACUUM METALLURGY. Materials Technology Series. Rointan F. Bunshah, ed. New York, Reinhold Publishing Corp., 1958. 484p.

The first part of this volume discusses vacuum equipment, including pumping systems, gages, leak detection, and trouble shooting. Part two describes the kinetics of vacuum metallurgy and kinetics in vacuum induction and vacuum arc melting. Part three presents various arcs and arc-melting processes. Part four describes induction melting of ingot products and cast shapes. The fifth part presents electron bombardment melting techniques. Part six discusses degassing in the liquid and solid states. Part seven is devoted to the distillation of metals. The last two parts discuss the metallurgical application of vacuum processing and analytical techniques. (W.L.H.)

THE REMOVAL OF GASEOUS IMPURITIES BY VACUUM ARC MELTING. Stanley J. Noesen (General Electric Research Lab., Schenectady, N. Y.). pp. 150-6 in "1957 Fourth National Symposium on Vacuum Technology Transactions."

The pressure of the non-condensable gases in a consumable electrode arc was measured. This pressure was obtained by utilizing a hollow molybdenum arc melting electrode. A pressure of 3000 microns was measured in the arc zone of the melting crucible while a pressure of less than 1 micron existed in the furnace body directly above the melting crucible. Using fairly impure raw materials, the degree of purification obtainable in this equipment was also determined. In the molybdenum alloy melted, it was found that oxygen was reduced from 780 ppm to 4 ppm, nitrogen was reduced from 190 ppm to 6 ppm and hydrogen was reduced from 130 ppm to < 1 ppm. The mode of purification is discussed. Finally, some observations are made concerning the criteria for choosing a particular vacuum system to do a specific vacuum arc melting job. (auth)

THE CHEMICAL PROCESSING OF USED REACTOR FUELS. I. L. Jenkins. New Scientist 4, 1443-5(1958) Dec. 4.

Two main types of liquid metal processes for dealing with metallic fuels were examined. In one system plutonium is separated from uranium; in the other, they are kept together. An apparatus used for small-scale processing experiments is described. The main advantage of liquid metal processes compared with aqueous solvent extraction methods are: a quick turn-round of fuel, much smaller volumes of active residues, and some easement of the criticality limitations. (J.H.M.)

LIQUID METALS TECHNOLOGY—Part I. C. F. Bonilla and others. (Chemical Engineering Progress Symposium Series, no. 20) New York, American Institute of Chemical Engineers, 1957. 84 p. $4.25.

Part I of this series on problems in liquid metals is comprised of most of the papers on this subject sponsored by the A. I. Ch. E. during 1956 and 1957.

5375 LM/TAB-6
MSA Research Corp., Callery, Penna.
LIQUID METALS TECHNOLOGY ABSTRACT BULLETIN FOR THE PERIOD NOVEMBER 1 TO JANUARY 15, 1958. Feb. 1, 1958. 23p.

FUSED SALT AND LIQUID METAL SYSTEMS 6.10

LIQUID METALS 6.10.02

EQUIPMENT 6.10.02.02

See also, under PLANTS AND EQUIPMENT,
HIGH-TEMPERATURE SYSTEMS--Furnaces (5.10.01),
Vessels (5.10.04),
TRANSFER OF FLUIDS--Pumps (5.16.03).

11820 ✓ ASAE-26
American-Standard. Atomic Energy Div., Redwood City, Calif.
MATERIALS FOR LIQUID METAL SYSTEMS. William Holman. Oct. 28, 1957. 32p. $6.30(ph), $3.00(mf) OTS.
Lecture presented at the Atomic Industrial Forum's Third Course on Reactor Materials, Stanford University, Stanford, California, July 9-19, 1957.
A review of progress in the use of liquid metals is presented. A number of liquid metals are compared, and the general problems of selecting container materials are considered. In addition, a discussion of corrosion mechanisms and corrosion testing methods commonly employed are examined as well as problems encountered with the liquid metals of greatest interest. (J.R.D.)

IMPROVEMENTS IN OR RELATING TO THE VACUUM MELTING OF HIGH-MELTING METALS AND ALLOYS BY MEANS OF AN ELECTRIC ARC. (to W. C. Heraeus G.m.b.H. Co.) British Patent 809,444. Feb. 25, 1959.
Two vacuum arc furnaces which make their own consumable electrodes are described. In one, Zr or Ti powder is mechanically pressed in the top of a tube at 2 to 10 kg/cm^2. As the powder compact rod moves down toward the arc chamber, it is degassed by argon sweeping and by vacuum pumps. In the evacuated arc chamber it melts in an arc formed by current supplied from below. The bottom of the water cooled arc chamber may be lowered by a mechanism as the arc-melted ingot forms. The second furnace design differs from the first in that prefabricated rods of Ti or Zr electrode are introduced into a tube at the top and are arc welded together in one chamber, then degassed and melted as before. (T.R.H.)

18031 AERE-Bib-120
United Kingdom Atomic Energy Authority. Research Group. Atomic Energy Research Establishment, Harwell, Berks, England.
PUMPS AND ELECTROMAGNETIC FLOWMETERS FOR LIQUID METALS: BIBLIOGRAPHY. May 1959. 28p. $0.63(BIS).
Supersedes AERE-Inf/Bib-93(4th Ed.).
This bibliography of unclassified literature supersedes AERE-Inf/Bib-93(4th edition), which was compiled in 1956. The scope has been widened to include references on mechanical pumps for liquid metals, as well as on the electromagnetic types. Recently declassified reports have been included. Patent literature and Progress Reports other than Final Reports on projects have been omitted. In the section on electromagnetic flowmeters, a number of general papers, which do not refer specifically to liquid metals, but which are, nevertheless, of considerable interest, have been included. Author and report number indexes have been added at the end of the bibliography. (auth)

20133
ELECTROMAGNETIC PUMPS. P. Fortescue (to UK Atomic Energy Authority). British Patent 804,621. Nuclear Eng. 4, 324(1959) July-Aug.-Sept.
A liquid metal duct is arranged axially within the poles of a three-pole circular iron yoke structure. A magnetic field is created transversely across parts of the duct section in turn by field coils on the yoke (with secondary windings and electrodes around the duct) excited by three-phase current and thus a longitudinal pumping action is effected on the liquid metal in the duct.

PROPERTIES 6.10.02.03

THE THERMODYNAMIC PROPERTIES OF DILUTE ALKALI METAL AMALGAMS. Harold L. Friedman and Kenneth Schug (Univ. of Southern California, Los Angeles). J. Am. Chem. Soc. 78, 3881-8(1956) Aug. 20.
The thermodynamic properties of ternary amalgams containing Na with K, Rb, Cs or Sr have been investigated by an equilibrium method. The results, together with independent data for Na amalgam, yield properties of the binary amalgams of K, Rb, Cs and Sr. When combined with

FUSED SALT AND LIQUID METAL SYSTEMS 6.10

LIQUID METALS 6.10.02

PROPERTIES 6.10.02.03

data from other sources, these provide information on the formation of the alkali metal amalgams from the elements and the deviations from Raoult's law of these solutions. For the process M(Hg, Hyp. 1 molal) = M^+(gas) + e^-(gas), ΔH decreases from Li to Cs, but ΔS increases strongly, becoming +30 e.u. for Cs. This and several other amalgam properties are consistent with the hypothesis that the alkali metals are ionized in the amalgams and that only the electron tends to enter the mercury phase from the gas phase. Then the positive ions tend to escape the amalgam, the

12739 ✓ ANL-5753
Argonne National Lab., Lemont, Ill.
A SURVEY OF METAL SOLUBILITIES IN LIQUID ZINC. M. W. Nathans. July 1957. 72p. Contract W-31-109-eng-38. $2.00(OTS).

A program was initiated on the development of a fuel decontamination process by means of fractional precipitation from metallic zinc solution. In order to gather background material on alloy systems of interest, a literature survey of the binary phase diagrams of zinc in the zinc-rich regions (>50 a/o zinc) was undertaken, as well as the crystallography of the solid phases which are expected to precipitate from solution, the latter for purposes of identification. 79 references. (auth)

1222
MEASUREMENT OF THE RELATIVE MOBILITY OF CATIONS IN MIXTURES OF OXIDE MELTS. Presented at the International Conference on Radioisotopes in Scientific Research, Sept. 9-20, 1957 at Paris. No. UNESCO/NS/RIC/69. V. I. Malkin and L. A. Schwarzman. London, Pergamon Press, Ltd., 1957. 8p.

ACTIVATION ENERGY VALUES FOR SPONTANEOUS DIFFUSION IN LIQUID METALS. K. A. Osipov (Baikov Inst. of Metallurgy, Academy of Sciences, USSR). Doklady Akad. Nauk S.S.S.R. 121, 1019-20(1958) Aug. 21. (In Russian)

An equation was formulated for expressing the activation energy values for spontaneous diffusion and viscosity of liquid Al, Fe, Mg, Na, Pb, and Sn. (R.V.J.)

6234
MISCIBILITY OF LIQUID METALS WITH SALTS. IV. THE SODIUM–SODIUM HALIDE SYSTEMS AT HIGH TEMPERATURES. M. A. Bredig and H. R. Bronstein (Oak Ridge National Lab., Tenn.). J. Phys. Chem. 64, 64-7 (1960) Jan.

The use of thermal analysis and of equilibration with a new method of sampling yielded improved data for consolute temperatures and compositions as well as for the solubilities of the metal in the molten salts in the lower temperature range. The consolute compositions of 28 mole % Na (at 1180°C), 50 mole % Na (1080°C), 52 mole % Na (1026°C), and 59 mole % Na (1033°C) for the NaF–Na, NaCl–Na, NaBr–Na, and NaI–Na systems, respectively, reflect the influence of the relative sizes of the metal and halide constituent on the symmetry of the phase diagrams. The depression of the melting point of the salt represented by the monotectic temperature and composition of the salt-rich phase corresponds to a positive deviation from Raoult's law similar to the one observed in the KX–K systems. A similar tentative interpretation, the association of the metal to diatomic molecules, is proposed. (auth)

5362 ✓
HEAT TRANSFER AND THERMOPHYSICAL PROPERTIES OF MOLTEN ALKALI METALS. I. I. Novikov, A. N. Solovyev, E. M. Khabakhpasheva, V. A. Gruzdev, A. I. Pridantsev, and M. Ya. Vasenia. Soviet J. Atomic Energy 4, 545-60(1956).

An investigation was undertaken of heat transfer to molten sodium during turbulent flow in a round copper or nickel tube. An interpolation formula was obtained. Experiments were conducted to determine thermal resistance between liquid sodium and a solid wall of copper, nickel, and stainless steel. Methods were developed for measurement of viscosity, temperature conductivity and density of molten metals. Results are given for measurements of these physical parameters for molten alkali metals (sodium, potassium, lithium and the eutectic solution of sodium and potassium) in a broad interval of temperatures. (auth)

FUSED SALT AND LIQUID METAL SYSTEMS 6.10

LIQUID METAL 6.10.02

Properties 6.10.02.03

5743 NRL-Memo-561
Naval Research Lab., Washington, D. C.
THERMAL AND RELATED PHYSICAL PROPERTIES OF MOLTEN MATERIALS. Progress Report No. 15 [for] August 1–October 31, 1955. B. E. Walker, C. T. Ewing, and R. R. Miller. Jan. 1956. 7p.

Heat content and heat capacity measurements have been made on salt "E" from 30 to 900°C. Measurements in the liquid range were at 500, 600, 700, 800, and 900°C. Results on heat capacity over this range varied from 0.45 to 0.41 cal/g 1°C. Preliminary measurements on heat content of salt "D" have been made. (For preceding period see NRL-Memo-516.) (auth)

THEORY 6.10.02.04

5591 NP-tr-330
HYDRODYNAMICS OF MOLTEN METALS. (Proceedings of the First Conference on the Theory of Casting Processes). (Gidrodinamika Rasplar Lennykh Metallor). (Trudy Pervogo Soveshchaniya po Teorii Litenynykh Protsessor). L. I. Fantalov and B. B. Gulyaeev (Gulyayev), eds. Translated from a publication of the Publishing House of the Academy of Sciences, Moscow. 1958. 384p. OTS.

Results of theoretical and experimental research on the dynamics of molten metals are presented. The viscosity properties of molten metals are examined along with the relationship of the liquid phase and the metallic aggregate formed from it. Metal flow in conduits, the causes of defects in castings, the effects of aluminum on steel, the relationship between the structural diagram of molten metal and its mechanical properties, and theoretical problems are also considered. (J.R.D.)

10637
THERMODYNAMICAL SIMILARITY AND THE VISCOSITY OF FUSED METALS. A. N. Solov'yev. Atomnaya Energiya 3, 550-2(1957).

An attempt was made to find a relationship between the thermodynamic and physical properties of a number of elements. The viscosity of fused Na, K, Li, Ru, Cs, Sn, Hg, Sb, Bi, and Ga was plotted against temperature. The curves fell into two groups, one for the alkali metals and the other for the rest of the metals. The hypothesis is made that the physical properties of similar metals could be deduced if the physical properties of one were accurately known. (J.S.R.)

3821 SO-2046
General Electric Co. Research Lab., Schenectady, N. Y.
SELF-DIFFUSION IN DILUTE BINARY SOLID SOLUTIONS, II. E. W. Hart, R. E. Hoffman, and D. Turnbull. Apr. 1956. 11p. Contract W-31-109-Eng-52. $1.80(ph OTS); $1.80(mf OTS). (56-RL-1530).

6396 AEC-tr-2900
THE POSSIBILITY OF USING SIMILARITY METHOD TO DETERMINE LIQUID-METAL PARAMETERS. I. M. Kirko and O. A. Lielausis. Translated by M. C. Wheeler from Fiz. Metal. i Metalloved. Akad. Nauk S.S.S.R., Ural. Filial 2, 563-4(1956). 2p.

7208
STRUCTURE OF LIQUID METALS. I. V. Radchenko. Uspekhi Fiz. Nauk 61, 249-76(1957) Feb. (In Russian)

Roentgenographic and neutronographic investigations showed that the atomic packing in liquid metals is related to the atomic packing in the solid state, however the character of such bond depends on the type of the metal. The coordination number of all metals either increases or changes. The indication of reduced coordination numbers for Pb, Tl, and In was not sufficiently substantiated due to the side maximum appearing on the atomic distribution curve. This could be an error incurred in developing the intensity distribution curves. The temperature relations of the intensity curves of K, Tl, Sn and other metals show that liquid metals retain their type of packing at considerably higher temperatures than the melting point. (R.V.J.)

2089
THERMAL DIFFUSION IN LIQUID METALS. F. R. Winter and H. G. Drickamer (Univ. of Illinois, Urbana). J. Phys. Chem. 59, 1229-30(1955) Dec.

Thermal diffusion measurements have been made on a series of liquid metal mixtures. The results are totally consistent with a previously presented theory. The separations depend on the "activation energy density" $\Delta U \neq /V$ of the components. It is also shown that the quantity $X(\partial\mu/\partial X)$ in the denominator is important for quantitative prediction of the separation. Results also are presented and discussed for three ternary mixtures. (auth)

HEAT TRANSFER 6.11

See also, under WASTE TREATMENT AND DISPOSAL, DECAY STORAGE--Heat Problems (8.02.01).

HEAT TRANSFER ENGINEERING. Hilbert Schenck, Jr. Englewood Cliffs, N. J., Prentice-Hall, Inc., 1959. 318p.

The book rests on two fundamental concepts: the first law of thermodynamics, referred to as the "energy balance," and various "rate equations" which are named for their originators. Starting with these ideas, the total structure of heat transfer from the basic design of a plane wall to the complete specifications of a compactly finned heat exchanger is built. Various modes of thermal energy transfer are covered chapter by chapter as separate segments of the complete science, then systems involving two or more of these basic mechanisms are discussed, and finally, whole thermal systems comprising several components are described. The last chapter outlines some of the principles of heat transfer test work for conjunction with experimental exercises. Sufficient examples are given to allow the reader not strong in dimensional methods to grasp the techniques. The formal solution of partial differential equations has been replaced, wherever possible, with approximated analytic solutions involving elementary integrals. Theory and design of heat exchangers and other complete units are given substantial space in the book. (C.J.G.)

Heat Transfer and Fluid Flow

13884 AD-135308

Coating and Chemical Lab., Aberdeen Proving Ground, Md.

SURVEY OF HEAT TRANSFER LITERATURE.

M. Levy. June 6, 1957. 56p. Project No. 593-28-001. (CCL-24).

The field of heat transfer is surveyed and a bibliography prepared of the recent available publications in books and technical journals. An extensive heat transfer literature search was conducted on four basic principles which may offer a means of effecting satisfactory cooling under anticipated conditions of new vehicular engines that will be much smaller and lighter and have greater power output than present engines. The four basic principles are surface phenomena, thermodynamics of chemical changes, thermodynamics of change of state, and thermodynamics of electromagnetic charges. The survey covers the period 1942-1956. (auth)

12095

HEAT AND MASS TRANSFER. Second Edition of Introduction to the Transfer of Heat and Mass. E. R. G. Eckert and Robert M. Drake, Jr. New York, McGraw-Hill Book Co., Inc., 1959. 543p.

Part A of this volume discusses heat conduction including the theory of heat conduction and the heat conduction equation, steady and unsteady heat conduction, and heat conduction with moving boundaries. Part B is devoted to heat transfer by convection including: flow along surfaces and in channels; forced convection in laminar flow, turbulent flow, and separated flow; special heat transfer processes; free convection; and condensation and evaporation. Parts C, D, and E discuss thermal radiation, transfer of mass, and heat exchangers. (W.L.H.)

6942 A/CONF.15/P/1288

ON HEAT TRANSFER BETWEEN A LIQUID-FLUIDIZED BED AND THE CONTAINER WALL.

E. Ruckenstein (Polytechnical Inst., Bucharest) and V. Shorr and G. C. Suciu (Oil and Gas Inst., Bucharest). 13p.

An experimental study concerning the heat transfer between a bed fluidized with oil or water and a wall is presented. The experimental results were correlated by means of nondimensional groups. Empirical equations were established to represent these results. The interpretation of the experimental results, as well as of some results obtained by other authors concerning the fluidization by air, was made by a model which improves the one proposed by Levenspiel and Walton. The equation obtained starting from this model is in better agreement with experiment than the one proposed by Levenspiel and Walton. (auth)

13094

HEAT TRANSFER IN BOILING. Heinz Kornbichler and Norbert Kretzinger. AEG Mitt. 48, 30-7(1958) Jan. (In German)

Heat transfer in boiling can only be calculated by means of empirically obtained formulas or diagrams. Although a large number of research results have been published, no comprehensive and interpretive work is found in the literature. It is shown that known numerical material will suffice for obtaining a number of regularities if the effect of a single parameter could be explained by more extensive experimental work. (tr-auth)

HEAT TRANSFER 6.11

6335 AEC-tr-2908
HEAT TRANSFER TO NON-NEWTONIAN FLUIDS FOR LAMINAR FLOW THROUGH TUBES. U. Grigull. Translated by F. Hudswell from Chem. Ing. Tech. 28, 553-6(1956). 10p.

13087 GAT-236
Goodyear Atomic Corp., Portsmouth, Ohio.
HEAT TRANSFER AND PRESSURE DROP IN A VERTICAL SQUARE TUBE. H. L. Foltz. July 14, 1958. 14p. Contract AT-(33-2)-1. $0.50(OTS).

A ¼-inch square vertical copper tube inside an 8-foot-long shell was found to transfer heat in much the same manner as a round tube of equal hydraulic diameter. The pressure drop from the bottom to the top of the tube was measured for each operation. Heat flux was uniform throughout the length of the tube up to a steam-to-Freon temperature difference of 20°F. The range investigated was approximately from 10° to 20°F. Data obtained from a single run above 20°F, indicating that heat flux is not uniform above 20°F, are in agreement with the results of previous studies. (auth)

5300
HEAT TRANSFER. E. R. G. Eckert, James P. Hartnett, T. F. Irvine, Jr., and P. J. Schneider (Univ. of Minnesota, Minneapolis). Ind. Eng. Chem. 49, 565-76(1957) March.

A literature review is presented on heat transfer. The material is divided into sections on: Heat Conduction; Channel Flow; Boundary Layer Flow; Flow with Separated Regions; Transfer Mechanism; Natural Convection; Free and Forced Convection from Rotating Surfaces; Transpiration and Mass Transfer Cooling; Change of Phase; Radiation; Heat Transfer Applications (measurement techniques; heat exchangers, nuclear reactors, aeronautics, thermal stress and shock). 219 references. (T.R.H.)

6325 BMI-1163
Battelle Memorial Inst., Columbus, Ohio.
VAPOR FORMATION AND BEHAVIOR IN BOILING HEAT TRANSFER. Richard A. Egen, David A. Dingee, and Joel W. Chastain. Feb. 4, 1957. 51p. Contract NObs-65057.

An experimental program was conducted to study the formation and distribution of vapor in a rectangular channel under boiling-heat-flux conditions at 2000 psia. In addition, the problem of preferential distributions was studied to determine its effect on the validity of the results of this program. Over the range of variables tested, there was no flow-rate effect, and the slip, where determinable, appeared to be constant. The void fraction is correlated with the quality and heat flux, in the vicinity of zero quality; the dependence upon heat flux decreases with increasing quality in this region. Inlet-temperature variation has no effect on the value of the void, providing the inlet temperature is below the temperature necessary for local boiling. (auth)

13889 TID-3305(Suppl. 1)
Technical Information Service Extension, AEC.
HEAT TRANSFER AND FLUID FLOW: A BIBLIOGRAPHY OF SELECTED REPORT LITERATURE.
James M. Jacobs, comp. June 1958. 435p. $6.00 (OTS).

A total of 2519 annotated references to the unclassified report literature is presented. Subjects covered under heat transfer and fluid flow include radioinduced heating, boiling, boilers, evaporators, pumps, heat exchangers, hydrodynamics, coolants and their properties, thermal and flow instrumentation, high-temperature materials, thermal properties of materials, and thermal insulation. Subjects covered less completely include thermodynamics, aerodynamics, high-temperature corrosion, corrosion specific to heat transfer systems, erosion, mass transfer, corrosion film formation and effects, coolant processing and radioactivity, radiation effects of heat transfer materials, and pertinent data of thermonuclear processes. Subject, report number, and author indexes are given. (auth)

NOTE ON THE ANALOGY BETWEEN MOMENTUM TRANSFER AND HEAT OR MASS TRANSFER FOR A HOMOGENEOUS ISOTROPIC TURBULENT FIELD.
Thomas J. Hanratty (Univ. of Illinois, Urbana).
A.I.Ch.E. Journal 4, 495-6(1958) Dec.

A mathematical analysis is presented for a fully developed velocity profile in a pipe by using Taylor's statistical description of turbulent diffusion for a homogeneous isotropic field as the basis of an analogy between momentum transfer and mass transfer. (W.L.H.)

FOR GASES AND VAPORS, GRAPHS GIVE TUBESIDE HEAT TRANSFER COEFFICIENTS FOR STREAMLINE AND TURBULENT FLOW. Ning Hsing Chen (M. W. Kellogg Co., Jersey City). Chem. Eng. 66, No. 1, 124-6(1959) Jan. 12.

Graphs are presented for quickly calculating tubeside heat transfer coefficients for gases and vapors in streamline and turbulent flow. (W.L.H.)

10982 ✓ WAPD-EM-2
Westinghouse Electric Corp. Atomic Power Div., Pittsburgh.
THE EFFECT OF FINS OR SPACERS ON HEAT TRANSFER, LOCAL BOILING AND PLATE STRESSES IN A SYSTEM WITH WATER FLOWING TURBULENTLY BETWEEN PARALLEL PLATES. J. T. Stiefel. Jan. 6, 1950. Decl. Mar. 25, 1957. 29p. $4.80(ph OTS); $2.70(mf OTS).

6698 ✓ HW-47088
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
THE DESIGN AND APPLICATION OF A HEAT TRANSFER ANALOGUE FOR RADIALLY SYMMETRICAL PROBLEMS. M. W. Cook. Jan. 30, 1957. 39p. Contract [W-31-109-Eng-52]. $7.80(ph OTS); $3.30(mf OTS).

An electrical heat transfer analogue of a typical HAPO waste tank has been designed, constructed, and tested. A typical steady state temperature profile obtained from this analogue is reported. A complete summary of both steady and transient state temperature profiles is presented and discussed in HW-47087. The analogue, which was constructed of Teledeltos conductive paper, is shown. Design and Construction details are discussed and this particular type of analogue is compared with other methods of solving the problem. The analogue comprises approximately 3000 individual resistance elements which were made by draft-

11669 ✓ ORNL-156
Oak Ridge National Lab., Tenn.
HEAT TRANSFER IN MANHATTAN DISTRICT AND ATOMIC ENERGY COMMISSION LABORATORIES: A CRITICAL SURVEY. W. B. Harrison. Oct. 1, 1948. Decl. Mar. 6, 1957. 274p. Contract W-7405-eng-26. $1.10(OTS).

An effort was made to study, correlate, and bring together in one volume the heat transfer data found in reports of the Manhattan Project and in the continuing activities of the Atomic Energy Commission. Information was categorized on the basis of its common nature such as the state of the medium, solid, liquid, gas; and the nature of the heat transfer, conduction, convection, turbulent flow, boiling. The physical properties of the mediums considered and references to source material are also given. (D.E.B.)

1958 HEAT TRANSFER AND FLUID MECHANICS INSTITUTE. Preprints of Papers. Held at University of California, Berkeley, California, June 19, 20, 21, 1958. Stanford, Calif., Stanford University Press, 1958. 270p.

Preprints of the twenty-one papers presented are given as a basis for discussion. The titles are: Theoretical Aerothermodynamics; Effect of a Contraction on Turbulence and Temperature Fluctuations; Experimental Investigation of Turbulent Pipe Flow with Coolant Injection; An Analysis of the Energy Separation in Laminar and Turbulent Compressible Vortex Flows; Mass Transfer Cooling with Combustion in a Laminar Boundary Layer; Considerations Related to the Quenching of Flames with Simple Kinetics; Wave Propagation in a Reacting Mixture; Criteria for Thermodynamic Equilibrium in Gas Flow; Compressible Couette Flow with Diffusion of a Reactive Gas from a Decomposing Wall;

10514 IGRL-T/W-12
CONTACT HEAT TRANSFER BETWEEN MACHINE COMPONENTS. E. P. Diban, N. M. Kondac, and I. T. Shvets. Translated by V. H. Brix from Izvest. Akad. Nauk S.S.S.R. 9, 63-79(1954). 20p.

4592 EES-5NX1609
Naval Engineering Experiment Station. Wave Mechanics Lab., Annapolis.
REPORT OF SHOCK TEST OF A HEAT EXCHANGER FOR BATTERY COOLING WATER MANUFACTURED BY ROSS HEATER AND MANUFACTURING CO., INC. C. B. Allison. Sept. 5, 1950. 6p.

Shock tests were made on a Ross heat exchanger for battery cooling water. Deformation of the mounting feet and stretching of the mounting bolts was sustained by the equipment as a result of shock. The manufacturer has proposed certain design changes to improve the shock resistance of the equipment. The unit with the proposed modifications is recommended for high-impact shock service. (auth)

6703 ✓ AGC-1310-3
Aerojet-General Corp., Azusa, Calif
EXPERIMENTAL STUDIES ON HEAT TRANSFER AND FLUID FLOW SYSTEMS. Quarterly Progress Report No. 3 [for] January 1 through March 31, 1957. M. E. Ellion, L. Green, Jr., and K. L. Nall. Apr. 30, 1957. 25p. Project 6-(1-9964). Contract AF33(616)-3767.

Fabrication of the test furnace for extended-surface heat-transfer studies is now completed, with the exception of the thermocouple to be employed in measuring high exit-gas temperatures, the furnace aft-closure, and the exhaust tube liners. The transparent-wall nozzle section for flow separation studies is now being fabricated. Fabrication of the spark shadowgraph and Schlieren systems is completed. A second, tentative model of the mechanism of heat transfer in the vicinity of the critical point has been formulated and is discussed. The instrumentation system which is required to control and measure the heat transferred to a flow of fluid near its critical point has been designed, and equipment is being procured. (auth)

6326 ✓ BMI-1167
Battelle Memorial Inst., Columbus, Ohio.
A STUDY OF ERROR EFFECTS IN MEASURING CYCLIC-TEMPERATURE HEAT-TRANSFER COEFFICIENTS. David A. Dingee and Joel W. Chastain. Feb. 15, 1957. 28p. Contract W-7405-eng-92. $0.25(OTS).

An experimental investigation of the errors associated with the cyclic-temperature method of obtaining convective heat-transfer coefficients was made. The models studied were a large single tube, a bundle of ½-in.-ID tubes, and a bed of randomly packed spheres. In order to measure the error attenuation due to velocity mixing, cyclic dye and cyclic ion pulses were used. It was found that most of the errors at the conditions under study could be explained by velocity mixing. The methods for measuring the attenuation errors were at best only qualitative and no real quantitative correction factor was found. The results do indicate that fluids of high density such as water are not suitable fluids for cyclic-temperature tests to obtain convective heat-transfer coefficients. In future tests, gases are recommended as coolants. It is felt that reliable heat-transfer coefficients can be obtained if gases are used. (auth)

LIQUID METAL PURIFIER. (to Babcock & Wilcox Co.) British Patent 809,584. Feb. 25, 1959.

A cold-trap device for removing oxides from liquid-metal systems is described. It consists of a heat exchanger at the top and a cold chamber at the bottom. The heat exchanger is so arranged that incoming hot metal warms up outgoing cooled metal. As hot metal flows through the cool region where oxides are precipitated out, heat from it passes through a barrier which is cooled by double-tube helical coils. The inner tube is for coolant and the outer tube contains N_2 for heat conductivity and to prevent contact of liquid metal and coolant. The particular arrangement of the invention and its other features are such that severe temperature gradients are avoided, and thermal stresses in the structure are reduced. (T.R.H.)

9150
HEAT TRANSPORT BY CONVECTION IN PRESENCE OF AN IMPRESSED MAGNETIC FIELD. Yoshinari Nakagawa (Univ. of Chicago). Phys. Fluids 3, 87-93(1960) Jan.-Feb.

The effect of an impressed magnetic field on heat transport by convection, which arises from instability in a layer of an electrically conducting fluid, bounded between two constant temperature surfaces, is examined. It is shown that such a field reduces the amount of heat transported by convection, and that when the strength of the magnetic field is increased, such reduction becomes proportional to $(\pi^2 Q)^{-1}$, where $Q = \sigma\mu^2 \cos^2\theta H^2 . d^2/\rho\nu$, d is the depth of the layer, ρ the density, H the strength of magnetic field, θ the inclination of the direction of H to the vertical, and σ, μ, and ν are the coefficients of electrical conductivity, magnetic permeability, and kinematic viscosity, respectively, for all types of the boundary conditions. It is also shown that in the neighborhood of the marginal state of stability, a simple formula characterizes the heat transport by convection. (auth)

9149
HEAT TRANSPORT BY CONVECTION. Yoshinari Nakagawa (Univ. of Chicago). Phys. Fluids 3, 82-6(1960) Jan.-Feb.

The character of heat transport by cellular convection, which arises beyond the marginal state of stability in a layer of fluid bound between two constant temperature surfaces is examined. It is shown that a simple equation characterizes the heat transport in the neighborhood of the marginal state of stability when the convection is steady, and its cellular pattern of motion is represented by the solutions of the linear theory. The results of the study include all three cases of boundary conditions, namely, when the bounding surfaces of the layer are both free, are both rigid, or one is free and the other is rigid. (auth)

HEAT TRANSFER 6.11

HEAT TRANSFER IN LIQUID METALS. P. S. Lykoudis and Y. S. Touloukian (Purdue Univ., Lafayette, Ind.). Trans. Am. Soc. Mech. Engrs. 80, 653-66(1958) Apr.

The problem of heat transfer in liquid metals is studied for the case of a fully developed turbulent flow in cylindrical pipes of constant diameter, the fluid under consideration being heated (or cooled) by a constant and uniform heat input at the wall. In the analysis, the similarity theory between heat and momentum transfer is used. A theory is presented which gives the ratio of eddy diffusivities for heat and momentum transfer as a function of the Prandtl modulus. It is shown that heat transfer in liquid metals can be analyzed universally in terms of a dimensionless parameter called the "generalized Peclet modulus" and defined by by the relation $N_{Pe}^* = (N_{Re}^*)(N_{Pr}^*) = [N_{Re}(f/2)^{1/2}](N_{Pr}\sigma)$. Temperature profiles are presented and the limiting cases for $N_{Pr} = 0$ are studied. (auth)

7237 ✓ CM-880

Wisconsin. Univ., Madison. Naval Research Lab.
HEAT CONDUCTIVITY IN POLYATOMIC, ELECTRONICALLY EXCITED, OR CHEMICALLY REACTING MIXTURES—III. J. O. Hirschfelder. Aug. 15, 1956. 42p. Contract NOrd-15884.

A survey is made of the theoretical problems involved in estimating the heat conductivity in a chemically reacting gas or gas mixture. New developments which have not been previously considered are presented. The larger the value of the entropy change in a chemical reaction, the larger is the maximum value of the specific heat of the reacting mixture and the coefficient of thermal conductivity of the reacting gas mixture; the deviation of the chemical composition from the local equilibrium (or thermodynamics of irreversible processes) solution is expressed very simply in terms of the higher temperature derivatives; the large Prandtl numbers of simple gases at very low temperature and polar gases at room temperature is supposed to be due to the distortion of the molecular distribution as the result of rotational-translation interactions. The Prandtl numbers for rough spheres and for rigid spheroids are found to be somewhat larger than would be expected on this account. Although the Prandtl number of different substances is the same value to within a few percent, the Prandtl number for mixtures may be twice as large or one-half as small as the Prandtl number of its components. An Eucken-type expression is derived for the coefficient of thermal conductivity of a polyatomic gas mixture and found to agree with experimental data. This formula involves the coefficients of self and ordinary diffusion for all of the molecular species. (auth)

UON EXCHANGE 6.12

EQUIPMENT 6.12.01

See also, this section,
RADIATION EFFECTS--Glass (6.15.04),
Metal (6.15.07),
and,
under PLANTS AND EQUIPMENT,
TRANSFER OF FLUIDS (5.16).

7137 ✓ AECU-3463

Tennessee. Univ., Knoxville.
AN EXPERIMENTAL STUDY OF THE PERFORMANCE CHARACTERISTICS OF A CONTINUOUS COUNTERCURRENT ION EXCHANGE COLUMN. G. M. Drake, Jr., and F. N. Peebles. Mar. 1957. 91p. For [Oak Ridge National Lab.] Contract W-7405-eng-26, Subcontract No. 783. $0.50(OTS).

The 4-in. glass pipe column used in these studies was 13 ft high over-all. The Dowex-50W resin particle diameter range was 840 to 1100 μ when wet. The feed solution varied from 4,000 to 15,000 ppm $CaCO_3$; the regenerant solution was 1 wt. % NaCl. Data are reported for liquid flow rates from 40 to 75 gph/ft^2 based on empty column cross section. Successful operation at 100 gph/ft^2 was obtained; this is near the flow rate for fluidization of the resin particles. It is concluded that operation of a continuous, countercurrent, moving dense bed, ion exchange column up to flow rates corresponding to fluidization is satisfactory. (T.R.H.)

12698 ✓ AECU-3540

Massachusetts Inst. of Tech., Oak Ridge, Tenn. Engineering Practice School.
HYDRAULIC TEST OF 36 INCH DIAMETER HIGGINS COLUMN. J. O. Cowles, R. W. Wolff, and D. A. Cameron. Dec. 20, 1956. 24p. For Oak Ridge Gaseous Diffusion Plant. Contract [W-7405-Eng-26]. $4.80(ph OTS); $2.70 (mf OTS).

A 36-in. Higgins ion exchange column was constructed in order that hydraulic tests of a commercial size column could be made. Information obtained in a test of the effect of head pressure and pulse time on the pulsing of this 36-in. column is given. A design is presented for a meter to measure the cumulative flow of resin over a period of operation. (W.L.H.)

ION EXCHANGE 6.12

EQUIPMENT 6.12.01

9726 ORNL-2504
Oak Ridge National Lab., Tenn.
DEVELOPMENTS IN CONTINUOUS ION EXCHANGE EQUIPMENT FOR AEC APPLICATIONS. J. T. Roberts. June 9, 1958. 45p. Contract W-7405-eng-26. $1.25(OTS).

Operational aspects of fixed-bed and continuous-countercurrent ion exchange are discussed, and several types of continuous ion exchange contactors are described. The ORNL-Higgins contactor is described in detail. (auth)

11595 ACCO-65
American Cyanamid Co. Atomic Energy Div. Raw Materials Development Lab., Winchester, Mass.
THE DEVELOPMENT OF FIXED SCREEN RESIN-IN-PULP DEVICES. W. D. Charles, D. F. Thorpe, G. W. Lower, David Kaufman, N. N. Schiff, C. S. Abrams, H. I. Viklund, Fred Howland, and E. S. Porter. Aug. 5, 1954. Decl. Mar. 15, 1957. 41p. Contract AT(49-1)-533. $6.30(ph OTS); $3.00(mf OTS).

A resin-in-pulp process was developed using a series of fixed-screen or Winchester cells. Various laboratory and pilot plant models were constructed and tested under simulated plant conditions. The fixed-screen device proved very effective in the recovery of U and/or V by a continuous, resin-in-pulp process. (auth)

8281 CF-56-6-74
Oak Ridge National Lab., Tenn.
THE THEORY AND SOME APPLICATIONS OF THE HYDRAULIC RAM AS APPLIED TO COUNTERCURRENT ION EXCHANGE. S. H. Jury. June 18, 1956. 22p. Contract [W-7405-eng-26]. $4.80(ph OTS); $2.70(mf OTS).

It has been shown on theoretical grounds that resin pressure in a hydraulic ram is distributed according to the equation

$$\ln \frac{\frac{4k}{D} P_{R1} + \frac{dp_L}{dZ} - \frac{g}{g_c} [\rho_R - (1-\alpha)\rho_L]}{\frac{4k}{D} P_{R2} + \frac{dp_L}{dZ} - \frac{g}{g_c} [\rho_R - (1-\alpha)\rho_L]} = \frac{4k}{D}(Z_2 - Z_1).$$

In a countercurrent ion exchange section of a column the critical fluidizing condition is $-(dp_L/dZ) = (g/g_c)[\rho_R -$

2337 CF-55-8-101
Oak Ridge National Lab., Tenn.
PLATE-TYPE GRAPHICAL AND ANALYTICAL ANALYSIS AND DESIGN OF COUNTERCURRENT ION EXCHANGE RECOVERY COLUMNS. S. H. Jury. Aug. 19, 1955. 20p. Contract W-7405-eng-26. $3.30(ph OTS); $2.40(mf OTS).

The plate-type graphical and analytical procedures for the analysis of data from and the design of continuous or semi-continuous countercurrent ion exchange columns are summarized. The ion exchange medium of such columns operates on an equivalent or heterovalent ion system in such a manner as to strip from a dilute feed liquor an ion of value and thus in the elution section of the column to produce a purified product solution which is considerably more concentrated. (auth)

1292 ORNL-1907
Oak Ridge National Lab., Tenn.
MECHANICAL FEATURES OF THE HIGGINS CONTINUOUS ION EXCHANGE COLUMN. I. R. Higgins. Oct. 25, 1955. 29p. Contract W-7405-eng-26.

The accessory equipment for the Higgins continuous ion-exchange contactor is described. Photographs of various systems used during the development work are shown. (auth)

INTERPRETATION AND CORRELATION OF ION EXCHANGE COLUMN PERFORMANCE UNDER NONLINEAR EQUILIBRIA. Nevin K. Hiester and Shirley B. Radding (Stanford Research Inst., Menlo Park, Calif.) and Richard L. Nelson, Jr. and Theodore Vermeulen (Univ. of California, Berkeley). A.I.Ch.E. Journal 2, 404-11(1956) Sept.

Various methods are known for interpreting experimental data for linear-equilibrium cases or constant-pattern breakthrough curves where either external or internal diffusion alone controls the rate in the ion exchange column. The present paper provides interpretive techniques for nonlinear equilibrium cases where the rate is controlled by a combined diffusion mechanism. The techniques can be applied whether or not the value of the equilibrium constant is known. A completely general correlational method is derived for adding the mass transfer resistances under conditions of nonlinear equilibrium. Published breakthrough results from numerous sources and from new measurements, evaluated by the techniques described, are used to determine the numerical constants for the general correlation. (auth)

ION EXCHANGE 6.12

EQUIPMENT 6.12.01

4191 ORNL-1310
Oak Ridge National Lab., Tenn.
A COUNTERCURRENT SOLID-LIQUID CONTACTOR FOR CONTINUOUS ION EXCHANGE. I. R. Higgins. Sept. 19, 1952. Decl. Jan. 5, 1956. 18p. Contract W-7405-eng-26. $3.30(ph OTS); $2.40(mf OTS).

A continuous countercurrent solid-liquid contactor has been developed which retains the desirable features of the conventional fixed bed and gains the advantages of countercurrent flow. It can be adapted to many solid-liquid mass-transfer processes such as ion exchange, silica gel adsorption, and ore leaching. (auth)

148 ORNL-1918
Oak Ridge National Lab., Tenn.
USE OF THE HIGGINS CONTINUOUS ION EXCHANGE CONTACTOR IN RECOVERING URANIUM FROM AQUEOUS SLURRIES. I. R. Higgins. Oct. 11, 1956. Decl. Sept. 9, 1957. 55p. Contract W-7405-eng-26. $1.50(OTS).

Flowsheets are presented for recovering U from unfiltered H_2SO_4—or carbonate-leached ore pulps, at a rate of 100 tons of ore per day, in a Higgins continuous ion-exchange contactor. In the experimental work on which the flowsheets were based, approximately 5 ft of Permutit SK-20 was required to maintain a U loss of less than 1% with resin U loadings of 40 to 50 g/liter and feed flow rates of 300 to 600 gal/hr/ft^2, or with 20 g/liter and a flow rate of up to 900 gal/hr/ft^2. When the loading was 30 g/liter or more, a higher column was required as the flow rate in-

10847 CF-58-8-53
Oak Ridge National Lab., Tenn.
A COMPARISON OF FIXED AND MOVING BED ION-EXCHANGE. C. W. Hancher. Aug. 20, 1958. 26p. ~~Contract [W-7405-eng-26].~~ $4.80(ph), $2.70(mf) OTS. (ORNL-CF-58-8-53)

The Higgins continuous ion exchange contactor and other continuous ion exchange contactors are superior to fixed bed ion exchange units. The operation, design and cost comparison of the two types of units are discussed. (auth)

19876
SEMICONTINUOUS COUNTERCURRENT APPARATUS FOR CONTACTING GRANULAR SOLIDS AND SOLUTION. C. W. Hancher and S. H. Jury (Oak Ridge National Lab., Tenn.). Chem. Eng. Progr. 55, Symposium Ser. No. 24, 87-99(1959).

Progress in engineering development and theoretical studies of countercurrent ion exchange equipment is summarized. (J.E.D)

EXCHANGERS 6.12,02

See also, this section,

RADIATION EFFECTS--Other Solids (6.15.09),

PLASTICS AND HIGH POLYMERS (6.15.10).

6501 A/CONF.15/P/101
RESIN POISONS IN ION EXCHANGE RECOVERY PROCESSES. D. A. Everest, E. Napier, and R. A. Wells (Chemical Research Lab., Teddington, Middx., Eng.). 19p.

The poisoning of anion-exchange resins in uranium recovery plants may be considered as falling into two main categories: poisoning by ionic species and poisoning by ions which undergo polymerization-type reactions in the resin phase. The detailed poisoning mechanism is discussed. (W.L.H.)

7051 A/CONF.15/P/1294
L'ACTION DES RADIATIONS IONISANTES SUR LES MACROMOLECULES SYNTHETIQUES POLYELECTROLYTIQUES. (The Chemical Modifications of the Exchange Resins Under the Action of Ionizing Radiation.) O. Costăchel and G. Furnică (Inst. Oncologique, Bucharest). 24p.

The modifications in the chemical and physical properties of various exchange resins exposed to ionizing radiation were studied. The characteristics considered were the total exchange capacity, the index of acidity, degree of absorption, and solubility. The effect of chemicals, such as cysteamine, was also determined. The results showed that the macromolecules of the resins show direct and indirect effects of ionizing radiation. The chemical and reticular structure is modified. The indirect effect of radiation can be diminished by the presence of chemical substances with an affinity for the radiolytic radicals formed in the water. (J.S.R.)

ION EXCHANGE 6.12

EXCHANGERS 6.12.02

ATTRITION AND RECOVERY 6.12.02.01

7053 A/CONF.15/P/1476
THE IRRADIATION OF ION EXCHANGERS IN JEEP.
K. A. Nater (Joint Establishment for Nuclear Energy Research, Kjeller, Norway). 20p.

Several organic and inorganic ion exchanger materials were irradiated in JEEP. Radiation effects are described and dependence on dose rate and quality of the radiation is discussed. By comparing the action of different kinds of ionizing radiation, possible reaction mechanisms are discussed. (auth)

3347 MITG-A111
Massachusetts Inst. of Tech., Watertown, Mass. Mineral Engineering Lab. and American Cyanamid Co. Atomic Energy Div. Raw Materials Development Lab., Winchester Mass.
CONSTRUCTION AND OPERATION OF AN AUTOMATIC DEVICE TO TEST RESIN LIFE. Fred N. Oberg. July 1, 1954. Decl. Oct. 19, 1955. 27p. Contracts W-7405-eng-85 and AT(49-1)-533. $0.25(OTS).

The design and construction of an automatic machine to test resin life in U ion exchange are described. The machine, at predetermined times, stops the flow of pregnant solutions, eluant, and wash waters and separates the products. The results of testing resin through many cycles are reported. (C.W.H.)

13035 AECU-3780
Massachusetts Inst. of Tech., Oak Ridge, Tenn. Engineering Practice School.
RESIN ATTRITION. G. R. Seiler, P. R. Ammann, and A. B. Newey. Apr. 9, 1958. 28p. [For Oak Ridge Gaseous Diffusion Plant. Contract W-7405-eng-26, Subcontract No. 70]. (KT-335). $4.80(ph OTS); $2.70 (mf OTS).

Uranium metal is produced by the bomb reduction of UF_4 with Mg. The slag from this reduction contains appreciable quantities of U, either in the metallic or the tetrafluoride form, in addition to the MgF_2. A process for recovering low enrichment U from this slag has gone into operation. A study of the causes of resin attrition and their relative magnitude both from an over-all point of view and with specific reference to the Higgins' Column and Dowex 21K resin is presented. Equipment was designed, assembled, and tested to investigate the effects of valve action, wall to resin and resin to resin friction, repeated chemical cycling, and column height. Certain mechanical properties of the

THE STABILITIES OF ION-EXCHANGE RESINS IN AQUEOUS SOLUTION ACTED ON BY Co^{60} γ-RAYS.
Tsuan Man-Wei, A. I. Chernova, and M. A. Proskurnin. Atomnaya Energ. 5, 573-5(1958). (In Russian)

The effects of Co^{60} γ radiation on the changes in the physico-chemical properties of ion exchange resins in aqueous solutions were investigated. Results are presented in tables and graphs. The dependence of resin dissolution on the radiation dose was found, and rapid changes were observed in the statistical capacity of the resins for sodium and uranyl ions. High radiation doses did not alter the capacity for sodium ions, while the uranyl ion capacity considerably increased. The presence of sodium chloride considerably slowed the process of resin dissolution. (R.V.J.)

ANALYTICAL PROCEDURES

12673 DP-218
Du Pont de Nemours (E. I.) & Co. Savannah River Lab., Augusta, Ga.
ANALYSIS OF USED ION EXCHANGE RESINS. Elizabeth W. Baumann. June 1957. 24p. Contract AT(07-2)-1. $0.25(OTS).

A scheme for analyzing used, mixed ion exchange resins is described. The methods are suitable for slightly radioactive resins. (auth)

7128
THE IRRADIATION OF UNSATURATED POLYESTER RESINS. A. Charlesby and V. Wycherley (Tube Investments Research Labs., nr. Cambridge). Intern. J. Appl. Radiation Isotopes 2, 26-34(1957) Apr.

10894
A RECOVERY SCHEME FOR POISONED ION EXCHANGE RESINS. Mayer B. Goren (Kerr-McGee Oil

ION EXCHANGE 6.12

EXCHANGERS 6.12.02

ATTRITION AND RECOVERY 6.12.02.01

Industries, Inc., Oklahoma City). Ind. Eng. Chem. 51, 539–42(1959) Apr.

Recovery of uranium by ion exchange process has led to resin poisoning problems stemming from irreversible sorption or deposition of substances present in minor quantities in leach liquor. In the Colorado Plateau area molybdenum, zirconium, titanium, and phosphate are the principal poisoning agents. Resins can be economically restored to their original loading and eluting characteristics by alternate treatment with strong acid and moderately strong alkali, the most efficient process involving alkaline treatment as a first step. The seemingly drastic treatment is without degradative effect on the resins, and the process is believed to be well suited to the restoration of ion exchange resins poisoned in a variety of processes. (auth)

16645 WAPD-PWR-PMF-364
Westinghouse Electric Corp. Atomic Power Div., Pittsburgh.
REGENERATION OF THE PWR CANAL WATER DEMINERALIZER RESIN. A. P. Zechella. 10p. PWR Project. Includes Appendices I, II, III, and PFS-Sketch No. 21. Contract AT-11-1-GEN-14. $1.80(ph), $1.80 (mf) OTS.

The regeneration of PWR canal water system demineralizer resin was investigated. The normal mode of handling spent demineralizer resin is underground storage. An operational procedure for the regeneration process is given which will allow reuse of the demineralizer and a saving of about $47,000 with a six month regeneration cycle. (J.R.D.)

8772
STABILITY OF THE CATION EXCHANGE RESINS. V. A. Nikashina, A. Kh. Breger, M. M. Senyavin, and A. V. Gordievskii (Inst. of Physics, Academy of Sciences, USSR, Moscow). Intern. J. Appl. Radiation and Isotopes 4, 201-9(1959) Jan.

The chemical stability to x, β, and γ radiation of air-dry cation exchange resins in the H form has been studied in connection with the use of ion-exchange resins for absorption and separation of radioactive substance mixtures. Variations in the exchange capacity, degree of swelling, and chemical stability of the resins have been investigated experimentally. The nature of these variations in properties of the cation-exchange resins investigated has been shown to be similar on exposure both to x and γ rays and to fast electrons. Decrease in exchange capacity and chemical stability and variations in swelling are observed after exposure. Most affected by irradiation are ion-exchange resins of aliphatic structure. A higher degree of degradation is observed on irradiation of cation-exchange resins in the presence of air. (auth)

DAMAGING EFFECTS OF RADIATION ON CHEMICAL MATERIALS. J. C. Bresee, J. R. Flanary, J. H. Goode, C. D. Watson, and J. S. Watson (Oak Ridge National Lab., Tenn.). Nucleonics 14, No. 9, 75-81(1956) Sept.

The effects of gamma radiation on protective coatings, gasket materials, asphalts and tars, ion exchange resins, and aqueous solutions are tabulated and discussed. (T.R.H.)

22008
CHANGES IN THE PROPERTIES OF ION-EXCHANGE RESINS PRODUCED BY IRRADIATION WITH IONIZING RADIATION. O. Costăchel, Gh. Furnică, and St. Grigorescu. Acad. rep. populare Romîne, Studii cercetări chim. 6, 67-74(1958). (Translated from Referat. Zhur. Khim. No. 2, 1959, Abstract No. 4080).

Changes in the exchange capacity, activity index, swelling in water, and in the solubility of ion exchange resins (IER) produced by exposure to x rays were investigated. The changes produced when dry and water-swollen IER are irradiated with x rays are quite different. Direct irradiation effects were established as well as an indirect hydroxylation effect which is proportional to the degree of swelling of the IER.

ION EXCHANGE 6.12

EXCHANGERS 6.12.02

NON-RESINS 6.12.02.02

See also, this section,

PLASTICS AND HIGH POLYMERS (6.15.10),

and,

under FISSIONABLE MATERIAL--RECOVERY,

ADSORPTION (3.03.01).

18956 NP-tr-254
LIQUID ANION EXCHANGERS. U. Schindewolf. Translated by R. C. Murray (U.K.A.E.A. Atomic Energy Research Establishment) from Z. Elektrochem. 62, 335-40 (1958). 15p. $3.30(ph), $2.40(mf) JCL.

The adsorption of metals by anion exchangers from solutions containing complex anions is discussed. It is also shown that liquid anion exchangers have the same adsorption properties as solid ones. Thus the exchange properties, determined by the active groups or fixed ions of the exchanger phase, do not depend on the physical state of the exchanger. The adsorption behavior agreement of the liquid anion exchangers which were investigated is emphasized. (J.R.D.)

6431 A/CONF.15/P/271
SYNTHETIC INORGANIC ION EXCHANGERS AND THEIR APPLICATIONS IN ATOMIC ENERGY. C. B. Amphlett (Atomic Energy Research Establishment, Harwell, Berks, Eng.). 12p.

Highly stable, granular, selective ion exchangers are being studied with a view to their application to the treatment of high-temperature pressurized water, chemical separations in intense radiation fields, and for normal separations where increased specificity is desirable. These materials include zirconium phosphate, arsenate, tungstate, molybdate, and oxide; oxides and phosphates of thorium and titanium, and tungsten oxide. The salts possess cation-exchange properties; tungsten oxide is a cation-exchanger, while the other oxides are amphoteric and may be used for either cation or anion exchange depending on the conditions. In the case of zirconium phosphate, the effect of variation in manufacturing method on the properties of the product has been studied on the 1 kg scale using standard

6458 A/CONF.15/P/1315
ETUDE SUR L'UTILISATION DES ARGILES ROUMAINES COMME ECHANGEURS D'IONS. DECONTAMINATION DES EAUX RADIOACTIVES. (A Study on the Use of Romanian Clays as Ion Exchangers). Gh. Ioanid, Gh. Mastacan, F. Bunus, M. Popesco, S. Stanciu, and A. Ciocănel (Inst. de Recherches Chimiques, Bucharest). 23p.

The behavior of some Romanian clays as ion exchangers for Sr^{90} and Cs^{137} was reviewed. A discussion is given of the research on the processing and regeneration possibilities of such clays as were found to be active. Their characteristics are further interpreted through a mineralogical study. (auth)

12482 CEA-tr-A-529
ECHANGEURS D'ANIONS LIQUIDES. (Liquid Ion Exchangers.) U. Schindewolf. Translated into French from Z. Elektrochem. 62, 335-40(1958). 20p.

This paper was previously abstracted from the original language and appears in NSA, Vol. 13, as abstract No. 1175.

6391 A/CONF.15/P/1683
PHENOMENES D'ADSORPTION D'ANIONS AUX BENTONITES. (Anion Adsorption Phenomena on Bentonites). Yolande Hendrickx (Univ. of Ghent). 15p.

A study of ion adsorption phenomena on bentonites was made with the help of radioactive tracers. Bentonite consists principally of montmorillonite. The experiments were made with Cr^{51}-labeled Na_2CrO_4 and I^{131}-labeled $NaIO_3$. The adsorption for sodium chromate was determined for a series of concentrations, and the results are graphed. Negative adsorption was noted for sodium iodate. The negative adsorption was interpreted as a result of the fact that H-bentonite retained a certain amount of water which was inaccessible to $NaIO_3$, causing an increase in the concentration of the iodate solution with respect to the standard solution. The results are tabulated. (J.S.R.)

10491
SYNTHETIC INORGANIC ION-EXCHANGE MATERIALS. I. ZIRCONIUM PHOSPHATE. C. B. Amphlett, L. A. McDonald, and M. J. Redman (Atomic Energy Research Establishment, Harwell, Berks, Eng.). J. Inorg. & Nuclear Chem. 6, 220-35(1958) June.

ION EXCHANGE 6.12

EXCHANGERS 6.12.02

NON-RESINS 6.12.02.02

Some of the properties of zirconium phosphate are given as an example of a class of very stable synthetic inorganic ion-exchange materials, which may be prepared in granular form suitable for column operations. It exhibits properties typical of a polyfunctional, weakly acid cation exchanger, with capacities up to ~5 mequiv/g in alkaline solution. Materials with a low phosphate: zirconium ratio possess in addition anion-exchange properties due to anions retained in the structure from the original zirconium salt. Cations may be exchanged rapidly and reversibly upon acid phosphate groups in

6355 A/CONF.15/P/1155
ETUDE DE QUELQUES ECHANGEURS MINERAUX UTILISABLES DANS L'EAU A HAUTE TEMPERATURE. (Study of Some Mineral Exchangers for Use in Water at High Temperature.) J. Huré, R. Platzer, and R. Bittel (Commissariat à l'Énergie Atomique, Paris); J. Fourré (Société le Carbone-Lorraine, France); and R. Wey (Univ. of Strasbourg). 45p.

The study of the use of ion exchangers at high temperatures was made with a view to the purification of water in reactors. Natural ion exchangers with mineral structures (clay of the montmorillonite type), natural mineral compounds so treated as to give them the properties of ion exchangers (activated graphite), and synthetic mineral compounds (zirconium phosphates and hydroxides and thorium hydroxide) were investigated. The preparation of the minerals is described, and the results obtained with them are discussed in detail.

10492
SYNTHETIC INORGANIC ION-EXCHANGE MATERIALS. II. HYDROUS ZIRCONIUM OXIDE AND OTHER OXIDES. C. B. Amphlett, L. A. McDonald, and M. J. Redman (Atomic Energy Research Establishment, Harwell, Berks, Eng.). J. Inorg. & Nuclear Chem. 6, 236-45 (1958) June.

The oxides ZrO_2, ThO_2, and TiO_2 behave as anion exchangers in acid and neutral solution and cation exchangers in alkaline solution, the pH at which their behavior changes depending upon the oxide itself and possibly also upon the nature of the exchanging ion. The oxides have been prepared in granular form and their reversible reactions with ions in solution have been studied; they are monofunctional exchangers, with appreciable rates of exchange, and display the expected

7597
ON ACID-BASES MECHANISM OF ABSORPTION ON SILICA GEL. A. V. Kisilev (Lomonosov Moscow State Univ.). Doklady Akad. Nauk S.S.S.R. 106, 1046-9(1956) Feb. 21. (In Russian)

14709 A/CONF.15/P/1832
Oak Ridge National Lab., Tenn.
ION EXCHANGE PROPERTIES OF HYDROUS OXIDES. K. A. Kraus, H. O. Phillips, T. A. Carlson, and J. S. Johnson. 29p. $0.50(OTS).

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

The large class of amorphous insoluble hydrous oxides has interesting ion exchange properties. These materials have high capacities and suitable exchange rates and may therefore be used in packed columns in typical chromatographic (ion exchange) manner. Hydrous oxides may exhibit either cation exchange or anion exchange properties, or both. The type of adsorption depends on the element on which the oxide is based, on the acidity of the medium and, to some extent, on the ion adsorbed. Though some of the most acidic oxides are soluble (e.g., P_2O_5) or easily dispersible (e.g., MoO_3, WO_3) their intrinsic cation exchange properties may be utilized by incorporating them in excess into an insoluble matrix, i.e., by preparing certain insoluble acidic salts. Some of the materials with cation exchange properties may be used even with extremely small columns for the separation of the alkali metals from each other or of the alkaline earths. The specificity of these compounds for cesium in acidic solutions permits the almost unique isolation of this element from practically all others. Those oxides with anion exchange properties also have unusual selectivities compared with the organic ion exchangers. Oxides based on Bi(III) have high selectivities for the heavier halides and others, such as the oxides of Zr(IV), Nb(V), and Ta(V) for remarkable selectivities for fluorides. (auth)

ION EXCHANGE 6.12

EXCHANGERS 6.12.02

NON-RESINS 6.12.02.02

/579
ANION EXCHANGE STUDIES. XIX. ANION EXCHANGE PROPERTIES OF HYDROUS ZIRCONIUM OXIDE. Kurt A. Kraus and Harold O. Phillips (Oak Ridge National Lab., Tenn.). J. Am. Chem. Soc. 78, 249(1956) Jan. 5.

17008
PROPERTIES OF A NEW STRONG BASE ANION EXCHANGER CONTAINING STRUCTURALLY-BOUND TERTIARY SULFONIUM CATIONS. S. Lindenbaum, G. E. Boyd, and G. E. Myers (Oak Ridge National Lab., Tenn.). J. Phys. Chem. 62, 995-9(1958) Aug.

Selectivity coefficients, rates of anion exchange, water absorption isotherms, and the absorptions of Zn(II) and Fe(III) from concentrated electrolyte solutions were measured using a new, methylene cross-linked, strong-base anion exchanger containing structurally-bound tertiary sulfonium cations. The selectivity coefficient scale, determined using thirteen anion salt forms, was nearly identical with that for quaternary ammonium type anion exchangers. The greater the water uptake at saturation vapor pressure by a given salt form the greater the bromide ion affinity. The tertiary sulfonium anion exchanger was appreciably more hydrated at low water vapor activities than the quaternary ammonium type, whereas the converse held for high water activities. The self-exchange of bromide ion in the sulfonium exchanger at 25° occurred with a self-diffusion coefficient of 1.18×10^{-7} sec^{-1}, which value is notably smaller than the coefficient observed with a comparable quaternary ammonium type exchanger at the same temperature. Micro-quantities of Zn(II) and Fe(III) were strongly absorbed from hydrochloric acid and lithium chloride solutions, and this absorption increased with electrolyte concentration in a manner similar to that found with quaternary ammonium type anion exchangers. (auth)

4264 AERE-M/R-2046
Gt. Brit. Atomic Energy Research Establishment, Harwell, Berks, England.
ON THE ION ADSORPTION PROPERTIES OF SYNTHETIC MAGNETITE. P. J. Anderson. Sept. 8, 1956. 29p.

An electrokinetic study has been made of magnetite dispersions in various electrolytes and ion adsorption isotherms calculated from the experimental results. It is shown that the magnetite surface possesses both cation and anion adsorption properties under appropriate conditions. The effects of pH upon the ζ-potential are interpreted in terms of a weak basic dissociation of surface groups. The significance of the results from the point of view of corrosion inhibition in water systems is discussed. (auth)

4373 AERE-C/R-1668
Gt. Brit. Atomic Energy Research Establishment, Harwell, Berks, England.
THE ABSORPTION OF INORGANIC SALTS BY NON-IONIC RESINS (A NEW ABSORPTIVE MECHANISM). J. Kennedy and H. Small. Nov. 1955. 13p.

Non-ionic insoluble (crosslinked) orthophosphate and phosphonate polymers show high absorption coefficients for UO_2^{2+}, Li, Co^{2+} and Fe^{3+} salts dissolved in alcoholic, alcoholic ether, and ketonic solvents. It is suggested that a Werner addition complex is formed between the metal and the O_2 of the resin phosphoryl group. (auth)

FLÜSSIGE ANIONENAUSTAUSCHER. (Liquid Anion Exchangers) U. Schindewolf (Massachusetts Inst. of Tech., Cambridge) and (Univ. of Michigan, Ann Arbor). Z. Electrochem. 62, 335-40(1958). (In German)

The solution of the hydrochloride of an organic long-chain amine in a water-immiscible solvent (liquid anion exchanger) acts as a strong anion exchanger. These liquid anion exchangers absorb or extract chloro-complexes of many metals from chloride-containing electrolytes with great anionic selectivity. The form of the distribution curve (partition coefficient vs. activity of chloride-containing carrier-electrolyte) is given by means of the aqueous phase reaction which leads to the formation of the absorbed complexes, and by means of the exchange reaction in the organic phase. The electrolyte effect observed in the case of strong anion exchangers (deviation from ideal behavior) is also characteristic of liquid anion exchangers. Varying the amine concentration in the organic phase presents a simple means of studying the influence of active groups on ion exchange. (tr-auth)

ION EXCHANGE 6.12

EXCHANGERS 6.12.02

NON-RESINS 6.12.02.02

9225
SYNTHETIC ION-EXCHANGERS. RECENT DEVELOPMENTS IN THEORY AND APPLICATION. G. H. Osborn. London, Chapman, and Hall Ltd., 1955. 419p.

1009
EFFECTS OF THE NATURE OF THE SURFACE ON THE SORPTION PROPERTIES OF SILICA GELS. I. E. Neĭmark, R. Yu. Sheĭnfaĭn, and L. G. Svintsova (Pisarzhevskiĭ Inst. of Physical Chemistry). Doklady Akad. Nauk S.S.S.P. 108, 871-4(1956) June 11. (In Russian)

Investigations were made of the hydrophilic nature of silica gel surfaces, their sorptive properties, and the effects produced by the chemical nature and porosity of silica gel surfaces in adsorption processes. To solve the problem the silica gel hydroxides were replaced by fluorite, the anions of which differ sharply from OH groups in their capacity to form hydrogen complexes. The sorption properties of silica gels with changed surfaces were compared to silica gels of identical structure but with hydroxide coated surfaces. Diagrams are given to show the sorption and desorption isotherms of methyl alcohol and benzoyl vapors on hydrophilic and fluorite silica gels of fine and large porosity. (R.V.J.)

7975
THE INTER-DIFFUSION OF TWO CHARGED PARTICLES (WITH PARTICULAR REFERENCE TO ION-EXCHANGE IN ZEOLITES). I. R. Beattie (King's Coll., London) and D. R. Davis (The Univ., Sheffield). Phil. Mag. (8) 2, 599-606(1957) May.

The general importance of ion exchange is mentioned, together with the work of previous investigators. The mechanism whereby two ions may change places is examined, and it is concluded that in many cases two ions diffusing in opposite directions in a charged substrate will diffuse independently of one another apart from any space charge effects. An equation is developed relating the amount of material exchanged during a reaction with the square root of the duration of the experiment. Practical applications of this equation are briefly discussed, as is the effect of immiscibility of solid solutions within the exchanger. (auth)

10490 NP-tr-101
THE SUPER-EQUIVALENT ADSORPTION OF CATIONS ON A NEGATIVELY CHARGED SURFACE OF MERCURY. A. N. Frumkin, B. B. Damaskin, and N. V. Nikolaeva-Fedorovich. Translated by Elmar Jarvesoo, (Amherst Coll., Mass.) from Doklady Akad. Nauk S.S.S.R. 115, 751-4(1957). 7p.

Measurements were made of differential capacity of cations on a negatively charged surface in the presence of an anion whose adsorption in the surface layer affects the magnitude of the differential capacity. These and other experiments are discussed in relation to the super-equivalent adsorption of cations in a surface. (T.R.H.)

6607
THE MAGNETOCHEMISTRY OF ADSORPTION OF IONS FROM SOLUTION. C. M. French and J. P. Howard (Queen Mary Coll., London). Trans. Faraday Soc. 52, 996-8(1956) July.

The adsorption from aqueous solutions of salts of some of the Pt and Pd group of elements, and of U on silica gel was studied. Magnetic measurements on the resulting complexes indicated that the electronic configuration for the U^{3+} and U^{4+} ions is unchanged on adsorption. The electronic configuration of the adsorbed ions of the Pt and Pd groups appears to be dsp^2 or d^2sp^3. The results in these two latter groups are, however, open to alternative explanation owing to the anomalous magnetic behavior of these elements. (auth)

19853 CF-58-5-108
PREPARATION OF ION EXCHANGE MEDIA BY SURFACE CHARGE REVERSAL OF SILICA GEL. Hans Kautsky and Hermann Wesslau. Translated by C. E. Schilling (Oak Ridge National Lab.) from Z. Naturforsch. 9b, 569-70(1954). 4p. $1.80(ph), $1.80(mf) OTS.

Conversion of an absorbent, silica gel, to an ion-exchange material of high capacity is described. This material would presumably be more radiation resistant than organic type exchangers. Thorium ion was used for creation of an anion-exchange material, phosphate ion for a cation—exchange material. Some application of this type material, or of the basic principles involved, appear quite possible in nuclear chemical processing. (auth)

ION EXCHANGE 6.12

EXCHANGERS 6.12.02

RESINS - DOWEX 6.12.02.03

137

THE EFFECT OF TEMPERATURE ON ION-EXCHANGE EQUILIBRIA. III. EXCHANGES INVOLVING SOME DIVALENT IONS. O. D. Bonner and Robert R. Pruett (Univ. of South Carolina, Columbia). J. Phys. Chem. 63, 1420-23(1959) Sept.

Ion-exchange equilibria in seven systems involving divalent ions have been investigated over the temperature range 0 to 97.5° using sulfonic acid type (Dowex-50) resins. In all exchanges between two divalent ions, the equilibrium constant decreases with increasing temperature, with a resulting negative value for ΔH^0. This is apparently characteristic of exchanges between ions of the same valence type since for all exchanges between two univalent ions negative ΔH^0 values have also been observed. With the exception of the cupric-magnesium system, the values of ΔS^0 for all exchanges between two divalent ions are positive. For the magnesium–hydrogen exchanges ΔH^0 and ΔS^0 are positive. The algebraic signs of these functions are identical with those of the cupric–hydrogen system already reported. (auth)

4376

EQUILIBRIUM SORPTION OF LANTHANIDES, AMERICIUM, AND CURIUM ON DOWEX-50 RESIN. J. P. Surls, Jr. and G. R. Choppin (Univ. of California, Berkeley). J. Am. Chem. Soc. 79, 855-9(1957) Feb. 20.

The equilibrium sorption of the tripositive lanthanides and americium and curium from 0.110m perchloric acid solutions on Dowex-50 cation-exchange resin has been studied at 25°. The relative sorbability decreased from lanthanum to dysprosium. From dysprosium to lutetium almost no change in sorbability was observed. Americium and curium sorbed with the same strength as lanthanides of comparable crystallographic radius. A correlation of ion-exchange sorbabilities and ionic conductance at infinite dilution has been made. By studying the effect of temperature on sorption of americium, promethium, thulium and lutetium heats and entropies were obtained. The reaction was found to have a considerable ΔC_p effect. Thermodynamic quantities calculated for americium and promethium differed from those of thulium and lutetium. (auth)

5276

A SELECTIVITY SCALE FOR SOME DIVALENT CATIONS ON DOWEX 50. O. D. Bonner and Linda Lou Smith (Univ. of South Carolina, Columbia). J. Phys. Chem. 61, 326-9 (1957) March.

Equilibrium studies involving Cu^{2+}, Zn^{2+}, Co^{2+}, Ni^{2+}, Mg^{2+}, Cd^{2+}, UO_2^{2+} and Pb^{2+} on Dowex 50 resins of approximately 4, 8 and 16% divinylbenzene content have been made at a constant ionic strength of approximately 0.1 M. These ions in addition to Ba^{2+}, Sr^{2+} and Ca^{2+} have been included in the same selectivity scale as the common univalent cations. Activity coefficient corrections have been made for the ions in the aqueous solution phase, and these corrections have been substantiated in some instances by the results of exchange reactions carried out at lower ionic strengths. It is observed that the order of decreasing activity coefficients for the nitrates of both the univalent and the divalent ions is the same as that of the selectivity scale. The characteristic maximum water uptake of the resin in these ionic forms is reported. (auth)

7136 ✓ AECU-3462

Tennessee. Univ., Knoxville.

A STUDY OF THE REACTION RATE FOR THE ION EXCHANGE SYSTEM DOWEX-50, CALCIUM, AND SODIUM UNDER FLOW CONDITIONS. N. T. Anderson and F. N. Peebles. Dec. 1956. 82p. For [Oak Ridge National Lab.]. Contract W-7405-eng-26, Subcontract No. 783. $0.45(OTS).

The ion exchange system Dowex-50, Ca, Na, was studied by measuring concentration changes in feed solution passed over a 1/8-in. thick, 4-in. diam. bed of Dowex-50. The mechanism is diffusion controlled and obeys the theory to a reasonable degree. (T.R.H.)

1764

THE BEHAVIOR OF URANYL AND NEPTUNYL IONS WITH DOWEX-50 CATION-EXCHANGE RESIN. J. C. Sullivan, Donald Cohen, and J. C. Hindman (Argonne National Lab., Lemont, Ill.). J. Am. Chem. Soc. 77, 6302-4(1955) Dec. 5.

The distribution coefficients of UO_2^{2+}, NpO_2^{+}, and NpO_2^{2+} between Dowex-50 resin and 1M $HClO_4$ (ionic strength = 1) have been measured as a function of time. Variations in the coefficients of neptunyl ions with time indicate that a reaction in addition to exchange is taking place. (C.W.H.)

ION EXCHANGE 6.12

EXCHANGERS 6.12.02

RESINS – DOWEX 6.12.02.03

3491 ✓ K-447
Carbide and Carbon Chemicals Co. K-25 Plant, Oak Ridge, Tenn.
SEPARATION OF COPPER, IRON, NICKEL AND URANYL IONS BY ION EXCHANGE COLUMNS. H. L. Bench, R. H. Capps, M. R. Skidmore, H. B. Weisblatt, and R. H. Winget. July 20, 1949. Decl. Dec. 27, 1955. 32p. (KLO-127). $6.30(ph OTS); $3.00(mf OTS).

The adsorption and elution characteristics of Cu, Fe, Ni, and uranyl ions on Dowex 50 synthetic exchange resin were studied with small columns. It was found possible to separate Cu and Ni from U adsorbed on a column of the Na form of Nalcite high capacity exchange resin (Dowex 50) using 5% ammonium citrate as an elutriant at pH 7, followed by the same elutriant at pH 3. Of the total U in the mixture, 83% was recovered in 160 ml of eluate. Sixty-six % was recovered free of contamination in 70 ml, 60% being concentrated in a 20 ml fraction of eluate. (auth)

136
THE EFFECT OF TEMPERATURE ON ION EXCHANGE EQUILIBRIA. II. THE AMMONIUM–HYDROGEN AND THALLOUS–HYDROGEN EXCHANGES. O. D. Bonner and Robert R. Pruett (Univ. of South Carolina, Columbia). J. Phys. Chem. 63, 1417-20(1959) Sept.

Ion-exchange reactions between ammonium and hydrogen and between thallous and hydrogen ion on Dowex-50 resins of 16% DVB content have been studied over the temperature range 0 to 97.5° while maintaining a constant solution ionic strength of approximately 0.1 M. The equilibrium constant and the standard free energy, enthalpy and entropy changes have been calculated for each exchange at each temperature. The differential free energy, enthalpy and entropy of exchange for each system is found to vary considerably with resin composition. One possible explanation of this variation is the assumption that the ion exchange resin in the hydrogen form is an acid which is similar in strength but probably slightly weaker than nitric acid. (auth)

5932
SOME CATION-EXCHANGE EQUILIBRIA ON DOWEX 50 AT 25°. O. D. Bonner, C. F. Jumper, and O. C. Rogers (Univ. of South Carolina, Columbia). J. Phys. Chem. 62, 250-3(1958) Feb.

Summaries of the results of ion-exchange equilibria and of maximum water uptakes at 25° of Dowex 50 resins of 4, 8, and 16% DVB content are given for the following systems: $NH_3OH^+-H^+$, $Ca^{+2}-Ni^{+2}$, $Mn^{+2}-Ni^{+2}$, $Ba^{+2}-Be^{+2}$, $Ce^{+3}-Ag^+$, $La^{+3}-Ce^{+3}$, $Ce^{+3}-Cr^{+3}$. (J.S.R.)

5295
CATION-EXCHANGE EQUILIBRIA INVOLVING SOME DIVALENT IONS. O. D. Bonner and Frances L. Livingston (Univ. of South Carolina, Columbia). J. Phys. Chem. 60, 530-2(1956) May.

Equilibrium studies involving Cu^{2+}, Ba^{2+}, Sr^{2+}, and Ca^{2+} on Dowex 50 resins of approximately 4, 8, and 16% divinylbenzene content have been made while maintaining a constant ionic strength of approximately 0.1. Selectivity coefficients have been measured at various resin loadings. The characteristic maximum water uptake of the resin in these ionic forms is reported. (auth)

9220
KINETICS OF NONAQUEOUS ION EXCHANGE SYSTEMS. HYDROGEN–BUTYLAMMONIUM EXCHANGE IN ETHANOL–WATER MIXTURES. Stafford Wilson and Leon Lapidus (Princeton Univ., N. J.). Ind. Eng. Chem. 48, 992-8(1956) June.

The adsorption of n-butylamine on the hydrogen form of Dowex 50 ion exchange resin was studied in a batch type reactor. Aqueous ethanol mixtures were used as solvents. Data were taken at varying amine and solvent concentrations, resin cross linkage, and resin particle size. The rate of reaction was found to be particle diffusion controlled and could be varied several thousandfold by changes of solvent composition. The use of theoretical equations originally derived for aqueous systems was found to correlate the rate data. Diffusivities calculated by the theoretical equations may be used to characterize the nonaqueous rate process. (auth)

ION EXCHANGE 6.12

EXCHANGERS 6.12.02

RESINS - DOWEX 6.12.02.03

6387 A/CONF.15/P/1600
CHEMICAL SPECIFICITY OF CATION EXCHANGE. I. CHARGE AND RADIUS EFFECTS WITH POLY-AMINO COBALTIC COMPLEXES. Bahattin Baysal (Univ. of Ankara). 13p.

Data are presented for the cation exchange equilibria in replacing La^{3+} from the cation resin Dowex-50 for five cobalt(III) complexes, three of tripositive charge (the trisethylenediamine complex, the hexamino, and the cisbisaquotetramino), on dipositive (the pentamino-chloro) and one unipositive (the tetraminoxalate). The studies extended over a range of mole fraction of cobalt complex from 0.05 to 0.85. (W.D.M.)

9746 USNRDL-TR-231
Naval Radiological Defense Lab., San Francisco.
ANION EXCHANGE STUDIES IN PHOSPHORIC ACID SOLUTIONS. E. C. Freiling, J. Pascual, and A. A. Delucchi. May 7, 1958. 15p.

The applicability of H_3PO_4 solutions to quantitative anion exchange separations was investigated by determining the equilibrium distribution coefficients of Cs, Sr, Ce(III), Zr(IV), Te(IV), Ce(IV), Np(IV), Nb(V), Mo(VI), and U(VI) between Dowex-2 resin in the phosphate form and various strengths of H_3PO_4 solutions. The results show that these elements fall into three groups. The first group, consisting of Cs and Te(IV), does not favor the resin phase to any appreciable extent even at 0.1N H_3PO_4. The group composed of Sr, Ce(III), and Ce(IV) are weakly adsorbed at low H_3PO_4 concentrations ($4 < K_d < 10$). The remaining elements are strongly adsorbed in these solutions ($K_d > 1000$). The adsorption of all elements studied decreases monotonically with increasing H_3PO_4 concentrations. (auth)

10477 USNRDL-TR-228
Naval Radiological Defense Lab., San Francisco.
ANION-EXCHANGE BEHAVIOR OF SEVERAL METAL IONS IN HCl, HNO_3, AND H_2SO_4 SOLUTIONS, WITH DOWEX-2. L. R. Bunney, N. E. Ballou, J. Pascual, and S. Foti. May 12, 1958. 14p.

Distribution coefficients were measured for Am, Ce, Mo(VI), Pa, Ru, Sr, Th, U(VI), Y, and Zr in the systems Dowex-2–HCl, Dowex-2–HNO_3, and Dowex-2–H_2SO_4. The acid concentrations ranged from 0.1N to concentrated. Sr, Y, Ce, and Am did not show any significant adsorption in any of the systems studied. A number of elements showed strong adsorption at low concentrations of H_2SO_4. Th, U(VI), and Zr show an adsorption maximum at approximately 8N HNO_3. (auth)

2767 USNRDL-TR-185
Naval Radiological Defense Lab., San Francisco.
QUANTITATIVE ION-EXCHANGE SEPARATIONS FOR SEQUENTIAL RADIOCHEMICAL ANALYSIS. L. Wish. Oct. 10, 1957. 27p. Project NS-081-001.

A method for a rapid separation and determination of Np, Pu, U, Zr, Nb, and Mo isotopes in mixed fission products is evolved from Dowex-2 anion-exchange equilibrium data of these elements in HCl, HCl–HF, and HNO_3. The fission products sample in concentrated HCl is added directly to the resin column, and no tracers or carriers or prior separations are required. The activities are eluted sequentially and determined directly in a gamma ray scintillation well counter or in a multi-channel gamma ray spectrometer. The yields are shown to be quantitative and the purity is equivalent to that obtained from the standard radiochemical procedure for Mo, Np, and Pu. The Zr and Nb fractions may contain gamma-ray impurities which are easily resolved by gamma ray spectrometry. The U is contaminated with the Te^{132}–I^{132} pair and usually requires further purification. (auth)

7136 AECU-3462
Tennessee. Univ., Knoxville.
A STUDY OF THE REACTION RATE FOR THE ION EXCHANGE SYSTEM DOWEX-50, CALCIUM, AND SODIUM UNDER FLOW CONDITIONS. N. T. Anderson and F. N. Peebles. Dec. 1956. 82p. For [Oak Ridge National Lab.]. Contract W-7405-eng-26, Subcontract No. 783. $0.45(OTS).

The ion exchange system Dowex-50, Ca, Na, was studied by measuring concentration changes in feed solution passed over a 1/8-in. thick, 4-in. diam. bed of Dowex-50. The mechanism is diffusion controlled and obeys the theory to a reasonable degree. (T.R.H.)

21999 CEA-tr-X-154
SÉPARATION DU PROTACTIMIUM D'AVEC LE THORIUM À L'AIDE DE RESINES ÉCHANGEUSES DE CATIONS. (Separation of Protactinium from Thorium by

ION EXCHANGE 6.12

EXCHANGERS 6.12.02

Means of Cation Exchange Resins.) K. Kumura, Y. Yokohama, H. Sans, and H. Mabuchi. Translated into French by Sen-Sik Minn from Bunseki Kagaku 6, 637-41 (1957). 13p.

The separation of Pa^{234} from its mother Th^{234} by cation exchange is described. About 0.9 g of Dowex 50-X 12 resin (100-150 mesh) was used in a column 1 $cm^2 \times 1.5$ cm high. The nitric acid solution of Th plus Pa is prepared and passed through the column. The Pa is eluted by 2 N HNO_3 or 0.5% oxalic acid. Distribution coefficients, elution curves, and decay curves are given. (T.R.H.)

RESINS – GENERAL 6.12.02.04

See also, this section,

PLASTICS AND HIGH POLYMERS (6.15.10).

18743 ✓ YAEC-115

Westinghouse Electric Corp. Atomic Power Dept., Pittsburgh.

EVALUATION OF ION EXCHANGE RESINS FOR USE IN THE YANKEE REACTOR. N. Michael and M. Bell. July 1959. 36p. For Yankee Atomic Electric Co. Contract AT(30-3)-222, Subcontract No. 1. $6.30(ph), $3.00(mf) OTS.

Radioactivity removal efficiencies, generally reported in terms of decontamination factors (DF), have been determined for aqueous corrosion and fission product solutions using mixed-bed ion exchange resins. Experiments show that many factors such as pH and constituent concentration affect the decontaminating ability of the resins. In carefully controlled laboratory tests, separate batches of radioisotopes often yield large differences in decontamination factors. The (H^+, OH^-) form of mixed cycle resins, which is capable of exchanging H–OH for acids, bases, and salts, gave DF values as low as 330 and as high as 1200. A typical alkali-releasing mixed cycle ion exchanger gave DF values between 7.7 and 95. Similarly, in a reactor application the decontaminating efficiency of the purification system may be expected to fluctuate with transient water conditions. Mixed-bed resin exhibits satisfactory thermal stability, as evidenced by the slight change in capacity on long exposure to moderately high temperatures. Eighteen weeks exposure to 140°F water, maximum temperature planned for ion exchange resin operation in the Yankee plant, caused only a 12% reduction in exchange capacity. Strongly basic anion resins were tested for their ability to remove boric acid from solution. With a 38 ppm boron concentration, the total capacity of the recommended anion resin for use in the Yankee reactor, Rohm and Haas XE-78, was found to be 4.6 lbs/cu ft. With more concentrated solution, the capacity of the resin increased; under alkaline conditions the capacity decreased. Stainless steel corrosion products, generated in a dynamic autoclave at 600°F, were circulated through a low-pressure purification system consisting of a demineralizer and/or filters. Several properties were determined: quantity of crud picked up until resin is exhausted; quantity of crud picked up by cellulose filters up- and down-stream of the resin bed; efficiency of resin regeneration; and affect of resin on corrosion products, rendering these more filterable. The experiments also showed that the elemental composition of stainless steel corrosion products is dependent upon conditions such as the oxygen content of the water. It was observed that the mechanism of crud pick-up by resin consists of a combination of ion exchange and filtration. Consequently, any calculations of resin service-life for a reactor purification system based solely on resin exchange capacity give an inaccurate and pessimistic estimate. (auth)

New Mexico. Univ., Albuquerque.

ION-EXCHANGE RESINS. A Bibliography of Unclassified References. Milton Kahn, Katheryn Lawson, and Kent J. Jones. July 1956. 180p. For Los Alamos Scientific Lab. Contract W-7405-Eng-36. (LA-2054UNM).

This bibliography contains 2088 references to unclassified work on ion-exchange resins. Information is included on the preparation and properties of resins, the preparation of inorganic and organic compounds, the separation of elements and biological materials, the determination of physicochemical properties of ions in solution, the purification of water, and on applications to analytical chemistry, pharmacy, medicine, and various industries. The references were obtained from *Chemical Abstracts*, 1947 to 1954, inclusive.

ION EXCHANGE 6.12

EXCHANGERS 6.12.02

RESINS – GENERAL 6.12.02.04

1435 JEN-45
Spain. Junta de Energia Nuclear, Madrid.
EL CAMBIO DE ION Y SU RELACION CON LA INDUSTRIA. I. QUIMICA DE LOS CAMBIADORES DE ION MÉTODOS DE EMPLEO. (Ion Exchange and Its Relation with Industry. I. Chemistry of Ion Exchangers. Methods of Use). B. Lopez Perez, A. Huriarte Hueda, and L. Gutierrez Jodra. 1958. 21p.

The latest trends in the preparation of modern synthetic ion exchangers obtained by the treatment of polymerization and polycondensation products are reviewed. The physical and chemical characteristics, especially the stability, of exchangers are discussed. The utilization of ion exchangers in basic operations is described and illustrated with the results obtained in its application to the hydrometallurgy of uranium. The life of such materials are also considered. The most important synthetic commercial exchangers and their uses and properties are tabulated. (tr-auth)

4760
RECENT ADVANCES IN THE PREPARATION AND USES OF ION-EXCHANGE RESINS. D. K. Hale (Chemical Research Lab., Teddington, Middlesex). Analyst 83, 3-9 (1958) Jan.

10096
THE ION EXCHANGE RESIN AND ITS USES IN THE METALLURGY OF URANIUM. R. De Leone. Energia nucleare (Milan) 3, 168-75(1956) June 15. (In Italian)

A brief survey is given of the principal characteristics of the ion exchange resins, and a description is given of equipment for the production of the raw salts of uranium using anion resins. (B.J.H.)

6605
THE BEHAVIOUR OF ION-EXCHANGE RESINS WITH MIXED SOLVENTS. C. W. Davies and B. D. R. Owen. J. Chem. Soc., 1676-80(1956) June.

Data are presented for the swelling of some ion-exchange resins in acetone–water and dioxan–water, and for the distribution of the solvents between the external and resin phases. The use of partition chromatography on ion-exchange columns is illustrated by some experiments with the alkali metal cations. (auth)

8298
THE RATE OF SWELLING OF ION-EXCHANGE RESIN PARTICLES IN DIFFERENT SALT SOLUTIONS. Carlos Pulido (Univ. of Uppsala, Sweden). Acta Chem. Scand. 10, No. 1, 49-55(1956).

ION EXCHANGE SEPARATIONS. CHROMATOGRAPHIC SEPARATIONS BASED ON ION CHARGE. James S. Fritz and Shirley K. Karraker (Iowa State Coll., Ames). Anal. Chem. 31, 921-3(1959) May.

A rapid chromatographic separation is based on the greater affinity of a cation exchange resin for a trivalent than for a bivalent metal ion. A solution of ethylenediammonium chloride serves as the eluting agent. Conditions are worked out for separation of zinc and lanthanum, and the effect of variables is studied. This type of separation should be applicable to a wide variety of analytical problems. (auth)

5292
ON THE ANION-EXCHANGE BEHAVIOR OF SOME QUADRIVALENT ACTINIDE ELEMENTS. Nobufusa Saito and Tatsuya Sekine (Univ. of Tokyo, Japan). Bull. Chem. Soc. Japan 30, 561-2(1957) July.

The adsorption of Th^{4+}, U^{4+}, and Np^{4+} on anion exchange resins was studied. The adsorption of Th^{4+} from 0.1M $(NH_4)_2SO_4$ solution was so complete no activity was detected in the effluent. The adsorbed Th^{4+} was easily eluted with 1N HCl. U^{4+} was adsorbed on a resin bed of sulfate solution containing Zn, which was used to reduce the uranyl ions. A green adsorption band, which was not affected by washing of the column with 0.01M $(NH_4)_2SO_4$ solution, appeared near the top of the column. The U^{4+} was eluted with 6N HCl. Equilibrium experiments were carried out on Th^{4+}. Column studies of the exchange behavior of Th^{4+} and Np^{4+} were carried out. Both ions were adsorbed from carbonate solutions. (J.S.R.)

5930
NEW ION EXCHANGE RESIN FOR URANIUM RECOVERY. A. H. Greer, A. B. Mindler, and J. P. Termini (Permutit Co., New York). Ind. Eng. Chem. 50, 166-70(1958) Feb.

A new resin, developed specifically for U recovery from H_2SO_4 leach liquors, is available commercially – Permutit SK for resin-in-column operation and Permutite SKB for resin-in-pulp operation. These resins have a fast U adsorption rate and a fast and a complete U elution rate. They have good physical and chemical stability which will mean a minimum of physical breakdown and only a small loss in U capacity. (W.L.H.)

ION EXCHANGE 6.12

EXCHANGERS 6.12.02

RESINS – GENERAL 6.12.02.04

9758

VARIABLE CAPACITY ANION EXCHANGE RESINS FROM QUATERNIZED DIMETHYLAMINOSTYRENE-STYRENE BEAD COPOLYMERS. Richard H. Wiley and J. M. Schmitt (Univ. of Louisville, Ky.). J. Am. Chem. Soc. 80, 1389-91(1958) Mar. 20.

A series of bead copolymers of dimethylaminostyrene and styrene cross-linked with 1–20% of divinylbenzene have been quaternized with methyl iodide at room temperature. Divinylbenzene cross-linked (10%) resins having exchange capacities of 1.47, 2.35, 2.80, and 3.00 meq/g are described. These capacity values are in agreement with theoretical values calculated on the basis of the copolymer composition and established 96–100% effective quaternization. Partially quaternized resins, quaternized by previously described techniques which give no more than 52% of the theoretical amount of quaternization, can be quaternized up to 88% of the theoretical amount by additional treatment with methyl iodide at room temperature. (auth)

3622

A SPECTROSCOPIC STUDY OF THE SORPTION OF METALLIC CATIONS BY OXIDIZED CELLULOSE. I. N. Ermolenko and R. G. Zhbankov. Kolloid. Zhur. 20, 429-35(1958) July-Aug. (In Russian)

The sorption of cations from dilute solutions containing UO_2^{2+}, Pb^{+2}, Cu^{2+}, Cd^{2+}, Ag^{+}, and Li^{+} has been investigated by means of a previously developed infrared spectroscopic method. It was found that active sorption takes place also from dilute solutions, and in case of a mixture both cations are sorbed even if they differ considerably in their affinity for carboxyl groups. Changes in concentration do not affect the degree of sorption, indicating a high sorption energy. It has been established that contrary to cellulose and dialdehyde cellulose the sorption of UO_2^{2+}, Pb^{2+}, and Ca^{2+} on the carboxyl groups of oxidized cellulose is associated with considerable increase in absorption in the 7 to 8μ region, varying with the cation species. (tr-auth)

RESINS – PHOSPHATE AND PHOSPHONATE 6.12.02.05

4360 AERE-C/R-1896

Gt. Brit. Atomic Energy Research Establishment, Harwell, Berks, England.

SEPARATION OF HEAVY METALS WITH ACID ALKYL PHOSPHATE AND PHOSPHONATE RESINS. J. Kennedy, R. V. Davies, and B. K. Robinson. Mar. 3, 1956. 6p.

Di-allyl phosphate resin in the H^{+} and Na^{+} form in conjunction with inorganic acids and di-sodium ethylenediamine tetraacetate as eluting agents has been successfully employed in the separation of UO_2^{2+} from alkaline earths, divalent transitional, trivalent Fe and lanthanide elements. Separation from tetravalent cations such as Zr and Th was not complete. (auth)

ION EXCHANGE SUBSTANCES BY SAPONIFICATION OF ALLYL PHOSPHATE POLYMERS. J. Kennedy (to U. S. Atomic Energy Commission). U. S. Patent 2,882,248. Apr. 14, 1959.

An ion exchange resin having a relatively high adsorption capacity for uranyl ion as compared with many common cations is reported. The resin comprises an alphyl-allyl hydrogen phosphate polymer, the alphyl group being either allyl or a lower alkyl group having up to 5 carbon atoms. The resin is prepared by polymerizing compounds such as alkyl-diallyl phosphate and triallyl phosphate in the presence of a free radical generating substance and then partially hydrolyzing the resulting polymer to cause partial replacement of organic radicals by cations. A preferred free radical generating agent is dibenzoyl peroxide. The partial hydrolysis is brought about by refluxing the polymer with concentrated aqueous NaOH for three or four hours.

17941

ION-EXCHANGE RESINS COMPRISING ALLYL ALPHA-AMINOALKYL PHOSPHONATE POLYMERS. J. Kennedy (to U. K. Atomic Energy Authority). British Application 14485/56. Atom No. 9, 27(1957) July.

These polymers are prepared by reacting the polymer of diallyl hydrogen phosphonate with an aliphatic ketone or aldehyde and a primary or secondary amine or with an aldehyde-imine, ketone-imine, or alpha-alkylolamine, and then hydrolyzing the product. The resins have a high affinity for cupric and ferric ions, and have a greater affinity for divalent transition metal ions than for uranyl ions.

ION EXCHANGE 6.12

EXCHANGERS 6.12.02

Resin - Phosphate and Phosphonate 6.12.02.05

ION-EXCHANGE PROPERTIES OF CELLULOSE PHOSPHATE. A. J. Head, N. F. Kember, R. P. Miller, and R. A. Wells (Chemical Research Lab., Teddington, Middlesex, Eng.). J. Chem. Soc. 3418-25(1958) Oct.

The ion-exchange properties of cellulose phosphate prepared by the urea-phosphoric acid method have been investigated. This material has been shown to possess a high affinity for certain cations, principally Th^{4+}, Ti^{4+}, U^{4+}, Ce^{4+}, Fe^{3+}, ZrO^{2+}, and UO_2^{2+}, which are adsorbed from 4N-acid. The influence of the preparative conditions and structure of the ester on its ion-exchange properties is described and discussed. (auth)

RESINS - THEORY 6.12.02.06

13716

ON ION EXCHANGE EQUILIBRIA. V. AN APPROXIMATE METHOD FOR CALCULATING THE PARTIAL MOLAR VOLUMES OF THE SPECIES IN ION EXCHANGERS. Erik Högfeldt (Brookhaven National Lab., Upton, N. Y.). Acta Chem. Scand. 12, 182-7(1958).

By formally regarding an ion exchange system as a binary mixture of the two resin forms, partial molar volumes can be determined using the intercept method of Roozeboom. This method was applied to the potassium-tetramethylammonium exchange on resins with DVB content 2, 10, and 26%, reported by Gregor, Gutoff, and Bregman. It is surprising to find that, to a first approximation all three systems have an ideal volume of mixing over the large range of variation, 1.3 to 18, in total molality of the resin. (auth)

16306

ANION-EXCHANGE STUDIES. XXIII. ACTIVITY COEFFICIENTS OF SOME ELECTROLYTES IN THE RESIN PHASE. Frederick Nelson and Kurt A Kraus (Oak Ridge National Lab., Tenn.). J. Am. Chem. Soc. 80, 4154-61(1958) Aug. 20.

Ratios Γ of activity coefficients of a number of electrolytes in the resin ($\gamma_{\pm(r)}$) to those in the aqueous phase ($\gamma_{\pm}$) are evaluated from water and electrolyte uptake data. At high concentrations $\Gamma = \gamma_{\pm(r)}/\gamma_{\pm}$ is remarkably constant but it varies substantially at low concentrations. Activity coefficients of HCl in the resin determined as a function of HCl activity and of cross-linking can be correlated by a concentrated electrolyte model of the exchanger with the assumption that Harned's rule of the linear variation of log $\gamma_{\pm}$ with composition holds at constant total molality. Similar considerations, with Harned's rule generalized to higher multi-component

13057 AEC-tr-3312

INTERRELATIONSHIP OF STRUCTURE AND EXCHANGE CAPACITY OF CATIONIC RESINS. G. J. Moralli. Translated by K. S. Bevis for Savannah River Lab. from J. recherches centre natl. recherche sci., Lab. Bellevue (Paris), No. 26, 277-89(1954). 24p.

Starting with ion exchange resins prepared from specific monomers, it is possible to study the ion exchange phenomenon by both a dynamic and a static method. Sulfonated phenol resins, having the sulfonic groups in the ortho position with respect to the phenolic hydroxyl group, have a higher capacity than the others, and one that is higher than the theoretical content of sulfonic groups. This fact proves that, in this case, there is an activation of the phenolic hydroxyl groups which also take part in the exchange. Some measurements of exchange selectivity, made on a mixture of radiosodium and radiopotassium, demonstrated that there is a great deal of interest in using resins of a known structure, preferably commercial resins. (auth)

ION-EXCHANGE EQUILIBRIA ON SINGLE BEADS. Erik Högfeldt (Brookhaven National Lab., Upton, N. Y.). Science 128, 1435-6(1958) Dec. 5.

Equilibria on single ion-exchange resin beads show that large differences may exist between beads from the same batch. They may be so large that a significant contribution to deviations from ideality can be due to this heterogeneity effect. Correlation between swelling and equilibrium properties shows that bead-to-bead variations are due to differences in cross-linking. (auth)

8735

ION-EXCHANGE DATA FOR THE ACTINIDE ELEMENTS IN NITRIC AND HYDROCHLORIC ACID SOLUTIONS. C. J. Hardy (Atomic Energy Research Establishment, Harwell, Berks, Eng.). pp. 357-62 in "Progress in Nuclear Energy. Series III. Process Chemistry. Volume 2."

ION EXCHANGE 6.12

EXCHANGERS 6.12.02

RESINS – THEORY 6.12.02.06

Ion-exchange data for the actinide elements in nitric and hydrochloric acid solutions are summarized. The selection of resin and operating conditions for the separation and purification of actinide elements and the structure of species in solution are discussed. (auth)

2987 ✓ ACCO-61
American Cyanamid Co. Atomic Energy Div. Raw Materials Development Lab., Winchester, Mass.
ION EXCHANGE STUDIES. PART I. EQUILIBRIUM CONSTANTS. PART II. NATURE OF URANIUM ADSORPTION ON IRA-400. T. L. O'Connor. July 28, 1954. Decl. Sept. 23, 1955. 27p. Contract AT(49-1)-533.

The relative affinities of chloride and bisulfate as compared to sulfate for IRA-400 have been studied. Both have a slightly higher affinity for the resin than sulfate as indicated by equilibrium constant determinations. Investigation on the nature of uranium adsorbed on IRA-400 has shown that the uranium is present principally as uranyl tri-sulfate complex. Some disulfate complex is also present and the ratio of the two complexes was found to be dependent upon uranium and sulfate concentrations, and pH. (auth)

6612
ADSORPTION OF COMPLEX ANIONS FROM URANYL SULPHATE SOLUTION BY ANION-EXCHANGE RESINS.
T. V. Arden and G. A. Wood (Chemical Research Lab., Teddington and Permutit Col. Ltd., London). J. Chem. Soc., 1596-1603(1956) June.

At pH values below pH 2.5 U is adsorbed from SO_4^{2-} solution by strong-base anion-exchange resins as a complex $UO_2(SO_4)_3^{4-}$. There is evidence that adsorption occurs by the formation of this ion on the resin by the addition of UO_2SO_4 to the resin sulfate, as well as by the normal exchange process. At pH values in excess of 2.5, the U loading of the resin increases, owing to the presence of increasing proportions of $U_2O_5(SO_4)_3^{4-}$ on the resin. (auth)

7148
ANION EXCHANGE IN URANYL SULPHATE SOLUTION. HIGH-CONCENTRATION EFFECTS AND RATE STUDIES.
T. V. Arden and Marion Rowley (Permutit Co., Ltd., London). J. Chem. Soc. 1709-13(1957) Apr.

The concentration of U and SO_4^{2-} in anion-exchange resins in equilibrium with concentrated solutions of UO_2SO_4, is higher than the saturation values for $UO_2(SO_4)_3^{4-}$, $UO_2(SO_4)_2^{2-}$, or $U_2O_5(SO_4)_3^{4-}$. The increase may be due to the presence of $U_2O_5(SO_4)_2^{2-}$, or more probably to the permeation of undissociated UO_2SO_4 into the resin, in addition to the normal exchange process. In dilute solution the rate of adsorption of U decreases with increasing pH, owing to the lower rate of diffusion through the resin of $U_2O_5(SO_4)_3^{4-}$ than $UO_2(SO_4)_3^{4-}$. (auth)

(J.S.R.)

6512 ✓ A/CONF.15/P/1113
AN ANALYSIS OF THE CHARACTERISTICS OF ANION EXCHANGE RESINS SUITABLE FOR THE URANIUM RECOVERY FROM ACID LEACH LIQUORS. R. E. Robinson, R. G. Velthuis, and E. T. Pinkney (Government Metallurgical Lab., Union of South Africa). 31p.

A brief outline of the ion exchange process for U extraction is presented with special reference to the aspects of interest in resin evaluation. The various properties of the resin which play an important role in the ion exchange operation are discussed in some detail. (W.L.H.)

PROCESSES AND METHODS 6.12.03

See also, this section,

RADIATION EFFECTS--Aqueous Solutions (6.15.01).

COUNTERCURRENT METHODS 6.12.03.01

COUNTERCURRENT ION EXCHANGE. T. A. Arehart, J. C. Bresee, C. W. Hancher, and S. H. Jury. Feb. 15, 1956. 39p. (CF-56-2-66)

A general discussion of countercurrent ion exchange is presented. Attention is focused on the operational features and test results from a semi-continuous ion exchange contactor, which has been the subject of recent investigations concerning the recovery of U from low grade western ores. A description is given of a 6 in. diameter contactor which

PROCESSES AND METHODS 6.12.03

Countercurrent Methods 6.12.03.01

was employed for experimental purposes. An engineering analysis follows wherein there is demonstrated a procedure for scale-up from a 6 to a 12 in. contactor. The operating characteristics and improvements made on the 12 in. contactor are outlined. Consideration is given to the chemistry of the uranyl ion and its bearing on the engineering analysis. The results of a mechanical resin attrition study under simulated contactor conditions is included.

Stanford Research Inst., Menlo Park, Calif.
SELECTED LIST OF REFERENCES REVIEWED DURING THE STUDY OF COUNTERCURRENT ION EXCHANGE. TECHNICAL REPORT NO. 8. Nevin K. Hiester and Russell C. Phillips. June 29, 1953. 20p. (AECU-2729).

This bibliography includes 216 references arranged alphabetically by authors.

8651 CEA-tr-R-348
L'ANALOGIE ENTRE L'ECHANGE ISOTOPIQUE DANS LES COLONNES A CONTRE-COURANT ET LES PROCESSUS D'ADSORPTION. (The Analogy between Ion Exchange in Counter-Current Columns and the Processes of Adsorption.) A. M. Rozen. Translated into French from Doklady Akad. Nauk S.S.S.R. 108, 122-5(1956). 13p.

The analogy between isotopic exchange in countercurrent columns and the processes of adsorption is discussed on the basis of the classical equation for mass transfer. As an example the reaction $H_2O^{18} + \frac{1}{2} CO_2^{16} = H_2O^{16} + \frac{1}{2} CO_2^{18}$ is used for the calculation of the isotopic exchange from the mass transfer equation. (J.S.R.)

ELUTION 6.12.03.02

See also, this section,

RADIATION EFFECTS--Aqueous Solutions (6.15.01).

ELUTION OF URANIUM FROM RESIN. Daniel C. McLean (to U. S. Atomic Energy Commission). U. S. Patent 2,877,089. Mar. 10, 1959.

A method is described for eluting uranium from anion exchange resins so as to decrease vanadium and iron contamination and permit recycle of the major portion of the eluate after recovery of the uranium. Diminution of vanadium and iron contamination of the major portion of the uranium is accomplished by treating the anion exchange resin, which is saturated with uranium complex by adsorption from a sulfuric acid leach liquor from an ore bearing uranium, vanadium and iron, with one column volume of eluant prepared by passing chlorine into ammonium hydroxide until the chloride content is about 1 N and the pH is about 1. The resin is then eluted with 8 to 9 column volumes of 0.9 N ammonium chloride–0.1 N hydrochloric acid solution. The eluants are collected separately and treated with ammonia to precipitate ammonium diuranate which is filtered therefrom. The uranium salt from the first eluant is contaminated with the major portion of the vanadium and iron and is reworked, while the uranium recovered from the second eluant is relatively free of the undesirable vanadium and irons. The filtrate from the first eluant portion is discarded. The filtrate from the second eluant portion may be recycled after adding hydrochloric acid to increase the chloride ion concentration and adjust the pH to about 1.

5576
A NEW ELUANT FOR THE SEPARATION OF THE ACTINIDE ELEMENTS. G. R. Choppin, B. G. Harvey, and S. G. Thompson (Univ. of California, Berkeley). J. Inorg. and Nuclear Chem. 2, 66-8(1956) Jan.

The use of ammonium α-hydroxy-isobutyrate as the eluting agent for the separation of the higher actinides from cation resin columns is described. (C.W.H.)

Refer also to abstract 5915.

6369 A/CONF.15/P/1424
CONTRIBUTIONS TO THE USE OF ORGANIC SOLVENT ELUANTS IN CATIONIC EXCHANGE. S. Ionescu, O. Constantinescu, D. Topor, and E. Gârd (Inst. of Atomic Physics, Bucharest). 12p.

The possibility of using a mixture of acetone with HCl and water as an actual eluant for several cations on resins is investigated. The behavior of Cu, Zn, Ce, La, uranyl, and Th cations on KU-2 and R-21 resins is discussed. (W.D.M.)

ION EXCHANGE 6.12

PROCESSES AND METHODS 6.12.03

ELUTION 6.12.03.02

7954 ✓

ION-EXCHANGE METHODS FOR THE QUANTITATIVE SEPARATION OF THE ALKALINE EARTHS, AND THEIR APPLICATION TO THE DETERMINATION OF Sr^{90} IN MILK ASH. Gwen M. Milton and W. E. Grummitt (Atomic Energy of Canada Ltd., Chalk River, Ontario). Can. J. Chem. 35, 541-51(1957) June.

A study has been made of the effects of column loading and eluting agent on the cation-exchange separation of the alkaline earths — Be, Mg, Ca, Sr, Ba, and Ra. The principal eluting agents tested were ammonium lactate, ammonium citrate, and HCl. Dowex-50 resin (Ion X, 300–400 mesh) was used throughout the investigation, and the column temperature was maintained at approximately 80°C. Under optimum conditions, involving the use of 0.25 g. of resin per milliequivalent of sample and with ammonium lactate eluant (0.55 M at pH 5 for Be, and 1.5 M at pH 7 for Mg, Ca, Sr, Ba, and Ra), a quantitative separation of all six elements in a single sample is possible in 5 hours. This procedure has been applied to the routine analysis of Sr^{90} in bulk milk ash samples, containing 20 mg. of Sr carrier, with yields of 85 to 95%. By the same method trace amounts of Sr naturally occurring in milk have also been determined. (auth)

ETHYLENE DIAMINE TETRAACETIC ACID AND CITRIC ACID AS ELUANTS IN ION EXCHANGE SEPARATION OF RARE EARTHS. F. W. Cornish, G. Phillips, and A. Thomas (Atomic Energy Research Establishment, Harwell, Berks, England). Can. J. Chem. 34, 1471-82(1956) Oct.

Studies of the distribution of rare earths between the cation exchange resin Zeokarb 225 and solutions of ethylene diamine tetraacetic acid (EDTA) show the existence of large separation factors between members of this series. However, column experiments show that the separation is poorer than with citric acid, probably because of a slow process occurring in the solution. The variation of column efficiency with distribution coefficient has been examined. Elution of europium with citric acid appears to be diffusion controlled, while that with EDTA is not. Further experiments with strontium indicate that its elution with both EDTA and citric acid is diffusion controlled. The results are in general agreement with Glueckauf's equation relating column variables with the height equivalent to a theoretical plate. Column irregularities appear to account for the very low diffusion constants calculated from this relation. (auth)

3119 ✓ ACCO-59

American Cyanamid Co. Atomic Energy Div. Raw Materials Development Lab., Winchester, Mass.

SYSTEMATIC ELUTION STUDIES. PART I. THE EFFECT OF SULFATE ACCUMULATION ON THE ELUTION OF URANIUM FROM STRONG BASE ION EXCHANGE RESINS WITH CHLORIDE. A. R. Kazanjian. [July 29, 1954]. Decl. Sept. 23, 1955. 22p. Contract AT(49-1)-533.

The increase in the volume of chloride eluate required for complete elution as the number of cycles of operation progresses has been studied. The build-up in solution of the concentration of sulfate has been found to be responsible for this phenomemon. It was found, also, that addition of ammonium sulfate to provide a concentration of above 100 g SO_4/l resulted in a reduction in the number of volumes of eluting solution required to a value only slightly greater than that required in the absence of sulfate. The effects of the presence of Na and $MgSO_4$ were similarly studied, but these salts led only to increasing the number of column volumes of eluate required. (auth)

2743 WIN-27

National Lead Co., Inc. Raw Materials Development Lab., Winchester, Mass.

DEVELOPMENT OF AN EFFICIENT ELUANT FOR REMOVAL OF MOLYBDENUM FROM ANION EXCHANGE RESINS. Kenneth P. Quinlan and Robert J. Barry. Aug. 1, 1956. 21p. Contract AT(49-6)-924. $3.30(ph OTS); $2.40(mf OTS).

When acid leach liquors obtained from U ores containing Mo are contacted with anion exchange resin, both U and Mo are adsorbed. The adsorbed Mo is not removed in the normal U elution process, and when the U-barren resin is recycled, the resin sites already occupied by Mo are unavailable for U adsorption. In succeeding cycles, more and more sites become occupied by Mo, and the resin capacity for U is seriously lowered. This rapid accumulation of an adsorbed species is known as resin "poisoning." The many disadvantages of such a situation are self-evident. (auth)

THE PRACTICAL APPLICATION OF CHROMATOGRAPHIC THEORY TO ANALYTICAL AND PREPARATIVE SEPARATIONS BY ION-EXCHANGE ELUTION. F. W. Cornish (Atomic Energy Research Establishment, Harwell, Berks, Eng.). Analyst 83, 634-42(1958) Nov.

ION EXCHANGE 6.12

PROCESSES AND METHODS 6.12.03

ELUTION 6.12.03.02

Data are given from which adequate operating conditions can be deduced for the separation of ionic species by ion-exchange elution. (auth)

1808

ION EXCHANGE PROCESSES. M. G. M. Atmore and R. R. Porter (to Transvaal and Orange Free State Chamber of Mines, Calcined Products, Pty., Ltd., (South Africa)). British Patent 775,529. Nuclear Eng. 2, 452(1957) Oct.

A process for the recovery of U in which relatively expensive ions are conserved for eluting purposes. The process is a three-stage process. In the first stage the ion exchange material does not carry the eluting ion. The second stage consists in the normal eluting step, which if followed by a third stage, i.e., a further eluting step in which the original eluting ion is displaced by the exchange ion. The exchange ion is conveniently the sulfate ion, the eluting ion, and the nitrate ion. The effluent recovered from the eluting ion recovery step may be concentrated and re-used

1304 ✓

ANION-EXCHANGE STUDIES. II. THE EFFECT OF CROSS-LINKAGE ON THE ELUTION OF SEVERAL TRANSITION ELEMENTS. Rolfe H. Herber, Kayacan Tonguc, and John W. Irvine, Jr. (Massachusetts Inst. of Tech., Cambridge). J. Am. Chem. Soc. 77, 5840-3(1955) Nov. 20.

A number of samples of the strongly basic anion exchange resin Dowex-1 have been studied with reference to their capacity, water content and elution behavior. Column experiments on the elution of Mn^{2+}, Co^{2+}, Cu^{2+}, and Zn^{2+} with HCl show that in each case retention of the anion complex is dependent on the cross-linkage of the exchanger. While 15 cm columns of 12% DVB Dowex-1 will effectively separate Mn^{52}–Na^{22} mixtures, 2 and 4% DVB resins will not effect this separation even in 12N HCl. A convenient instrumental arrangement for determining elution behavior of ion-exchange resins is described. (auth)

USE OF SPARINGLY-SOLUBLE SALT-FORMING REAGENTS AS ELUTING AGENTS IN CATION-EXCHANGE PROCESSES. Hideo Yamatera (Osaka City Univ.). J. Inorg. & Nuclear Chem. 7, 299-300(1958) Oct.

Reagents that form sparingly soluble salts with the cation to be removed were examined as to their suitability as eluting agents in the cation-exchange processes. In every case tested, the reagents proved to be effective, and in some cases were found to be of practical use. It was demonstrated that the perchlorate ions promote elution of the Cs ion. Experiments were also performed to confirm the effectiveness of nitric acid for elution of Sr and Ba, of $(NH_4)_2CO_3$ and Na_2CO_3 for elution of rare earths and Sr, and of $(NH_4)_3SO_4$ for elution of Sr. The results and probable explanation parallel that for Cs. (T.B.A.)

ION EXCHANGE STUDY OF THIOCYANATE COMPLEXES OF THE ACTINIDES AND LANTHANIDES. J.P. Surls and G.R. Choppin (Univ. of California). Journ. Inorg. Nucl. Chem. 4, 62-73, 1957.

The elution behavior of 3-valent actinides, lanthanides and yttrium with ammonium thiocyanate solution from Dowex 1 anion exchange resin was studied. The lanthanides and yttrium eluted sooner than the actinides under all the conditions studied. Neither lanthanides not actinides showed a simple elution sequence with atomic number. Using Dowex 50 resin, however, the actinides eluted in the usual sequence of decreasing atomic number. The actinides eluted sooner than the lanthanides of comparable ionic radius, indicating stronger complexing of the actinides with thiocyanate.

195

ETHYLENE DIAMINE TETRAACETIC ACID AND CITRIC ACID AS ELUANTS IN ION EXCHANGE SEPARATION OF RARE EARTHS. F. W. Cornish, G. Phillips, and A. Thomas (Atomic Energy Research Establishment, Harwell, Berks, England). Can. J. Chem. 34, 1471-82(1956) Oct.

Studies of the distribution of rare earths between the cation exchange resin Zeokarb 225 and solutions of ethylene diamine tetraacetic acid (EDTA) show the existence of large separation factors between members of this series. However, column experiments show that the separation is poorer than with citric acid, probably because of a slow process occurring in the solution. The variation of column efficiency with distribution coefficient has been examined.

ION EXCHANGE 6.12

PROCESSES AND METHODS 6.12.03

Elution 6.12.03.02

Elution of europium with citric acid appears to be diffusion controlled, while that with EDTA is not. Further experiments with strontium indicate that its elution with both EDTA and citric acid is diffusion controlled. The results are in general agreement with Glueckauf's equation relating column variables with the height equivalent to a theoretical plate. Column irregularities appear to account for the very low diffusion constants calculated from this relation. (auth)

GENERAL 6.12.03.03

7345

ANION-EXCHANGE STUDIES. XXVII. ABSORBABILITY OF A NUMBER OF ELEMENTS IN HCl–HF SOLUTIONS. Frederick Nelson, Richard M. Rush, and Kurt A. Kraus (Oak Ridge National Lab., Tenn.). J. Am. Chem. Soc. 82, 339-48(1960) Jan. 20.

Nineteen elements including some from groups 2, 3, 4, 5, and transition elements were studied at constant 1 M HF and variable HCl concentration (0.1 to 11 M). The data are compared with adsorbabilities in HCl solutions, not containing HF. A large group of elements which are nonadsorbable from HCl solutions are also not adsorbed from HCl–HF mixtures. This group includes even such highly charged ions as Al(III), Y(III), Eu(III), Th(IV), and V(IV). Be(II) shows some adsorption at low HCl concentration in the presence of 1 M HF. The adsorption functions of many elements adsorbable from HCl solutions show little change on addition of HF. The adsorption functions may be substantially altered as shown for Sc(III), Ti(IV), Fe(III), Ga(III), Ge(IV), Sn(II) and (IV), and Sb(III) and (V). From the adsorption functions in HCl and HCl–HF, estimates of the extent of fluoride complexing are obtained. Application of the adsorption data to separations is discussed and illustrated with typical cases. (auth)

ION EXCHANGE ISOLATION PROCESSES. F. R. Bruce. May 1, 1957. p. 303-31 of SYMPOSIUM ON THE REPROCESSING OF IRRADIATED FUELS HELD AT BRUSSELS, BELGIUM, MAY 20-5, 1957. 1165p. (TID-7534(Bks. 1-3))

Ion exchange processes have been developed which both concentrate the fissionable material and give additional separation from corrosion and fission products. The processes were developed specifically for the Pu product from the Purex and Redox processes and the U from the Thorex process. However, they have application to Pu and U isolation from other processes. These two processes have been demonstrated on the pilot plant and full plant scale. 12 references.

2455 CNLM-1802-3

Pratt and Whitney Aircraft Div., United Aircraft Corp., Middletown, Conn.

THE ION-EXCHANGE BEHAVIOR OF ALUMINUM, CHROMIUM, COBALT, MOLYBDENUM, NICKEL, NIOBIUM, TANTALUM, TITANIUM, TUNGSTEN, VANADIUM, AND ZIRCONIUM. A Bibliography. E. A. Cernak, comp. Oct. 9, 1959. 42p.

This bibliography contains 311 references on the separation, determination, and behavior of aluminum, chromium, cobalt, molybdenum, nickel, niobium, tantalum, titanium, tungsten, vanadium, and zirconium in ion exchange reactions. The references are arranged chronologically in twelve classifications. The sources used in the compilation are Abstracts of Classified Reports, 1948–Aug. 1959; Chemical Abstracts, 1945–Sept. 10, 1959, Nuclear Science Abstracts, 1948–Sept. 15, 1959; and the Card Catalog. (J.E.D.)

11142

ION EXCHANGE TECHNOLOGY. F. C. Nachod and Jack Schubert, eds. New York, Academic Press Inc., 1956. 660p.

The following subjects are discussed: ion exchange resins, equipment and process design, mass transfer and equilibria, principles of deionization with ion exchange resins, electrochemical operations, ion exclusion, economic considerations, prediction of fixed-bed performance with a computing machine, ion exchange technology in water treatment, catalysis with ion exchange resins, application in hydrometallurgy, chromium recovery from plating solutions, recovery from plating solutions, recovery of Cu from rayon wastes, the isolation in quantity of individual rare earths of high purity by ion exchange, processing radioisotopes by ion exchange, treatment of radioactive wastes, sugar refining and by-product recovery, organic chemical purification by ion exchange, and pharmaceutical and biological products. (J. E. D.)

ION EXCHANGE 6.12

PROCESSES AND METHODS 6.12.03

GENERAL 6.12.03.03

10103
ION EXCHANGE IN THE ATOMIC ENERGY PROGRAM. Robert Kunin and Albert F. Preuss (Rohm & Haas Co., Philadelphia). Ind. Eng. Chem. 48, 30A-5A(1956) Aug.
Ion exchange processes for the recovery of U from ores and ore solutions are described. Other uses of ion exchange in atomic energy programs are briefly outlined. (C.W.H.)

0299
ION EXCHANGE. FIVE CONTRIBUTIONS TO NUCLEAR TECHNOLOGY. 1. URANIUM EXTRACTION. 2. URANIUM PURIFICATION. 3. REACTOR WATER TREATMENT. 4. ZIRCONIUM PURIFICATION. 5. WASTE DISPOSAL. L. D. Roland (Permutit Co., Ltd.). Atomics 8, 131-5(1957) Apr.

4374
ION EXCHANGE. Henry C. Thomas and Galen R. Frysinger (Yale Univ., New Haven, Conn.). Ann. Rev. Phys. Chem. 7, 137-66(1956).
Ion exchange theory and applications are reviewed. The topics included are: Equilibria in the Ion Exchange Process; Distribution of the Solvent; Cation Exchanges; Anion Exchanges; the Nonexchange Ion and Neutral Molecules;. Kinetics in Ion Exchange; Inorganic Ion Exchangers; Separations Produced by Ion Exchange; Membranes of Ion Exchangers; Catalysis Produced by Ion Exchangers. (T.R.H.)

2478
SEPARATION OF SUBSTANCES PRESENT IN NEUTRON-IRRADIATED URANIUM. (to United Kingdom Atomic Energy Authority). British Patent 823,761. Nov. 18, 1959.
An ion-exchange chromatographic process for irradiated U is described. The U in HNO_3 solution is passed through a phenol formaldehyde resin column where U, Pu, and fission products are chromatographically adsorbed. The U is eluted by 0.2 M H_2CO_4, the fission products are removed by 0.6 M H_3PO_4 1 M in HNO_3, the Pu is then eluted by 0.8 M H_3PO_4 1 M in HNO_3. (T.R.H.)

CONCENTRATION AND PURIFICATION OF URANIUM, PLUTONIUM, AND NEPTUNIUM BY ION EXCHANGE IN NUCLEARLY SAFE EQUIPMENT. F. W. Tober. 19p. (A/CONF.15/P/520) Du Pont
Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.
The initial products from the processing of irradiated fuel elements are usually relatively dilute solutions of U, Pu, or Np which must be concentrated and further purified. Ion exchange has proved to be a useful method for accomplishing these objectives. Several isotopes which are, or accidentally could be, present in the process streams are fissionable. All the equipment, therefore, must be designed to prevent the accumulation of fissionable material which could generate an excessive amount of neutrons by multiplication. Even with the limitations imposed to provide nuclear safety, the processing can be done economically by ion exchange because a large batch of product can be absorbed, purified, and eluted within a short period of time. Nuclear safety is easily obtained by using resin beds with small diameters, or by using a composite column that consists of a series of shallow beds separated sufficiently to avoid interaction of the neutrons from the individually safe beds. Rapid processing is obtained by using the maximum flowrates that are practical. The rates of processing are directly proportional to the cross-sectional area but are independent of the depth of resin in the ion exchange column. General requirements that are common to all the processes are discussed, together with separate discussions of processes for U, Pu, and Np. Typical chemical flowsheets are presented. The process data needed to develop ion exchange processes and the basic chemistry that illustrates specific problems are described.

5272
ION EXCHANGE. Robert Kunin, Francis X. McGarvey, and Ann L. Farren (Rohm & Haas Co., Philadelphia). Ind. Eng. Chem. 49, 507-13(1957) March.
A literature review on ion exchange theory, applications, techniques, materials, and apparatus is presented. 274 references. (T.R.H.)

4763
RADIOCHEMICAL SEPARATIONS BY ION EXCHANGE. Kurt A. Kraus and Frederick Nelson (Oak Ridge National Lab., Tenn.). Ann. Rev. Nuclear Sci. 7, 31-46(1957).

ION EXCHANGE 6.12

PROCESSES AND METHODS 6.12.03

GENERAL 6.12.03.03

6366
PROCESS FOR REMOVAL OF CATIONS FROM AQUEOUS SOLUTION. E. Gleuckauf and P. J. O. Roberts (to U. K. Atomic Energy Authority). British Patent 781,726. Nuclear Eng. 3, No. 23, 90(1958) Feb.

The aqueous solution containing cations is brought into contact with a solid anion exchange substance, insoluble in water and having, at the exchange positions, anions which will form insoluble compounds with the cations. All metal ions except those of alkali metals may be precipitated. These cations may also be separated from each other, or from those of the alkali metals. The precipitate formed on the anion exchange substance may be removed by washing with a suitable aqueous acid or complexing agent. Anion's mentioned are hydroxide, carbonate, sulfide, or chromate.

7149
ION EXCHANGE AND ITS APPLICATIONS. London, Society of Chemical Industry, 1955. 173p.

The twenty papers read at conference at London University, England, April, 1954 are presented. Some of the topics discussed are: structure, synthesis, and properties of ion exchange resins; ion exchange equilibria and kinetics; operation of ion exchange columns; and continuous ion-exchange. The analytical, research, and industrial applications of ion exchange are treated. (T.R.H.)

11127
ION EXCHANGERS IN ORGANIC AND BIOCHEMISTRY. Calvin Calmon and T. R. E. Kressman, eds. New York, Interscience Publishers, Inc., 1957. 770p.

The first two parts of this volume are concerned with the fundamentals of ion exchange and the techniques and apparatus used in carrying out the exchange and separation of ions. The third part deals fully with its application to specific aspects of organic and biochemistry. (W.L.H.)

2328 CF-50-9-80
Oak Ridge National Lab., Tenn.
CONTINUOUS ION EXCHANGE COLUMN INVESTIGATIONS. S. H. Jury. Aug. 31, 1950. Changed from OFFICIAL USE ONLY Oct. 3, 1956. 17p. Contract [W-7405-eng-26]. $3.30(ph OTS); $2.40(mf OTS).

A continuous column was constructed and used to investigate fluidization characteristics, ion exchange parameters, and physical properties of resin beds. (T.R.H.)

8297
EXTRACTION FROM SLURRIES BY ION-EXCHANGE RESINS. E. A. Swinton and D. E. Weiss (C.S.I.R.O., Melbourne, Australia). Australian J. Appl. Sci. 7, 98-112(1956) Mar.

A pulsating technique is described which extends the use of the conventional ion-exchange procedure to the direct adsorption of soluble constituents from suspensions of finely divided solids, without the necessity for prior filtration. Slurries containing up to 40% by weight of solids have been passed through a bed of ion-exchange resin by pulsating the flow. It has been established that the process of displacement and ion exchange is as efficient as in conventional procedures using the same resin. An application of the process to the extraction of uranium ores is briefly described. (auth)

3576 ISC-1056
Ames Lab., Ames, Iowa.
ION-EXCHANGE SEPARATION OF METALS BY A SINGLE-PASS METHOD. Richard T. Oliver and J. S. Fritz. June 1958. 44p. Contract W-7405-eng-82. $1.25(OTS).

A single-pass method for the ion-exchange separations of binary mixtures of metals is described, and experimental applications are presented. The method consists of complexing each of the components in the mixture with a separate complexing agent at a pH sufficient to ensure maximum coördination of the metals. The complexing agents are chosen such that the metal complexes formed are of opposite charge. The mixture is then passed through an ion-exchange resin which absorbs one species completely, allowing the other to be collected in the effluent. An anion- or cation-exchange resin is used. Sulfosalicylic acid was used to form a

23133 CF-54-7-12(Rev.)
Oak Ridge National Lab., Tenn.
DETERMINATION OF THE EFFICIENCY OF AN ION EXCHANGE SYSTEM IN SEPARATING THE LITHIUM ISOTOPES (PRELIMINARY STUDY). R. E. Blanco, A. H. Kibbey, and J. T. Roberts. July 22, 1954. Decl. Feb. 9, 1959. 26p. $4.80(ph), $2.70(mf) OTS.

The lithium isotopic separation factors for the ion exchange systems studied appear to be quite low, of the

ION EXCHANGE 6.12

PROCESSES AND METHODS 6.12.03

GENERAL 6.12.03.03

magnitude of 1.003. Any possible effects on the separation factor attributable to concentration or the presence of complexing agents were within the limits of experimental error. The HTU for lithium ion exchange is low, i.e., a fraction of an inch in the absence of a complexing agent, at a flow rate of 1 ml/min/cm^2 and approximately proportional to the flow rate. In the presence of a complexing agent the HTU was larger and again was proportional to the flow rate. The efficiency of the system therefore cannot be improved by increasing the throughput by means of faster flow rates. This fact plus the low separation factor make it seem unlikely that lithium highly enriched in Li^7 may be economically produced by ion exchange at the present time, even in a continuous contactor. (auth)

22069
ION EXCHANGE STUDIES OF THE ACTINIDE ELEMENTS. Gregory R. Choppin (Florida State Univ., Tallahassee). J. Chem. Educ. 36, 462-5(1959) Sept.
A review is given on ion exchange studies of the actinide elements. References are contained to each of the specific processes. (C.J.G.)

THEORY 6.12.04

See also, this section,

ADSORPTION (6.01),
CHELATION (6.02),
COMPLEX FORMATION (6.05).

5264 ✓
THE SEPARATION OF SUBSTANCES WITH VERY SIMILAR PROPERTIES BY DISPLACEMENT METHODS USING ION EXCHANGERS. I. GENERAL STUDY. II. THE METHOD OF DEVELOPMENT BY DISPLACEMENT. Bernard Tremillon (Ecole de Physique et de Chimie, Paris). Bull. soc. chim. France, 502-11(1958). (In French)

THEORY 6.12.04

From the theory it is possible to foresee a bilogarithmic symmetrical form for displacement isochrones, both in frontal analysis and in development by displacement. The claculation is justified by experiment, but it is impossible to foresee exactly the spread of these isochrones since the height equivalent theoretical plate varies considerably not only with the mechanical operating conditions of the columns but also with the kinetics of the exchange reaction, and therefore with the nature of the ions exchanged. It seems to be possible nevertheless to maintain it reasonably within limits lower than a few millimeters. This is one of the great advantages of the ion exchange chromatography method over most of the other methods of countercurrent separation. The closer the exchange constant is to unity, the greater is the spread of the isochrone. Any quantitative forecast on the subject is submitted to a preliminary experimental determination of the height equivalent theoretical plate. In any case it can be affirmed that very quantitative exchange reactions are not necessary in order to obtain exceedingly sharp separations. (auth)

5252 ✓ HW-59709
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
ANNULAR FIXED-BED ION-EXCHANGE STUDIES. G. A. Nicholson. May 12, 1959. 29p. Contract W-31-109-Eng-52. OTS.
The effect of gas formation in the bed on the operating and mass transfer characteristics of both a simple and an annular fixed-bed ion-exchange column was studied. Standard methods were used in correlating the pressure drop and fluidization data. Other methods were used in studying the fluid channeling and/or resin loading characteristics of the simple and annular columns, respectively. (auth)

1454 ✓ CEA-tr-A-581
RÉACTIONS ANORMALES DANS LES ÉCHANGEURS D'IONS. (Anomalous Reactions in Ion Exchangers). R. Griessbach. Translated into French from Chimia (Switz.) 11, No. 2, 29-41(1957). 33p.
Anomalies in the behavior of ion exchangers and their interest to science are discussed. (T.R.H.)

ION EXCHANGE 6.12

THEORY 6.12.04

6398 √ A/CONF.15/P/1818
BEHAVIOUR OF CATIONIC EXCHANGE BETWEEN METALS AND ELECTROLYTIC SOLUTIONS.
F. Barreira (Comissão de Estudos da Energia Nuclear, Lisbon). 15p.

Metals and ionic solutions labelled with radioisotopes were used on the experimental study of cationic exchange. Experiments were performed with metallic zinc and silver, contacting with solutions of its own salts and solutions of other metal salts. Solutions of zinc sulfate and sodium sulfate and chloride were used with activated metallic zinc; cupric and zinc salts were employed with activated metallic silver. In all the systems studied the exchange of cations was observed with the only exception of metallic silver in contact with solutions of zinc salts. Results are discussed according to a proposed general theory of cationic exchange. On the system silver/cupric sulfate the influence of the surrounding gaseous atmosphere was studied. There is no difference when experiments are performed using argon instead of air.

13818 CF-58-8-36
Oak Ridge National Lab., Tenn.
A CHEMICAL ENGINEERING DEVELOPMENT PROGRAM—AN INVESTIGATION OF THE KINETIC MECHANISMS OF URANYL SALT ION EXCHANGE. S. H. Jury. Aug. 11, 1958. 17p. Contract [W-7405-eng-26.] $3.30 (nh OTS); $2.40(mf OTS).

Recent literature concerning uranyl salt complex chemistry and ion exchange was reviewed in an effort to develop the present state of understanding of the equilibria and kinetic mechanisms involved. In the light of this, a development program is discussed which hopefully would lead to further enlightenment. Various kinetic mechanisms of sorption and elution are proposed and a comprehensive mathematical development is given for one such sorption mechanism. (auth)

17004
MASS TRANSFER BEHAVIOR IN FIXED BEDS. James O. Osburn (State Univ. of Iowa, Iowa City). Chem. Eng. 67, No. 18, 143-5(1958) Sept. 8.

Design methods for fixed-bed operations such as gas chromatography, ion exchange, and absorption are presented. The length of the packed bed and its effect on the outlet concentration are of primary interest. Simple adsorption is used to remove a substance from a fluid stream, while elution or chromatographic separations are used for substances adsorbed on a bed of adsorbent. These principles are considered along with application of theoretical plate methods to fixed-bed operations. (J.R.D.)

21994 CEA-tr-A-608
ECHANGE D'IONS PAR FOCALISATION. I. PRINCIPES ET THEORIE. (Ion Exchange by Focusing. I. Principle and Simple Theory). E. Schumacher. Translated into French Helv. Chim. Acta 40, 221-7 (1957). 13p.

In a study of ion exchange apparatus a new technique for resin columns was found. A cation which takes part in the exchange equilibrium is not stopped in a stationary manner when an electric field is applied to the column. The theory and parameters of the technique are discussed, and applications in rapid analyses of cations, separation of trace radioelements, studies of kinetics of complex formation, separation of rare earths, and perhaps, isotope separation are given. (T.R.H.)

11124 √ AEC-tr-2910
ION EXCHANGERS IN ANALYTICAL CHEMISTRY. Developments During The Last Year. U. Schindewolf. Translated by K. S. Bevis from Angew. Chem. 69, 226-36 (1957). 27p.

The efficiency of ion-exchange processes determined by the equilibrium constants of ions which are affected by various factors such as the elutriant and complexing agent is discussed. An extensive table which correlates numerous separation methods for inorganic ions with literature references is given, and several special applications of ion exchangers are described. (tr-auth).

A LABELED ATOM STUDY OF THE INVERSION OF ADSORPTION SERIES IN ULTRA-MICROCONCENTRATION RANGE. S. Yu. Elovich and L. G. Kuz'mina (Moscow Inst. of Physical Chemistry). Kolloid. Zhur. 18, 268-75(1956) May-June. (In Russian)

A chromatographic separation of ions on organic ion exchangers may be followed by inversion of adsorption series.

ION EXCHANGE 6.12

THEORY 6.12.04

Adsorption statistics and dynamics were studied with Cs^{137}, Sr^{89}, and Ce^{141} radioisotopes and HCl solutions of various concentrations. The theory of the process has been developed and generalizations were shown to be in agreement with experimental facts. (tr-auth)

THE APPLICATION OF SOME ION EXCHANGE THEORIES TO THE ADSORPTION OF TRACE ELEMENTS. Herman Schuyler Gile. June 1955. Decl. Feb. 20, 1956.

A thesis submitted to Oregon State College in partial fulfillment of the requirements for the degree of Master of Science.

Breakthrough curves and distribution ratios were obtained experimentally for the adsorption of Pu^{4+}, Cs, Sr, and Ce as trace elements from a $UO_2(NO_3)-HNO_3$ matrix system onto Dowex-50 resin at several column lengths and flow rates. The exchange zone method of Michaels, the plate theory of Hermann, Mayer, and Thompkins, and the kinetic diffusion design theory of Vermuelen and Hiester which were applied to the data obtained involve an equilibrium parameter which is the distribution ratio and a kinetic parameter which is related to the height of a transfer unit. The parameters were calculated for each of the breakthrough curves. All 3 theories gave similar results for a given case; differences which were noted seemed to be related to the magnitude of the distribution ratio. The height of a transfer unit was independent of the flow rate when the distribution ratio was moderate; when the distribution ratio was large, the height of a transfer unit was dependent on flow rate and in most cases the resin was not completely utilized. 22 references.

MASS TRANSFER RATES IN ION EXCHANGE. W. A. Selke, Y. Bard, A. D. Pasternak, and S. K. Aditya (Columbia Univ., New York). A.I.Ch.E. Journal 2, 468-70(1956) Dec.

The rates of both the liquid-phase mass transfer and the internal-diffusion steps in ion exchange were studied by means of shallow-bed experiments. The mass transfer coefficients obtained fitted the general correlations for other packed-bed operations when the Schmidt group was evaluated with experimentally determined ionic counterdiffusivities. An incremental calculation of the diffusion rates within the particles yielded a value of the counterdiffusivity in the resin phase. A general design procedure based on these findings is proposed. (auth)

4375

SURFACE CHEMISTRY AND CATALYSIS. L. H. Reyerson (Univ. of Minnesota, Minneapolis). Ann. Rev. Phys. Chem. 7, 383-402(1956).

Physical adsorption, chemisorption, and surface catalysis are the subjects included in this review. (T.R.H.)

8653 CEA-tr-A-255

CINÉTIQUE DE L'ÉCHANGE D'IONS. (Kinetics of Ion Exchange.) F. Helfferich. Translated into French by B. Moreau from Angew. Chem. 68, 693-8(1956). 25p.

The diffusion of ions to the interior of an exchanger or across a film of adherent liquid determines the velocity of ion exchange. The application of Fick's first law leads to a complete theory of isotopic exchange which is confirmed by experiment. For the exchange of ions of different mobilities or valence, it is still difficult from the mathematical viewpoint to consider the diffusion potential which is formed. In addition, the effect of the activity and swelling coefficients can be determined only qualitatively. A decisive role on the velocity of exchange is played by the coefficients of self-diffusion in the exchanger. The experimental results, the dependence which exists between the valences, the size of the ions, the structure and concentration in solid ions of the exchanger, and the concentration of the contacting solution are discussed. 28 references. (tr-auth)

17012 NP-tr-147

THEORY OF ION EXCHANGE DYNAMICS. IV. EQUATION OF MOTION OF A STEADY FRONT. V. V. Rachinskii. Translated by M. C. Wheeler (U.K.A.E.A. Atomic Energy Research Establishment) from Zhur. Fiz. Khim. 31, 444-51(1957). 14p.

The theory of the dynamics and kinetics of ion exchange with a regime of parallel transfer of a sorption wave front is considered. The steady front motion equation was tested by the use of a radiochromatographic method and found to be in agreement with the experimental results. (J.R.D.)

ION EXCHANGE 6.12

THEORY 6.12.04

8309
THEORY OF ION EXCHANGE DYNAMICS. DYNAMICS AND KINETICS OF ION EXCHANGE FOR THE CASE OF LINEAR ISOTHERM. V. V. Rachinskiĭ, and O. M. Todes (Timiryazev Agricultural Academy). Zhur. Fiz. Khim 30, 407-16(1956) Feb. (In Russian)

An asymptotic solution has been obtained for the differential equation of ion exchange. The theory has been proved experimentally with the aid of the isotope method. It has been confirmed that the center of the sorption wave front moves with constant speed and that the front spreads out proportionally to $\sqrt{t}$. The values were checked on the rate constant of ionic sorption of labeled ions of Na and Rb on the swelling resin. The constants are of the order 1 sec^{-1}. (tr-auth)

STUDIES OF HIGH POLYMERS BY RADIOACTIVE METHOD. I. SELF-DIFFUSION OF IONS IN ION EXCHANGERS. Shintaro Sugai and Jiro Furuichi (Hokkaido Univ., Sapporo, Japan). J. Phys. Soc. Japan 10, 1032-40(1955) Dec.

In order to study kinetic exchange reaction and self-diffusion of ions in ion exchangers, the rate of ion exchange was measured by a radioactive tracer method similar to that employed by A. Langer and Pitts' general theory of radioactive ion exchange was applied, with attention given to the rate determining factor of reaction in each case, to analyze the results. When concentrated external solution is utilized, diffusion of cation in cation exchangers and diffusion of anion in anion exchangers was suggested to be the controlling factor, whereas in the case of the use of dilute external solution, the rate of exchange is probably controlled by surface diffusion. In the former case, diffusion coefficients, activation energy of diffusion, and activation entropy of diffusion were calculated, and from the results obtained, the mechanism of diffusion in the exchange resins was discussed. (auth)

ION TRANSFER (MEMBRANES) 6.13

See also, this section,

SPECIAL PROCESSES--Excer (6.17.08),
Flurex (6.17.10).

3254
STUDY ON GENERAL CHARACTERISTICS AND DISTRIBUTION OF ION CONCENTRATION IN ION EXCHANGE-ELECTROLYTIC EQUIPMENT. J. Shimokawa and G. Nishio (Japan Atomic Research Inst., Tokyo). J. Atomic Energy Soc. Japan 1, 225-33(1959). (In Japanese)

An electrolytic deionization apparatus in which spherical ion exchangers and permselective ion exchange membranes were included was made for the treatment of radioactive aqueous wastes. The general characteristics of the ion exchange-electrolytic equipment are discussed in comparison with the ion exchange column and the ion exchange membrane equipment. Equations for the distribution of ion concentrations in the deionization cell of the apparatus were derived as a function of feed concentration, flow rate, electric voltage, current, current efficiency, equivalent conductivities of ion exchangers and solutions, volume fraction of particles, and cell geometry. Radioactive Na^{24} tracers were utilized to measure actual ion concentrations in the deionization cell. The experimental data for low solid wastes indicate that the electrolytic process incorporating resin particles and resin membranes is superior to the simple process using permselective membranes only. (auth)

11648 ORNL-1812
Oak Ridge National Lab., Tenn.
THE ELECTROCHEMICAL UTILIZATION OF ION EXCHANGE MEMBRANES IN AEC OPERATIONS. E. J. Parsi. Jan. 27, 1955. Decl. Feb. 23, 1957. 34p. Contract W-7405-eng-26. $0.35(OTS).

The feasibility of concentrating acids and bases, of preparing acid-deficient salt solutions, and of preparing

ION TRANSFER (MEMBRANES) 6.13

acids and bases by the electrochemical utilization of ion-exchange membranes was demonstrated. The concentration of hydrofluoric acid by electrodialysis techniques was shown to be a simple, compact, and inexpensive operation. Monobasic aluminum nitrate was prepared economically and continuously in an electrolytic cell which used ion-exchange membranes. Purified alkali hydroxide was obtained from its sulfate salt in a similar electrodialysis-membrane cell. Separation of alkali metals in an ionic fractionation still was shown to be possible, but separation factors were small. Separation of hydrogen and deuterium by electromigration through a palladium diaphragm was investigated briefly. (auth)

1829

ON THE TRANSPORT NUMBER FOR ION-EXCHANGE RESIN MEMBRANES. Yoshio Oda and Tadashi Yawataya (Research Lab., Asahi Glass Co., Ltd., Benten-cho Tsurumi-ku, Yokohama). Bull. Chem. Soc. Japan 29, 673-9(1956)Aug.

The cation transport numbers through cation-exchange resin membranes were determined experimentally by two methods, first, computation from membrane potential measurement, and second from analytical tracing of the concentration change which takes place, on current passage, in the solution separated by the membrane. The former, which is considered as "Hittorf transport number", is found to be far smaller than the latter, the true transport number. The difference between the two sorts of transport number was accounted for on the basis of the concept of convective conduction within the pores of ion-exchange resin. The electroosmotic water transference through the membrane was discussed on comparing the results of its volumetric- and gravimetric measurements. (auth)

14673 A/CONF.15/P/502
Ionics, Inc., Cambridge, Mass.
APPLICATION OF ION TRANSFER MEMBRANES IN NUCLEAR CHEMICAL PROCESSING. E. A. Mason and E. J. Parsi. 12p. $0.50(OTS).

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

A broad review of the properties of membranes and their variety of potential applications in nuclear chemical processing is presented. The properties of ion transfer membranes which are important in determining the selection and application of membranes to various processes are summarized, and a series of brief descriptions (including data) of applications in nuclear processing which have already been subject to investigation are presented. In ion transfer membranes, ion "exchange" in the more conventional sense does not occur, and so chemical regeneration is not required. Descriptions of chemical structures along with data on the electrical resistance, transport efficiencies, and water transfer are included. Stabilities of various membrane types in a variety of chemical solutions found in the nuclear industry and to beta and gamma irradiation are discussed. Examples of applications are: (1) in the recovery and concentration of hydrofluoric acid from a waste stream, (2) concentration of lithium and sodium hydroxide solutions, (3) conversion of lithium and sodium chlorides and sulfates to the corresponding hydroxides, (4) preparation of acid deficient solutions of aluminum, thorium, and uranium nitrates, (5) separation of light and heavy water and of potassium and ammonium ions (as typical of the separation of like cations), (6) removal of nitric acid, and aluminum nitrate from radioactive waste solutions, (7) and in the electrolytic reduction of uranyl solutions in the production of uranium tetrafluoride. (auth)

PRECIPITATION 6.14

See also, under FISSION PRODUCT, POISON, AND RADIOISOTOPE REMOVAL, PRECIPITATION (2.37),

and,

under WASTE TREATMENT AND DISPOSAL, PRECIPITATION (8.05.07).

731

SLOW PRECIPITATION PROCESSES. APPLICATION OF PRECIPITATION FROM HOMOGENEOUS SOLUTION TO LIQUID-SOLID DISTRIBUTION STUDIES. Louis Gordon (Syracuse Univ., N. Y.). Anal. Chem. 27, 1704-7(1955) Nov.

PRECIPITATION 6.14

The direct addition of a precipitant to a solution results temporarily in a heterogeneity of conditions. In the vicinity where the precipitant has been introduced, the formation of the solid phase takes place under conditions such that the solution concentrations vary between very wide limits. Therefore, results of coprecipitation studies obtained with conventional precipitation procedures may also vary markedly. Precipitation from homogeneous solution offers an ideal technique for controlling the rate and mode of addition of a precipitant. It permits a slow precipitation process, which allows near equilibrium to be established between the surface of the solid and the solution. It is thereby possible to determine the nature and extent of coprecipitation. Applications of this technique are described in which Doerner-Hoskins' distribution coefficients have been obtained for systems containing barium-radium mixtures. Other coprecipitation studies are also described, particularly some which have revealed that the extent of coprecipitation is negligible except during the initial and final stages of the precipitation process. (auth)

12521 CEA-tr-R-626
SUR LES MÉTHODES PERMETTANT D'ÉTABLIR LE MÉCANISME DE LA COPRÉCIPITATION DES RADIOÉLEMENTS AVEC DES SELS PEU SOLUBLES. (The Methods Permitting the Establishment of the Mechanism of The Coprecipitation of Radioelements with Slightly Soluble Salts.) A. P. Ratner. Translated into French from Zhur. Obshchei Khim. 26, 949-51(1956). 10p.

This paper was previously abstracted from the original language and appears in NSA, Vol. 11, as abstract No. 98.

2472
CO-CRYSTALLIZATION OF RADIOACTIVE SUBSTANCES WITH VARIOUS PRECIPITANTS. V. I. Grebenshchikova. Khim. Nauka i Prom. 4, 456-64(1959). (In Russian)

Data on co-crystallization of small amounts of radioactive substances with precipitant are analyzed and the equilibrium between solid and liquid phases is discussed. (R.V.J.)

PRECIPITATION OF METAL FROM SALT SOLUTION BY REDUCTION WITH HYDROGEN. F. A. Schaufelberger (Ciba States Ltd., New York). J. Metals 8, 695-704(1956) May.

Early work on chemical precipitation of metals from metal salt solutions is reviewed. The chemistry and thermodynamics of precipitating copper, nickel, cobalt, and cadmium metals by reaction with hydrogen are discussed. Mechanisms of metal precipitation, nucleation, growth, and agglomeration are reviewed, as well as some solubility phenomena of gases and solids at elevated temperatures. Experimental data presented deal mainly with the reaction of copper sulfate solution with H_2. (auth)

10407 AEC-tr-3271
THE SEPARATION PROCEDURE IN A SINGLE DROP. Herbert Weiss. Translated by Lydia Venters (Argonne National Lab.) from Mikrochim. Acta 140-7(1954). 10p.

A new simple method is given for separating ions or groups of ions in a single drop. Two pieces of equipment have been developed for this purpose. One serves for simple precipitations on filter paper with gaseous reagents. An apparatus, designated as a ring furnace, is designed to wash the soluble materials from a spot and to concentrate them in a sharply bounded circular zone, where they can be detected. The usefulness of the procedure is demonstrated by typical examples. (auth)

6544 A/CONF.15/P/2413
THREE-DIMENSIONAL PRESENTATION OF PRECIPITATION OF URANYL ION IN AQUEOUS SYSTEM OF SODIUM CARBONATE AND BARIUM CHLORIDE. B. Težak, H. Füredi, and M. Branica (Inst. Ruder Boškovic, Zagreb and Univ. of Zagreb). 12p.

Equal volumes of sodium carbonate and barium chloride solutions of varying concentrations were mixed. The systems were kept at 25°C for 24 hours and then filtered. The quantity of precipitated uranyl ion was determined polarographically. Electron microscope photographs illustrate the appearance of the complex precipitate. (W.D.M.)

RADIATION EFFECTS 6.15

AQUEOUS SOLUTIONS 6.15.01

See also, this section,

SOLVENT EXTRACTION--Radiation Damage to Solvents (6.16.03).

ALPHA 6.15.01.02

22088
RADIATION CHEMICAL REACTIONS IN NITRATE SOLUTIONS. N. A. Bakh, V. I. Medvedoskii, A. A. Revina, and B. D. Bitiukov (Inst. of Physical Chemistry Academy of Sciences, U.S.S.R.). p. 39-43 of "Radiation Chemistry of Aqueous Solutions (Inorganic and Organic Systems)."

Results are reported from an investigation of the products formed by the radiolysis of nitrate solutions. The hydrogen peroxide and oxygen formed were determined polarographically. A comparison was made of the total amounts of radiation oxidation and reduction products. The investigations showed that in oxygen-free, neutral aqueous nitrate solutions the radiation reactions gave only nitrite, hydrogen, hydrogen peroxide, and oxygen, and that the ratio of the yields of these products depended on the concentration of the solution and dose strength. By choosing suitable concentrations, the evolution of molecular hydrogen was eliminated and under these conditions the formation of nitrate balanced that of hydrogen peroxide and oxygen. (C.H.)

RADIOLYSIS OF WATER BY PARTICLES OF HIGH LINEAR ENERGY TRANSFER. THE PRIMARY CHEMICAL YIELDS IN AQUEOUS ACID SOLUTIONS OF FERROUS SULFATE, AND IN MIXTURES OF THALLOUS AND CERIC IONS. Marc Lefort and Xavier Tarrago (Faculté des Sciences, Paris). J. Phys. Chem. 63, 833-6(1959) June.

In order to measure the primary chemical yields of water radiolysis, mixtures of thallous and ceric ions in aqueous 0.8 N H_2SO_4 solutions were irradiated by plutonium α particles. Absolute dosimetry measurements were carried out. A G-value of 5.5 ± 0.1 was found for ferrous sulfate oxidation. From this value and from results obtained on the ceric system, the following values were determined: $G_{OH} = 0.50$, $G_{HO_2} = 0.11$, $G_{H_2O_2} = 1.45$, $G_H = 0.60$ $G_{H_2} = 1.57$, and $G_{H_2O} = 3.62$. However, these yields depend on the concentration of thallous ions which inhibit the intratrack reaction $H_2O_2 + OH \rightarrow H_2O + HO_2$. An explanation also is given for the low yield (3.6) obtained for the oxidation of ferrous sulfate in deaerated solutions. (auth)

9207
THE ACTION OF α-PARTICLES ON SOLUTIONS CONTAINING FERROUS AND CUPRIC IONS. D. M. Donaldson and N. Miller (Edinburgh Univ.). Trans. Faraday Soc. 52, 652-9(1956) May.

6452 A/CONF.15/P/1232
DETERMINATION DES RENDEMENTS DE RADIOLYSE DE L'EAU PAR LES RAYONS α, A L'AIDE DU SYSTEME $V^{V}/^{IV}$. (Determination of Yields in the Radiolysis of Water by α-Rays by Means of the V^{V}/V^{IV} System.) J. Pucheault and C. Ferradini (Univ. of Paris). 15p.

Under irradiation by Po^{210} α particles, the relative concentrations of VO_2^+ and of VO^{2+} in acid solution (0.8 N H_2SO_4) tend to concentrations corresponding to a stationary state between the oxidized and reduced forms. The initial rates of reduction or oxidation in absence of air, the rate of formation of peroxide VO_3^+, and the rate of appearance of gaseous products lead to the assignment of different values to the radical and molecular yields for the radiolysis of water by α rays. There is no particular agreement between these values and those calculated from the results for other solutes which, as a rule, do not agree among themselves. These disagreements have led to the hypothesis that, there should exist a well delineated region in which the following back-reactions take place: (a) $OH + H_2O_2 \rightarrow HO_2 + H_2O$; (b) $OH + H_2 \rightarrow H + H_2O$; and (c) $H + H_2O_2 \rightarrow OH + H_2O$. Upon this basis, one is able to explain why the radical and molecular yields depend on whether the system in solution, under the given experimental conditions, is a good scavenger for OH radicals (S_{OH}), H atoms (S_H) or both ($S_{OH,H}$). The G_a, G_b, G_c amounts which modify the yields are calculated by comparing various results with those obtained for vanadium. Using those G values, the calculated oxidation or reduction yields are found to be in agreement with the results concerning several solute systems (vanadium salts, ceric sulfate in presence and in absence of air, and ferrous sulfate in presence of air at various intensities, in absence of air, and in presence of copper). The maximum calculated yields for the decomposition of

RADIATION EFFECTS 6.15

AQUEOUS SOLUTIONS 6.15.01

Alpha 6.15.01.01

water is $G_{-H_2O} = 4.1$ (for $S_{OH,H}$). An attempt is made to give a physical interpretation of the origins of the suggested second region; the limitations of the theoretical treatment are discussed. (auth)

GAMMA AND X 6.15.01.02

6471 A/CONF.15/P/1617
ON THE MECHANISM OF OXIDATION OF AERATED FERROUS SULPHATE SOLUTIONS BY GAMMA RAYS. Yaacov Gilat and Gabriel Stein (Hebrew Univ., Jerusalem). 4p.

No agreement exists regarding the exact mechanism and relative importance of the elementary steps in the radiation-chemical oxidation of ferrous ions in aerated solutions. The dependence of the reaction of pH may furnish important information in this respect. Observations for the case where the pH was varied and the ionic strength was held constant and for the case where acid concentration was held constant and the ionic strength was varied are reported. (W.D.M.)

6469 A/CONF.15/P/1517
PRIMARY PRODUCTS IN THE IRRADIATION OF AQUEOUS SOLUTIONS WITH X- OR γ-RAYS. E. Hayon and J. Weiss (Univ. of Durham, Newcastle-upon-Tyne, Eng.), D. Armstrong (Univ. of Saskatchewan, Saskatoon, Can.), E. Collinson and F. S. Dainton (Univ. of Leeds, Eng.), and D. M. Donaldson and N. Miller (Edinburgh Univ.). 30p.

The aim of radiation chemical studies is to identify and to measure the yield of the various primary products which are formed under varying conditions. The results of indirect action on various solutes in dilute aqueous solution are presented and discussed. Solutes considered are single organic solutes (mono- and trichloroacetic acids), polymerizable monomer (acrylamide), formic acid, ferric and cupric ions, and oxygen. The results are broadly consistent with the diffusion theory of Magee and co-workers. (W.D.M.)

HYDROGEN PEROXIDE YIELD IN WATER IRRADIATED WITH X RAYS. J. Bednář and J. Teplý (Militärtechnische Akademie, Brno.). Collection Czechoslov. Chem. Communs. 24, 127-34(1959) Jan. (In German)

The hydrogen peroxide yields obtained by the action of 150-kev x radiation on air-saturated solutions of KBr were determined. The yields of the reactions $2\ H_2O \rightarrow H_2O_2 + H_2$ and $2\ H_2O \rightarrow H_2O_2 + 2\ H$ are strongly pH dependent. A hypothesis on the various mechanisms for the formation of hydrogen peroxide by these reactions is presented. (tr-auth)

12292 ✓
HYDROGEN FORMATION IN CONCENTRATED AQUEOUS SODIUM SULFATE SOLUTIONS BY COBALT-60 GAMMA-RAY RADIATION. H. A. Mahlman (OakRidge National Lab., Tenn.). J. Chem. Phys. 28, 1256-7 (1958) June.

The hydrogen yields is degassed concentrated $NaSO_4$ solutions from gamma irradiation with a 900-c Co^{60} source are tabulated for various molarities, pH values, and dose ranges. It is concluded that the total energy absorbed by the solution was effective in decomposing the water and that the bisulfate and sulfate ions are not effective hydrogen atom scavengers. (L.T.W.)

13030
MEASUREMENT OF HYDROGEN PEROXIDE FORMATION BY UV AND IONIZING RADIATION IN WATER SOLUTIONS. K. Damaschke (Bundesanstalt für Materialprüfung, Berlin-Dahlem). Z. Naturforsch. 13b, 322-6(1958) May. (In German)

The formation of H_2O_2 by UV and ionizing radiation under various experimental conditions was followed by means of the electrochemical determination of H_2O_2. The method of anode measurement permits 2×10^{-8} mole/l of H_2O_2 to be detected and recorded continuously. Zinc oxide was used for the absorption of the UV radiation. The quantity of H_2O_2 formed is directly proportional to the ZnO concentration. A linear dependence between the H_2O_2 produced and the irradiation time as well as the radiation intensity was found. With x radiation, the quantity of H_2O_2 formed at constant voltage is proportional to the irradiation dosage (max time). The formation rate of H_2O_2 up to 2000 μ g/l shows a linear

RADIATION EFFECTS 6.15

AQUEOUS SOLUTIONS 6.15.01

GAMMA AND X 6.15.01.02

dependence on the O_2 concentration. Co^{60} and Ir^{192}, used as gamma radiation sources, show a proportionality between the peroxide formed and the exposure time on the one hand and the radioactivity of the source used on the other. For the formation of H_2O_2 in water solutions, the secondary electrons formed or the photoelectrons are the determinant as they are in the radiation of solutions containing ZnO. An investigation was also made using Sr^{90} as a beta source and it showed that here also peroxide was formed in small quantities. (tr-auth)

14662 A/CONF.15/P/952
Argonne National Lab., Lemont, Ill.
RADIATION DECOMPOSITION OF WATER UNDER STATIC AND BUBBLING CONDITIONS. Sheffield Gordon and Edwin J. Hart. 12p. $0.50(OTS).
Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

Operation of Borax III (Argonne Experimental Boiling Water Reactor) reveals much higher water decomposition rates than are found in non-boiling water moderated reactors. Hydrogen and oxygen in low concentrations appear in the effluent steam. Normally, in water moderated reactors, decomposition of water is not a serious problem since there is a large reserve γ-ray flux available for promoting the recombination reaction: $H_2 + H_2O_2 = 2H_2O$. But during shaking or boiling, the hydrogen escapes from the aqueous phase and the radiation induced decomposition of hydrogen peroxide leads to oxygen evolution: $2H_2O_2 = 2H_2O + O_2$. The kinetics of non-boiling irradiated water has been studied and reported in the extensive investigations of Allen and co-workers. Borax III experiments show, however, that the effectiveness of the recombination reaction is reduced appreciably during boiling. (auth)

4342
CHEMICAL ACTION OF IONISING RADIATIONS IN SOLUTION. PART XIX. SOME ASPECTS OF THE REDUCTION OF METHYLENE-BLUE BY X-RAYS IN AQUEOUS SYSTEMS. E. Hayon, G. Scholes, and J. Weiss (Univ. of Durham, Newcastle upon Tyne, 1). J. Chem. Soc. 301-11 (1957) Jan.

4347
DIRECT AND INDIRECT ACTION OF IONIZING RADIATIONS ON AQUEOUS SOLUTIONS. I. STUDY OF THE DIRECT ACTION ON PERCHLORIC ACID. Maurice Cottin. J. chim. phys. 53, 903-16(1956) Nov.-Dec. (In French)

The chemical modifications induced in aqueous solutions by ionizing radiations are due either to a direct action in which the changed molecule is that which directly absorbs the energy or to an indirect action in which the molecule is changed by the action of the free radicals formed in the solvent. The direct action was studied by Co γ irradiation in concentrated solutions of perchloric acid or sodium perchlorate. The perchlorate ions are decomposed, as much in the absence of air, with the formation of chlorides and chlorates. The yields of these two products are proportional to the concentration of the perchloric solutions and independent of the nature of the physical state of the solution and of the presence of dissolved oxygen. A mechanism is proposed to explain the observed phenomena. The energy utilized to decompose a perchlorate ion is evaluated at about 25 ev. (tr-auth)

X-RAY INDUCED $Tl^{+++}-Tl^{+}$ EXCHANGE IN 0.8 N H_2SO_4. George E. Challenger and Burton J. Masters (Los Alamos Scientific Lab., N. Mex.). J. Am. Chem. Soc. 78, 3012-18 (1956) July 5.

The $Tl^{+}-Tl^{3+}$ isotopic exchange reaction in 0.8 N H_2SO_4 solutions undergoing irradiation with 50 kvp x-rays was studied. Throughout the range of the measurements, the radiation-induced exchange rate was found to be independent of thallous concentration, 0.503-order dependent upon thallic concentration, and 0.670-order dependent upon irradiation intensity. The results are explained in terms of a proposed mechanism involving the formation of the intermediate species Tl^{2+} at an appreciable steady-state concentration in the irradiated solutions. The Tl^{2+} intermediate is presumed to exchange rapidly with Tl^{+} and somewhat less readily with Tl^{3+}. In the absence of radiation, ferrocyanide ion was found to induce an exchange in $Tl^{+}-Tl^{3+}$ solutions, indicating

RADIATION EFFECTS

9208
EFFECT OF γ RADIATION ON THE EXCHANGE BETWEEN TRIVALENT AND HEXAVALENT CHROMIUM IONS IN AQUEOUS ACID SOLUTIONS. Marc Lefort and Michel Lederer. Compt. rend. 242, 2458-61(1956) May 14. (In French)

RADIATION EFFECTS 6.15

AQUEOUS SOLUTIONS 6.15.01

GAMMA AND X 6.15.01.02

The use of Cr^{51} has allowed proof of a γ induced exchange between the III and VI valences of Cr. Proof is thus given of an oxidation reaction of the Cr^{III} ions by the OH radicals arising from the radiolysis of H_2O. This reaction only partially offsets with the reduction of the Cr^{VI} ions observed as a total phenomenon. (tr-auth)

10072
ON THE OXIDATION OF FERROUS IRON IN SULFURIC ACID SOLUTION UNDER OXYGEN PRESSURE BY Co^{60} γ RADIATION. H. Le Bail and J. Sutton. J. chim. phys. 53, 430-2(1956) May. (In French)

Solutions of Mohr salts 2×10^{-2}M in 5N sulfuric acid were irradiated with Co^{60} γ rays under oxygen pressures of 6 and 14 atmospheres. The yield, G, the number of ferric ions produced by 100 ev absorbed in the solutions does not differ from that found for 10^{-3}M Mohr salts solutions in 0.8N sulfuric acid in equilibrium with air, but in the presence of benzene or metals, the yield G increases in variable proportions. (tr-auth)

1274
PEROXIDE YIELD IN THE γ-IRRADIATION OF AIR-SATURATED WATER. Augustine O. Allen and Richard A. Holroyd (Brookhaven National Lab., Upton, N. Y.). J. Am. Chem. Soc. 77, 5852-5(1955) Nov. 20.

The peroxide yield decreases in air-saturated γ-irradiated water on the addition of Br^- ion at pH = 2 and 5. These results confirm previous investigations by Sworski (J. Am. Chem. Soc. 76 4687(1954)). (C.W.H.)

22093
THE EFFECT OF LOW ENERGY (LESS THAN 0.7 Mev) PROTONS ON AQUEOUS SOLUTIONS. II. DECOMPOSITION OF OXALIC ACID. Ivan Draganich and Miomir Simich (Boris Kidrich Inst. of Nuclear Studies, Belgrade). p.63-4 of "Radiation Chemistry of Aqueous Solutions (Inorganic Systems)."

In connection with our systematic study of the behavior of aqueous oxalic acid solutions under the action of Co^{60} γ radiation and the radiation of a nuclear reactor, we were also interested in the behavior of this system under the action of low-energy protons. Data are presented on the effect of the number of protons striking the solution, their energy, and the intensity of the proton beam on the radiolysis yield. (auth)

22099
THE EFFECT OF X-RADIATION ON DILUTE AQUEOUS SOLUTIONS OF ORGANIC MATERIALS. V. I. Duzhenkov and P. I. Dolin (Physical Chemistry Inst., Academy of Sciences, U.S.S.R.). p. 87-90 of "Radiation Chemistry of Aqueous Solutions (Inorganic and Organic Systems)."

The effect of a series of organic substances on the yield of molecular products from water radiolysis was investigated. Results are reported from studies on benzene, heptane, caproic acid, acetic acid, acetone, hexyl alcohol, and ethyl alcohol. Emphasis was placed on the effect of the various additives on hydrogen evolution. From an examination of the data it was concluded that for substances in the same class of organic compound the efficiency of their interaction with OH, HO_2, and H radicals increases as the length of the carbon atom chain increases. Caproic acid reacted with radicals more efficiently than acetic. The same relationship was found between hexyl and ethyl alcohols. With the same chain length, hydrocarbons were found to have the lowest reactivity, acids greater, and alcohols even greater reactivity. (C.H.)

6465 A/CONF.15/P/1422
ACCELERATION DES REACTIONS RADICALAIRES SOUS L'EFFET DES RADIATIONS IONISANTES. I. SYNTHESE DE L'ACIDE CYANHYDRIQUE A PARTIR DU METHANE ET DE L'AMMONIAC. (The Acceleration of Radical Reactions under the Action of Radiation). R. Mihail and J. Herscovici (Inst. de Recherches Chimiques, Bucharest). 17p.

The effect of ionizing radiations from Co^{60} on the reaction between methane and ammonia was studied between 500 and 950°C in the presence of a platinum catalyst. The reaction leads to the formation of HCN by a radical mechanism in the course of which the methyl and hydrazine radicals appear. By increasing the concentration of the active components, ionizing radiation increases the conversion of ammonia to HCN. (tr-auth)

RADIATION EFFECTS 6.15

AQUEOUS SOLUTIONS 6.15.01

BETA (ELECTRONS) 6.15.01.03

6481 A/CONF.15/P/2020
ABSORPTION OF BETA PARTICLES IN SOLUTIONS. A. K. Hilmi and S. Abid Husain (Bagdad Pact Nuclear Centre). 6p.

The absorption of beta particles in three groups of different solutions, namely, acetates, chlorides, and nitrates was studied. The dependence of beta absorption on atomic number was found to apply to these solutions in the same way as it applied to pure elements and organic liquids. Identical absorption curves were obtained when plotted in terms of $Z^{4/3}/A$. The principle was used to determine the concentration of barium in barium acetate and of lead in lead acetate and lead nitrate. (auth)

12294
REDUCED YIELD IN DILUTE SOLUTIONS IRRADIATED AT VERY HIGH DOSE RATES. Franklin Hutchinson (Yale Univ., New Haven). Radiation Research 9, 13-23 (1958) July.

The irradiation of dilute methylene blue solutions with a high-intensity electron beam producing 10^4 r/μsec pulse gives less radiation-induced decolorization than the same dose given at lower rates. This decrease, which does not appear to have been previously observed, is to be expected from the mechanism involving H and OH radicals usually used to explain the effects of ionizing radiations on dilute solutions. On the basis of this mechanism a simple theory of the rate dependence is derived. From a comparison of theory and experiment it is concluded that the OH radical is extremely likely to react when it collides with a methylene blue molecule, the observed reaction rate being somewhere between 25 and 100% of the maximum possible if a reaction took place on every encounter. (auth)

5240
THE RADIATION CHEMISTRY OF FERROUS CHLORIDE SOLUTIONS. Harold A. Schwarz (Brookhaven National Lab., Upton, N. Y.). J. Am. Chem. Soc. 79, 534-6(1957) Feb. 5.

The radiation induced oxidation of Fe^{2+} in air-saturated 0.4 M HCl differs from the oxidation in 0.4 M H_2SO_4 in that the Fe^{3+} production is not linear with dose. The initial yield is essentially the same, $G_{(FeIII)}$ (ions oxidized per 100 e.v. absorbed) being 15.8, but the kinetics indicate a competition between Fe^{3+} and O_2 for H atoms. The effect of the competition is to decrease the yield, and the effect increases with increasing Cl^- concentration. The ratios of the rate constant for H atoms reacting with Fe^{3+} and O_2 at the various Cl^- concentrations have been separated into the ratios for the individual species present, giving $k_{Fe^{+++}}/k_{O_2} = 0.004$, $k_{FeCl^{++}}/k_{O_2} = 0.23$ and $k_{FeCl_2^+}/k_{O_2} = 0.48$. (auth)

9053 SCL-T-130
QUANTITATIVE STUDIES OF RADIO-CHEMICAL REACTIONS. III. OXIDATION OF FERROUS SULPHATE BY BETA PARTICLES. (Etudes Quantitatives des Reactions Radiochimiques. III. Oxydation du Sulfate Ferreux par les Particules Beta.) D. M. Donaldson and N. Miller. Translated by M. I. Weinreich from J. chim. phys. 52, 578(1955). 4p.

The effect of beta particles, emitted by dissolved phosphorus, on an 0.8N sulfuric acid solution of ferrous sulfate in equilibrium with the air was studied. The absolute yield of the ferrous ion oxidation was measured and found to be 15.4 ± 0.8 ferric ions per 100 ev absorbed. (J.S.R.)

6481 A/CONF.15/P/2020
ABSORPTION OF BETA PARTICLES IN SOLUTIONS. A. K. Hilmi and S. Abid Husain (Bagdad Pact Nuclear Centre). 6p.

The absorption of beta particles in three groups of different solutions, namely, acetates, chlorides, and nitrates was studied. The dependence of beta absorption on atomic number was found to apply to these solutions in the same way as it applied to pure elements and organic liquids. Identical absorption curves were obtained when plotted in terms of $Z^{4/3}/A$. The principle was used to determine the concentration of barium in barium acetate and of lead in lead acetate and lead nitrate.

THEORY 6.15.01.04

214
ACTIVITY CONCEPT IN RADIATION CHEMISTRY. H. A. Mahlman (Oak Ridge National Lab., Tenn.). J. Chem. Phys. 31, 993-5(1959) Oct.

RADIATION EFFECTS 6.15

AQUEOUS SOLUTIONS 6.15.01

THEORY 6.15.01.04

The decomposition of aqueous solutions by ionizing radiations has been interpreted by assuming an initial solvent decomposition into H atoms and OH radicals. Radical recombination gives rise to the observed molecular products H_2 and H_2O_2. Substantiating this hypothesis, it was demonstrated that reaction of solutes with the radicals depress the yields of the molecular products. The linear dependence of the molecular yields on the cube root of the solute molarity applies very well for dilute solutions of strong electrolytes, however, in more concentrated solutions considerable deviation is observed. It is in this region that the activity of an electrolyte manifests itself, and therefore the cube root of the activity is the criterion rather than the molarity. For this study particular attention was addressed to the depression of hydrogen yields by aqueous sodium nitrate solutions. Alteration of the molecular hydrogen yield was demonstrated by varying the scavenging solute concentration and the activity of the scavenging solute. The addition of three different salts representing uni-univalent, di-univalent, and tri-univalent electrolytes was used to alter the ionic strength of the solutions. (auth)

6482 A/CONF.15/P/2022
STUDIES IN THE RADIATION CHEMISTRY OF AQUEOUS SOLUTIONS. M. A. Proskurnin and Y. M. Kolotyrkin (L. Y. Karpov Inst. of Physical Chemistry, U.S.S.R.). 37p.

It is generally considered that the yields of radiation-chemical reactions in aqueous solutions are determined by the initial formation of approximately 4 pairs of radicals (H + OH) per 100 ev. New data are presented which indicate that it is possible to involve short-lived particles in reactions (radicals, ions, and excited molecules) whose role should not be overlooked and whose reaction capacity proves to be considerable. Studying the reaction of oxidation of Fe^{2+} in a vacuum and in an atmosphere of N_2O is a compelling reason for giving up the generally accepted Krenz–Dewhurst's scheme. T. Rigg's, G. Stein's, and J. Weiss' assumption regarding the oxidizing action of H_2^+ has not been confirmed in experiments on oxidation of Sn^{2+} and Ti^{3+} ions in a vacuum. The above contradictions are eliminated by the new assumptions. (auth)

22086
RADIATION CHEMISTRY OF AQUEOUS SOLUTIONS (INORGANIC AND ORGANIC SYSTEMS). Part II of Proceedings of the First All-Union Conference on Radiation Chemistry, Moscow, 1959. (In English Translation). New York, Consultants Bureau, Inc., 1959. 80p. $50.00.

Topics discussed include the effect of KBr and KCl concentrations on the yield of molecular products of radiolysis in aqueous solutions; radiation chemical reactions in nitrate solutions; the effect of ionizing radiation on concentrated aqueous solutions of inorganic salts; the formation and reactions of oxygen compounds of iron during radiolysis of aqueous solutions; radiation processes in solutions of tetravalent uranium; the effect of low-energy protons on aqueous solutions; the effect of accelerated electrons on $KMnO_4$ solutions; the effect of gamma radiation on colloidal solutions of cobalt, nickel, silver, and gold sulfides; simultaneous polarographic determination of oxygen and hydrogen peroxide during their formation by radiation; the kinetics of hydrogen peroxide decomposition by gamma radiation; the oxidation of aqueous benzene solutions under the effect of gamma radiation; the effect of x radiation on dilute aqueous solutions of organic materials; conjugated oxidation-reduction processes in the radiolysis of aqueous solutions; and the kinetics of the decoloration of an aqueous methylene blue solution under the effect of x rays. (C.H.)

CHEMICAL EFFECTS OF RADIATION. F. S. Dainton (Univ. of Leeds, Eng.). Radiation Research Suppl. No. 1, 1-25(1959).

The principal features of the radiation chemistry of aqueous systems are presented in chart form. While crudities and oversimplifications are obvious, the chart serves to focus attention on the major features. Included are a critical history of recent researches on the radiation chemistry of aqueous systems, chemical methods of determining G values of primary products a review of recent experimental results concerning the yields of the primary products and a discussion of the influence of the state of aggregation on results. 41 references. (C.H.)

RADIATION EFFECTS 6.15

AQUEOUS SOLUTIONS 6.15.01

THEORY 6.15.01.04

14660 A/CONF.15/P/915
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
RADIATION SENSITIVITY AND CHEMICAL STRUCTURE. D. R. Kalkwarf. 11p. $0.50(OTS).
Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.
The radiation sensitivity of a compound in aqueous solution is determined in many cases by its reactivity with the free radicals and other products formed by the irradiation of water. In order to examine the nature of radiation sensitivity, a method was devised for measuring relative reaction rates of compounds with the radicals formed in aerated water. Each compound was allowed to compete with the dye eriogiaucine for the radicals formed in the same aqueous solution during radiolysis. The ratio of the rate constants for these reactions was then calculated from the measured bleaching rate of the dye, assuming both competitive reactions were bimolecular. For a large number of compounds,

4343
CHEMICAL EVIDENCE OF TRACK EFFECTS IN THE RADIOLYSIS OF LIQUIDS. Adolphe Chapiro (Laboratoire de Chimie Physique de la Faculté des Sciences de Paris, France). Radiation Research 6, 11-26(1957) Jan.

EVOLUTION OF RADIATION CHEMISTRY. WATER AND AQUEOUS SOLUTIONS. Memoire No. 6. Maria do Carmo Anta. Lisbon, Junta de Energia Nuclear, 1957. 16p. (In French)
After a review of the principal works in radiation chemistry which led to the consideration that the radiolysis of water is an electrolysis without electrodes, the Fricke theory of "activated water" and the present theory on free radicals are discussed. The most recent theories are applied to the interpretation of the mechanism of the oxidation of ferrous sulfate in aqueous solutions. A detailed analysis is made of the utilization of this reaction for chemical dosimetry. (auth)

9204
EFFECT OF SOLUTE CONCENTRATION IN THE IRRADIATION OF AQUEOUS SOLUTIONS BY IONIZING RADIATIONS. G. R. A. Johnson, G. Scholes, and J. Weiss (King's Coll., Newcastle upon Tyne, England). Nature 177, 883-4 (1956) May 12.

United Kingdom Atomic Energy Authority. Research Group. Atomic Energy Research Establishment, Harwell, Berks, England.
SELECTED ABSTRACTS OF ATOMIC ENERGY PROJECT UNCLASSIFIED REPORT LITERATURE IN THE FIELD OF RADIATION CHEMISTRY and BIBLIOGRAPHY OF THE PUBLISHED LITERATURE. (Papers Noted to March 1956). PART 1. THEORY, INTERPRETATIONS, WATER AND AQUEOUS INORGANIC SYSTEMS. R. W. Clarke, comp. Jan. 1956. 98p. (AERE-C/R-1575(1)).
There is a total of 446 references to report and published literature. Author and subject indexes are included.

22089
SOME DATA ON THE EFFECT OF IONIZING RADIATION ON CONCENTRATED AQUEOUS SOLUTIONS OF INORGANIC SALTS. A. M. Kabakchi, V. A. Gramolin, and V. M. Erokhin. p.45-8 of "Radiation Chemistry of Aqueous Solutions (Inorganic and Organic Systems)."
Using concentrated Na, K, NH_4, Sr, Ba, Pb nitrate and Na, K, and Ba chloride solutions as examples, we established that the radiation yields of nitrite ion in the first cast and free chlorine in the second do not depend on the nature of the cation. The yield of radiation chemical reaction products in the above solutions increases noticeably with an increase in solute concentration. This increase is practically independent of the type of radiation. (auth)

6453 A/CONF.15/P/1233
HETEROGENEOUS CATALYSIS IN RADIATION CHEMISTRY. M. Haissinsky and M. Duflo (Univ. of Paris). 9p.
The effects of small quantities (3 to 5 mg/cm^3) of various semiconductors on the radiochemical transformations produced by Co^{60} γ rays or 20-kev x rays (tungsten target) in aqueous solution were studied. Several experiments were also carried out with platinum black and palladium black. The results of oxidation of uranous sulfate and potassium iodide, and the reduction of ferric o-phenanthroline, and the bleaching of methylene blue are discussed. (auth)

RADIATION EFFECTS 6.15

ON ELECTRONIC COMPONENTS 6.15.02

GENERAL 6.15.02.01

12186 NP-7365(Vol. 3)(Paper 22)
Convair, Fort Worth, Tex.
PREDICTING THE PERFORMANCE OF IRRADIATED ELECTRONICS SYSTEMS BY SIMULATION ON THE ANALOG COMPUTER. V. C. Brown and N. M. Peterson. Paper 22 [of] THIRD SEMI-ANNUAL RADIATION EFFECTS SYMPOSIUM [HELD IN ATLANTA], OCTOBER 28-30, 1958. VOLUME 3. AIRCRAFT SYSTEMS AND MATERIALS PAPERS. 15p. (FZM-1159).

The method of simulating electronic systems on analog computers for the purpose of studying the effects of radiation is described, and a comparison is made between the results obtained by the simulation technique and data obtained in an actual irradiation. The results show that, by combining proper statistical methods with computer simulation, the performance of electronics systems under radiation may be successfully predicted. (auth)

12196 NP-7365(Vol. 4)
Lockheed Nuclear Products, Marietta, Ga.
THIRD SEMI-ANNUAL RADIATION EFFECTS SYMPOSIUM [HELD IN ATLANTA], OCTOBER 28-30, 1958. VOLUME 4. ELECTRONICS AND SEMI-CONDUCTORS PAPERS. 311p. (NR-51(Vol. 4)).

Thirteen papers are presented on the radiation effects in semiconductors and electronic equipment. (W.D.M.)

EFFECT OF HIGH INTENSITY RADIATION ON ELECTRONIC PARTS AND MATERIALS. C. P. Lascaro and A. L. Long. Elec. Mf. 119-21, 306(1958) Sept.

The results of a study of the effect of neutron and gamma radiation on electronic components used in communication equipment are reported. (auth)

15676 AD-208788
Stevens Inst. of Tech., Hoboken, N. J.
UNCLASSIFIED LITERATURE SURVEY ON THE EFFECTS OF NUCLEAR RADIATION TO ELECTRON TUBE MATERIALS. Quarterly Report [for] May 31, 1958 to September 1, 1958. E. R. Johnson. 17p. SC Project No. -112B. Contract DA-36-039-SC-73146.

Glass tubes containing boron when irradiated with neutrons show a sharp rise in temperature attaining a maximum of 537°C. The rise in temperature is proportional to the boron content. Reduction of the cathode by CO liberated from the walls of glass tubing is discussed. Nonex glass electron tubes when irradiated for a total integrated dose of 10^{16} nvt show no cracks or fissures. (auth)

15675 AD-202691
Stevens Inst. of Tech., Hoboken, N. J.
UNCLASSIFIED LITERATURE SURVEY OF THE EFFECTS OF NUCLEAR RADIATION TO ELECTRON TUBE MATERIALS. Quarterly Report Period, March 1, 1958 to May 31, 1958. E. R. Johnson. 16p. DA Project No. 3-99-13-000. Contract DA-36-039-SC-73146.

Bombardment of glass surfaces with high energy radiation will release CO_2, the amount depending upon the mass of the glass exposed. It is reported that alumina ceramics show no structural changes after exposure to an integrated dose of 10^{20} nvt. Borosilicate glass containing 8% boron shows large cracks and fissures after exposure to 5×10^{15} nvt. (auth)

15157 A/CONF.15/P/802
General Atomic Div., General Dynamics Corp., San Diego, Calif.
ELECTRICAL EFFECTS OF HIGH-INTENSITY IONIZING RADIATION ON NONMETALS. Victor A. J. van Lint and P. H. Miller, Jr. 11p. $0.50(OTS).

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

Experiments were performed utilizing a 7-Mev electron linear accelerator. Ionization rates up to 5×10^8 r per sec during 10-μ pulses were achieved. In this environment typical resistors became shunted by 3×10^4 to 10^5 ohms, capacitors leaked their stored charge in 10 to 100 μ, and semiconductor junctions became shorted in the back direction for up to ten minority-carrier lifetimes unless the excess carriers were swept across the junctions by large currents. Theories are developed to explain the effects in resistors and semiconducting devices. Further detailed experiments are being performed. (auth)

RADIATION EFFECTS 6.15

ON ELECTRONIC COMPONENTS 6.15.02

GENERAL 6.15.02.01

7362 ✓
THE EFFECT OF RADIATION DAMAGE ON THE ELECTRONIC PROPERTIES OF SOLIDS. E. W. J. Mitchell (Univ. of Reading). Brit. J. Appl. Phys. 8, 179-89(1957) May.

An outline is given of the way in which the concentration of defects introduced by irradiation may be estimated. The topics which are discussed are the effects of irradiation on the electron mobility in metals, the carrier concentration in germanium and the optical absorption in quartz and diamond. The emphasis in the discussion of results is on their contribution to the study of defects in solids. (auth)

8215 ✓ NP-6322
Admiral Corp., Chicago.
THE EFFECTS OF NUCLEAR RADIATION ON ELECTRONIC COMPONENTS. PHASE 1. Scientific Report No. 7. Apr. 1, 1957. 507p. Contract AF33(616)-3091.

A variety of electronic components were tested before, during and after exposure to nuclear radiation. The irradiation facilities included the CP-5 reactor, where most of the irradiations were performed, the MTR, and a Co^{60} facility. The components, some of which were under normal operating conditions, were irradiated for several days. The radiation environment varied considerably, but typical values for the CP-5 were 2×10^6 r/hr for gammas, 5×10^9 epi-Cd neutrons/(cm^2sec) and 10^{12} thermal neutrons/(cm^2sec). Electron tubes tested consisted of the following: 2C40, 2K50, 3JP12, 371-B, 5703, 5719, and 6198. The 2C40 UHF triode showed some change in plate current in-pile. Five of the six active tubes survived post irradiation. The 2K50 thermally tuned klystrons failed in-pile; post irradiation tests indicated glass-to-metal seal fractures. Both 3JP12 cathode ray tubes survived irradiation, some envelope discoloring occurred. All of the 371-B high

4743
ANNEALING OF COLD-WORKED COPPER BY ELECTRON IRRADIATION. C. J. Meechan (Atomics International, North American Aviation, Inc., Canoga Park, Calif.). J. Appl. Phys. 28, 197-200(1957) Feb.

Copper wires were cold worked at room temperature to approximately a 15% reduction in area and were then irradiated at temperatures between 100 and 150°C with 1.25-Mev electrons. The residual resistivity was observed to decrease as a function of exposure at temperatures above 100°C. The higher the temperature at which the irradiation was performed, the greater was the rate of resistivity decrease. From these data, it is concluded that one of the primary defects produced by electron irradiation becomes mobile in the temperature range, 100 to 150°C. It is suggested that interstitials and vacancies produced by the irradiation initiate a process which results in the annihilation of dislocations. From an analysis of the temperature dependence of the rate of decrease, a value for the activation energy for vacancy migration in copper has been deduced: 1.28 ± 0.10 ev. (auth)

6149 ✓ D2-1662
Boeing Airplane Co., Seattle.
GAMMA IRRADIATION OF STAR-TRACKER COMPONENTS. J. A. Barton and H. L. Steele. Jan. 11, 1957. 49p.

To determine the effects of radiation on the operation of a star-tracking system, the components of the system were subjected to gamma radiation in a Co^{60} facility. The components were both optical and electronic. Tests were performed on these components and the radiation-induced changes noted. Among the system components were crown and flint glass, silica, mirror surface, telescope assembly, photomultiplier tube, capacitor and a voltage regulator. Transmission, luminescence, and reflectance tests were conducted on the optical components, and capacitance and conductivity tests on electrical components. (D.E.B.)

DAMAGING EFFECTS OF RADIATION ON ELECTRONIC COMPONENTS. R. D. Shelton and J. G. Kenney (Admiral Corp., Chicago). Nucleonics 14, No. 9, 66-9(1956) Sept.

The effects of radiation on conductors, insulators, resistors, capacitors, semiconductors, and vacuum tubes are discussed qualitatively. (T.R.H.)

10641
WHAT ARE EFFECTS OF RADIATION ON ELECTRONIC COMPONENTS? Nucleonics 14, No. 7, 33-5(1956) July.

Phototubes, bias cells, and dry rectifiers may fail to operate in intense fields of neutrons and gammas. But sensitive circuits made of selected components will function normally. (auth)

RADIATION EFFECTS 6.15

ON ELECTRONIC COMPONENTS 6.15.02

GENERAL 6.15.02.01

10465 WADC-TR-56-465
Wright Air Development Center. Materials Lab., Wright-Patterson AFB, Ohio.
ELECTRICAL PROPERTIES OF IRRADIATED POLYMERS. Period Covered: March 1955 to May 1956. Ralph E. Woodard. June 1957. 31p. Project title: MATERIALS ANALYSIS AND EVALUATION TECHNIQUES. Task title: NUCLEAR RADIATION APPLICATIONS TO ENGINEERING MEASUREMENTS. (AD-130801).

A brief discussion is given of the electronic nature of solids as it applies to insulators and polymers. The fundamental theory of nuclear radiation effects is discussed and shown to be associated with the laws of energy dissipation of these radiation particles within a material. A survey is given on the role that nuclear radiation plays in polymer kinetics. Work of various investigators on the electrical properties of irradiated polymers is reported. An analogy is drawn between photoconductivity in semi-conductors and nuclear radiation induced conductivity in certain polymers. The instantaneous nature of this induced conductivity is The components tested included carbon composition and deposited carbon-film resistors, Ge and Si crystal rectifiers, high vacuum power and high voltage rectifiers, gaseous type power rectifiers, miniature and general-purpose triodes, cathode ray oscilloscope tubes, and mica and ceramic capacitors. For the purpose of planning future experiments with camera tubes, material tests of various camera tube parts were performed. The RC type, carbon composition resistors underwent slight decreases in resistance. At initial reactor power increase, the resistance readings showed a distinct decrease and at an integrated thermal flux of 1.01×10^{18} there is no evidence of failure in any of the resistors. At the end of the test, the resistance values varied from a plus 1 to a minus 10% change. RF type, deposited-carbon resistors underwent an early decrease in resistance but recovered and stabilized near the initial value. There were no failures in this group. The 1N58 Ge rectifiers suffered drastic changes in backward and forward current characteristics before reaching an integrated thermal flux of 10^{18} neutrons/cm^2. The Si rectifiers showed similar variations. All 6 of the 5R4WGB power rectifiers tested showed over a tenfold apparent increase in emission current before 1.6×10^{17} NVT was reached. The tubes burned out when the normal voltage associated with the standard test circuit was applied. All the 1Z2 high voltage rectifiers operated throughout the test period without showing serious deviations. Of the 6 0Z4 power gaseous rectifiers tested in the reactor, one operated for the entire test and the remaining five either failed or showed erratic operation. All 6 of the 6C4 miniature triodes operated satisfactorily to 7×10^{17} NVT, but three of the six failed before 10^{18} NVT by interelement short circuit. The 6 3-in. cathode ray oscilloscope tubes operated throughout the test with negligible change in cathode current. Post-irradiation inspection will permit an evaluation of damage to phosphor and glass face plates. (auth)

6028
USE OF THERMOCOUPLES IN A RADIATION FIELD. Hubert P. Yockey (Oak Ridge National Lab., Tenn.). Phys. Rev. 101, 1426(1956) Feb. 15.

The radiation damage effects on an iron–constantan thermocouple are briefly discussed. It is concluded that such thermocouples give satisfactory temperature indication for most radiation field applications. (B.J.H.)

Refer also to abstracts 5889 and 5932.

8710 AD-82829
Admiral Corp., Chicago.
THE EFFECTS OF NUCLEAR RADIATION ON ELECTRONIC COMPONENTS. Scientific Report No. 2. R. D. Shelton, ed. Jan. 1, 1956. 95p. Contract AF33(616)-3091.

During this period several major improvements were made in the method of testing and instrumentation, in addition to the irradiation of selected electronic components. Integrated thermal fluxes were monitored by inserting small Co wires with the components being tested. The cobalt wires were then calibrated by comparison with irradiated gold foils. During this period a group of polyethylene-insulated wires was compared to a similar group of conductors using polyvinyl-chloride as the insulator. These were compared in a test designed to maximize any leakage effects present. The result of this test showed the polyethylene insulation to be superior as an insulator material while undergoing irradiation in the reactor. Tests were made on full-sized sample quantities of types 6AK5 and 1AD4 vacuum tubes, with the result that these tube types were able, in general, to operate throughout the irradiation period of the tests without failure. A group of electrolytic capacitors was tested. They were found to fail after approximately 1.4×10^{17} NVT by becoming

RADIATION EFFECTS 6.15

ON ELECTRONIC COMPONENTS 6.15.02

GENERAL 6.15.02.01

short-circuited. A single type 5U4G rectifier tube was placed in the reactor and connected to an external power supply in such a way that it operated in normal rectifier service under full load conditions. This tube operated throughout the test run without a significant change in its operation. A group of four oil-impregnated paper capacitors was tested without showing signs of failure. (For preceding period see AD-77763.) (auth)

8034
THE EFFECT OF REACTOR IRRADIATION ON ELECTRICAL INSULATION. J. C. Pigg, C. D. Bopp, O. Sisman, and C. C. Robinson (Oak Ridge National Lab., Tenn.). Communication and Electronics, No. 22, 717-23(1956) Jan.

The presence of a radiation field subjects the insulation of a circuit to environmental conditions which both accelerate the normal processes of deterioration and introduce new characteristics to the insulation behavior. The resistance of an insulation under irradiation drops to some value which is a function of the flux level and maintains this value as irradiation proceeds. Eventually the insulation resistance begins to drop again. This final resistance drop can be designated the breakdown of the insulation. The breakdown irradiation dosage for several insulations studied is shown. The change in insulation resistance in the presence of a

7050V AD-77763
Admiral Corp., Chicago.
THE EFFECTS OF NUCLEAR RADIATION ON ELECTRONIC COMPONENTS. Scientific Report No. 1. E. R. Pfaff and R. D. Shelton. Oct. 10. 1955. 62p. Contract AF33(616)-3091.

A selection of electronic components has been irradiated in the CP-5 research reactor at Argonne National Laboratory. The components were subjected to in-pile operation and testing, and the results of this preliminary testing program have been used to provide guidance and background for the formulation of a more extensive and exacting program, the purpose of which is to determine the effects of nuclear radiation on electronic components. The principal effort has been directed toward planning and exploratory tests which would define the problems of component testing, handling and storing of radioactive components, facility planning, radiation monitoring, and data processing. The components tested included wires, tors with a 10^{-9} fd capacity showed small rate effects and more or less permanent damage, the latter tending to diminish somewhat after irradiation. Carbon and wire-wound resistors of approximately 6000 ohms were affected respectively less than 8% and 1% by an irradiation to an integrated thermal flux of 10^{18} nvt. Vacuum tube testing was made difficult by the presence of the radiation field. Large leakages between plate voltage supply lines and high impedance grid circuits resulted in anomalous readings before the trouble was found and corrected. Post irradiation tests indicated that, although most tubes will operate in some manner to an integrated thermal flux of 10^{18} nvt, they seem more susceptible to damage by handling. Several tubes operated to 10^{18} nvt, but were found to have open filaments when the post irradiation tests began. (auth)

SEMICONDUCTORS 6.15.02.02

2780
HOW RADIATION AFFECTS SEMICONDUCTOR DEVICES. Michael F. Wolff. Electronics 32, No. 48, 55-7(1959) Nov. 27.

Radiation effects on semiconductors are discussed. Protection can be increased through proper choice of transistors, diodes, and special circuits. The advantages of these devices for semiconductors under neutron and electron bombardment are discussed. (C.J.G.)

662
EFFECT OF RADIATION ON SEMICONDUCTORS. J. C. Pigg and C. C. Robinson (Oak Ridge National Lab., Tenn.). Elec Mfg. 116-24(1957) Apr.

When high-energy particle bombardment is part of the environment, circuit designs employing semiconductors must include consideration of the effects of crystal lattice disorder. Changes in characteristics of typical semiconductor components when subjected to various radiation levels are described. (auth)

3372 AD-125193
Air Force Inst. of Tech., Wright-Patterson AFB, Ohio.
IRRADIATION OF P-N JUNCTION DIODES (thesis). William Matscher. Mar. 1957. 51p.

In an attempt to determine the effects of gamma radiation on germanium p-n junction diodes, the back characteristics of type 1784 diodes were measured while they

ON ELECTRONIC COMPONENTS 6.15.02

SEMICONDUCTORS 6.15.02.02

were exposed to a gamma intensity of 3.5×10^5 r/hr from a Co^{60} source. The electrical properties of germanium diodes are known to be structure sensitive. The relative changes produced in the body and the surface as a result of gamma irradiation is a prime object of this report. Diodes with treated and untreated surfaces were used to gain information on the surface effects. (auth)

665
EFFECT OF IRRADIATION ON THE HOLE LIFETIME OF N-TYPE GERMANIUM. O. L. Curtis, Jr., J. W. Cleland, J. H. Crawford, Jr., and J. C. Pigg (Oak Ridge National Lab., Tenn.). J. Appl. Phys. 28, 1161-5(1957) Oct.

The minority carrier lifetime of Ge is very sensitive to certain types of irradiation. The results of irradiating large single crystal samples of n-type Ge with fast neutrons and Co^{60} gamma rays are presented and discussed. Lifetime was determined from a transient measurement of the decay of holes following an injection pulse. The effect of initial carrier concentration has been considered. According to the James-Lark-Horovitz model, four energy levels are introduced by irradiation into the forbidden band of Ge, one above and three below the center of the band. Using the upper level, a one level recombination model serves to explain the experimental results approximately.

2571 AD-125271
Inland Testing Labs., Morton Grove, Ill.
RESEARCH TO DETERMINE THE EFFECTS OF NUCLEAR RADIATION ON SEMICONDUCTOR ELECTRONIC COMPONENTS. Scientific Report No. 2 (Second Quarter). M. A. Xavier. Feb. 18, 1957. 157p. Contract AF33(616)-3776.

The results of tests to determine the effects of radiation on semiconductor electronic components are presented. The results on diodes are discussed briefly with reference to an equivalent circuit. Effects of irradiations on transistors yielded relatively consistent behavior. (auth)

670
THE STRUCTURAL DEFECTS IN GERMANIUM MONOCRYSTALS IRRADIATED BY BETA PARTICLES AND FAST NEUTRONS, AND THE INFLUENCE OF THESE DEFECTS ON ELECTRON-HOLE RECOMBINATION. Presented at the International Conference on Radioisotopes in Scientific Research, Sept. 9-20, 1957 at Paris. No. UNESCO/NS/RIC/197a. V. S. Vavilov, L. S. Smirnov, A. V. Spitzin, V. M. Patskevitch, and M. V. Chukichev. London, Pergamon Press, Ltd., 1957. 10p.

9156 / KAPL-1742
Knolls Atomic Power Lab., Schenectady, N. Y.
SUMMARY OF FAST NEUTRON IRRADIATION STUDIES ON GERMANIUM. W. A. Smith, Jr., J. E. Noonan, and K. Minassian. Apr. 26, 1957. 35p. Contract W-31-109-Eng-52. $0.30(OTS).

A summary is given of studies made at KAPL to determine the energy dependence of fast neutron effects on the electrical properties of Ge. Irradiations were made by using neutrons produced in positive ion reactions. A nonlinear relationship between neutron energy and the number of interstitial atoms produced per neutron is indicated for the energy range 0.7 to 4.8 Mev. Material preparation techniques are discussed and methods of determining neutron response characteristics of Ge materials for dosimetry applications are presented. (auth)

9506 ✓
ON THE DISORDERING MECHANISM BY α PARTICLES IN GERMANIUM. A. Ascoli, M. Asdente, and E. Germagnoli (CISE Labs., Milano). Energia nucleare (Milan) 4, 131-8 (1957) Apr.

Changes in electrical conductivity due to α-particle irradiation of n-type Ge are considered. From these measurements the number of carriers removed from the conduction band is deduced and information is obtained concerning the spectrum of electron levels which are introduced within the gap. (auth)

9160
ON THE ATOMIC DISPLACEMENTS PRODUCED BY α-PARTICLES IN GERMANIUM. A. Ascoli, M. Asdente, and E. Germagnoli (Lab. CISE, Milano). Nuovo cimento (10) 5, 1145-52(1957) May.

The number of negative carriers which are removed from the conduction band by irradiation of germanium single crystals with P^{210} α particles is measured from the initial rate of decrease of conductivity in n-type germanium. It has been found that (79 ± 5) electrons are removed by a

RADIATION EFFECTS 6.15

ON ELECTRONIC COMPONENTS 6.15.02

SEMICONDUCTORS 6.15.02.02

5.30 Mev α particle. The possibility of comparing this result with the theory of atomic displacements by high energy charged particles is discussed. (auth)

7365 ✓
EFFECTS OF IRRADIATION UPON DIODES OF THE SILICON JUNCTION TYPE. R. Gorton (E.M.I. Engineering Development, Ltd., Feltham, Middlesex). Nature 179, 864 (1957) Apr. 27.

4985 KAPL-1455
Knolls Atomic Power Lab., Schenectady, N. Y.
IRRADIATION OF GERMANIUM BY FAST MONOENERGETIC NEUTRONS. J. W. Moyer, W. A. Smith, Jr., and O. L. Cunningham. May 25, 1956. 33p. Contract W-31-109-Eng-52. $0.30(OTS).

The effect of monoenergetic fast neutrons (from a Van de Graaff accelerator) upon the electrical resistivity and the Hall coefficient of different germanium materials was studied. Significant changes in carrier concentrations were observed in the case of the gold-doped materials. The observed increases of carrier concentration in the p-type gold-doped germanium samples were contrary to the changes caused by higher integrated fluxes of fast neutrons from a pile source. Further experiments are planned. (auth)

12161 ✓ APEX-462
American Nuclear Science Corp., New York.
THE EFFECT OF REACTOR RADIATION AND TEMPERATURE ON SILICON JUNCTION DIODES.
A. Friedman, L. M. Slater, H. K. Alan Kan, and L. M. Sharpe. Feb. 1959. 173p. To General Electric Co. Aircraft Nuclear Propulsion Dept. Contracts AF33(600)-38062 and AT(11-1)-171. $3.00(OTS).

The effects of high temperature (up to 300°C) and reactor radiation on silicon junction diode forward and reverse characteristics, switching characteristics, noise, Zener voltage, and Zener slope characteristics were measured using three positions in the BNL Reactor. Eleven types of commercial silicon junction diodes were irradiated. The response to irradiation was found to vary greatly among diodes even of the same type. (W.D.M.)

4738 AERE-T/R-630
Gt. Brit. Atomic Energy Research Establishment, Harwell, Berks, England.
A PRELIMINARY INVESTIGATION OF THE EFFECT OF IRRADIATION ON THE HALL COEFFICIENT OF AN INTRINSIC SEMICONDUCTOR. D. Luffman. Mar. 1951. Decl. Nov. 26, 1956. 12p.

A relation is found between the Hall coefficient and the number of electron traps in an intrinsic semiconductor of the graphite type. On a very simple model, and on the assumption that each vacancy produced by irradiation functions as an electron trap, the electron trap density can later be related to neutron damaging flux. It is hoped that the calculations will help to elucidate the effect of irradiation of graphite by a neutron flux. (auth)

9347 ✓
ON THE DEPTH OF MODERATE ENERGY ION PENETRATION INTO MATTER. M. M. Bredov and N. M. Okuneva. (Inst. of Semiconductors, Acad. of Sciences U.S.S.R.). Doklady Akad. Nauk S.S.S.R. 113, 795-6(1957) Apr. 1. (In Russian)

Measurements were made of the 4-kev Cs ion penetration into Ge. The radioactive isotopes Cs^{134} with 2.2 year half life was added to common Cs to determine the depth of penetration. The activity of irradiated samples was measured after dipping into the standard solutions of hydrogen peroxide. It was found that Cs ions with energy 4 kev penetrated into Ge on the order of 0.1 μ. (R.V.J.)

GASES 6.15.03

9247 ✓
DECOMPOSITION OF CARBON DIOXIDE BY IONIZING RADIATION. PART II. P. Harteck and S. Dondes (Rensselaer Polytechnic Inst., Troy, N. Y.). J. Chem. Phys. 26, 1727-33(1957) June.

Research on CO_2 was conducted at pressures from 1 to 50 atm, and on liquid carbon dioxide at 10°C, near the critical temperature. Also a novel method was developed in using the direct impact of the fission fragments on the gases (uranium-235 in contact with the gases on bombardment with thermal neutrons). The action of inhibitors other than NO_2 to stop the back reaction of the decomposed carbon dioxide was also investigated. (auth)

RADIATION EFFECTS 6.15

GASES 6.15.03

5563
KINETICS AND PRODUCTS OF THE POLYMERIZATION OF ISOBUTENE UNDER THE ACTION OF α RAYS. W. Mund, Ch. Guidée, and J. Vanderauwera. Bull. classe sci. Acad. roy. Belg. 41, No. 8, 805-15(1955). (In French).

The constant pressure polymerization of isobutene irradiated by radon α particles has given coherent and reproducible manometric data. The combination of these with the cryoscopy of the produce (interpreted by taking the dissolved gas into consideration) as well as with the measurement of the small quantity of hydrogen formed, suggests $C_4H_8 = C_4H_7 + H$ and $C_4H_8 + H = C_4H_7 + H_2$ as the initiative mechanism of the chains. (tr-auth)

5561
PROTON TRANSFER IN THE RADIATION CHEMISTRY OF GASEOUS AMMONIA. Antoine B. Zahlan and Benjamin P. Burtt (Syracuse Univ., N. Y.). J. Chem. Phys. 24, 478(1956) Feb.

The reactions of NH_3^+ and NH_2^+ with NH_3 in the gas phase by proton transfer during alpha irradiation are suggested by recent observations of the effects of pressure and intensity on the net ion yield. (C.W.H.)

4336
CHEMICAL STATE OF RADIO-IODINE IN NEUTRON-IRRADIATED PERIODATE AFTER THERMAL TREATMENT. A. H. W. Aten, Jr., G. K. Koch, G. A. Wesselink, and A. M. de Roos. J. Am. Chem. Soc. 79, 63-6(1957) Jan. 5.

The distribution of radioactive iodine over the iodide–iodine, iodate and periodate fractions is determined in neutron-irradiated potassium periodate. The main activity is found in the iodate fraction with minor contributions in the other fractions. If the activated solid is heated, the iodide–iodine activity disappears below 100°. Above this temperature most of the activity goes into the periodate fraction but part of it remains in the iodate fraction. (auth)

12293
THE X-RAY DECOMPOSITION OF NITROUS OXIDE. Benjamin P. Burtt and John F. Kircher (Syracuse Univ., N. Y.). Radiation Research 9, 1-12(1958) July.

The ion yield for the decomposition of N_2O by x rays has been investigated at 50, 200, and 555 mm of pressure at extremely low intensities by the direct method. The effect of changes in pressure and in applied electric field have been studied. The reactions between the products NO and O_2 were not measurable at room temperature but proceeded immediately when a trap was immersed in liquid nitrogen to solidify the N_2O. By means of these reactions it was possible to determine the relative amounts of the products formed and hence the amount of N_2O decomposed per ion pair. The N_2O ion yield was 4.85 molecules decomposed per ion pair at 555 and 200 mm, but increased to 6.87 as the pressure was lowered to 50 mm. This increase was due to an ion recombination-induced reaction not important at the higher pressures. The yield of O_2 was about 1.3 and

12827
W FOR HEAVY PARTICLES. Lewis W. Cochran (Univ. of Kentucky, Lexington). Health Phys. 1, 34-8(1958) June.

The values of W, the average energy expended per ion pair produced in a gas, are listed for many pure gases. The change of W with small amounts of contaminant gases, the determination of an effective W for gas mixtures, and the variation of W with velocity for very low velocity particles are considered. Values of W for low-energy, high-mass recoil atoms are compared with the values obtained with alpha particles in the same gases. (auth)

14655 A/CONF.15/P/1769
Rensselaer Polytechnic Inst., Troy, N. Y.
RADIATION CHEMISTRY OF GASES. P. Harteck and S. Dondes. 10p. $0.50(OTS).

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

The kinetic radiation equilibrium has only been obtained with two systems, nitrogen–oxygen, and carbon dioxide. In the case of the nitrogen–oxygen system, the end results are nitrogen, oxygen, nitrogen dioxide, and nitrous oxide, regardless of the initial material. Thus, if nitrous oxide or a 66.33 mixture of nitrogen to oxygen is the starting material, the concentrations of the reactants and products at the kinetic radiation equilibrium are identical. Analogous results are obtained with nitric oxide of a 50:50 mixture of nitrogen to oxygen and with nitrogen dioxide and a 33.66 mixture of nitrogen to oxygen. The carbon dioxide equilibrium yields carbon dioxide, carbon monoxide, and oxygen. (auth)

RADIATION EFFECTS 6.15

GASES 6.15.03

126
CHEMICAL EFFECTS OF HOT ATOMS. Louis Monchick, Koichi Funabashi, and John L. Magee (Univ. of Notre Dame, Ind.). J. Chem. Phys. 27, 734-9(1957) Sept.

A model is presented for the study of the chemical effects which result from the passage of energetic atoms through liquid or gaseous media. The particular case of energetic Br atoms in CH_3Br is treated in detail. The average rate of energy loss, the range, and the number of C−Br bonds broken in primary events have been calculated. The track structure and the nature of chemical effects are discussed. (auth)

IONIZATION OF ACETYLENE MIXTURES AND OTHER MIXTURES BY Pu^{239} α-PARTICLES. Harold J. Moe, T. E. Bortner, and G. S. Hurst (Oak Ridge National Lab., Tenn.). J. Phys. Chem. 61. 422-5(1957) Apr.

The ionization produced when Pu^{239} α particles were completely absorbed in binary gas mixtures having acetylene as one of the components was measured. The W value (electron volts per ion pair) was computed from the known energy and the indirectly measured ionization for various percentages of the two gases in the mixture. The W values for varying proportions of a binary gaseous mixture were then compared to W_m as determined by the equation $1/W_m = (1/W_1 - 1/W_2)Z + 1/W_2$ where $Z = P_1/(P_1 + aP_2)$ and W_m is the W value for a mixture of two gases having partial pressures P_1 and P_2 and W values W_1 and W_2, respectively. Good agreement was found when the following values were used for the empirical constant "a": $N_2-C_2H_2$, 0.265; $CO_2-C_2H_2$, 0.927; $CH_4-C_2H_2$, 0.386; N_2-CH_4, 0.617; He−C_2H_2, 0.058; C_2H_2−A, 3.42; C_6H_6−A, 5.376; CH_4−A, 2.00. The W values for mixtures of neon with acetylene would not yield to this interpretation. (auth)

THE KINETIC RADIATION EQUILIBRIUM OF AIR. P. Harteck and S. Dondes (Renssalaer Polytechnic Inst., Troy, N. Y.). J. Phys. Chem. 63, 956-61(1959) June.

A study was made of the irradiation of nitrogen−oxygen mixtures near and at the kinetic radiation equilibrium, i.e., the steady state of formation and decomposition of the constituents. Air and two to one nitrogen to oxygen mixtures showed that under proper irradiation conditions, all the oxygen is consumed to form nitrogen dioxide and nitrous oxide and, in some cases, a small surplus of nitric oxide remained. The kinetics of the nitrogen−oxygen system at the equilibrium is explained. (auth)

GLASS 6.15.04

11478 ✓
IRRADIATION EFFECTS IN BOROSILICATE GLASS. Bernhard Gross. Phys. Rev. 107, 368-73(1957) July 15.

Irradiation of thick samples of borosilicate glass with high-energy electrons introduces space charges into the dielectric, the decay of which is extremely slow at room temperature. The charged dielectric shows a behavior similar to that of an electret; charges are induced on adjacent conductors and short-circuit discharge currents are observed during heating. The nature of the charges of the dielectric, annealing effects, and discharge mechanism were investigated. It is shown that the dielectric contains charges of two types: a negative charge constituted by arrested irradiation electrons and a positive compensation charge. (auth)

1947 V
FORMATION OF COLOR CENTERS IN GLASSES EXPOSED TO GAMMA RADIATION. N. J. Kreidl and J. R. Hensler (Bausch and Lomb Optical Co., Rochester, N. Y.). J. Am. Ceram. Soc. 38, 423-32(1955) Dec.

Glasses exposed to gamma radiation develop a visible absorption band similar to the F-band developed in alkali halide crystals. The function of cerium in preventing coloration of glass was studied in detail in an ultraviolet-transmitting phosphate base glass in which its characteristic absorption could be measured. When visible absorption was thus prevented, radiation still caused a strong absorption was thus prevented, radiation still caused a strong absorption in the ultraviolet which was associated with the

3845 ✓ NYO-3784
Bausch and Lomb Optical Co., Rochester, N. Y.
IRRADIATION DAMAGE TO GLASS. N. J. Kreidl and J. R. Hensler. Nov. 15, 1955. 28p. Contract AT(30-1)-1312. $4.80(ph OTS); $2.70(mf OTS).

RADIATION EFFECTS 6.15

GLASS 6.15.04

13790
NEUTRON IRRADIATION EFFECTS IN BOROSILICATE GLASS AND THEIR DETECTION BY ULTRASONIC ATTENUATION AND VELOCITY MEASUREMENTS. Rohn Truell and John deKlerk (Brown Univ., Providence, R. I.) and Paul W. Levy (Brookhaven National Lab., Upton, N. Y.). J. Appl. Phys. 29, 225-6(1958) Feb.

The damage or irradiation effects arising from the reaction of slow neutrons with B^{10} were detected and observed in a boron glass by means of high-frequency ultrasonic attenuation. The glass used (Corning 7070) contained 28% by weight of B_2O_3, or approximately 8% B. Irradiation for more than an hour in the Brookhaven Reactor caused the glass samples to crack. Reactor irradiation of 15, 30, 45, and 60 minutes produced both attenuation and velocity changes that could be measured in the frequency range from 10 to 50 Mc/sec. The results show an attenuation increase of approximately 50%

11150
BIREFRINGENCE IN NEUTRON-IRRADIATED BORON GLASS. C. Mylonas and R. Truell (Brown Univ., Providence, R. I.) and P. W. Levy (Brookhaven National Lab., Upton, N. Y.). J. Appl. Phys. 29, 864-5(1958) May.

Samples of boron glass were irradiated (slow flux approximately 2×10^{12} nv) in the Brookhaven Reactor for studies of the resulting ultrasonic attenuation and velocity changes. The plates invariably cracked after about an hour's irradiation; after irradiation for several hours they fell to pieces. Examined in polarized light incident normally on their largest faces, the plates showed little birefringence, but in an end-to-end viewing they exhibited relative retardation of several wave lengths. The disappearance of the birefringence in the neighborhood of certain stress-relieving cracks proved

11152
RESISTANCE OF INDUSTRIAL GLASS AGAINST THE EFFECT OF GAMMA RAYS. S. M. Berkhovskikh. Steklo i Keramika No. 1, 10-14(1958). (In Russian)

The resistivity of various types of industrial glass to the effects of gamma radiation was studied. The light transparency of the glasses was determined before and after irradiation. The color characteristic of the glasses was also determined. The results obtained are tabulated. (J.S.R.)

11270
GLASS TECHNOLOGY IN THE NUCLEAR FIELD. Nuclear Eng. 2, 333-4(1957) Aug.

13586
GAMMA RAYS FROM THE PROTON BOMBARDMENT OF SILICON. Eiji Okada, Iwao Miura, Tetsuo Wakatsuki, and Yasuo Hirao (Osaka Univ.). J. Phys. Soc. Japan 13, 541-4(1958) June.

The gamma-ray spectrum from silicon bombarded by 5.7-Mev protons has been studied with a scintillation spectrometer. The following gamma rays due to inelastic scattering of protons were observed: 1.78 Mev (Si^{28} 1st—ground), 1.28 Mev (Si^{29} 1st—ground), 2.03 Mev (Si^{29} 2nd—ground), 2.43 Mev (Si^{29} 3rd—ground), 2.24 Mev (Si^{30} 1st—ground). Excitation curves for the 1.78-Mev gamma rays have been measured in the proton energy range from 2.2 to 5.7 Mev. Resonances were found at 2.9, 3.5, 4.4, 5.0, and 5.4 Mev. At 5.4 Mev, the angular distribution of the 1.78 Mev gamma rays was measured. By comparison with theory, the corresponding compound level of P^{29} at 7.9 Mev was assigned $d_{3/2}$. The cross section for this process is 250 mb. (auth)

20582 AD-209698
Chatham Electronics. Div. of Tung-Sol Electric Co., Livingston, N. J.
DEVELOPMENT AND EVALUATION OF ELECTRON TUBE GLASSES RESISTANT TO RADIATION DAMAGE. Semi-annual Progress Report No. 1 [for] Period: May 1 to November 1, 1958. 23p. DA Project No. 3-99-00-100. Contract DA-36-039-SC-75065.

Objectives of the program are to investigate effects of radiation on known soft and hard glasses used in electron tubes and to develop and evaluate suitable glasses which will withstand certain radiation levels without damage. Dummy bulbs of G8, G12, 7720, and 7052 glass were exposed to a neutron bombardment of an integrated thermal flux of 1016 nvt. Enclosures using the same glasses were made with lead-in wires sealed-in. (W.D.M.)

RADIATION EFFECTS 6.15

GLASS 6.15.04

EFFECT OF NEUTRON IRRADIATION ON CRYSTALLINE AND GLASSY INORGANIC SOLIDS. J. H. Crawford, Jr. (Oak Ridge National Lab., Tenn.). p.531-50 in "Progress in Nuclear Energy. Series V. Metallurgy and Fuels. Volume 2."

Research progress since the Geneva Conference in the field of radiation effects in non-metallic systems is surveyed. Particular attention is given to the recent developments in the study of structural and electronic changes in diamond and the silica system that result from fast neutron bombardment. The experimental findings for these systems indicate that inhomogeneous distribution of neutron-produced lattice defects is an important factor in determining the observed changes in physical properties. Optical and magnetic evidence of lattice or network imperfections produced by both fast neutrons and Co^{60} γ rays for fused silica has been reviewed in some detail and correlated with similar information obtained on quartz crystals. New information on radiation induced phase transformations in non-metal systems and radiation effects in ionic crystals is also summarized. (auth)

GRAPHITE 6.15.05

14638 ✓ A/CONF.15/P/1778

Argonne National Lab., Lemont, Ill.; Brookhaven National Lab., Upton, N. Y.; and General Atomics Div., General Dynamics Corp., San Diego, Calif.

RADIATION EFFECTS ON THE OXIDATION RATE AND ON OTHER CHEMICAL PROPERTIES OF GRAPHITE. G. R. Hennig, G. J. Dienes, and W. Kosiba. 12p. $0.50(OTS).

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

Irradiation by fast particles changes most physical and chemical properties of graphite by producing various lattice defects such as displaced atoms, vacancies, dislocations, etc. Attempts have been made to evaluate the contributions of different types of defects to the property changes. Such evaluations have proven rather successful for physical properties and will be extended

4323

EFFECT OF IRRADIATION ON THE LOW-TEMPERATURE SPECIFIC HEAT OF GRAPHITE. W. DeSorbo and W. W. Tyler (General Electric Research Lab., Schenectady, N. Y.). J. Chem. Phys. 26, 244-7(1957) Feb.

The specific heat of heavily irradiated Acheson-type graphite has been measured from 13° to 300°K. Throughout this temperature range, the specific heat of the irradiated graphite is appreciably larger than that of unirradiated graphite of similar type. This difference is explained by assuming that interplanar atoms or clusters of atoms, give rise to modes of low energy which contribute to the specific heat at lower temperatures than do the normal lattice modes. The entropy of the irradiated sample at 298.16°K is 1.492 ± 0.009 cal/g-atom deg compared with 1.372 ± 0.005 cal/g-atom deg for the unirradiated graphite. (auth)

2649 NAA-SR-196

North American Aviation, Inc., Downey, Calif.

THE THERMOELECTRIC POWER OF GRAPHITE DEPENDENCE ON TEMPERATURE, TYPE AND NEUTRON IRRADIATION. W. P. Eatherly and N. S. Rasor. Nov. 21, 1952. Decl. Oct. 18, 1955. 31p. Contract AT-11-1-Gen-8.

The thermoelectric power of nine grades of artificial and natural graphites has been measured in the temperature region 4.2 to 300°K. The effect of neutron exposure on the temperature dependence in this interval has also been determined, including Hanford irradiations up to 2000 Mwd/ct. An analytical expression for the thermoelectric power has been derived, employing a two-

11101 ✓

THE EFFECT OF DISPLACED ATOMS AND IONIZING RADIATION ON THE OXIDATION OF GRAPHITE. W. L. Kosiba and G. J. Dienes (Brookhaven National Lab., Upton, N. Y.). Advances in Catalysis 9, 398-405(1957).

The graphite-oxygen reaction on irradiated and unirradiated samples has been studied over the 250 to 450° temperature range. The irradiated samples were exposed to about 4×10^{20} neutrons/cm^2 in the reactor prior to oxidation. The exposure, which produces about 2% displaced atoms at room temperature, increased the oxidation rate relative to an unirradiated sample by about a factor of 6. The oxidation rate of untreated samples in the presence of gamma-rays alone (200,000 r/hr) was hardly altered, al-

RADIATION EFFECTS 6.15

GRAPHITE 6.15.05

though a significant increase was observed at the higher intensity of 610,000 r/hr (at 300°). The reaction rate of a sample previously irradiated in the reactor and oxidized in the presence of gamma-rays (200,000 r/hr) at 300° was higher by an additional factor of about 3, i.e., a factor of about 18 relative to unirradiated specimens. It is concluded that displaced atoms exert a large influence on the rate of this heterogeneous gas-solid reaction. When, in addition, ionizing radiation is present during the reaction the rate is further increased, probably because of the ionization of oxygen molecules. (auth)

9724

A CHEMICAL MODEL OF RADIATION DAMAGE IN GRAPHITE. G. R. Hennig (Argonne National Lab., Lemont, Ill.). Nuclear Sci. and Eng. 3, 514-28(1958) May.

It is shown that the production rate of electron traps during fast neutron bombardment of graphite can be determined by comparing its properties with those of chemically doped graphite. A value of 9×10^{-5} trap per equivalent megawatt day unit of reactor bombardment is obtained. This comparison also shows how the scattering of the carriers is affected by bombardment. The validity of the conclusions which are reached depends upon certain assumptions concerning anisotropy and heterogeneity, which are explored in rather considerable detail. (auth)

5234

SURFACE PROPERTIES OF IRRADIATED GRAPHITE. C. N. Spalaris, L. P. Bupp, and E. C. Gilbert (General Electric Co., Richland, Wash. and Oregon State Coll., Corvallis). J. Phys. Chem. 61, 350-4(1957) March.

The surface characteristics of neutron irradiated graphite have been investigated. Studies include measurements of surface area, density, heat of adsorption and pore size distribution. The results obtained indicate that the magnitude of these properties decreases with increasing irradiation exposures. It has been proposed that the graphite crystallites in expanding upon neutron irradiation, occupy the voids in their immediate vicinity, thus causing the changes in surface properties. (auth)

LUBRICANTS 6.15.06

7492 NP-7297

Midwest Research Inst., Kansas City, Mo.
RESEARCH ON HIGH TEMPERATURE METALLO-ORGANIC AND INORGANIC BASE FLUIDS. Quarterly Progress Report No. 3 [for] October 16, 1958–January 31, 1959. L. W. Breed. 39p. M.R.I. Project No. 2194-C. Contract AF33(616)-5718.

Data for 22,240 inorganic and metallo-organic compounds have been collected, the structures coded, and all information entered on IBM punch-cards. An analysis of the data to find correlations among liquid range, thermal stability, melting point, boiling point, formula weight, and structural features of the compounds is now being carried out. In the report a discussion of the limitations and possibilities of such a data correlation program is included. (auth)

1708

SPECIAL LUBRICANTS FOR BRADWELL. Nuclear Power 2, 427-8(1957) Oct.

The work at Shell's Co^{60} facility on radiation effects on oils is reported, along with the development of special radiation resistant lubricants. (M.H.R.)

METALS 6.15.07

NEUTRONS 6.15.07.01

RADIATION DAMAGE TO METALS AND ASSOCIATED PROBLEMS. THE EFFECT OF NEUTRON IRRADIATION ON THE MECHANICAL PROPERTIES OF METALS. M. J. Makin (Atomic Energy Research Establishment, Harwell, Berks, England). J. Inst. Metals 84, 101-3(1956) Aug.

2183

RADIATION DAMAGE TO METALS AND ASSOCIATED PROBLEMS. IRRADIATION TECHNIQUES FOR FISSILE MATERIALS. O. S. Plail (Atomic Energy Research Establishment, Harwell, Berks, England). Bull. Inst. Metals 3, 122-6(1956) Oct.

RADIATION EFFECTS 6.15

METALS 6.15.07

NEUTRONS 6.15.07.01

Investigations have been carried on at Harwell into the effects of neutron irradiation on both fissile and non-fissile materials. In general, the more difficult problems of technique are associated with the fissile irradiations, and this paper is concerned mainly with the problems thus encountered. The subject can be dealt with under two main headings. First, there is the sample preparation and actual irradiation (i.e., the exposure of specimens to neutron bombardment) and, secondly, the subsequent examination of the samples when they are themselves highly radioactive. (auth)

5886 NMI-1165
Nuclear Metals, Inc., Cambridge, Mass.
EFFECTS OF RADIATION DAMAGE ON PRECIPITATION-HARDENING ALLOYS WITH SPECIAL REFERENCE TO COPPER–IRON ALLOYS. Alvin Boltax. Oct. 18, 1956. 21p. Contract AT(30-1)-1565. $0.20(OTS).

Studies of the effects of neutron irradiation on precipitation-hardening alloys were carried out on a large number of different alloy systems. Most of this work was concerned with radiation-induced precipitation from supersaturated solid solutions. "Irradiation aging" was observed in Cu–Be alloys at room temperature and in Ni–Be alloys at 300°C. With these experiments as a basis, prerequisites were suggested for "irradiation aging" which involve the atomic number of the principal constituent of the alloy, the activation energy of diffusion, and the critical particle size. It was suggested that the neutron irradiation caused precipitation by accelerating diffusion through the introduction of vacancies and interstitials. Comparatively few experiments have been carried out on the

8828 ✓
STUDY OF STRUCTURAL CHANGES PRODUCED BY NEUTRON IRRADIATION. S. T. Konobeevskii, N. F. Pravdyuk, K. P. Dubrovin, B. M. Levitskii, L. D. Panteleev, and V. M. Golyanov. Atomnaya Energiya 4, 34-44(1958) Jan. (In Russian)

Measurements were made of the electrical resistance of neutron-irradiated U–9 wt.% Mo alloy, and x-ray-diffraction and microscopic analyses were carried out to determine the structural changes in the specimen. Preliminary annealing yielded specimens with eutectic structure $\alpha + \gamma'$ of various grain sizes. The rate of diffusion under irradiation of the annealed alloy is inversely proportional to the grain area. In homogeneous specimens (γ phase) radiation induced quick alteration of properties and structure of the alloy. Observations of irradiation effects indicated a certain disorder during γ phase transition

12930 IGR-TN/C-651
Gt. Brit. Culcheth Labs., Culcheth, Lancs, England.
IRRADIATION OF STEELS. T. J. Heal. Aug. 1957. 12p.

The effects of neutron irradiation on the properties of steels to be used in reactor construction are described, and suggestions for the modification of the present irradiation programs in the light of recent knowledge are made. (auth)

4739 CF-56-11-1
Oak Ridge National Lab., Tenn.
RECENT DATA ON THE EFFECTS OF NEUTRON IRRADIATION ON STRUCTURAL METALS AND ALLOYS. R. G. Berggren and J. C. Wilson. Jan. 30, 1957. 31p. Contract W-7405-eng-26. $4.80(ph OTS); $2.70(mf OTS).

Data are presented from tensile and notched-bar impact tests on several carbon steels, a carbon steel weldment, a nickel steel, a high-strength steel, and a high-purity iron which were irradiated in the LITR or the MTR. It is shown that neutron irradiation increases the yield and tensile strength of structural steels, decreases their ductility, and may change the behavior of the steels to such an extent that they become unfamiliar materials for structural components. (B.J.H.)

13200 ✓ CP-1355
Chicago. Univ. Metallurgical Lab.
RADIOACTIVITY INDUCED IN COMMERCIAL MATERIALS. L. Seren, W. Sturm, W. Moyer, G. Miller, and W. Jordan. Feb. 12, 1944. Decl. Feb. 15, 1957. 9p. (A-2002). $1.80(ph OTS); $1.80(mf OTS).

Some structural metals (2S Al, 72S Al, Cu, Fe, steel, Pb, and Bi) were neutron irradiated, and the decay of the induced β and γ activities was followed. (L.T.W.)

RADIATION EFFECTS 6.15

METALS 6.15.07

Neutrons 6.15.07.01

8827 ✓ AEC-tr-3210
THE INFLUENCE OF NEUTRON IRRADIATION ON MARTENSITE TRANSORMATIONS. A. I. Zakharov and O. P. Maximova. Translated by S. J. Rothman from Doklady Akad. Nauk S.S.S.R. 114, 1195-8(1957).

The preliminary neutron irradiation of various steels significantly affected the austenite stability with respect to the martensite transformation. The change of stability was found to assume a different character in various materials. (J.E.D.)

12624 ✓ KAPL-954(Del.)
Knolls Atomic Power Lab., Schenectady, N. Y.
THE EFFECT OF IRRADIATION ON THE MECHANICAL PROPERTIES OF METALS: THE PRESENT STATUS OF THE PROGRAM. J. R. Low, Jr., and C. A. Bruch. July 1, 1953. Decl. with deletions Feb. 25, 1957. 19p. Contract W-31-109-Eng-52. $3.30(ph OTS); $2.40(mf OTS).

A program for investigating the effect of neutron bombardment on the mechanical properties of metals on a pre-irradiation-postirradiation basis is well under way. The irradiation variables to be studied include nv, nvt, and temperature. The group of metals to be studied includes high-purity Ni, Cu, Fe, Mo, Zr, and commercial purity Ti and Type 347 stainless steel. Specimens are already in the process of being irradiated and tested. Most of the remotely operable testing equipment has been built and is in operating condition. Some of the first test results obtained are included. (auth)

GENERAL 6.15.07.02

3834 ✓ NAA-SR-4301
Atomics International Div., North American Aviation, Inc., Canoga Park, Calif.
ATOMISTIC INTERPRETATION OF RADIATION EFFECTS IN METALS. A. Sosin. Dec. 1, 1959. 34p. Contract AT-11-1-GEN-8. OTS.

The present status of atomistic interpretations of radiation damage in metals is reviewed. Detailed consideration is given to radiation effects and associated lattice defect phenomena in copper, since copper has received the most extensive experimental and theoretical study. The various mechanisms of defect production are presented and models for point defect recovery are considered. The case of radiation-hardening is then concluded to be principally due to more extended radiation-induced defects such as displacement spikes. Finally a particular radiation damage problem, fuel swelling, is discussed with emphasis on its lattice defect implications. (auth)

12194 NP-7365(Vol. 3)(Paper 32)
General Atomic Div., General Dynamics Corp., San Diego, Calif.
MEASUREMENT OF THE RANGE OF RECOIL ATOMS. R. A. Schmitt and R. A. Sharp. Paper 32 [of] THIRD SEMI-ANNUAL RADIATION EFFECTS SYMPOSIUM [HELD IN ATLANTA], OCTOBER 28-30, 1958. VOLUME 3. AIRCRAFT SYSTEMS AND MATERIALS PAPERS. 9p.

An important problem in the interpretation of radiation damage and sputtering phenomena is the evaluation of the range of an atom which moves through a lattice after having received an initial energy of 10 to 100 kev. An experiment is described in which atoms with initial energies in this range were produced by irradiating suitable targets with high-energy bremsstrahlung gamma rays, and the products of photonuclear reactions, such as photoneutron (γ,n) transmutations, were observed. The photon spectrum of the gamma rays was continuous from 0 to 24 Mev. The energy spectra of evaporated neutrons were centered at about 1.5 Mev. (W.D.M.)

18102 ✓ KAPL-M-JWB-3
Knolls Atomic Power Lab., Schenectady, N. Y.
STATUS OF THE PREDICTION OF ACTIVITY BUILDUP IN INCONEL SYSTEMS. John W. Bartlett. July 20, 1959. 40p. Contract [W-31-109-Eng-52]. $7.80(ph), $3.30(mf) OTS.

Test programs, data, and associated information required to obtain a reasonably reliable prediction of the rate of buildup of radiation dose rates resulting from deposited long-lived activity in Inconel systems are given. Current theories on mechanisms of crud activation and transport and influential parameters are discussed in detail, in order to emphasize the importance of system variables and the present lack of fundamental knowledge of these factors. Methods used to predict activity buildup are briefly outlined. Available data from all known sources are reviewed, and it is concluded that

RADIATION EFFECTS 6.15

METALS 6.15.07

GENERAL 6.15.07.02

these data do not resolve any of the inherent uncertainties associated with attempts to use stainless steel data to predict activity buildup in an Inconel system. In general, then, it is felt that no estimate of precision could be attached to a prediction of Inconel activity buildup which is based on data and information now available. (auth)

10171 ✓ AECU-3684
Illinois. Univ., Urbana.
IRRADIATION DAMAGE AT LOW TEMPERATURES IN THE NOBLE METALS. Technical Report No. 13. J. S. Koehler, F. Seitz, and R. O. Simmons. May 1958. 28p. Contract AT(11-1)-182. $6.30(ph OTS); $3.00 (mf OTS).

The nature of the damage produced in noble metals by nuclear radiation and the changes which occur during thermal annealing below 60°K are described. Experimental data and interpretation of the damage are summarized. (auth)

RADIATION DAMAGE STUDIES OF SEVEN NON-FISSIONABLE METALS. Presented at Nuclear Engineering and Science Conference, held at Chicago, March 17 to 21, 1958. Preprint 37, Session 26. C. A. Bruch and W. E. McHugh (Knolls Atomic Power Lab., Schenectady; N. Y.). New York, American Institute of Chemical Engineers, 1958. 69p.

Seven metals were irradiated under a variety of irradiation exposure conditions in three types of nuclear reactors. Measurements were made of both the exposure conditions and the resulting changes in properties. All of the metals experienced the same general effects, including increases in strength, hardness, and electrical

1410 ✓
A SURVEY OF IRRADIATION EFFECTS IN METALS. J. W. Glen (Royal Society Mond Lab., Cambridge, England). Advances Phys. 4, 381-478(1955) Oct.

The effects of irradiation on the properties of metals are reviewed. Changes in electrical resistance, mechanical properties, allotropic forms, and crystal structure of metals are tabulated. Similar effects in non-metals are discussed. The theory of radiation damage is summarized. 230 references. (C.W.H.)

10616 ✓ NP-6759
United Kingdom Atomic Energy Authority. Industrial Group. Culcheth Labs., Culcheth, Lancs, England.
BRITTLENESS IN METALS. PREPRINTS OF PAPERS FOR A CONFERENCE TO BE HELD AT CULCHETH LABORATORIES ON 1st NOVEMBER 1957. Oct. 1957. 64p.

Papers are presented on: theory of brittle fracture in steel and its application to radiation embrittlement; the transition temperature in notched specimens; mechanical properties of neutron-irradiated steels; effects of radiation on steels; radiation testing of steels; possible effects of radiation on H_2 embrittlement of steels; a survey of H_2 embrittlement of steels; ductile-brittle transition in non-ferrous b.c.c. metals; cleavage fracture in hexagonal metals; and the ductility problem of reactor Be. (T.R.H.)

7042 ✓ A/CONF.15/P/80
THE MECHANICAL PROPERTIES, EMBRITTLEMENT AND METALLURGICAL STABILITY OF IRRADIATED METALS AND ALLOYS. M. J. Makin, D. R. Harries, R. E. Smallman (Atomic Energy Research Establishment, Harwell, Berks, Eng.), and A. T. Churchman (Associated Electrical Industries, Ltd., Aldermaston, Berks, Eng.). 26p.

The change in the tensile properties on irradiation of a number of metals of use as reactor constructional materials is described in detail. Materials tested include titanium, zirconium, Zircaloy-2, molybdenum, tungsten, mild steel, nickel, copper, and certain magnesium alloys. In general, the results indicate an increase in yield and ultimate strength and a decrease in elongation to fracture. (auth)

11282
DEFECTS IN CRYSTALLINE STRUCTURE AND PROPERTIES OF METALS AND ALLOYS. I. Ya. Dekhtyar. Uspekhi Fiz. Nauk 62, No. 2, 99-128(1957) June. (In Russian)

RADIATION EFFECTS 6.15

METALS 6.15.07

GENERAL 6.15.07.02

Studies were made of various atomic defects in the crystalline lattice of metals (voids, dislocations) which formed during crystallization of liquid metals, cold and thermal treatments, or strong irradiation. Investigations of metal and alloy properties show that the same type defects effect diffusion, electroconductivity, plastic deformation, and other processes. (R.V.J.

BENEFICIAL EFFECTS OF RADIATION ON METALS. H. A. Saller (Battelle Memorial Inst., Columbus, Ohio). Nucleonics 14, No. 9, 86-8(1956) Sept.

A qualitative discussion of the improvements effected in the properties of reactor fuels, cladding and structural materials, and of non-reactor structural materials, semiconductors, and catalysts by irradiation is presented.

16806

HOW RADIATION DAMAGES METALS. PART II. EFFECTS ON MECHANICAL PROPERTIES. M. J. Makin. Atomics and Nuclear Energy 9, 318-22(1958) Sept.

Experimental data are given on radiation damage in metals, and theoretical interpretations of the cause of the damage are discussed. (W.D.M.)

THE EFFECTS OF IRRADIATION UPON METALS. Frederick Seitz (Univ. of Illinois, Urbana). Trans. Met. Soc. AIME 215, 354-67(1959) June.

The information concerning the generation and the properties of the point imperfections (vacant lattice sites and imperfections) in the noble metals, particularly copper, which is gained from the studies of the effects produced by bombardment is surveyed. It is pointed out that the studies have now reached a stage at which it can be stated with considerable assurance that interstitial atoms migrate at temperatures well below that of liquid nitrogen with activation energies near 0.1 ev. Lattice vacancies can be generated with less energy under equilibrium conditions; however, their activation energy for motion is nearer 0.75 ev. The major part of the effects observed to date in pure monatomic metals at present appears to be ascribable to interstitials and vacancies. (auth)

8858

EFFECTS OF RADIATION ON MATERIALS. Julius J. Harwood (Office of Naval Research, Washington). Metal Progress 71, No. 6, 72-4(1957) June.

Discussions are presented of changes in metallic properties by extreme irradiation by neutrons and gamma rays. These are explainable by displacement of atoms in the crystalline lattice or introduction of new atoms. Certain new organic compounds of quasi-crystallinity were produced by intense radiation. (W.L.H.)

7366

THE DISPLACEMENT OF ATOMS IN SOLIDS BY RADIATION. T. M. Fry (Atomic Energy Research Establishment, Harwell, Eng.). Nuovo cimento (10) 4, Suppl. No. 3, 1329-34(1956).

Experiments performed on metals to investigate the influence of irradiation on structure-sensitive properties are reviewed. Ways in which the lattice can recover its initial degree of perfection are discussed. (M.H.R.)

RADIATION DAMAGE IN FERROUS METALS. D. E. Thomas (Westinghouse Electric Corp., Pittsburgh). J. Metals 11, 523-7(1959) Aug.

In iron and ferrous materials, irradiation increases strength and decreases ductility. Yield stress, in general, is increased proportionately more than the ultimate stress, accompanied by a reduced strain-hardening capacity and decreased uniform elongation. In stainless steels, property changes generally appear to saturate at total exposure of 10^{19} or 10^{20} nvt. Effects of irradiation on various low-alloy steels are presented. (C.J.G.)

RADIATION EFFECTS OF HIGH-ENERGY ELECTRONS IN METALS. C. E. Dixon (Atomics Div., North American Aviation, Inc., Canoga Park, Calif.). p.475-87 in "Progress in Nuclear Energy. Series V. Metallurgy and Fuels. Volume 2."

Several electron-irradiation experiments of metals are described. These include determinations of displacement energy in copper and nickel, acceleration of the ordering process in copper–gold alloys, acceleration of a phase transformation in a copper–iron alloy, increase in Young's modulus, hardness and flow stress in copper and electrical resistivity changes in copper at various irradiation temperatures. The usefulness of

RADIATION EFFECTS 6.15

METALS 6.15.07

General 6.15.07.02

these kinds of experiments in studying the nature of radiation damage and in adding to knowledge of metal physics is discussed. It is concluded that electron irradiation experiments have improved understanding of the effects of a simple distribution of interstitials on the properties of metals. More experiments will be required before precise predictions of radiation effects can be made. Particular needs are for studies of displacement cross-sections as a function of incident particle energy and of the interactions between lattice defects and impurities and dislocations. (auth)

GAMMA 6.15.07.03

INVESTIGATIONS INTO THE DOMAIN OF THE RADIATION CHEMISTRY OF ORGANOMETALLIC COMPOUNDS. GAMMA RADIATIONS IN A REACTION OF METALLIC TIN WITH HALOGEN ALKYLS. L. V. Abramova, N. I. Sheverdina, and K. A. Kocheshkov (Karpov Research Inst. of Physics and Chemistry). Doklady Akad. Nauk S.S.S.R. 123, 681-4(1958) Dec. 1. (In Russian)

Interactions between γ-irradiated alkyl halides and metallic tin were studied. The reaction between alkyl bromides and tin takes place at standard temperatures with the yield up to 55 M per 100 ev, while RBr and tin form only traces of R_2SnX. The alkyl iodides react easily under irradiation with yields reaching 60 M per 100 ev. Correlations of published and obtained data show that alkyl chlorides and tin (in absence of catalyzer) do not form any tin-organic compound; under gamma irradiation or thermal interactions alkyl bromides form tin-organic compounds while alkyl iodides form tin-organic compounds in all mentioned interactions. (R.V.J.)

11149

RADIATION DAMAGES IN METALS AND SEMICONDUCTORS AFTER γ-IRRADIATION. S. Yu. Atomnaya Energiya 3, 562(1957).

The effects of gamma radiation on germanium and copper were investigated. The germanium was irradiated in the temperature range from liquid nitrogen temperature to room temperature. The irradiation of n-Ge causes a decrease of the "carrier number." After a long irradiation n-Ge is converted into p-Ge. An investigation of the temperature dependence of the Hall effect showed that two acceptor levels are formed in the n-Ge. One is 0.2 ev lower than the conductivity band and the other is 0.26 ev higher than the limit of the valence band. The irradiation of p-Ge causes a decrease of the concentration of "holes." Irradiating a copper monocrystal for 50 hours at room temperature with a gamma intensity of 4×10^{10} γ/cm^2 sec raises

6424 A/CONF.15/P/63

THE EFFECT OF IONISING RADIATION ON SOLID CATALYSTS. E. J. Gibson, R. W. Clarke, T. A. Dorling, and D. Pope (Gt. Brit. Dept. of Scientific and Industrial Research). 14p.

Some results obtained during a study of the irradiation of iron oxide catalysts with the gamma radiation from Cobalt-60 are presented. The main interest has been in the use of these catalysts for reactions involving carbon monoxide and hydrogen. It was found that after a dose of gamma radiation of the order of 1 to 5×10^7 rep, some iron oxide catalysts show a long-lived increase in activity for the synthesis of hydrocarbons from carbon monoxide and hydrogen at 280°C and 10 to 20 atm. pressure. The increased activity persists to the end of the tests, some of which lasted 300 hours. The product obtained using the irradiated catalyst did not differ significantly from that obtained using the untreated catalyst. Not all iron oxide catalysts for this reaction can be affected in this way, and some differences between the catalysts are discussed. Measurements of electrical conductivity, oxo-electron emission, and other properties were made on some of the catalysts tested, and the information so far obtained is discussed in relation to the catalytic activity of the samples. (auth)

4742 WADC-TR-56-467

Wright Air Development Center. Materials Lab., Wright-Patterson AFB, Ohio.

EFFECTS OF NUCLEAR RADIATION ON STRUCTURAL ADHESIVE BONDS. Period Covered: March 1955 to April 1956. Robert S. Arlook and Douglas G. Harvey. Aug. 1956. 52p. Project title: ANPP DEVELOPMENT SUPPORT PROJECT. Task title: SHIELDING MATERIALS AND RADIATION EFFECTS.

RADIATION EFFECTS 6.15

Metals 6.15.07

Gamma 6.15.07.03

A group of nine commercially available metal-to-metal adhesives were investigated to determine the effect of γ radiation upon their strength properties. Standard 2024-T3 alclad Al alloy lap joint specimens were prepared and three series of exposures were made at the MTR γ facility. The approximate dosages were 9×10^7, 3×10^8, and 9×10^8 r. Tensile shear and bend tests were conducted at room temperature, at 180°F, and at 260°F. Two adhesives were also tested at 500°F. Fatigue tests were performed at room temperature. Only one adhesive was essentially unaffected by γ radiation at all testing temperatures. The remaining adhesives were affected to varying degrees. (auth)

ORGANIC FLUIDS 6.15.08

See also, this section,

SOLVENT EXTRACTION--Radiation Damage to Solvents (6.16.03).

ALCOHOLS 6.15.08.01

6284 ✓
CHEMICAL ACTION OF IONISING RADIATIONS IN SOLUTION. PART XX. ACTION OF X-RAYS (200 kv) ON ETHANOL IN AQUEOUS SOLUTION. G. G. Jayson, G. Scholes, and J. Weiss (Univ. of Durham, Newcastle upon Tyne). J. Chem. Soc. 1358-68(1957) Mar.

The action of x rays (200 kv) on ethanol in aqueous solution in the presence of oxygen gave only acetaldehyde; in deaerated solutions, butane-2:3-diol was also produced. Quantitative studies have been carried out under various experimental conditions and the effects of pH and concentration of the solute have been examined. A mechanism for the radiation chemistry of aqueous-ethanol solutions in the presence and in the absence of oxygen has been proposed. The detailed study of this system has led to some new aspects regarding the modes of formation of hydrogen peroxide from OH radicals in aqueous systems. In deaerated ethanol solutions, a feature of interest is the importance of the dehydrogenation of ethanol by hydrogen atoms: $CH_3 \cdot CH_2 \cdot OH + H \rightarrow CH_3 \cdot CH(OH) + H_2$. (auth)

13033 ✓
EFFECTS OF IONIZING RADIATIONS ON POLYVINYL ALCOHOL. I. BREAKDOWN OF MAINCHAIN AND FORMATION OF CARBONYL GROUPS. Akibumi Danno (Univ. of Tokyo). J. Phys. Soc. Japan 13, 609-13(1958) June.

Polyvinyl alcohol in the form of film and powder were irradiated in air by gamma rays from a Co^{60} source, and by deuterons accelerated by a cyclotron. Weight changes, viscosity, solubility, and infrared absorption spectra of irradiated samples were measured as a function of absorbed dose. The reduction of the molecular weight of the irradiated specimens was observed suggesting that the effect was one of degradation. Infrared absorption spectra showed the formation of the carbonyl groups in the irradiated specimen. (auth)

13034
EFFECTS OF IONIZING RADIATION ON POLYVINYL ALCOHOL. II. CHANGES OF PHYSICAL PROPERTIES OF THE IRRADIATED SPECIMEN CAUSED BY HEAT TREATMENT. Akibumi Danno (Univ. of Tokyo). J. Phys. Soc. Japan 13, 614-17(1958) June.

The physical properties of polyvinyl alcohol irradiated by deuterons or gamma rays were found to change remarkably by heat treatment. Irradiation causes the decrease of the molecular weight and the increase of the carbonyl groups. When these specimens are heated above 120°C, however, the solubility diminishes considerably and even gel formation occurs for highly irradiated ones. Infrared absorption spectra revealed that the number of carbonyl groups formed by the irradiation decreased greatly as a result of heat treatment. Some discussion are given on the process of gel formation. (auth)

2032 UCRL-Trans-370
DECOMPOSITION OF METHYL ALCOHOL BY Co^{60} γ-RAYS. Y. Yizuka, Y. Ouchi, K. Hirota, and G. Kusumoto. Translated for Univ. of Calif. Radiation Lab., Berkeley from Nippon Kagaku Zasshi 78, 129-31(1957). 6p.

RADIATION EFFECTS 6.15

ORGANIC FLUIDS 6.15.08

ALCOHOLS 6.15.08.01

Deuterated methyl alcohol was decomposed by γ rays, and the deuterium composition of the resulting hydrogen gas was analyzed. From the results obtained, the possible reaction mechanism leading to the formation of the hydrogen molecule was examined, and it was concluded that the initial step was a removal of the hydrogen atom from the hydroxyl group. The resulting hydrogen atom then attacks another methyl alcohol molecule and takes a hydrogen atom from the methyl group, thus forming a hydrogen molecule. (auth)

THE CHEMICAL EFFECTS OF γ-RADIATION ON ORGANIC SYSTEMS. PART II. THE ACTION OF RADIATION ON BENZYL ALCOHOL ALONE OR MIXED WITH ACETONE OR BROMOBENZENE. G. A. Swan and D. Wright (Univ. of Durham, Newcastle Upon Tyne, Eng.). J. Chem. Soc. 4673-80 (1958) Dec.

Irradiation of benzyl alcohol with γ rays gave rise to benzaldehyde, dibenzyl, 1:2-diphenylethanol, and meso- and racemic hydrobenzoin. In the presence of acetone, the last two compounds were produced in considerably greater energy yield. The products formed by the irradiation of a mixture of benzyl alcohol and bromobenzene were also investigated. (auth)

16287

RADICAL AND MOLECULAR YIELDS IN THE γ-IRRADIATION OF LIQUID METHANOL. G. E. Adams and J. H. Baxendale (Univ. of Manchester, Eng.). J. Am. Chem. Soc. 80, 4215-19(1958) Aug. 20.

Fe(III) salts and benzoquinone are reduced during the γ-irradiation of methanol and the extent of reduction is a measure of G(radicals). These solutes also decrease $G(H_2)$, $G(CH_4)$, and G(glycol) while at the same time increasing $G(CH_2O)$. The observations are accounted for by the oxidants reacting with the radicals H, CH_2OH, and CH_3 and the results are analyzed to give G(H), $G_m(H_2)$, $G(CH_2OH)$, $G(CH_3)$, $G_m(CH_2O)$, G_m(glycol), and $G_m(CH_4)$. Appreciable differences in G(H) and $G(CH_3)$ are observed between pure methanol and methanol which is 0.1 N in sulfuric acid. (auth)

7729

RADIOLYSIS OF LIQUID METHANOL BY Co^{60} GAMMA-RADIATION. Gisuke Meshitsuka and Milton Burton (Univ. of Notre Dame, Ind.). Radiation Research 8, 285-97(1958) Apr.

3272 UCRL-3317

California. Univ., Berkeley. Radiation Lab.

THE RADIOLYSIS PRODUCTS FROM ETHYL ALCOHOL; EFFECT OF TOTAL ENERGY INPUT ON THE RADIOLYSIS PRODUCTS. Amos S. Newton and W. R. McDonell. Feb. 21, 1956. 9p. Contract W-7405-eng-48. $0.15(OTS).

Data are presented on the effect of increasing bombardment on the yields of some products formed in the helium ion irradiation of liquid C_2H_5OH. The energy input was varied from 0.029 to 2.7×10^{22} ev/ml. Hydrogen, total carbonyl products, and vicinal glycols decrease markedly with increasing bombardment. The addition of acetaldehyde or hexene-1 causes a marked decrease in the yield of H_2 but smaller changes in other products. The results are discussed in terms of mechanisms involving charge or excitation exchange and radical traps. (auth)

BENZENES 6.15.08.02

8468 NP-8361

Polish Academy of Sciences. Inst. of Nuclear Research. Warsaw.

THE POSSIBILITY OF RADIATION NITRATION OF AROMATIC HYDROCARBONS. Report No. 123/ChR. R. Broszkiewicz, S. Minc, and Z. Zagórski. Dec. 1959. 3p.

A solution of benzene in water containing calcium nitrate was irradiated with gamma rays in a closed aluminum vessel. Nitrobenzene was produced in considerable yield. Equations are proposed for the reaction. (C.J.G.)

3663 CEA-tr-R500

NATURE DU PRODUIT INSOLUBLE FORMÉ LORS DE L'OXYDATION RADIOLYTIQUE DU BENZÈNE DANS L'EAU. (Nature of the Insoluble Product Formed During the Radiolytic Oxidation of Benzene in Water.) E. V. Barelko, L. I. Kartacheva, and M. A. Proskournine. Translated from Doklady Akad. Nauk S.S.S.R. 116, 74-7(1957). 8p.

RADIATION EFFECTS 6.15

ORGANIC FLUIDS 6.15.08

BENZENES 6.15.08.02

The white suspension which is formed during the irradiation of benzene in water was studied. The suspension was separated from the solution, its molecular weight was determined by cryoscopy, the elementary composition was determined by combustion analysis, the spectrum of alcohol solutions in the ultraviolet was studied, and a colorimetric analysis of its solutions was made. The results showed that the weight of the suspension increases linearly with the irradiation dose. The spectrum has a maximum at λ = 250 to 255 mμ. The elementary analysis gave C = 74.4%, O = 16%, and H = 7 to 8%. The molecular weight was 183 ± 3. The results led to the conclusion that the suspension is probably a dioxide derivative of diphenol. The probable reaction mechanism for the radiolytic oxidation of benzene in water solutions is discussed. (J.S.R.)

7545

EFFECT OF IODINE ON THE RADIOLYSIS OF BENZENE. Robert H. Schuler (Brookhaven National Lab., Upton, N. Y.). J. Phys. Chem. 60, 381-2(1956) Mar.

The radiation yield for hydrogen production from benzene was found to be independent of iodine concentration except at very high I_2 concentrations. The mechanism of benezene radiolysis is discussed. (C.W.H.)

THE CHEMICAL EFFECTS OF γ-RADIATION ON ORGANIC SYSTEMS. PART I. THE SYSTEMS PYRIDINE-BROMOBENZENE AND ANILINE-BROMOBENZENE. G. A. Swan and P. S. Timmons (Univ. of Durham, Newcastle Upon Tyne, Eng.). J. Chem. Soc. 4669-72(1958) Dec.

Gamma irradiation of a mixture of pyridine and bromobenzene afforded 2-, 3-, and 4-phenylpyridine, as well as pyridine hydrobromide. From an irradiated mixture of aniline and bromobenzene, 2- and 4-aminodiphenyl, azophenine, and aniline hydrobromide were isolated. These reactions are apparently initiated by homolysis of bromobenzene under the influence of γ rays. (auth)

9203

CHEMICAL ACTION OF IONIZING RADIATIONS IN SOLUTION. PART XV. EFFECT OF MOLECULAR OXYGEN IN THE IRRADIATION OF AQUEOUS BENZENE SOLUTIONS WITH X-RAYS. M. Daniels, G. Scholes, and J. Weiss (Univ. of Durham, Newcastle upon Tyne, England). J. Chem. Soc., 832-4(1956) Apr.

The chemical effects of x-rays (200 kv) on aqueous benzene have been investigated further with particular reference to the nature of the products (in addition to phenol) formed in the presence of dissolved O_2. Under these conditions, small amounts of an aldehyde, probably mucondialdehyde, are produced; it is suggested that this is produced via the intermediary formation of organic peroxy-radicals. (auth)

22122

THE EFFECT OF γ-RADIATION ON MIXTURES OF BENZENE AND CARBON TETRACHLORIDE WITH FLUORINE-CONTAINING COMPOUNDS. A. V. Zimin, S. V. Churmanteev and A. D. Verina (L. Ia. Karpov Physicochemical Inst.). p.209-11 of "The Radiation Chemistry of Simple Organic Systems."

It was established that during the action of γ-radiation on mixtures of CCl_4 with inorganic fluorides and on mixtures of C_6H_6 and very simple fluorocarbons fluorination of CCl_4 and C_6H_6 occurs. The fluorination of CCl_4 proceeds by the substitution of chlorine atoms by fluorine atoms, while the presence of oxygen in the mixture increases the yield of fluorinated products. The fluorination of benzene proceeds by the substitution of hydrogen atoms by fluorine-containing radicals, which increases the number of carbon atoms in the fluorinated molecule. (W.L.H.)

637

THE CHEMICAL STATE OF F^{18} FROM THE FAST NEUTRON IRRADIATION OF FLUOROBENZENE. A. H. W. Aten, Jr., Beatrix Koch, and J. Kommandeur (Amsterdam, Holland). J. Am. Chem. Soc. 77, 5498-9(1955) Nov. 5.

Fluorobenzene, in the presence of HF (<1%), was irradiated with fast neutrons. The fraction of the resultant F^{18} activity found in the organic compound was 0.36. (C.W.H.)

RADIATION EFFECTS 6.15

ORGANIC FLUIDS 6.15.08

Benzenes 6.15.08.02

22123

THE PROSPECTS OF PREPARING HEXACHLOROCYCLOHEXANE USING IONIZING RADIATION. P. V. Zimakov, E. V. Volkova, and L. A. Krasnousov. p.213-15 of "The Radiation Chemistry of Simple Organic Systems."

Results of experiments on the chlorination of benzene under the action of various forms of radiation over the temperature range of −80 to 40°C are presented. (W.L.H.)

22098

OXIDATION OF AQUEOUS BENZENE SOLUTIONS UNDER THE EFFECT OF γ-RADIATION. E. V. Barelko, L. I. Kartasheva, P. D. Novikov, and M. A. Proskurnin (L. Ia. Karpov Physicochemical Inst.). p. 81-5 of "Radiation Chemistry of Aqueous Solutions (Inorganic and Organic Systems)."

Results are reported from an investigation of the oxidation of benzene under the action of large doses of radiation. The theory of the process is discussed. During the irradiation of aqueous solutions of benzene in vacuum both normal phenol and high-molecular phenolic compounds, which precipitated, were formed. The total benzene consumption in this case was approximately 2.0 to 2.5 molecules/100 ev, while the phenol yield was a total of 0.5 molecules/100 ev. A possible scheme is presented for the precipitate. Results are also reported on the formation of phenol in an oxygen atmosphere both in the presence and absence of ferrous ions. (C.H.)

22127

THE EFFECT OF IONIZATION DENSITY ON THE RADIOLYSIS OF HYDROCARBONS. I. V. Vereshchinskii and N. A. Bakh (Inst. of Physical Chemistry, Academy of Sciences, U.S.S.R.). p.233-5 of "The Radiation Chemistry of Simple Organic Systems."

A series of investigations devoted to the radiation-oxidation of organic compounds is presented. Benzene and cyclohexane containing molecular oxygen were sealed in ampules and irradiated in a heavy water reactor. Radiation chemical yields of benzene and cyclohexane radiolysis products are presented. (W.L.H.)

GENERAL 6.15.08.03

5271

EFFECTS OF PHASE ON REACTIONS INDUCED BY RADIATION IN ORGANIC SYSTEMS. T. O. Jones, R. H. Luebbe, Jr., J. R. Wilson, and J. E. Willard (Univ. of Wisconsin, Madison). J. Phys. Chem. 62, 9-15(1958) Jan.

This paper summarizes some of the data in the literature on the effects of phase on chemical reactions induced in organic systems by light, ionizing radiation and nuclear transformations. It also presents new data which show that: (1) the ratio of HI to I_2 produced in the radiolysis of pure isopropyl iodide with Co^{60} γ-rays is about tenfold higher in the solid phase at −190° than in the liquid at room temperature; (2) the yield of iodine from the radiolysis of isopropyl iodide in either the liquid or solid is dependent on the past history of irradiation of the sample in the other phase; (3) when the spectrum of ethyl iodide glass at −190° is examined after exposure of the solid to 2537 A. light, little or no change is observed as a result of the irradiation, but when the glass is then melted and immediately refrozen, an absorption peak appears at 3700 A.; a similar effect is caused by the self-irradiation of tritiated ethyl iodide glass; (4) the ratio of HI to I_2 produced by the photolysis of ethyl iodide is approximately ten-fold higher in the glass at −190° than in the liquid at room temperature. (auth)

CHEMICAL BEHAVIOR OF HOT ATOMS IN NUCLEAR TRANSFORMATION. An. N. Nesmeyanov and E. A. Borisov. Uspekhi Khim. 28, 133-61(1958) Feb. (In Russian)

A review is given of the current data on the hot atom chemistry in organic systems. The behavior of the product atoms from slow neutron capture, the effects of the temperature and composition on the retention of the radioactive atom in (n,γ) reactions, and the relation of the mass and initial recoil energy of the particles on the retention in elastic collisions are discussed. (R.V.J.)

A CORRELATION BETWEEN MASS SPECTRA AND RADIOLYSIS DATA. John G. Burr (Atomics International, Canoga Park, Calif.). J. Phys. Chem. 61, 1483-4(1957) Nov.

Four examples are given of an apparent similarity between the mechanisms of certain types of processes which can be observed in the data from mass spectra and in the data from liquid phase radiolysis of isotopically labeled organic molecules. The possible significance of this correlation is discussed. (auth)

RADIATION EFFECTS 6.15

ORGANIC FLUIDS 6.15.08

GENERAL 6.15.08.03

4344
RADIATION POLYMERIZATION OF LIQUIDS. Frank E. Hoecker and Ivan Watkins (Univ. of Kansas, Lawrence). Radiology 68, 257(1957) Feb.

14658 ✓ A/CONF.15/P/797
Esso Research and Engineering Co., Linden, N. J.
RADIATION INITIATED SULFOXIDATION OF HYDROCARBONS. James F. Black and Edmund F. Baxter, Jr. 10p. $0.50(OTS).

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

Mechanism studies on the gamma radiation initiated reaction of SO_2 and O_2 with hydrocarbons to form sulfonic acids are discussed. These studies are of theoretical interest because they demonstrate G values ranging as high as 7000 for certain feed stocks; show that certain types of hydrocarbons are efficient inhibitors for this reaction; reveal steric hindrance effects which throw light on the structure of the chain carrying agents, and present evidence for an unusual type of chain reaction with delayed branching which is not explosive due to inherent quenching characteristics. The studies are of practical interest since they could lead to a radiation initiated process for the manufacture of new classes of water or oil soluble detergents. Conversions

8474
CARBOXYLATIONS EFFECTED BY IONIZING RADIATION. B. C. McKusick, W. E. Mochel, and F. W. Stacey (E. I. du Pont de Nemours and Co., Wilmington, Del.). J. Am. Chem. Soc. 82, 723-5(1960) Feb. 5.

Irradiation of a mixture of a hydrocarbon and carbon dioxide with high-energy electrons gives carboxylic acids. For instance, toluene is converted to phenylacetic acid. The radiation energy is used inefficiently (G = 0.01 – 0.1). Formic acid can be used in place of carbon dioxide to obtain carboxylic acids. Irradiation of a mixture of a hydrocarbon and hydrogen cyanide leads to nitriles. (auth)

10452 ✓
HIGH TEMPERATURE RADIATION CHEMISTRY OF HYDROCARBONS. P. J. Lucchesi, B. L. Tarmy, R. B. Long, D. L. Baeder, and J. P. Longwell (Esso Research and Engineering Co., Linden, N. J.). Ind. and Eng. Chem. 50, 879-84(1958) June.

Radiation-induced cracking of pure hydrocarbons and mixtures is a long-chain reaction at 320° to 510°C. Product distributions are similar to those obtained with thermal cracking. The radiation yields (10^4 to 10^5) increase with increasing temperature, are lower in the liquid than the vapor phase, and increase at lower intensity. Radiocracking results from both cobalt-60 and pile work can be qualitatively explained by assuming the Rice-Herzfeld radical mechanism. Close agreement between calculated and predicted radiation yields indicates that atmospheric pressure radiocracking is an ordinary radical chain reaction. This gives new insight into the mechanism of thermal decomposition of hydrocarbons, which is believed to be a chain process of low chain length. (auth)

6463 A/CONF.15/P/1350
EFFECTS OF γ-RADIATION ON POLYMER REACTIONS. Seizo Okamura, Tomonobu Manabe, Shigeru Futami, Tatsuo Iwasaki, Akio Nakajima, Kenji Odan, Hiroshi Inagaki, and Ichiro Sakurada (Kyoto Univ.). 52p.

Results are reported from an investigation of the effects of gamma radiation on polymer reactions. It was concluded that the polymerization process is initiated by primary radicals released from the monomer or the solvent by irradiation. The initiation depends on the number of radicals to be formed per 100 ev (G-value) of the radical-producing substances. The G-values of mixtures of benzene-chlorbenzene and of benzene-carbon were determined. The effect of air during irradiation is discussed for nylon, polyvinyl alcohol, dimethylsiloxane oil, and polymethylmethacrylate. (C.H.)

6456 A/CONF.15/P/1287
HYDROCHLORURATION DU CAOUTCHOUC NATUREL DISSOUS SOUS L'ACTION DES RAYONNEMENTS IONISANTS. (The Addition of Hydrochlorides to Dissolved Natural Rubber under the Action of the γ Rays of Co^{60}). D. Stefanesco, Cl. Nicolau, and Eugénie Gârd (Inst. de Physique Atomique, Bucharest). 12p.

The effect of gamma radiation on natural rubber dissolved in carbon tetrachloride or monochlorobenzene

RADIATION EFFECTS 6.15

ORGANIC FLUIDS 6.15.08

GENERAL 6.15.08.03

was studied. As a result of the gamma irradiations hydrochloric rubber was obtained. Energies released by the radiation source, approximately 10^{18} to 10^{19} ev, were sufficient to bond the dissolved rubber. With these energies the reaction proceeds quantitatively to the formation of bonded hydrochloric rubber, which can not be obtained by any chemical method. (tr-auth)

7132 ✓
THE PREPARATION OF STABLE FREE RADICALS IN SOLUTION BY MEANS OF IONISING RADIATION. A. J. Swallow (Tube Investments Research Labs., Cambridge). J. Chem. Soc. 1553-5(1957) Apr.

Irradiation of a mixture of an easily reduced and an easily oxidised substance gives rise to a coupled oxidation–reduction. Methylene-blue has been irradiated in the presence of ethanol, and in strongly acid solution is reduced to the same stable semiquinone free radical as is produced by $TiCl_3$. Thionine also gives some free radical under these conditions, but attempts to prepare triphenylmethyl and diphenylpicrylhydrazyl by irradiation have not succeeded. Reasons are given. (auth)

THE RADIATION CHEMISTRY OF ORGANIC SUBSTANCES. E. Collinson (Univ. of Leeds, England) and A. J. Swallow (Univ. of Cambridge, England). Chem. Revs. 56, 471-568 (1956) June.

A review of the radiation chemistry of organic substances is presented; it includes only reactions induced by x- and γ-rays, electrons produced in machines, β-particles from nuclear disintegration, protons, deuterons, α-particles, and neutrons, giving rise to extranuclear changes. (T.R.H.)

RADIATION CHEMISTRY. F. S. Dainton (Univ. of Leeds, England). *Ann. Rev. Nuclear Sci.* 5, 213-40(1955).

Radiation chemistry of organic compounds and aqueous systems is reviewed. The theories of primary processes are discussed. 140 references.

6450 ✓ A/CONF.15/P/1215
PEROXYDATION DE COMPOSES ORGANIQUES INDUITE PAR LES RAYONNEMENTS IONISANTS. (Radiation Induced Peroxidation of Organic Compounds.) M. Durup, J. Durup, F. Kieffer, and M. Magat (Univ. of Paris). 27p.

The oxidation and peroxidation of organic compounds by gamma radiation were studied under three aspects: formation of peroxides, radiochemical degradation of high polymers in aerated solutions, and transformations by anti-oxidants of the β-naphthol type. The mechanism and kinetics for the peroxidation of cumene (isopropyl benzene) were determined. The degradation of polystyrolene in aerated solutions of CCl_4 or $CHCl_3$ by the action of gamma rays was followed. A hypothesis on the formation mechanism and the nature of the different peroxides of polystyrolene was presented. The transformation which β-naphthol undergoes during its irradiation in aerated solutions of hexane was investigated. The results of the study showed that it is possible to describe the majority of the reactions as classical radical reactions. (J.S.R.)

7099 ✓
FLUORESCENCE EFFICIENCIES OF ORGANIC COMPOUNDS. Milton Furst, Hartmut Kallmann, and Felix H. Brown (New York Univ.). J. Chem. Phys. 26, 1321-32 (1957) May.

The relative fluorescence efficiencies of a large number of organic compounds of different structures were measured in solution under high-energy and ultraviolet excitations. P/R values of the more efficient solutes under high-energy excitation are given. Where possible at room temperature the fluorescence under high energy was also determined for the compound in solid form. A considerable parallelism is found between the relative fluorescence efficiencies in solution under both modes of excitation. Oxygen effects were measured and found to be greater on the solvent than on the solute, but do not greatly influence the relative efficiencies. Changes in fluorescence produced by substituents which do not considerably alter the absorption spectrum are discussed. (auth)

4110 WADC-TR-57-465
Stanford Research Inst., Menlo Park, Calif.
EFFECTS OF HIGH-ENERGY, HIGH-INTENSITY ELECTROMAGNETIC RADIATION ON ORGANIC LIQUIDS. [Period covered] May 1956 to April 1957. E. M. Kinderman. Sept. 30, 1957. 55p. Project title: MATERIALS

RADIATION EFFECTS 6.15

ORGANIC FLUIDS 6.15.08

GENERAL 6.15.08.03

explainable in terms of the greater inductive effects possible with the branch chain and benzyl groupings. Greater radiation stability is observed with higher molecular weight compounds. This effect may be explained in terms of the mechanisms of energy absorption and disintegration by the molecule. Comparison of the radiolytic with mass spectrographic data shows some correlation between the modes of molecular disintegration induced in the two cases. The correlation is best when conditions of radiolysis are similar to those in the mass spectrograph, that is when radiolysis occurs in an essentially low pressure condition through activated species whose half-life is short and whose diffusion constants or reaction times are long. Both electron and gamma radiation was used. The former received emphasis because of its speed and convenience in the test program. Comparison of the effects of the two types of radiation in six cases shows some quantitative differences in radiolytic yield. The products identified are the same, and the same structural effects are evident. The available data do not permit definite statements of equivalence between electron and gamma effects, although this is indicated. (auth)

ANALYSIS AND EVALUATION TECHNIQUES. Task title: EFFECTS OF NUCLEAR RADIATION. Contract AF33 (616)-3738.

WADC-TR-57-141, "A Comparison of High-Energy Electron and Gamma Irradiation Effects on Organic Liquids," was issued June 26, 1957.

A series of organic esters, hydrocarbons, and miscellaneous compounds were subjected to radiation. A quantitative measurement of the amounts and kinds of radiation products produced was made. From the observed products and their amounts, some inferences were made as to the stability of these materials and the influence that their structure had on their stability. Radiolysis of organic liquids is influenced by structure. Molecules containing branch chain and benzyl groupings are more susceptible to radiolytic damage than are molecules containing only straight chain groupings. This greater damage may be

THE RADIATION CHEMISTRY OF ORGANIC SUBSTANCES. E. Collinson (Univ. of Leeds, England) and A. J. Swallow (Univ. of Cambridge, England). *Chem. Revs.* 56, 471-568 (1956).

A review of the radiation chemistry of organic substances is presented; it includes only reactions induced by x- and γ-rays, electrons produced in machines, β-particles, and neutrons, giving rise to extranuclear changes. 637 references.

22121

SOME PROBLEMS IN THE RADIATION CHEMISTRY OF MIXTURES OF ORGANIC SUBSTANCES. Kh. S. Bagdasar'ian, T. S. Nikitina, and V. A. Krongauz (L. Ia. Karpov Physicochemical Inst.). p.203-7 of "The Radiation Chemistry of Simple Organic Systems."

The deviation from radiation chemical additivity was investigated either by direct determination of the yield of primary radicals or by determination of the rate of decomposition of the substance in dilute solution under conditions allowing accurate control of secondary reactions. (W.L.H.)

6473 A/CONF.15/P/1619

THE RADIATION CHEMISTRY OF UNSATURATED HYDROCARBONS IN CHLOROFORM. THE SYSTEM CHLOROFORM-BIALLYL. I. Eliezer, H. J. G. Hayman, and G. Stein (Hebrew Univ., Jerusalem). 4p.

The reaction kinetics and the products obtained when oxygen-free chloroform solutions containing unsaturated hydrocarbon compounds are irradiated with x rays were investigated. In particular, the behavior of biallyl in chloroform was studied. It was shown that strictly zero order kinetics apply, by determination of the degree of unsaturation as a function of the total dose. In 1% solution, no evidence for chain reactions was obtained. After very high doses both double bonds of biallyl were found to have added chlorine containing radicals. The mixture of the chlorinated hydrocarbons thus produced has been studied by gas chromatography and fractional distillation. The elementary analysis of the fractions obtained is given and the formation of chlorinated teleomers discussed. (auth)

EFFECT OF RECOIL ENERGY ON THE CHEMICAL CONSEQUENCES OF NUCLEAR ACTIVATION PROCESSES. ACTIVATION OF SOLUTIONS OF IODINE IN HYDROCARBONS. Charles E. McCauley and Robert H.

RADIATION EFFECTS 6.15

ORGANIC FLUIDS 6.15.08

HALIDES 6.15.08.04

Schuler (St. Peter's Coll., Jersey City, Brookhaven National Lab., Upton, N. Y., and Mellon Inst., Pittsburgh). J. Phys. Chem. 62, 1364-8(1958) Nov.

Comparative studies of the chemical consequences of activation of the halogens by the (n,γ) and (n,2n) processes have been extended to solutions of iodine in hydrocarbons. In contrast to the alkyl iodide system where no difference in chemistry between the two modes of activation was observed, (n,2n) activation results in this case in an appreciably higher organic yield. The observed increase in organic activity is attributed to the greater local disruption caused by the more energetic recoil atoms. (auth)

7962 ✓
EFFECTS OF STRUCTURE, PRODUCT CONCENTRATION, OXYGEN, TEMPERATURE AND PHASE ON THE RADIOLYSIS OF ALKYL IODIDES. Evalyn O. Hornig and John E. Willard (Univ. of Wisconsin, Madison). J. Am. Chem. Soc. 79, 2429-34(1957) May 20.

7963 ✓
QUANTITATIVE TREATMENT OF THE ELEMENTARY PROCESSES IN THE RADIOLYSIS OF ALKYL IODIDES BY γ-RAYS. Robert J. Hanrahan and John E. Willard (Univ. of Wisconsin, Madison). J. Am. Chem. Soc. 79, 2434-40 (1957) May 20.

8341 ✓ ISC-855
Ames Lab., Ames, Iowa.
CHEMICAL EFFECTS OF PHOTONUCLEAR REACTIONS IN THE PROPYL BROMIDES. A. E. Richardson and A. F. Voigt. Dec. 1956. 86p. Contract W-7405-eng-82. $2.25(OTS).

When nuclear reactions occur in atoms in molecules chemical changes follow as a result of the recoil of the product atoms. The nature of these chemical changes was studied intensively for a number of systems but in most cases the magnitude of the recoil energy was about the same. In the present studies use was made of a much greater recoil energy than that normally used. The reaction $Br(\gamma,n)Br^*$ gives a recoil bromine atom with an energy of the order of 10^5 ev, compared to 10^2 ev for the product of the $Br(n,\gamma)Br^*$ reaction which is usually used. The chemical nature of the products resulting when the propyl bromides were irradiated with neutrons and 70-Mev gamma rays was studied by established techniques. It is possible to differentiate between the reactions which occur while the recoiling bromine atom still is highly energetic, or "hot," and those which occur after it has been slowed down to thermal energies. The products of the hot reactions show little dependence on the initial recoil energy. In the case of the thermal reactions, the higher energy recoil produces a greater number of products which are different from the original molecules. This can be attributed to the presence of a higher concentration of free radical fragments which are produced in slowing down the higher energy recoiling atom. These fragments combine with the radioactive bromine after it has reached thermal energies to form a greater variety and amount of new products. (auth)

6247
THE NON-HOMOGENEOUS REACTIONS FOLLOWING NEUTRON CAPTURE IN ORGANIC BROMIDES. Miriam Milman and P. F. D. Shaw (Clarendon Lab., Oxford). J. Chem. Soc. 1325-32(1957) Mar.

An attempt is made to treat quantitatively the kinetics of the reactions occurring between radicals and inorganic radio-bromine compounds formed near the site of neutron capture. Allowance for the dilution of the reactants by diffusion has been made by considering these to be held uniformly in a sphere of liquid whose radius increases with time. By making reasonable assumptions regarding the rates of the reactions involved, curves have been obtained which fit the experimentally determined variation of retention with bromine concentration in ethyl bromide, ethylene dibromide, and tri- and tetra-bromoethane. (auth)

6244 ✓
THE SZILARD-CHALMERS REACTION IN ETHYL BROMIDE. Miriam Milman and P. F. D. Shaw (Clarendon Lab., Oxford). J. Chem. Soc. 1303-10(1957) Mar.

The nature of the reactions following neutron capture by the bromine atom in ethyl bromide has been investigated by analyzing the products at different free bromine concentrations. The products from the processes associated

RADIATION EFFECTS 6.15

ORGANIC FLUIDS 6.15.08

HALIDES 6.15.08.04

with the slowing down of the energetic atoms consist largely of compounds which could only be produced from highly fragmented ethyl bromide molecules. By contrast, the product from the ensuing diffusion-dependent reactions consists only of ethyl bromide. (auth)

6245

THE SZILARD-CHALMERS REACTION IN ETHYLENE DIBROMIDE. Miriam Milman, P. F. D. Shaw, and I. B. Simpson (Clarendon Lab., Oxford). J. Chem. Soc. 1310-17 (1957) Mar.

The retentions of radio-bromine produced by neutron capture in purified ethylene dibromide are not reproducible. This can be largely explained by the presence of vinyl bromide which is formed, on storage and by radiolysis, by dissociation of the parent compound. When present, vinyl bromide causes increased retentions by exchanging with radio-bromine atoms produced by neutron capture; subsequent addition of bromine or hydrogen bromide gives increased yields of active tribromoethane or ethylene dibromide respectively. Analysis of the products from the neutron irradiation of bromine-ethylene dibromide mixtures, shows that the predominant diffusion-controlled reactions result in the formation of ethylene dibromide and vinyl bromide, which is subsequently saturated to give tribromoethane. (auth)

6246

THE NATURE OF THE DIFFUSION-CONTROLLED REACTIONS FOLLOWING NEUTRON CAPTURE IN ALKYL BROMIDES. Miriam Milman and P. F. D. Shaw (Clarendon Lab., Oxford). J. Chem. Soc. 1317-25(1957) Mar.

The variation of retention caused by adding 1:2-dibromoethylene to ethyl bromide, ethylene and ethylidene dibromide, and 1:1:2-tri- and 1:1:2:2-tetra-bromoethane has been investigated in an attempt to elucidate the nature of diffusion-controlled reactions undergone by radicals resulting from neutron capture in these bromides. It is concluded that a large proportion of the radio-bromine atoms, which diffuse from the site of neutron capture, is probably produced by reaction of organic radicals with HBr* and Br_2* formed by high-energy processes. (auth)

12688

RADIATION-INDUCED EXCHANGE OF CHLORINE WITH CARBON TETRACHLORIDE. John W. Schulte (Los Alamos Scientific Lab., N. Mex.). J. Am. Chem. Soc. 79, 4643-8 (1957) Sept. 5.

The chemical and virtual changes in CCl_4 containing Cl_2 under the influence of Co^{60} γ rays have been investigated. Two reactions are observed: the exchange of Cl_2 with CCl_4, and the decomposition of CCl_4 to form C_2Cl_6 and Cl_2. The rates of both reactions are independent of the chlorine concentration and directly proportional to the dose rate. The former reaction is interpreted as a measure of the "radical yield" in the system, and it is observed to occur with an efficiency corresponding to 3.5 ± 0.35 molecules of chlorine being brought into exchange with CCl_4 for every 100 e.v. absorbed. The latter reaction is interpreted as a measure of molecular yield and takes place with an efficiency of 0.80 ± 0.06 molecule of C_2Cl_6 and Cl_2 being formed for every 100 e.v. absorbed. Spontaneous exchange and exchange under the influence of sunlight and ultraviolet light also were noted. (auth)

5564

CHEMICAL ACTION OF ALPHA RAYS ON CARBON TETRACHLORIDE. W. Mund, P. Huyskens, and J. Debaisieux. Bull. classe sci. Acad. roy. Belg. 41, No. 9, 929-36(1955).

The experimental study of the chemical transformation undergone by CCl_4 in the gaseous phase under the action of α rays was undertaken, being confined however to a qualitative identification concerning one of the products (hexachloroethane). As to the chlorine, it was titrated and it was established that it is formed with an ionic yield of about 0.12. (tr-auth)

1854 HW-32405(Del.)

General Electric Co. Hanford Atomic Products Operation, Richland, Wash.

RADIATION STABILITY OF CARBON TETRACHLORIDE. R. E. Olson and K. L. Adler. July 12, 1954. Decl. with deletions Feb. 26, 1957. 14p. Contract [W-31-109-Eng-52]. $3.30(ph OTS); $2.40(mf OTS).

The decomposition of CCl_4 containing TBP exposed to γ radiation is roughly proportional to the total radiation exposure. Excessive corrosion rates due to high Cl^- ion concentrations may be expected on stainless steel equipment exposed to solutions which have been in contact with CCl_4 at high radiation intensities. Corrosion may be particularly severe in equipment operated at high temperatures and in the presence of hydrogen ion. (auth)

RADIATION EFFECTS 6.15

ORGANIC FLUIDS 6.15.08

HEXANES 6.15.08.05

A COMPARISON OF THE DECOMPOSITION OF HEXANE AND CYCLOHEXANE BY DIFFERENT TYPES OF RADIATION. Harold A. Dewhurst and Robert H. Schuler (General Electric Research Lab., Schenectady, N. Y. and Brookhaven National Lab., Upton, N. Y.). J. Am. Chem. Soc. 81, 3210-12(1959) July 5.

Hexane and cyclohexane have been irradiated with radiations having energy loss parameters from 0.02 to 5 ev/A, and the liquid products have been examined by gas chromatographic and infrared methods. In this preliminary study no change in over-all decomposition is observed which can be attributed directly to a variation in the linear energy transfer of the radiation. (auth)

5272

RADIATION CHEMISTRY OF ORGANIC COMPOUNDS. II. n-HEXANE. H. A. Dewhurst (General Electric Research Lab., Schenectady, N. Y.). J. Phys. Chem. 62, 15-20(1958) Jan.

The formation of gas and liquid products in the 800-kvp electron radiolysis of n-hexane liquid has been studied as a function of the energy absorbed, temperature and the presence of solutes. The hydrogen yield decreased with temperature to a limiting value in the solid state. The same limiting yield was attained in the presence of solutes. The yield of unsaturated product, mainly trans-vinylene was independent of temperature but was found, in some cases to decrease in the presence of solutes, notably oxygen and iodine. The yield of intermediate and dimer product was found to depend on temperature as well as the solute present. The results are discussed in terms of ion-molecule, radical and so-called molecular processes. (auth)

ELECTRON AND γ-RAY RADIOLYSIS OF n-HEXANE. H. A. Dewhurst and E. H. Winslow (General Electric Co., Schenectady, N. Y.). J. Chem. Phys. 26, 969-70(1957) Apr.

KINETIC STUDY OF THE RADIOCHEMICAL OXIDATION OF CYCLOHEXANE IN THE LIQUID PHASE. Monique Brun and Roger Montarnal. Compt. rend. 247, 2361-4(1958) Dec. 22. (In French)

The radiochemical oxidation of cyclohexane in the liquid phase proceeds by a classical chain reaction with characteristics easy to determine because of the independence between the initial step and the later steps. The law $I^{1/2}$ was verified, the activation energy of propagation was measured, certain characteristics of the chain reaction were calculated, and the effect of very radiosensitive solvents (sensitizers) was determined.

10069

ACTION OF Co^{60} RADIATION ON γ-HEXACHLOROCYCLOHEXANE AND CONSIDERATIONS ON THE RADIOSENSITIVITY. Jules Duchesne, Andre Monfils, and Joseph Depireux. Compt. rend. 243, 259-61(1956) July 16. (In French)

OTHER 6.15.08.06

10464 KAPL-1768

Knolls Atomic Power Lab., Schenectady, N. Y.

THE ULTRAVIOLET ABSORPTION SPECTRA ON DIPHENYL, MONO-ISOPROPYL DIPHENYL, AND IRRADIATED MONO-ISOPROPYL DIPHENYL. D. Dutine and J. T. Porter, II. Mar. 28, 1957. 18p. Contract W-31-109-Eng-52. $0.20(OTS).

This report presents the curves for the ultraviolet absorption spectra of diphenyl and mono-isopropyl diphenyl. The spectra of the products resulting from the irradiation of the latter are also reported. (auth)

1264 56-RL-1623

General Electric Co. Research Lab., Schenectady, N. Y.

RADIATION CHEMISTRY OF ORGANIC COMPOUNDS. I. SATURATED ALIPHATIC HYDROCARBONS. H. A. Dewhurst. Sept. 1956. 21p.

A series of n-alkane liquids, pentane to hexadecane, were irradiated with 800 kvp electrons. The H_2 yield, $G(H_2) \sim 4.9$, was found to be independent of the length of the C atom length, whereas the methane yield decreased with increase in the length of the C atom chain. Infrared analysis has shown that the unsaturation produced is largely trans vinylene with a smaller amount of vinyl. Gas-liquid partition chromatography has shown that three primary product groups are formed; a dimer group, an intermediate group between the dimer and parent, and a degradation group. These results show that C-C bond scission is an important process, contrary to conclusions based only on gas analysis. (auth)

RADIATION EFFECTS 6.15

ORGANIC FLUIDS 6.15.08

OTHER 6.15.08.04

12697
RADIATION STABILITY OF COPPER PHTHALOCYANINE. Mark T. Robinson and Gilbert E. Klein (Oak Ridge National Lab., Tenn.). J. Phys. Chem. 61, 1004(1957).

THE FORMATION OF POLYMERS DURING THE IRRADIATION OF 2,4,4-TRIMETHYL-1-PENTENE. M. Grabriel de Gaudemaris. Compt. rend. 247, 2131-4 (1958) Dec. 10. (In French)

A high dose of electron irradiation of 2,4,4-trimethyl-1-pentene produces, with a radiochemical yield G = 6.5, well-defined polymers. The speed of the polymerization is bound to the dose administered by a relation of the order of 1, the relation being verified with good precision. The dimer and trimer, easily separated, are obtained selectively. (tr-auth)

EFFECT OF ADDITIVES ON THE RADIATION INDUCED POLYMERIZATION OF ISOBUTENE. R. Worrall (Tube Investments Research Labs., Hinxton, Eng.) and A. Charlesby (Royal Military Coll. of Science, Shrivenham, Berks, Eng.). Intern. J. Appl. Radiation and Isotopes 4, 84-8(1958) Dec.

The polymerization of certain monomers under the influence of high-energy radiation is believed to proceed by an ionic mechanism. The yield is found to be greatly increased in the presence of fine powders of some metallic oxides, such as zinc oxide and silica. It is reduced by the presence of polyethylene powder, in which radicals are produced by radiation. Data on the effect of zinc oxide concentration, dose, and radiation intensity are given. The increased yield is due to an increased initiation rate. (auth)

13028
CHEMICAL ACTION OF IONISING RADIATIONS IN SOLUTION. PART XXI. THE ACTION OF ^{60}Co γ-RADIATION ON AQUEOUS SOLUTIONS OF ETHYLENE AT DIFFERENT PRESSURES. P. G. Clay, G. R. A. Johnson, and J. Weiss (Oxford Univ.). J. Chem. Soc. 2175-91(1958) June.

Cobalt-60 irradiation of water saturated with ethylene (1 atm.), in the absence of oxygen, yields aldehydes, mainly acetaldehyde and n-butyraldehyde, and hydrogen peroxide, together with an oily polymer. From water, saturated with different ethylene-oxygen mixtures, the irradiation products are: acetaldehyde, formaldehyde, glycollaldehyde, hydrogen peroxide, and relatively small amounts of an organic hydroperoxide. Their formation at different ratios of [ethylene]/[oxygen], at different pH values, and in the presence of added ferrous salt has been studied. Irradiations of aqueous ethylene-oxygen solutions at higher pressures (up to 10 atm.) showed that the products and their yields, under these conditions, are not markedly different from those at lower pressures. (auth)

HIGH-ENERGY METHYL IONS FROM PROPANE UNDER ELECTRON IMPACT. Henry E. Stanton (Argonne National Lab., Lemont, Ill.). J. Chem. Phys. 30, 1116-17(1959) Apr.

Four C_3H_8 + e reactions were studied, and each is examined to explain the formation of CH_3^+ ions possessing appreciable kinetic energies (~2 ev). A hypothesis is advanced to explain the break-up of the C_3H_8 molecule to give the high-energy of CH_3^+ ions, i.e., the recoil fragment is emitted without charge and if this fragment subsequently dissociates, the products with masses above 14 appear mainly as neutrals. (T.B.A.)

6285
PROTECTION OF SULFHYDRYL GROUPS AGAINST IONIZING RADIATION. F. E. Littman, E. M. Carr, and J. K. Clauss (Stanford Research Inst., Menlo Park, Calif.). Science 125, 737-8(1957) Apr. 19.

Results indicate that the sulfhydryl groups may be protected against ionizing radiation by masking the radiosensitive groups by specific chemical reactions and by the addition of free radical acceptors. (W.L.H.)

123 AEC-tr-3059
THE INFLUENCE OF GAMMA RAYS ON THE OXIDATION OF CETANE. M. A. Proskurnin, Yu. L. Khmel'nitskii, E. V. Barelko, A. T. Slepneva, and I. I. Melekhonova. Translated by V. Beak from Doklady Akad. Nauk S.S.S.R. 112, 886-9(1957). 7p.

RADIATION EFFECTS 6.15

ORGANIC FLUIDS 6.15.08

Others 6.15.09.05

10819 ✓ CF-56-8-198
Oak Ridge National Lab., Tenn.
RADIATION DAMAGE TO FREON. M. D. Silverman, B. O. Heston, and P. S. Rudolph. Aug. 27, 1956. 7p. Contract [W-7405-eng-26]. $1.80(ph OTS); $1.80(mf OTS).

A radiation experiment on Freon-11 was made to determine the decomposition products and their amounts under conditions approximating those anticipated for the HRT. A Van de Graaff accelerator was used for this decomposition study, as it provides a 100-fold more intense radiation source than the 1100 Curie Co^{60} source which was not available for prolonged continuous exposures and which was experimentally inadequate. (auth)

5190 ✓ NP-6232
Mine Safety Appliances Co., Callery, Penna.
IN-PILE TESTING OF MONO-ISOPROPYL BIPHENYL. Technical Report No. 55. M. J. McGoff and J. W. Maustellar. Feb. 28, 1957. 34p. Contract NObs-65426.

Mono-isopropyl-biphenyl (MIPB) was circulated at 600°F through a loop in the Brookhaven reactor and investigated for stability in a fast flux of 1.6×10^{11} neutrons/cm^2-sec. The average specific decomposition of MIPB was ~3% per 10^{17} NVT fast flux (>1.6 mev) and compares favorably with the damage rate reported for biphenyl. The gassing rate of MIPB was 15 to 20 ml STP/g of decomposition product being higher than biphenyl by ~ 50%. Hydrogen and methane were the principle decomposition gases. No fouling of heat transfer surfaces was observed. Activity level of the MIPB after 175 days exposure was 0.02 μc/g, starting from 0.10 μc/g: 50% of the initial activity was Cl^{38} (auth)

5242 ✓
RADIOCHEMICAL DEGRADATION OF DL-METHIONINE. Jaroslav Kolouseк, Jindřich Liebster, and Arnošt Babický (Academy of Sciences, Prague). Nature 179, 521-3(1957) March 9

4522 NP-7074
Rochester, N. Y. Univ.
RADIOLYSIS OF CH_3COOCH_3 AND CH_3COOCD_3 BY COBALT-60 γ-RAYS. P. Ausloos and C. N. Trumbore. [1958]. 22p. Contract AF18(600)-1528.

Results of studies on the radiolysis of liquid and gaseous CH_3COOCH_3 and CH_3COOCD_3 by Co^{60} gamma radiation are reported. Yields of the volatile radiation decomposition products were studied as a function of temperature, dose, and the presence of added radical scavengers. A comparison of the isotopic hydrogen, methane, ethane, and dimethyl ether species present in these products provides information about possible primary processes, hot radical effects, and radical disproportionation and recombination reactions within the liquid cage. Recombination of methyl radicals originating from the same parent molecule as well as abstraction reactions between radicals in the same cage are postulated to explain the non-statistical yields of CH_3CD_3 and the high yields of CH_3D and CH_3OCD_3 in the liquid phase. (auth)

445 ✓
RADIATION INDUCED CHANGES IN THE STRUCTURE OF POLYISOBUTYLENE. P. Alexander, R. M. Black, and A. Charlesby (Chester Beatty Research Inst., Institute of Cancer Research, Royal Cancer Hosp., and Atomic Energy Research Establishment, Harwell, Berks, England). Proc. Roy. Soc. (London) A232, 31-48(1955). Oct. 11.

Polyisobutylene suffers random main-chain fracture when exposed to high radiation. Identical results are produced by electron and γ radiation and the average energy absorbed per break amounts to 20 eV at 20°C. The unsaturation produced is proportional to the number of breaks. A new absorption band appears in the infra-red at

OTHER SOLIDS 6.15.09

See also, under CHEMISTRY AND PHYSICS OF IMPORTANT ELEMENTS,

PLUTONIUM--Irradiation Effects (I.28.06.03),

URANIUM--Irradiation Effects (I.43.06.03).

RADIATION EFFECTS 6.15

OTHER SOLIDS 6.15.09

GENERAL 6.15.09.01

RADIATION EFFECTS ON SELECTED STRUCTURAL MATERIALS, W. E. Bost, Nov. 1959. (TID-3538).

13624 KAPL-M-PHK-2
Knolls Atomic Power Lab., Schenectady, N. Y.
REMARKS ON THE INTERACTION OF RADIATION WITH THE WALLS OF VESSELS AS IT AFFECTS THE RADIATION CHEMISTRY OF THEIR CONTENTS. Philipp H. Klein. May 8, 1956. 8p. Contract W-31-109-Eng-52. $3.30(ph OTS); $2.40(mf OTS).

8207 NP-7316
Sylvania-Corning Nuclear Corp., Bayside, N. Y.
BIBLIOGRAPHY ON EFFECTS OF IRRADIATION ON SOLIDS. (Fifth Edition—1958). Helen C. Friedemann. 85p.

The fourth edition of this report was issued as NP-6237.

The bibliography contains 958 references, divided into three parts: theoretical aspects, effects on metals, and effects on nonmetals. Literature on biological effects, as well as on effects on liquids and gases, has not been included. It deals with metals, alloys, inorganic dielectrics, semiconductors, organic and polymeric materials, and especially the materials which are used in nuclear reactor components, such as fuel elements, moderators, coolants, control materials, and shielding. The extensive subject index at the end of the bibliography will be useful to provide ready reference to the literature. (auth)

8033 NP-5939
Penn-Texas Corp., New York.
EFFECT OF IRRADIATION ON SOLIDS. A BIBLIOGRAPHY (2ND EDITION). Helen C. Friedemann, comp. Mar. 30, 1956. 29p.

12020
THE PROBLEM OF RADIATION INFLUENCE OF SOLID BODIES. K. Lintner and E. Schmid (Univ. of Vienna). Nukleonik 1, 29-40(1958) Apr.

A survey is given of the effects of corpuscular radiation on the properties of crystalline substances. 53 references. (J.S.R.)

INVESTIGATION OF FREE RADICALS FORMED IN SOLIDS SUBJECTED TO BOMBARDMENT BY FAST ELECTRONS. Yu. N. Molin, A. T. Koritskii, N. Ya. Buben, and V. V. Voevodskii (Inst. of Chemical Physics, Academy of Sciences USSR). Doklady Akad Nauk S.S.S.R. 123, 882-3(1958) Dec. 11. (In Russian)

An attempt was made to find the short-life-time radicals in solid materials bombarded by fast electrons. Radicals with life-time period of a few minutes were determined by the electron paramagnetic resonance method. (R.V.J.)

8201 MRL-39
Watertown Arsenal. Materials Research Lab., Mass.
IRRADIATION EFFECTS ON CRYSTAL SURFACES. John J. Antal. June 1958. 14p.

A review is made of the work published or otherwise reported concerning the effects of radiation on the crystal structure of solid surfaces. The reviewed work is not extensive and is of recent origin. An analysis of the various experimental techniques which are available for surface structure studies is made with regard to their applicability in irradiation effects studies. (auth)

14765 A/CONF.15/P/679
Oak Ridge National Lab., Tenn.
RADIATION STABILITY OF NONMETALS AND CERAMICS. J. H. Crawford, Jr. and M. C. Wittels. 27p. $0.50(OTS).

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

Recent studies in the United States of the effects of radiation on nonmetallic systems with potentiality for reactor applications are surveyed. In as much as structural integrity is one of the more important practical considerations, much attention has been given the extent and nature of changes in crystal structure and density of a wide variety of refractory inorganic solids after prolonged reactor exposure. Where possible, single crystals are used but in certain cases ceramic bodies and powders have also been examined in some detail. Significant recent results are the discoveries that in several systems a crystal structure transition into a higher energy phase occurs on exposures in excess of 10^{19} fast systems. Optical absorption, magnetic susceptibility and paramagnetic resonance, properties which lend themselves to correlation with the nature of defects, have been investigated in some detail for several systems:

RADIATION EFFECTS 6.15

OTHER SOLIDS 6.15.09

GENERAL 6.15.09.01

silica, magnesia, alumina, and certain of the alkali halides. Another tool that has proved effective in studies of radiation induced defect structures is low temperature thermal conductivity. Both electronic properties and thermal conductivity indicate an inhomogeneous distribution of structural damage as a result of fast neutron bombardment. Studies have also been directed toward developing refractory fuel elements and shield materials. Thermally stable bodies containing uranium and thorium and refractory cladding materials are being evaluated with regard to structural integrity and fission gas retention in an attempt to develop elements which will operate at temperatures well above those to which current technology is limited by metallic fuels. (auth)

920 AERE-M/TN-52
United Kingdom Atomic Energy Authority. Research Group. Atomic Energy Research Establishment, Harwell, Berks, England.
SELECTED UNCLASSIFIED ABSTRACTS ON RADIATION DAMAGE IN SOLIDS (1945–1957). O. Flint, comp. June 1958. 50p.
Approximately 110 abstracted references to published literature are given. (T.R.H.)

THE EFFECTS OF RADIATION ON SOLIDS. Yu. N. Sokurskii. Atomnaya Energ. 6, 403-25(1959) Apr. (In Russian)
A review is given of reports presented at the Second International Conference on Peaceful Applications of Nuclear Energy (Geneva, 1958) on the effects of radiation on solids. Analysis is made of experimental work on the quantitative evaluation of the radiation-induced defects in materials and on the neutron radiation effects on fissionable uranium, plutonium, and their alloys. New data are presented on the effects of large burnup (up to 2 at.%) on the dimensions and shape of fabricated uranium and its alloys. Data are also presented on the effects of temperature and radiation dose as well as other parameters on the mechanical properties of steel and other construction materials. Results are given from investigations of radiation effects on the nonmetallic materials BeO, UO_2–BeO, UO_2–ThO_2, graphite, and others. (tr-auth)

THE EFFECTS OF RADIATION ON SOLIDS. H. G. Van Bueren, A. H. Cottrell, J. Heijboer, and G. Mayer. Gravenhage, Netherlands, Reactor Centrum Nederland, 1957. 77p. (In English and Dutch)
A series of four articles are presented on the effects of radiation on solids. Lattice imperfections in crystals are discussed as a general introduction to the characteristic properties of physical point and line imperfections such as can occur in irradiated metallic, ionic, and valence crystals. Ionizing radiations dissipate most of their energy in solids by exciting electrons. The effect of ionizing radiation on organic high polymers is discussed from the fundamental point of view. The effects of fast neutrons on some physical properties of graphite are described. (J.S.R.)

10635 NRL-4807
Naval Research Lab., Washington, D. C.
THE DISORDERING OF POLYATOMIC SOLIDS BY NEUTRONS. E. G. Harris. July 6, 1956. 11p. Project No. NR-665-010.
A general method is outlined for calculating the number of vacant lattice sites or interstitial atoms produced in a polyatomic solid by neutron radiation. It is assumed that the colliding atoms are in the energy range for which the orbital picture is valid and are of sufficiently low energy for no appreciable fraction of their energy loss to occur in inelastic collisions. It is found that for energies much greater than the binding energies of the atoms in the lattice, the number of displaced atoms is a linear function of the neutron energy. The fraction of the energy of an incident neutron used in the production of displacements is found to be approximately one-half and to be very insensitive to the composition of the solid. If the energy required to displace an atom is the same for the atomic species in the solid, then equal numbers of each species are displaced. The theory is also extended to include charged particle radiation. (auth)

RADIATION EFFECTS ON SOLIDS, INCLUDING CATALYSTS. Ellison H. Taylor (Oak Ridge National Lab., Tenn.). J. Chem. Educ. 36, 396-407(1959) Aug.
Resistance, damage, and ionization effects due to irradiation of solids are discussed in relation to various solids. Alterations of solids by radiolytic reactions in liquids, by simultaneous irradiation, and by influence of prior irradiation are briefly presented. Influence of prior irradiation upon catalytic activity is described in detail. (C.J.G.)

RADIATION EFFECTS 6.15

OTHER SOLIDS 6.15.09

HALIDES 6.15.09.02

13006
THE EFFECT OF PRESSURE ON COLOR CENTERS IN ALKALI HALIDE CRYSTALS. W. G. Maisch and H. G. Drickamer (Univ. of Illinois, Urbana). Phys. and Chem. Solids 5, 328-36(1958).

The absorption spectra of both x-irradiated and additively colored alkali halides were studied under pressure up to 50,000 atm. These experiments were conducted at room temperature and, with a few exceptions, only those bands occurring in or near the visible region of the spectrum were studied. Frequency shifts and bleaching effects, the latter induced both by radiation and by plastic deformation, were observed. An apparently new band, intimately related to the F center and appearing at the expense of the F band, has been studied. Several related experiments were run at 1 atm. It is shown that atmospheric oxidation is one of the mechanisms responsible for the bleaching of plastically deformed crystals. (auth)

8850 ✓
IMPERFECTIONS IN THE STRUCTURE OF NEUTRON-IRRADIATED LITHIUM FLUORIDE: SUBMICROSCOPIC CAVITIES. Marianne Lambert and Andre Guinier. Compt. rend. 244, 2791-4(1957) June 3. (In French)

Central diffusion of x rays revealed some cavities in neutron-irradiated LiF. Their dimensions and concentration were determined as a function of radiation intensity and thermal annealing. (auth)

RADIATION-INDUCED CHANGES OF DIMENSIONS, INDEX OF REFRACTION, AND DISPERSION OF LITHIUM FLUORIDE. William Primak (Argonne National Lab., Lemont, Ill.). Phys. Rev. 112, 1075-83(1958) Nov. 15.

Deuteron, electron, gamma ray, and x-ray irradiated specimens were examined by a photoelastic method and by interferometric methods. In addition to the increase in physical dimensions, there were found an increase in refractive index and an increase in dispersion, and these were measured. The results agree with the classical dispersion formulas if it is assumed that the effects are caused by a change in the refractivity of the base material due to the introduction of vacancies and holes and a change in the refractivity due to the F centers and the 450 centers, the size of the vacancies being about that of the absent ions and the oscillator strength of the F centers being near unity. (auth)

NITRATES 6.15.09.03

10026 ✓
RADIOLYSIS OF SOLID NITRATES. C. J. Hochanadel and T. W. Davis (Oak Ridge National Lab., Tenn.). J. Chem. Phys. 27, 333-4(1957) July.

4514
RADIATION DAMAGE IN LANTHANUM MAGNESIUM NITRATE CRYSTALS CONTAINING RADIOACTIVE ISOTOPES. B. Bleaney, W. Hayes, and P. M. Llewellyn (Clarendon Lab., Oxford). Nature 179, 140-2(1957) Jan. 19.

Observations were made of the paramagnetic resonance spectrum of lanthanum magnesium nitrate containing two different radioactive isotopes, Am^{241} and Pm^{147}. Data concerning the spectrum are tabulated. (B.J.H.)

Refer also to abstracts 4551 and 4766.

13791 ✓
CHEMICAL EFFECTS IN FISSION PRODUCT RECOIL. III. THE DECOMPOSITION OF POTASSIUM NITRATE. D. Hall and G. N. Walton (Atomic Energy Research Establishment, Harwell, Berks, Eng.). J. Inorg. & Nuclear Chem. 6, 288-94(1958) July.

The effect of the recoil of fission products from uranium foils into an adjacent layer of potassium nitrate was investigated. Under these conditions about 10.5 nitrite ions are formed per 100 ev of recoil energy absorbed. This is considerably greater than the values observed for other forms of radiation and the reasons for this are discussed. (auth)

OXIDES 6.15.09.04

8217 ✓
ELECTRON-BOMBARDMENT-INDUCED DISSOCIATION OF ALKALINE EARTH OXIDES. P. Wargo and W. G. Shepherd (Univ. of Minnesota, Minneapolis). Phys. Rev. 106, 694-703(1957) May 15.

RADIATION EFFECTS 6.15

OTHER SOLIDS 6.15.09

Oxides 6.15.09.04

The oxygen evolution induced by electron bombardment of alkaline earth oxides has been directly observed by means of an omegatron mass spectrometer. Threshold energies for the process have been determined as 9.5 ± 0.5 ev, 140 ± 0.5 ev, and 16.9 ± 0.5 ev for BaO, SrO, and MgO, respectively. It is found that the efficiency of the process is in all cases proportional to the square of the current density at the target. It is suggested that a two-step electron excitation in the solid is involved. In the case of SrO, the use of a radioactive isotope of strontium provided a monitor for the release of the metal from the target. The energy dependence of the evolution of oxygen and strontium suggests that there are two processes, one predominating at low energies and the other at higher energies. An evolution of oxygen at a threshold energy of approximately 5 ev was observed for a variety of targets. The behavior of this evolution suggested that a desorption process was involved. (auth)

8216

IRRADIATION DAMAGE IN SINGLE CRYSTALS OF MAGNESIUM OXIDE. F. P. Clarke (Atomic Energy Research Establishment, Harwell). Phil. Mag. (8) 2, 607-27 (1957) May.

Single crystals of magnesium oxide have been subjected to ultra-violet, x, and neutron irradiation. The bands produced by these radiations, their optical and thermal bleaching and associated luminescence are discussed. Taking into account the data of other workers, schemes for ultra-violet activation and luminescence are proposed. The ultra-violet activation is attributed to the freeing of positive holes, perhaps with exciton formation as an intermediary process, and the subsequent trapping of both electrons and positive holes at negative and positive ion vacancies. The role of the various impurities is discussed and it is concluded that they do not directly give rise to the optical absorption bands observed. The luminescence scheme assumes an optical band width of over 7.0 ev. It is then shown that the various luminescence photon energies correspond to values which are to be expected on the assumption that the optical absorption energies represent the heights of bands measured from the top of the valence band. (auth)

OTHERS 6.15.09.05

7364

FAST NEUTRON EFFECTS IN TETRAGONAL BARIUM TITANATE. M. C. Wittels and F. A. Sherrill (Oak Ridge National Lab., Tenn.). J. Appl. Phys. 28, 606-9(1957) May.

Tetragonal $BaTiO_3$ is transformed into cubic $BaTiO_3$ as a result of irradiation with fast neutrons at a temperature of approximately 100°C. Sufficient atomic displacements are produced by bombardment with an integrated fast neutron flux of 1.8×10^{20} n/cm^2 so that tetragonal single crystals (c_0 = 4.0349 A and a_0 = 3.9923 A) expand anisotropically to form perovskite-type cubic single crystals with a_0 = 4.0824 A. The irradiated material remains cubic to the lowest temperature of measurement, 78°K, showing none of the low temperature phase transitions of unirradiated $BaTiO_3$, and after annealing at 1000°C the crystal remains cubic but with a reduced lattice parameter. X-ray, thermal, and optical methods were employed in studying these effects. (auth)

12690

NEUTRON IRRADIATION OF SOME CRYSTALLINE SALTS OF PHOSPHORIC, PHOSPHOROUS, AND HYPOPHOSPHOROUS ACIDS IN VACUUM. Philip A. Sellers, Takuya R. Sato, and Harold H. Strain (Argonne National Lab., Lemont, Ill.). J. Inorg. and Nuclear Chem. 5, 31-47(1957).

The radioactive products obtained by the neutron plus gamma irradiation of phosphorus compounds depend upon the conditions of the irradiation and upon the nature of the compounds irradiated. In vacuum, where easily oxidized, neutron activation products such as elemental phosphorus or phosphine should be recoverable. The principal radioactive irradiation product of anhydrous Na_2HPO_4 was phosphate, which was accompanied by traces of phosphorus and several unidentified products. The principal radioactive irradiation products of anhydrous Na_2HPO_3 and NaH_2PO_2 were phosphite and hypophosphite accompanied by a little phosphate and phosphorus. Neither phosphine nor diphosphine was detected as an irradiation product of these salts. The principal neutron irradiation products of PH_3 were H_2 and radioactive PH_3 and P. As shown by analysis, most of the radioactive products were formed in chemically detectable quantities, but the specific activity of these products was greater than that of the irradiated substances

RADIATION EFFECTS 6.15

OTHER SOLIDS 6.15.09

Other 6.15.08.06

themselves. In vacuum, gamma rays alone scarcely altered Na_2HPO_4, but they converted Na_2HPO_3, NaH_2PO_2, and PH_2 into the same products formed by neutrons plus gamma rays. In the reactor where the gamma rays accompanying the neutrons are very intense, the final neutron irradiation products of Na_2HPO_4, Na_2HPO_3, and NaH_2PO_2 in vacuum were the primary neutron activation products, the gamma-ray alteration products of these primary activation products, and the neutron activation products of the gamma-ray modifications of the original salts. Slight alteration of the phosphate by neutrons plus gamma rays indicates that the dissipation of the nuclear energy after the absorption of a neutron does not disrupt the chemical bonds of the recoiling atoms in nearly all instances. The formation of similar products by gamma irradiation and by neutron plus gamma irradiation of phosphite and hypophosphite indicates that the gamma-induced reactions and the neutron-induced reactions have similar but not necessarily identical mechanisms. (auth)

1849 KAPL-1301
Knolls Atomic Power Lab., Schenectady, N. Y.
SOME FACTORS IN THE RESISTANCE OF CRYSTALS TO RADIATION DAMAGE. C. W. Tucker, Jr. and P. Senio. Mar. 24, 1955. Decl. Oct. 6, 1955. 17p. Contract W-31-109-Eng-52.

X-ray-diffraction studies of radiation damage in crystals indicate that the following factors are important in determining the radiation stability of a given material: homogeneity of damage, particle size, cleavage or fracture strength, grain boundaries, temperature, and bond size. (C.W.H.)

1599 KAPL-1403
Knolls Atomic Power Lab., Schenectady, N. Y.
EFFECT OF IRRADIATION ON HOT-PRESSED BORON CARBIDE. W. D. Valovage. Nov. 15, 1955. 45p. Contract W-31-109-Eng-52.

Hot-pressed boron carbide was irradiated in vacuo, in helium, and in sodium to determine the quantity of He released and the physical stability of this material after burnups as high as 36% of the B^{10} atoms. The quantity of He released, in terms of the percentage of generated He, was determined as a function of burnup and temperature. It was found that extensive physical damage occurs, beginning with loss of hardness and cracking after very low burnup and increasing with burnup until complete granulation results after 36% burnup. At each level of burnup investigated, those specimens irradiated in Na were damaged more extensively than specimens in vacuo or in He. (auth)

2925
RADIATION-INDUCED AMORPHISM IN DIAMOND. Paul W. Levy and Otto F. Kammerer (Brookhaven National Lab., Upton, N. Y.). Phys. Rev. 100, 1787-8(1955) Dec. 15.

In order to investigate the possibility that one might find evidence for amorphous carbon or graphite in diamond severely damaged by irradiation, 270-mesh diamond was annealed at 500°C for 2 hrs and then irradiated in the MTR. X-ray-diffraction patterns of both the irradiated and a control sample are shown. The pattern from the irradiated diamond shows the continuous, almost structureless pattern of an amorphous material. (B.J.H.)

6459 A/CONF.15/P/1316
OXYDATION DE LA PARAFFINE TECHNIQUE ROUMAINE SOUS L'EFFET DES RADIATIONS DU Co^{60}.
(The Oxidizing of the Paraffins under the Action of Gamma Radiation). I. Drimus (Inst. Polytechnique, Bucharest), G. Ioanid, A. Dragut, P. Vasilesco (Inst. de Recherches Chimiques, Bucharest), and V. Dumitresco (Inst. de Physique Atomique, Bucharest). 29p.

A study was made of the effects of gamma radiation on the oxidizing reaction in various types of paraffins. The oxidation experiments were made at normal pressure and at temperatures between 130 and 150°C. The optimum dose for initiating oxidation was determined. The kinetics of the reaction were studied. The separation of the reaction products, variation in the conversion percentage with the dose, and methods of determining the relation between the dose and speed of reaction were also investigated. Particular attention was given to the low-volatility reaction products. The economic aspects of the reaction were also considered. (J.S.R.)

RADIATION EFFECTS 6.15

PLASTICS AND HIGH POLYMERS 6.15.10

ADHESIVES 6.15.10.01

11755 NP-6097
Stanford Research Inst., Menlo Park, Calif.
LITERATURE SURVEY OF RADIATION OF ADHESIVES AND RELATED MATERIALS. Literature Survey Report No. 1. Report No. 2. E. M. Kinderman and S. B. Radding. June 15, 1956. 75p. SRI Project No. SU-1726. Contract AF33(616)-3632.

Papers on polymerization by irradiation and irradiation of polymers with emphasis on formation, improvement, and degradation of polyethylene by radiation are abstracted for this bibliography. This work is directed at recording the latest advances in the rapidly changing field. (T.R.H.)

5246 HW-44092
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
PLASTICS AND ELASTOMERS FOR USE IN RADIATION FIELDS. I. EFFECTS OF GAMMA IRRADIATION. Robert Harrington. Nov. 30, 1956. 41p. Contract W-31-109-Eng-52. $0.30(OTS).

The results obtained from exposing 48 plastics and elastomeric materials over a dose range of 3×10^6 r to 9×10^7 r are summarized. Spent reactor fuel elements were used as the source of γ radiation. Among the materials evaluated, a styrene-butadiene rubber (PR 408-70) appeared to be the least affected. Two acrylic materials (Acrylon BA-12 and EA-5) and two vinyl chloride materials (Vyflex L-10 White and Black) appeared almost equally as good as the styrene. Teflon was the poorest of the materials, being severely damaged at 3×10^6 r. Kel-F Elastomer and a butyl type (Hycar 2202) became soft and tacky, indicating their poor suitability for use in radiation fields. The acrylics, neoprenes, nitriles, and ethylenes appeared to be about equally damaged as classes, with some overlapping with respect to the individual materials within the classes. The silicone elastomers were generally damaged more severely, although the phenyl types were affected less. Although elongation is a sensitive property and provides a fairly good index of the degradation of the materials, a study of the effects on several properties is necessary to obtain the true condition of the materials. (auth)

12692 NP-6430
Stanford Research Inst., Menlo Park, Calif.
THE EFFECT OF NUCLEAR RADIATION ON STRUCTURAL ADHESIVES AND PLASTICS. Bimonthly Report No. 1 [for] June 7, 1957 to August 15, 1957. R. Y. Mixer. Aug. 15, 1957. 7p. SRI Project SU-2228. Contract AF-33 (616)-5369.

Some experiments to determine the effects of radiation on structural adhesives and laminating resins are described. The effects of p-quaterphenyl and triphenyl phosphate in protection of Epon VIII resin from radiation was studied. (See also NP-6244.) (T.R.H.)

GENERAL 6.15.10.02

6495 ✓ A/CONF.15/P/2294
O MEKHANIZME SSHIVANIYA POLIMERNYKH TSEPEI POD DEISTVIEM γ IZLUCHENIYA. (On the Mechanism of Cross-Linking of Polymer Chains Under Gamma-Irradiation.) A. N. Pravednikov, Ying Sheng Kang, and S. S. Medvedev (U.S.S.R.). 9p.

In studying cross-linking processes in polyethylene caused by gamma radiation over a wide range of temperature, it was established that below the vitrification temperature T_v the cross-linking rate is temperature independent. The cross-linking of polymer chains resulting from the interaction of thermal hydrogen atoms depends on the degree of mobility of polymer chain segments and the reactivity of hydrogen atoms in the main or side chains. In polymer radiolysis, transfer reactions and recombination reactions are accompanied by the conversion of the tetrahedral structure into a plane structure. This conversion is easier if the polymer chain has side chains. For polymers with side chains no change in the cross-linking rate is observed when the temperature passes T_v. Heating of straight-chain irradiated polymers produces additional cross-linking. Partial destruction is observed when polystyrene is irradiated at 135°C or when it is pre-irradiated at room temperature and then heated to 140°C. (J.S.R.)

6463 ✓ A/CONF.15/P/1350
EFFECTS OF γ-RADIATION ON POLYMER REACTIONS. Seizo Okamura, Tomonobu Manabe, Shigeru Futami, Tatsuo Iwasaki, Akio Nakajima, Kenji Odan, Hiroshi Inagaki, and Ichiro Sakurada (Kyoto Univ.). 52p.

RADIATION EFFECTS 6.15

PLASTICS AND HIGH POLYMERS 6.15.10

GENERAL 6.15.10.02

Results are reported from an investigation of the effects of gamma radiation on polymer reactions. It was concluded that the polymerization process is initiated by primary radicals released from the monomer or the solvent by irradiation. The initiation depends on the number of radicals to be formed per 100 ev (G-value) of the radical-producing substances. The G-values of mixtures of benzene-chlorbenzene and of benzene-carbon were determined. The effect of air during irradiation is discussed for nylon, polyvinyl alcohol, dimethylsiloxane oil, and polymethylmethacrylate.

13019 ✓ AERE-I/M-45
United Kingdom Atomic Energy Authority. Research Group. Atomic Energy Research Establishment, Harwell, Berks, England.
A LITERATURE SURVEY OF THE EFFECTS OF HIGH ENERGY RADIATION ON VARIOUS PLASTICS. P. R. Sewell. July 1957. 39p.
A literature survey of the effects of radiation on various plastics was prepared from about three hundred unclassified papers and reports. (J.E.D.)

6439 A/CONF.15/P/963
FORMATION OF RADICALS BY ULTRAVIOLET AND IONIZING RADIATIONS AND THEIR REACTIONS IN PLASTICS. Günther O. Schenck, Werner Meder, and Martin Pape (Univ. of Göttingen, Ger.). 30p.
Irradiation was performed on plastic films in which several component systems to be studied were dissolved. The plastics were irradiated with electrons (150 kev to 15 Mev), x rays, and γ rays, as well as with quartz-mercury ultraviolet rays. The observation of the radical formation was usually made spectrokinetically using a Beckman recording spectrophotometer DK-2. (W.L.H.)

6438 ✓ A/CONF.15/P/962
RADIATION CHEMICAL REACTIONS IN ORGANIC SYSTEMS: REACTIONS ON MACROMOLECULAR SUBSTANCES AND THE SULFINATION OF HYDROCARBONS. A. Henglein, K. Heine, et al. (Univ. of Cologne). 29p.

Graft polymers by γ radiation. Upon irradiation of a polymer in the presence of a vinyl compound by γ rays free, macro-radicals resulting from the decomposition of the polymer initiate the polymerization of the monomer. Systems investigated comprised 3 components. A definite form of a polymer dips into an "inert" fluid having no swelling or dissolving capacity for the polymer. The monomer vinyl compound is dissolved in the fluid in an adequate concentration. Before irradiation the vinyl compound slowly diffuses into the polymer. Because the γ rays affect the whole system, those parts of the polymer are grafted which contain the infiltrated monomer. Furthermore, homo-polymerization of the monomer in the solution takes place. Depending on the diffusion before irradiation, there results a grafting of the surface layer of the polymer (surface grafting) or a grafting extending to the interior of the polymer (bulk or total grafting). Oxygen cancels the homo-polymerization in the solution. Oxygen diffuses from the solution into the polymer and inhibits polymerization in a thin layer just under the surface. In this case, grafting is effected in a layer in the interior of the polymer (imbedded grafting). The kinetics of these different graft processes was studied for several systems and the properties of the resulting products were investigated. Degradation and cross-linking of macromolecules in solution: For the degradation by γ rays of the main chain of polymethylmethacrylate, different mechanisms are operative: direct and indirect radiation effects and energy transfer between excited states of the polymer and of the solvent. The prevailing mechanism depends on the solvent. The degradation may be essentially decreased either by defined mixtures of solvents or by the addition of certain compounds in very small concentration. Polystyrene in solution undergoes at the same time a degradation of the main chain and an intermolecular cross-linking. With increasing concentration of the polymer, the cross-linking increases at a higher rate than the degradation. Cross-linking depends on the solvent, i.e., "bad" solvents give an improved cross linking and vice versa. Even small amounts of substances known as inhibitors for radical chain polymerizations suppress cross-linking of polystyrene. Sulfination of organic compounds: Organic substances undergo reactions with sulfur dioxide under the influence of γ rays and high-energy electrons. Hydrocarbons, for instance, form sulfonic and sulfinic acids, sulfones, and sulfoxides. G-values for several substances undergoing reaction with sulfur dioxide were determined and also the quantities of the resulting compounds. (auth)

RADIATION EFFECTS 6.15

PLASTICS AND HIGH POLYMERS 6.15.10

GENERAL 6.15.10.02

14661 A/CONF.15/P/948
Argonne National Lab., Lemont, Ill.
RADIATION CHEMISTRY IN RIGID ORGANIC MATERIALS: THE DETECTION OF INTERMEDIATES. Max S. Matheson. 22p. $0.50(OTS).

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

A discussion of the radiation chemistry of rigid noncrystalline systems is presented. Emphasis is centered on the use of a rigid matrix to trap reactive fragments, which are identified and studied for electron spin resonance. The hyperfine structure of ESR helps identify the radical or ion produced and gives information on the electronic structure of the fragment. The materials used in the paramagnetic resonance studies included several of the simpler hydrocarbons, alcohols and ethers, methyl alkyl compounds, and various plastics. (J.R.D.)

8857 WADC-TR-56-534(Pt. II)
Stanford Research Inst., Menlo Park, Calif.
NUCLEAR RADIATION EFFECTS ON STRUCTURAL PLASTICS AND ADHESIVES: PART II. EXTENSION OF LITERATURE SURVEY. R. Y. Mixer and Shirley B. Radding. Mar. 15, 1957. 37p. Project title: ANPP SUPPORT. Task title: RADIATION EFFECTS. Contract AF33(616)-3632.

An abstract bibliography on radiation effects on polymers, polymerization, and organic materials is continued. (See also NP-6244.) (T.R.H.)

6368 ✓ A/CONF.15/P/1423
GREFFAGE DES POLYCONDENSATS SOUS L'EFFET DES RADIATIONS IONISANTES. (The Addition of Polycondensed Macromolecules Under the Action of Ionizing Radiation.) R. Mihail, J. Herşcovici, and B. Hlevca (Inst. de Recherches Chimiques, Bucarest). 14p.

A study was made of the behavior of the polycondensed macromolecules under the action of ionizing radiation (gamma rays of Co^{60}) and in the presence of various monomers. The polycondensed macromolecules studied were: polyethyleneterphthalate, the polyamides, the unsaturated polyesters, and cellulose acetate. As a monomer, use was made of acrylonitrile, styrene, methyl methacrylate, ethylene, etc. In order to facilitate the work, some monomers were tagged with C^{14}. A study was made of the rate of formation of the added copolymer, and an attempt was made at determining its position on the macromolecule. (auth)

12093
BENEFICIAL EFFECTS OF RADIATION ON POLYMERS. Arthur Charlesby (Tube Investments Research Labs., Cambridge, England). Nucleonics 14, No. 9, 82-5(1956) Sept.

The effects of radiation on physical properties of polymers, with a view to possible commercial applications, are discussed. (T.R.H.)

13794 ✓
CHEMICAL EFFECTS IN THE IRRADIATION OF POLYMERS IN THE SOLID STATE. J. Weiss (Univ. of Durham, Eng.). J. Polymer Sci. 29, 425-32(1958) June.

The action of ionizing radiations on polymers leads, in general, to degradation and, in certain cases, to some further polymerization (crosslinking). It is known, on general physical grounds, that the energy of the radiation is converted into ionization and excitation of the molecules present in the system. The question arises as to how the radiation-produced primary species enter into the mechanism of the subsequent chemical processes. It is suggested that electronic processes are of considerable importance in the radiation-induced reactions of solid polymers; this is discussed with reference to various physical and chemical data on irradiated polymers (radiation-induced electrical conductivity, paramagnetic resonance, etc.). A unified picture for the mechanism of the action of ionizing radiations is proposed, based on the zone theory of solids. (auth)

5812 ✓
STIMULATION OF BRANCHED CHAIN REACTIONS BY THE ACTION OF PENETRATING RADIATION IN THE INITIAL PERIOD OF THE DEVELOPMENT PROCESS. N. M. Emanuel. Doklady Akad. Nauk S.S.S.R. 111, 1286-9(1956) Dec. 21. (In Russian)

RADIATION EFFECTS 6.15

PLASTICS AND HIGH POLYMERS 6.15.10

GENERAL 6.15.10.02

Effects of γ radiation in stimulating the chain reaction in the oxidation of paraffin and other hydrocarbones were studied. Diagrams of stimulated oxidation curves and kinetic curves of products originated in chain reaction with degenerated branching are shown. (R.V.J.)

3374 CF-54-4-221(Del.)
Oak Ridge National Lab., Tenn.
RADIATION DAMAGE TO ELASTOMERS, LUBRICANTS, FABRICS AND PLASTICS. Apr. 15, 1954. Decl. with deletions Feb. 19, 1957. 18p. Contract [W-7405-eng-26]. $3.30(ph OTS); $2.40(mf OTS).

Elastomers, plastics, fabrics, gaskets, insulators, organic materials, and lubricants were irradiated to evaluate the changes in physical properties for various total doses in rep and to determine the usefulness of the material in the irradiated condition. (L.T.W.)

7056 ✓
ON THE MECHANISM OF RADIOCHEMICAL DETERIORATION OF POLYMERS. Adolphe Chapiro. J. chim. phys. 53, 306-7(1956) Mar. (In French).

13796 ✓
THE EFFECTS OF IONIZING RADIATION ON NATURAL AND SYNTHETIC HIGH POLYMERS. Frank A. Bovey. New York, Interscience Publishers, Inc., 1958. 297p.

A critical review is presented treating significant basic research in the field of radiation effects on polymers. The material is divided into chapters on: properties of ionizing radiations, chemical effects of ionizing radiation, general radiation chemistry of polymers, statistical treatment of radiation-induced crosslinking and scission, hydrocarbon polymers, acrylate and methacrylate polymers and miscellaneous oxygen-containing addition polymers, chloro- and fluoro-polymers, diolefin polymers, condensation polymers, and natural polymers and derivatives. (T.R.H.)

11107 ✓ NYO-7498
Pennsylvania State Univ., University Park.
EFFECT OF RADIATION ON DYNAMIC PROPERTIES OF HIGH POLYMERS. Progress Report [for] July 1, 1956 to June 30, 1957. J.A. Sauer. July 1, 1957. 25p. Contract AT(30-1)-1858. $4.80(ph OTS); $2.70(mf OTS).

Dynamic mechanical measurements, together with nuclear magnetic resonance studies and observations of specific volume, were carried out over a wide temperature range for a series of polyethylene specimens both unirradiated and irradiated. The irradiations were carried out in the Brookhaven Reactor, the Penn. State Research Reactor, and a Co^{60} source. A considerable amount of new information has been obtained and reported in the technical literature. Some investigations have also been made on various types of polyamides and polyolefins. Irradiation has been shown to be a useful tool for studying transitions in polymers, and studies on the relative effects of different types of irradiation are now in progress. (auth)

MECHANISM OF HIGH ENERGY RADIATION EFFECTS IN POLYMERS. Robert Simha and Leo A. Wall (New York Univ., New York and National Bureau of Standards, Washington). J. Phys. Chem. 61, 425-30(1957) Apr.

A series of elementary processes for cross-linking and scission reactions based on free radical intermediates in a homogeneous system are proposed. The kinetic evaluation under several approximations leads to expressions for the amounts of volatiles, unsaturation, cross-links and scissions as functions of time and intensity of radiation. The dependence of the rates on intensity is linear only in special cases. Isotopic substitution with deuterium should decrease the production of unsaturation. Hydrogen yield, on the other hand, can remain constant if certain conditions are fulfilled. The observed isotopic differences in polystyrene may indicate an additional step for the capture of atoms. This is suggested to occur through the phenyl ring. Qualitative considerations that seem to favor free radical over ion intermediates in polymers are mentioned. (auth)

103 ✓
HOW RADIATION CHANGES POLYMER MECHANICAL PROPERTIES. C. D. Bopp and O. Sisman (Oak Ridge National Lab., Tenn.). Nucleonics 13, No. 10, 51-5(1955) Oct.

RADIATION EFFECTS 6.15

PLASTICS AND HIGH POLYMERS 6.15.10

Consideration is given to mechanisms of deformation caused by radiation-induced cross linking in high polymers. Stress-strain curves are given for reactor-irradiated nylon, natural rubber, vinyl chloride acetate, lucite, and butyl rubber. (B.J.H.)

7203 NP-6642
Brooklyn. Polytechnic Inst.
RESEARCH AND DEVELOPMENT ON ORIENTED POLYMERS FOR RADIATION RESISTANT APPLICATION. Quarterly Progress Report. C. G. Overberger, F. M. Beringer, A. Borchert, E. Davidson, H. M. Gladstone, J. E. Mulvaney, and Eli M. Pearce. Mar. 1958. 34p. Project No. 7-(8-7340). Contract AF33(616)-5253.

The preparation of a number of polyvinyl cycloalkanes is presented. The preparation of polymers containing the trifluoromethyl group and the polymerization of small-ring olefins are discussed. Heterogeneous alkali metal initiated polymerization is also discussed. (For preceding period see NP-6517.) (W.L.H.)

12091
DAMAGING EFFECTS OF RADIATION ON PLASTICS AND ELASTOMERS. Robert Harrington (Hanford Atomic Products Operation, Richland, Wash.). Nucleonics 14, No. 9, 70-4(1956) Sept.

The results of experiments with gamma radiation on some 50 plastics and elastomers are tabulated and discussed. (T.R.H.)

11555 APEX-261
General Electric Co. Aircraft Nuclear Propulsion Dept., Cincinnati.
RADIATION DAMAGE TO ELASTOMERS, PLASTICS, AND ORGANIC LIQUIDS. C. G. Collins and V. P. Calkins. Sept. 1956. 245p. Contracts AF-33(038)-21102 and AT-(11-1)-171. $1.25(OTS).

Experimental data on radiation damage to elastomers, plastics, and organic liquids are compiled. Preliminary considerations, factors in radiation damage, dosage conversion, calculation methods, and relation of dosage to viscosity are included. (T.R.H.)

128
RADIATION POLYMERIZATION. Presented at International Conference on Radioisotopes in Scientific Research held Sept. 9-20, 1957 at Paris. No. UNESCO/NS/RIC/22. S. S. Medvedev. London, Pergamon Press, Ltd., 1957. 11p.

POLYETHYLENE 6.15.10.03

GAMMA-RADIATION EFFECTS ON POLYETHYLENE PROPERTIES. B. I. Saxhin, A. M. Lobanov, et al. (High Polymers Inst., Leningrad). Zhur. Tekh. Fiz. 28, 1991-8(1958) Sept. (In Russian)

Results are presented of studies of the infrared spectra, x-ray pictures, and mechanical and dielectric losses of polyethylene exposed to γ radiation. Macromolecular merging and changes in the structure were observed at 80×10^6 r, which caused mechanical and dielectric losses. (tr-auth)

4351
MELTING BEHAVIOR OF IRRADIATED POLYETHYLENE. Malcolm Dole and W. H. Howard (Northwestern Univ., Evanston, Ill.). J. Phys. Chem. 61, 137-9(1957) Feb.

The specific heat of a sample of polyethylene exposed to 50 megarep of γ radiation as well as one exposed to approximately 336 megarep of pile radiations has been measured from −20 to 140°. In the case of the 50 megarep sample a slight depression of the maximum melting temperature of about 1.5° was observed, although no flow of the polyethylene occurred above this temperature. In all other respects the specific heat of this sample was identical with that of unirradiated polyethylene. In the case of the pile irradiated sample, the crystallinity decreased 5.7%. No depression of the maximum melting point could be observed, probably because of a change in the character of the melting. The G-value for hydrogen evolution was estimated to be 3.75. (auth)

DYNAMIC MECHANICAL PROPERTIES OF IRRADIATED POLYETHYLENE. E. Butta and A. Charlesby (Univ. of Pisa, Italy and Tube Investments Research Labs., Hinxton Hall, Eng.). J. Polymer Sci. 33, 119-28(1958) Dec.

Dynamic mechanical properties of eight samples of polyethylene irradiated to various extents have been de-

RADIATION EFFECTS 6.15

PLASTICS AND HIGH POLYMERS 6.15.10

POLYETHYLENE 6.15.10.03

termined over a temperature range from −70 to +280°C and a frequency range from 10^3 to 2×10^4 cycles/sec. At room temperature, minimum values in Young's modulus and maximum values in damping factor are observed for a critical radiation dose. Three transition points T_g, T_s, and T_m are exhibited by the sound velocity-temperature curves; these may be associated with the transition from the glass state, the softening of the amorphous regions, and the melting of the crystallites, respectively. With increasing dose, T_m decreases and T_g increases, while T_s shows a minimum for a radiation dose equal to the critical value. At temperature above the melting point of the crystallites T_m, lightly crosslinked samples show rubber-like elasticity, but highly crosslinked samples show the same dynamic mechanical behavior as highly crosslinked amorphous materials. Results are discussed and explained on the basis of the partial crystallinity of polyethylene and of the changes that irradiation causes on the molecular structure. (auth)

12296

INHIBITION ON CRYSTALLIZATION IN POLYETHYLENE SUBSEQUENT TO GAMMA IRRADIATION. T. F. Williams, H. Matsuo, and M. Dole (Northwestern Univ., Evanston, Ill.). J. Am. Chem. Soc. 80, 2595-6(1958) May 20.

The hypothesis was confirmed that irradiation of polyethylene at room temperature and doses below 29×10^{26} e.v.g.$^{-1}$ causes no change in crystallinity but that on fusion and solidification subsequent to the irradiation, the crystallinity is decreased. The post-irradiation fusion also causes additional vinyl decay and vinylene growth, and it is thought that long branches and gel structure are important in inhibiting crystallization. (J.R.D.)

IRRADIATED POLYETHYLENE AND PRODUCTS THEREFROM. (to General Electric Co. (U. S. A.)). British Patent 785,505. Nuclear Eng. 3, 186(1958) Apr.

Polyethylene which has been treated with high energy electrons and, in certain applications, maintained at elevated temperatures (125 to 175°C) continuously for very long periods, has shown a degradation as evidenced by a pitted surface and excessive flow. Further, when irradiated polyethylene has remained in contact with copper for 25 to 50 hours at such or higher temperatures, a reaction appears to occur causing "greening" of the copper. A green film develops on the surface which reduces the adhesion of the polyethylene to the copper core. Incorporation of stabilizers has been insufficient. The interposition of a resinous coating protecting the copper has solved the problem.

14611 A/CONF.15/P/826

Continental Can Co., Inc., Chicago.

MODIFICATION OF POLYMERS BY IONIZING RADIATION: VULCANIZATION AND GRAFT COPOLYMER FORMATION. Robert B. Mesrobian. 10p. $0.50(OTS).

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

The preparation, properties, and application of two classes of graft copolymers obtained with the aid of gamma-rays are described; graft copolymers of polyethylene containing styrene which are converted to permeaselective ion exchange membranes having important usage for the removal of salt from water by the method of multiple chamber membrane electrodialysis and the simultaneous grafting and vulcanization of natural rubber with several vinyl monomers. The latter method not only serves to minimize the dose required to effect gelation of network chains but also enables the preparation of vulcanizates having a broad range of mechanical and chemical properties. (auth)

8260

THE RADIATION CHEMISTRY OF HYDROCARBON POLYMERS: POLYETHYLENE, POLYMETHYLENE, AND OCTACOSANE. A. A. Miller, E. J. Lawton, and J. S. Balwit (General Electric Research Lab., Schenectady, N. Y.). J. Phys. Chem. 60, 599-604(1956) May.

Polymethylene, polyethylene, and octacosane were irradiated with high-energy (800 kev) electrons and crosslinking, changes in unsaturation, and gas evolution were measured. The only type of unsaturation formed is trans-vinylene and this is produced in approximately equal yields in all three hydrocarbons. About 40% of the hydrogen evolved comes from this reaction, the remainder arising from crosslinking. Volatile hydrocarbons result from C−C scissions near the chain-ends and also at the short branches in polyethylene. The results indicate that in the unbranched hydrocarbons,

RADIATION EFFECTS 6.15

PLASTICS AND HIGH POLYMERS 6.15.10

POLYETHYLENE 6.15.10.03

polymethylene and octacosane, permanent C–C scissions do not occur at random throughout the hydrocarbon chain. In polyethylene the evidence is not as clear but the tentative

10460 ✓
EFFECT OF PILE IRRADIATION ON THE DYNAMIC MECHANICAL PROPERTIES OF POLYETHYLENE.
C. W. Deeley, D. E. Kline, J. A. Sauer, and A. E. Woodward (Pennsylvania State Univ., University Park). J. Polymer Sci. 28, 109-20(1958) Feb.

The effects of pile irradiation on the damping and resonant frequency of a "high pressure" polyethylene were investigated over a temperature range from 80 to 550°K in the audiofrequency region using an apparatus which drives rod-like specimens in their transverse modes. Using irradiation dosages which result in approximately 4% cross-linking or greater, the mechanical loss peaks found at about 165, 265, and 355°K in polyethylene are altered. Definite changes in the resonant frequency or modulus-temperature plots also

EFFECT OF TEMPERATURE UPON THE CROSS-LINKING OF POLYETHYLENE BY HIGH-ENERGY RADIATION.
R. M. Black (British Insulated Callender's Cables, Ltd., London). Nature 178, 305-6(1956) Aug. 11.

6273 NP-6599
Battelle Memorial Inst. Radiation Effects Information Center, Columbus, Ohio.
THE EFFECT OF NUCLEAR RADIATION ON METALLO-ORGANIC COMPOUNDS AND ON POLYETHYLENE. Technical Memorandum No. 3. N. J. Broadway. Feb. 15, 1958. 6p. Project No. 2133. Contract AF33(616)-5171.

16539 ✓ WAPD-P-151
Westinghouse Electric Corp. Atomic Power Div., Pittsburgh.
THE EFFECT OF GAMMA RADIATION ON THE DIELECTRIC BREAKDOWN STRENGTH OF POLYETHYLENE. W. L. Lawrence and S. Mayburg. Jan. 15, 1952. Decl. Oct. 14, 1958. 13p. Contract AT-11-1-GEN-14. $3.30(ph), $2.40(mf) OTS.

The dielectric breakdown strength of polyethylene was found to decrease during gamma irradiation. The results obtained during irradiation are in agreement with estimates made from data on the stopping of fast electrons in matter by the equation $F = -(1/e)(dE/dx)$, where F is the breakdown field, e the electronic charge and, $-dE/dx$ is the energy loss per cm for the Compton electron which initiates the breakdown avalanche. (auth)

14646 ✓ A/CONF.15/P/805
Brookhaven National Lab., Upton, N. Y.
THE CONVERSION OF HIGH ENERGY RADIATION TO STORED CHEMICAL ENERGY AND ITS UTILIZATION IN THE FORMATION OF GRAFT COPOLYMERS. D. S. Ballantine, A. Glines, and D. J. Metz. 12p. $0.50 (OTS).

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

Radiation-produced trapped free radicals in organic materials are discussed. The formation of trapped free radicals in thin films of polyethylene and their subsequent use in initiating the graft polymerization of styrene is described in detail. (T.R.H.)

14647 ✓ A/CONF.15/P/818
Northwestern Univ., Evanston, Ill.
THE RADIATION CHEMISTRY OF A TYPICAL MACROMOLECULE, POLYETHYLENE. Malcolm Dole, T. F. Williams, and A. J. Arvia. 14p. $0.50 (OTS).

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

By making a careful study of the infrared absorption spectra, the formation of ring-links or the cyclization of polyethylene in the solid state under the action of ionizing radiations was discovered. This effect as well as the formation of vinylene unsaturation is probably largely the result of ionic reactions inasmuch as the radiation yield is practically the same at liquid nitrogen temperature as at room temperature. Such an effect appears to depend upon the crystallinity of the polyethylene. A new discovery has been made concerning the influence of molecular hydrogen pressure upon the rate of hydrogen gas production during irradiation. Increase of hydrogen pressure lowers the yield, possibly due to a reaction involving chain scission and hydrogenation of the free radicals produced to form methyl groups. This and other phenomena are discussed. (auth)

RADIATION EFFECTS 6.15

PLASTICS AND HIGH POLYMERS 6.15.10

RUBBER 6.15.10.04

TECHNICAL INFORMATION FOR THE APPLICATION OF HYPALON RUBBER IN CHEMICAL SEPARATIONS PLANTS. J. H. Kleinpeter. Mar. 5, 1957. 9p. (HW-48884)

Hypalon rubber is an ozone-resistant chlorosulfonated polyethylene elastomer. The outstanding resistance of this material to O_2 and oxidizing environments prompted its evaluation for possible use in chemical separations plants. Hypalon rubber was found to be resistant to 60% HNO_3 and 50% NaOH at room temperature, but not suitable in boiling solutions of HNO_3 of any concentration. Organic solvents cause a rapid swelling and severe damage to Hypalon, and exposure to air at temperatures of 100°C and over cause the material to harden. Hypalon was not seriously affected when exposed to γ radiation below 10^8 r

Exposure to 5×10^7 r γ radiation seemed to improve its tolerance to solvents but decrease its room temperature resistance to 60% HNO_3.

1946

EFFECTS OF RADIATION ON ORGANOPOLYSILOXANES. E. L. Warrick (Mellon Inst., Pittsburgh). Ind. Eng. Chem. 47, 2388-93(1955) Nov.

Vulcanization of organopolysiloxane elastomers by radiation gave rubbers with superior high-temperature properties. The resistance of these elastomers to radiation cross linking is discussed. (C.W.H.)

7360 WADC-TR-55-58(Pt. III)
Goodrich (B. F.) Co. Research Center, Brecksville, Ohio.
A STUDY OF THE EFFECTS OF NUCLEAR RADIATIONS ON ELASTOMERIC COMPOUNDS AND COMPOUNDING MATERIALS. [Period Covered]: January 1, 1956 to December 31, 1956. John W. Born, Dwain E. Diller, and Eugene H. Rowe. Dec. 1956. 54p. Project title: ANPP DEVELOPMENT SUPPORT PROJECT. Task title: RADIATION EFFECTS. Contract AF33(616)-2308.

Research followed four main lines: the development of Anti-Rads to prevent radiation damage; infrared and mass spectral analyses to relate radiation stability to molecular structures of elastomers; stress relaxation studies to define the mechanisms of radiation damage; and measurements of dynamic mechanical properties of representative rubber compounds after irradiation. Emphasis upon applied testing of end-item materials increased. The best Anti-Rad extended the retention of tensile strength and ultimate elongation by natural rubber tread stock more than tenfold. The screening study is providing criteria for the selection of superior Anti-Rads. A new technique of mass spectral analysis promises to aid greatly in determining what molecular structural features impart radiation stability. Stress relaxation measurements detail the important role of oxygen in radiation damage. Dynamic test data are given for eight representative rubber compounds. (See also WADC-TR-55-58(Pt. II.) (auth)

12694 RIA-55-3641
Rock Island Arsenal Lab., Ill.
PHYSICAL PROPERTIES OF GAMMA RADIATION CURED ELASTOMERS. Z. T. Ossefort. [Oct. 1955]. 27p. (AD-121406).

The results obtained indicate that the treatment of vulcanized elastomers with high-energy irradiation results in degradative reactions, the severity of which depend upon the radiation dosage. Treatment of unvulcanized rubbers, on the other hand, by similar radiation produces a high state of cure in many of these rubbers. The state of cure is dependent upon the radiation exposure, and optimum cure differs for each elastomer. The vulcanizates thus obtained have excellent age resistance and appear to have a better balance between low temperature flexibility and oil resistance than similar vulcanizates obtained by conventional means. (auth)

OTHERS 6.15.10.05

γ-IRRADIATION OF POLYMETHYL METHACRYLATE AND POLYSTYRENE. L. A. Wall and D. W. Brown (National Bureau of Standards, Washington). J. Phys. Chem. 61, 129-36(1957) Feb.

The number of scissions produced in polymethyl methacrylate for a given dose of γ irradiation is decreased measurably by the presence of air, small amounts of benzene, and by lowering the temperature to −196°. Irradiation in air produced labile structures in the polymer, probably peroxides, which decompose and cause further scissions. The decomposition of these structures is accelerated by traces of t-butylcatechol, hydroquinone and dimethylaniline. The cross-linking reaction in polystyrene caused by γ irradiation is also decreased when temperatures are lowered

RADIATION EFFECTS 6.15

PLASTICS AND HIGH POLYMERS 6.15.10

OTHERS 6.15.10.05

to −196°. Studies of partially deuterated styrenes show little correlation between cross-link formation and site of deuteration. This observation, plus the fact that cross-links exceed the number of hydrogen molecules produced, suggests that the phenyl ring is involved in cross-linking. Comparison of the results with deuterated polystyrene and deuterated polyethylene indicates a difference between the two materials in the mechanisms of irradiation-induced processes. (auth)

7055

EFFECT OF γ RADIATION ON SOLID STATE POLYMERS. II. DETERIORATION OF METHYL POLYMETHACRYLATE AND CELLULOSE ACETATE. Adolphe Chapiro. J. chim. phys. 53, 295-305(1956) Mar. (In French).

The effect of γ rays on methyl polymethacrylate and cellulose acetate was studied. A progressive decrease of fusion temperature with dose was found. The study of the coloring and absorption spectra of the methyl polymethacrylate show the existence of a definite displacement of the absorption region in the ultraviolet towards the visible and the appearance of an absorption band at 5,050 A. This last disappears slowly at 20° and very rapidly at 80°. The significance of these results is discussed and the different chemical processes which might explain the radiochemical deterioration of the polymers are examined. (tr-auth)

10071

ON CERTAIN CHARACTERISTICS OF THE RADIOCHEMICAL POLYMERIZATION OF ACRYLONITRILE. A. Prevot-Bernas and J. Sebban- Danon. J. chim. phys. 53, 418-21(1956) May. (In French)

In the case of polymerization initiated by γ rays, an attempt was made to determine exactly the law relating the speed of polymerization to the radiation intensity for different degrees of advancement of the reaction. The transfer constant of acrylonitrile was also estimated. (tr-auth)

10451

CERTAIN FEATURES IN THE RADIATION DECOMPOSITION OF POLYMERS. A. B. Taubman and L. P. Yanova (Inst. of Physical Chemistry, Academy of Sciences, USSR). Doklady Akad. Nauk S.S.S.R. 118, 991-3(1958) Feb. 11. (In Russian)

Investigations were made of the gas formation and its effects on the decomposition of polytetrafluoroethylene, polymethylmethacrylate, and polyethylene polymers during fast electron beam irradiation at various temperatures. (R.V.J.)

6423 A/CONF.15/P/51

THE EFFECT OF FAST ELECTRONS AND FAST NEUTRONS ON POLYPHENYLS AT HIGH TEMPERATURES. W. G. Burns, W. Wild, and T. F. Williams (Atomic Energy Research Establishment, Harwell, Berks, Eng.). 24p.

Biphenyl, ortho-, meta-, and para-terphenyls, and a commercial mixture of these (Santowax-R) have been subjected to fast electron beams and to irradiation in BEPO at temperatures between 250 and 400°C. Gas production and polymer formation were measured, and in some cases the changes in a variety of physical properties were studied. No great differences of stability were observed among these compounds. The initial yields of gas and polymer increased with rise in temperature, the effect on gas production being more pronounced. On continued irradiation the instantaneous rate of polymer formation decreased. Striking decreases were produced by the deliberate addition of polymer, suggesting that the latter contained a protective agent. The two types of radiation did not produce equivalent effects. Differences in relative amounts of products were observed. The initial radiation yields of all products were markedly greater using pile radiation. These yields increased more rapidly with rise in temperature than did those obtained with electron irradiation. The concept that equal energy inputs from differing types of radiation cause equal damage does not seem to hold for this class of compound. (auth)

8345 HW-54994

General Electric Co. Hanford Atomic Products Operation, Richland, Wash.

IRRADIATION OF THE BIPHENYL, ORTHO-, META-TERPHENYL EUTECTIC. D. R. de Halas. Feb. 17, 1958. 14p. Contract W-31-109-Eng-52. $0.50(OTS).

RADIATION EFFECTS 6.15

PLASTICS AND HIGH POLYMERS 6.15.10

The ternary eutectic of 55 per cent ortho-, 20 per cent meta-terphenyl and 25 per cent biphenyl has been irradiated in an in-reactor facility. Some of the physical property changes during irradiation have been measured. The stability of the two terphenyls and the biphenyl towards radiolytic damage are about equal, and only slightly greater than that measured for monoisopropylbiphenyl in previous experiments. (auth)

POLYMERIZATION OF VINYL ACETATE BY HIGH ENERGY RADIATION. Seizo Okamura, Takao Yamashita and Toshinobu Higashimura (Department of Textile-chemistry, Faculty of Engineering, Kyoto Univ., Japan). Bull. Chem. Soc. Japan 29, 647-8(1956) Aug.

4346
ACTION OF GAMMA RAYS ON POLYMERS IN THE SOLID STATE. III. IRRADIATION OF POLYVINYL CHLORIDE. Adolphe Chapiro. J. chim. phys. 53, 895-902(1956) Nov.-Dec. (In French)

A study was made of the action of ionizing radiation on polyvinyl chloride in the form of filaments and transparent films. If the irradiation is made in the presence of air, the polymer filaments are degraded while in vacuum a reticulation of the polymer is observed. The radiochemical yield of this reticulation is very small. The spectra of the irradiated films show characteristic absorption bands whose position is modified if the polymer includes stabilizers. The color and the absorption spectra of the irradiated polymer are different according to whether the irradiation is made in the presence of air or in vacuum. Towards the end of the irradiation a slow evolution of the coloration and spectrum with time was ascertained. An interpretation of these observations is proposed. (tr-auth)

2529 MND-RP-1256-2
Martin Do. Nuclear Div., Baltimore.
"METALLO-ORGANIC" POLYMERS. A. J. Restanino. Dec. 1956. 4p.

Presented before First Winter Meeting American Nuclear Society, Washington, D. C., December 10-12, 1956.

The effect of gamma radiation on the rate of polymerization of metal acrylates is described. The composition and properties of some newly developed metallo-organic polymers are presented. (W.L.H.)

16988
DEGRADATION OF POLYSTYRENE IN DILUTE AERATED SOLUTION UNDER THE INFLUENCE OF GAMMA RADIATION. Jean Durup (Laboratoire de Chimie Physique, Paris). J. Polymer Sci. 30, 533-6 (1958) July. (In French)

The degradation of polystyrene in chloroform solution under the influence of γ-rays in the presence of air has been studied by a viscometric method. The kinetic analysis of the degradation velocity during and after irradiation indicates an oxidative degradation scheme, in which two parallel processes occur with two intermediates, one of a short life-time (oxygenated radical), the other relatively stable at ordinary temperatures (hydroperoxide). The results of irradiation of polystyrene in other solvents are discussed in terms of this mechanism. (auth)

3377 NP-6496
Battelle Memorial Inst. Radiation Effects Information Center, Columbus, Ohio.
THE EFFECT OF NUCLEAR RADIATION ON FLUORINATED POLYMERS IN DIFFERENT ENVIRONMENTS. R. I. Leininger. Oct. 31, 1957. 3p. Project 2133. Contract AF-33(616)-5171.

Data are presented on the irradiation of fluorinated polymers in air, argon, and jet turbine oil at 400°F by Co^{60} radiation. (W.L.H.)

9892 HW-35284
Hanford Atomic Products Operation, Richland, Wash.
FLEXURE TESTS ON IRRADIATED TEFLON BELLOWS. P. B. McCarthy. Feb. 1, 1955. Changed from OFFICIAL USE ONLY Feb. 2, 1956. 4p. Contract W-31-109-Eng-52. $1.80(ph OTS); $1.80(mf OTS).

Although the published literature states that teflon is not damaged by radiation exposures below 10^6r, the results of flexure tests indicate that the radiation exposure limit for teflon bellows which are to be subjected to continuous flexing conditions is approximately 10^5r. Below this radiation exposure, other effects such as machining tolerance, teflon purity, and stress assume more importance to the life expectancy of the bellows than the effects of radiation. (auth)

RADIATION EFFECTS 6.15

SURVEYS AND THEORY 6.15.11

BIBLIOGRAPHIES 6.15.11.01

Armed Services Technical Information Agency Reference Center, Library of Congress, Washington, D. C.
THE EFFECTS OF RADIATION ON MATERIALS: A REPORT BIBLIOGRAPHY. Feb. 1956. 17p. (AD-89451; ARC-3694).

This bibliography is a reproduction of catalog cards covering government-sponsored research on the effects of radiation on alkali halide crystals, crystals and semiconductors, glass and optical materials, metals and alloys, organic compounds, packaging materials, photographic film and emulsions, plastics and elastomers, and miscellaneous materials. Approximately 117 references are included.

13022 NP-7509
Battelle Memorial Inst. Radiation Effects Information Center, Columbus, Ohio.
MONTHLY ACCESSION LIST 22 [ON RADIATION EFFECTS DATA]. Apr. 15, 1959. 44p. Project No. 2133. Contract AF33(616)-5171.

5100 NP-7199
Battelle Memorial Inst. Radiation Effects Information Center, Columbus, Ohio.
MONTHLY ACCESSION LIST NO. 19 [ON RADIATION EFFECTS DATA]. Jan. 15, 1959. 17p. Project No. 2133. Contract AF33(616)-5171.

3368 NP-7100
Battelle Memorial Inst. Radiation Effects Information Center, Columbus, Ohio.
MONTHLY ACCESSION LIST NO. 18 [ON RADIATION EFFECTS DATA]. Dec. 15, 1958. 33p. Project No. 2133. Contract AF33(616)-5171.

17871 NP-6936
Battelle Memorial Inst. Radiation Effects Information Center, Columbus, Ohio.
MONTHLY ACCESSION LIST NO. 15 [ON RADIATION EFFECTS DATA]. Sept. 15, 1958. 30p. Project No. 2133. Contract AF33(616)-5171.

710 NP-7987
Battelle Memorial Inst. Radiation Effects Information Center, Columbus, Ohio.
MONTHLY ACCESSION LIST NO. 28 [ON RADIATION EFFECTS DATA]. Oct. 15, 1959. 19p. Project No. 2133. Contract AF33(616)-6564.

13789 NP-6837
Battelle Memorial Inst. Radiation Effects Information Center, Columbus, Ohio.
MONTHLY ACCESSION LIST NO. 13 [ON RADIATION EFFECTS DATA]. July 15, 1958. 20p. Project No. 2133. Contract AF33(616)-5171.

The accession list includes the following categories: general background; electrical and electronic systems; polymeric materials; fuels, lubricants, and hydraulic fluids; ceramic materials; metals and alloys; organic chemicals; facilities; dosimetry; and a miscellaneous section. (J.R.D.)

12011 NP-6786
Battelle Memorial Inst. Radiation Effects Information Center, Columbus, Ohio.
MONTHLY ACCESSION LIST NO. 12 [ON RADIATION EFFECTS DATA]. June 15, 1958. 19p. Project No. 2133. Contract AF33(616)-5171.

A compilation of articles which, in most cases, have been abstracted is presented. The list is divided into articles concerning: general background, electrical and electronic systems, polymeric materials, fuels, lubricants and hydraulic fluids, ceramic materials, metals and alloys, inorganic chemicals, organic chemicals, facilities, experimental techniques, and a miscellaneous section. (J.R.D.)

11146 NP-6766
Battelle Memorial Inst. Radiation Effects Information Center, Columbus, Ohio.
MONTHLY ACCESSION LIST NO. 11 [ON RADIATION EFFECTS DATA]. May 15, 1958. 25p. Project No. 2133. Contract AF-33(616)-5171.

7574 NP-6638
Battelle Memorial Inst. Radiation Effects Information Center, Columbus, Ohio.
ACCESSION LIST NO. 9 [ON RADIATION EFFECTS DATA]. Mar. 15, 1958. 40p. Project No. 2133. Contract AF33 (616)-5171.

RADIATION EFFECTS 6.15

SURVEYS AND THEORY 6.15.11

BIBLIOGRAPHIES 6.15.11.01

7573 NP-6627
Battelle Memorial Inst. Radiation Effects Information Center, Columbus, Ohio.
ACCESSION LIST NO. 8 [ON RADIATION EFFECTS DATA]. Feb. 15, 1958. 51p. Project No. 2133. Contract AF33(616)-5171.

5078 NP-6560
Battelle Memorial Inst. Radiation Effects Information Center, Columbus, Ohio.
ACCESSION LIST NO. 7 [ON RADIATION EFFECTS DATA]. Jan. 15, 1958. 35p. Project No. 2133. Contract AF33(616)-5171.

1759 NP-6461
Battelle Memorial Inst. Radiation Effects Information Center, Columbus, Ohio.
ACCESSION LIST NO. 4 [ON RADIATION EFFECTS DATA]. Oct. 15, 1957. 57p. Project No. 2133. Contract AF33(616)-5171.

657 NP-6449
Battelle Memorial Inst. Radiation Effects Information Center, Columbus, Ohio.
ACCESSION LIST NO. 3 [ON RADIATION EFFECTS DATA]. Sept. 15, 1957. 40p. Project No. 2133. Contract AF33(616)-5171.

11473 ✓ NP-6405
Battelle Memorial Inst. Radiation Effects Information Center, Columbus, Ohio.
ACCESSION LIST NO. 2 [ON RADIATION EFFECTS DATA]. Aug. 15, 1957. 32p. Project No. 2133. Contract AF33(616)-5171.

11472 ✓ NP-6391
Battelle Memorial Inst. Radiation Effects Information Center, Columbus, Ohio.
ACCESSION LIST NO. 1 [ON RADIATION EFFECTS DATA]. July 15, 1957. 12p. Project No. 2133. Contract AF33(616)-5171.

5742 SC-4046(M)(Add. 5)
[Sandia Corp., Albuquerque, N. Mex.].
UNCLASSIFIED PORTION OF RADIATION EFFECTS BIBLIOGRAPHY. Addendum 5. Dec. 1, 1957. 7p. $1.80(ph OTS); $1.80(mf OTS).

5741 SC-4046(M)(Add. 4)
[Sandia Corp., Albuquerque, N. Mex.].
UNCLASSIFIED PORTION OF RADIATION EFFECTS BIBLIOGRAPHY. Addendum 4. Sept. 1, 1957. 8p. Contract [AT(29-1)-789]. $1.80(ph OTS); $1.80(mf OTS).

5740 SC-4046(M)(Add. 3)
[Sandia Corp., Albuquerque, N. Mex.].
UNCLASSIFIED PORTION OF RADIATION EFFECTS BIBLIOGRAPHY. Addendum 3. Aug. 1, 1957. 6p. Contract [AT(29-1)-789]. $1.80(ph OTS); $1.80(mf OTS).

6959 ✓ SC-4046(M)
Sandia Corp., Albuquerque, N. Mex.
UNCLASSIFIED PORTION OF RADIATION EFFECTS BIBLIOGRAPHY. S. T. Landrith, comp. Mar. 1, 1957. 52p. Contract [AT-(29-1)-789]. $9.30(ph OTS); $3.60 (mf OTS).

5667 ✓ NP-6237
Penn-Texas Corp., New York and Martin (Glenn L.) Co., Baltimore.
EFFECT OF IRRADIATION ON SOLIDS: A BIBLIOGRAPHY. Fourth Edition. Helen C. Friedemann, comp. Mar. 1957. 65p.
The Second Edition of this bibliography was issued as NP-5939.

19654 ✓ NP-7825
Battelle Memorial Inst. Radiation Effects Information Center, Columbus, Ohio.
MONTHLY ACCESSION LIST NO. 26 [ON RADIATION EFFECTS DATA]. Aug. 15, 1959. 25p. Project No. 2133. Contract AF33(616)-5171.

20589 SC-4046-B(M)
Sandia Corp., Albuquerque, N. Mex.
UNCLASSIFIED PORTION OF RADIATION EFFECTS BIBLIOGRAPHY. S. T. Landrith, comp. Dec. 1, 1958.

RADIATION EFFECTS 6.15

SURVEYS AND THEORY 6.15.11

BIBLIOGRAPHIES 6.15.11.01

101p. Contract AT-(29-1)-789. $15.30(ph), $5.40(mf) OTS. Supersedes SC-4046A(M).

A bibliography is presented of all unclassified documents and all photostats of periodical literature collected in the Radiation Effects Program File of the Radiation Effects Section. Approximately 1600 references. (D.E.B.)

SURVEYS 6.15.11.02

12533

PROCEEDINGS OF THE SECOND UNITED NATIONS INTERNATIONAL CONFERENCE ON THE PEACEFUL USES OF ATOMIC ENERGY, HELD IN GENEVA, 1 SEPTEMBER–13 SEPTEMBER 1958. VOLUME 29. CHEMICAL EFFECTS OF RADIATION. Geneva, United Nations, 1958. 481p. $14.50.

The papers presented at this session of the conference were on the following subjects; irradiation of water and aqueous solutions, irradiation of organic substances and solutions, irradiation of polymers, irradiation of various materials, general studies, utilization of fission product energy, and radiation sources. (W.L.H.)

9332 AD-201416

Netherlands. Technisch Documentatie Centrum voor de Krijgsmacht, Hague.

INVLOED VAN RADIOACTIEVE STRALING OP MATERIALEN. (Effect of Radioactive Radiation on Materials.) J. van Galen, ed. June 10, 1958. 48p. (TDCK-13939).

A survey is presented on the changes in properties caused by radiation. The physical background of radiation damage is briefly discussed, and radiation effects are illustrated with lattice faults in crystals. A description of the various radiation effects, the various kinds of radiation, and their effects on a number of materials is given. Abstracts are presented of articles dealing with radiation effects on metals, reactor materials, chemical compounds, electrical components, semiconductors, and photographic materials. (J.S.R.)

CHEMISCHE REAKTIONEN IONISIERENDER STRAHLEN (RADIATION CHEMISTRY). (Chemical Reactions of Ionizing Radiation (Radiation Chemistry). Hermann Mohler, ed. Frankfurt am Main, Verlag H. R. Sauerlander and Co., 1958. 299p.

A series of lectures given at the University of Basel on production of chemical reactions by high energy radiation and sponsored by several industrial firms has been expanded into a book for the chemist and methods engineer. The first chapter is an introduction to the reactions of high-energy radiation with matter. Radiation sources is the subject of the second chapter with a consideration of Van de Graaff generators, linear accelerators, and reactors and radioactive sources. The third chapter, on dosimetry, discusses conventional, practical, chemical, and calorimetric dosimetry. The fundamentals of chemical radiation effects are reviewed in the fourth chapter. In the fifth chapter effects of atomic radiation on polymers are considered. Radiation-induced reactions of potential industrial importance are reviewed in the final chapter. (J.S.R.)

SYMPOSIUM ON RADIATION EFFECTS ON MATERIALS. VOLUME I. Presented at the Second Pacific Area National Meeting, Los Angeles, Calif., September 20, 1956. ASTM Special Technical Publication No. 208. Philadelphia, American Society for Testing Materials, 1957. 194p. $4.75.

Sixteen papers are presented in this volume. They cover the subjects of theory, radiation facilities and mechanics of testing, fuel and graphite materials, and structural materials including organics. (W.L.H.)

SYMPOSIUM ON RADIATION CHEMISTRY. Academy of Sciences of the USSR, Division of Chemical Science, Moscow, 1955. N. A. Bakh, ed. New York, Consultants Bureau, 1956. 229p. $100.00.

Papers devoted to the results of experimental investigations on radiation chemistry, carried out in the USSR in recent years, are presented. The papers are divided into two parts: those relating to transformations in aqueous solutions and those relating to transformations in organic substances. (J.S.R.)

RADIATION EFFECTS 6.15

SURVEYS AND THEORY 6.15.11

SURVEYS 6.15.11.02

SYMPOSIUM ON RADIATION EFFECTS ON MATERIALS. VOLUME 3. Presented at the Sixty-first Annual Meeting, American Society for Testing Materials, Boston, Mass., June 24, 1958. ASTM Special Technical Publication No. 233. Philadelphia, American Society for Testing Materials, 1958. 168p. $4.25.

Twelve papers are presented in this volume. They cover the subjects of dosimetry, irradiation facilities and techniques, and studies on radiation effects. (W.L.H.)

1758 NARF-57-19T(Vol. 4)
Convair, Fort Worth, Tex.
FIRST SEMIANNUAL 125A RADIATION EFFECTS SYMPOSIUM, MAY 22-23, 1957. 187p. (FZK-9-118(Vol. 4)).

Topics discussed include: a description of the Battelle Radiation Effects Information Center; the effects of high-energy, high-intensity radiation on organic fluids; the effects of nuclear radiations on rubber-like materials; the effects of radiation on semi-conductors; the effects of irradiation on the performance of electronic equipment; the design of electronic systems to operate in a nuclear environment; the performance of closed circuit television equipment for remote viewing in high radiation fields; the effects of radiation on the performance of commercial electric motors insulated with Alkanex; the effects of radiation on the stability of operating components of a jet engine control system; and determination of the body radiation dose received while working in a multiple source radiation field typical of that encountered in radioactive systems maintenance. (C.H.)

THE EFFECTS OF RADIATION ON MATERIALS. J. J. Harwood, Henry H. Hausner, J. G. Morse, and W. G. Rauch, eds. New York, Reinhold Publishing Corporation, 1958. 361p.

Papers given at a Colloquium on "The Effects of Radiation on Materials," sponsored by the Office of Naval Research jointly with the Martin Co., at Johns Hopkins Univ., Baltimore, March, 1957, are presented. These papers deal with metals, alloys, inorganic dielectrics, semiconductors organic and polymeric materials, and materials for reactor components including fuel elements, moderators, coolants, and shielding materials. Theories and concepts of radiation effects, radiation sources, and measurements of radiation, and the known effects of radiation on the physical, metallurgical, mechanical, corrosion, and electrical properties are presented. (W.D.M.)

RADIATION CHEMISTRY. F. S. Dainton (Univ. of Leeds, England). *Ann. Rev. Nuclear Sci.* 5, 213-40(1955).

Radiation chemistry of organic compounds and aqueous systems is reviewed. The theories of primary processes are discussed. 140 references.

22080
PRIMARY ACTS IN RADIATION CHEMICAL PROCESSES. Part I of Proceedings of the First All-Union Conference on Radiation Chemistry, Moscow, 1957. (In English Translation). New York, Consultants Bureau, Inc., 1959. 31p. $25.00.

Topics discussed include the first stages of radiation chemical reactions in gases, investigation of the ion-molecular elementary act of a radiation chemical process, space-time distribution of radicals and yield of molecular products in water radiolysis in the presence of acceptors, electron paramagnetic resonance of radicals obtained from H_2O and H_2O_2, and the kinetics and mechanisms of nitrogen oxidation under electron impact. (C.H.)

THEORY 6.15.11.03

8398 ✓ JLI-650-3-7
Johnston (William H.) Labs., Inc., Lafayette, Ind.
ION-MOLECULE REACTIONS. H. M. Rosenstock, C. R. Mueller, M. B. Wallenstein, Marvin L. Vestal, Audrey Tory, Diana Rivers, and W. H. Johnston. Oct. 1959. 99p. Contract AT(11-1)-650. OTS.

Experimental techniques for studying ion–molecule reactions are discussed. A survey of all reactions previously reported is presented, and some qualitative observations concerning these processes are made. The theory of microscopic and macroscopic cross section calculations is considered. Elastic scattering, the potential function, ion clustering, and the decomposition of a polyatomic ion–molecule complex along competing reaction paths are theoretically treated. The role of ion–molecule reactions

RADIATION EFFECTS 6.15

SURVEYS AND THEORY 6.15.11

THEORY 6.15.11.03

in irradiated gaseous systems is discussed relative to hydrogen reactions, the radiolysis of acetylene, methane, and ethylene, the polymerization of terminal olefins and isobutene, and the oxidation of nitrogen. Application of the theory of ion—molecule reactions to advances in radiation chemistry and in high-level radiation technology is discussed. (C.J.G.)

16284
RADIATION CHEMISTRY. Milton Burton (Univ. of Notre Dame, Ind.). Record. Chem. Progr. (Kresge-Hooker Sci. Lib.) 19, 13-21(1958).
The development of radiation chemistry is reviewed. Illustrative examples cited include the following: the radiation chemistry of water, hydrocarbons, and other organic compounds; radiation-induced cross-linking of polymers; and the radiation-induced nitrogen—oxygen reaction. (C.H.)

4334
RADIATION CHEMISTRY. C. J. Hochanadei and S. C. Lind (Oak Ridge National Lab., Tenn.). Ann. Rev. Phys. Chem. 7, 83-106(1956).
Studies of reactions induced by absorption of high-energy radiation are reviewed with special emphasis on mechanisms. (T.R.H.)

7551
RADIATION CHEMISTRY. F. S. Dainton (Univ. of Leeds, England). Ann. Rev. Nuclear Sci. 5, 213-40(1955).
Radiation chemistry of organic compounds and aqueous systems is reviewed. The theories of primary processes are discussed. 140 references. (C.W.H.)

BASIC MECHANISMS. D. S. Billington (Oak Ridge National Lab., Tenn.). Nucleonics 14, No. 9, 54-8(1956) Sept.
Fast neutrons and fission fragments produce 5 types of changes in solids: vacancies, interstitial atoms, very small transient high temperature regions or thermal spikes, impurity atoms, and ionization. The effects of β- and γ-radiation are trivial in comparison. Only qualitative information is available since generalities are few and research facilities unavailable. (T.R.H.)

14649 A/CONF.15/P/916
University of Notre Dame, Notre Dame, Ind.
A CONSIDERATION OF ELEMENTARY PROCESSES IN RADIATION CHEMISTRY. Milton Burton, William H. Hamill, and John L. Magee. 21p. $0.50(OTS).
Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.
Mechanistic details of chronology and geometry of energy absorption and dissipation and their chemical consequences as influenced by composition, physical state, and density are discussed. An adequate kinetic mechanism must account for all intermediate species and events. The immediate effects produced by charged particles involve energy loss by electrons, their reaction with molecules or positive ions, ion-molecule reactions and charge neutralization processes. The consequences of electronic excitation include migration, transfer and

8344
ON CERTAIN REACTION-MECHANISMS OF HOT ATOMS. I. G. Campbell (Inst. of Nuclear Research, Warsaw). Nukleonika 3, 43-52(1958). (In Polish)
An account is given of some important features of hot atom reactions with atoms and molecules of the environment. An analysis is made of Libby's billiard ball hypothesis, and other theories of chemical bonds by hot, epithermal, and cooled atoms are outlined. (tr-auth)

7049 A/CONF.15/P/998
ON THE THEORY OF RADIATION DAMAGE AND RADIATION HARDENING. Alfred K. Seeger (Max-Planck-Inst. für Metallforschung, Stuttgart and Technischen Hochschule, Stuttgart). 52p.
A theory of radiation damage of nonfissionable material is developed, which attempts mainly to explain the phenomena of radiation hardening. It is shown that these can be accounted for quantitatively in the following way: each fast knock-on atom creates a disturbed "zone" at the end of its trace; these zones harden the material, since they have to be cut by dislocation lines; and this gives the observed temperature dependence of the critical shear stress of neutron-irradiated copper crystals and also the observed annealing behavior of radiation hardening. The recovery of electrical resistivity after irradiation is interpreted as follows (using copper as an example): the low temperature recovery (at about 40°K) after neutron bombardment is due to atomic rearrangements in the "zones," whereas after electron bombardment it is due to the recombination of

RADIATION EFFECTS 6.15

SURVEYS AND THEORY 6.15.11

THEORY 6.15.11.03

6565 AEC-tr-2891
THEORY OF LATTICE DEFECTS. II. IRRADIATION OF SOLID CRYSTALLINE MATERIALS WITH BEAMS OF PARTICLES ("RADIATION DAMAGE"). A. Seeger. Translated from Handbuch der Physik. Crystal Physics, Pt. 1, 7, 431-50(1955). 20p.
A study of the effects of fast neutrons and other fast particles on the mechanical properties of solid bodies is presented. (W.L.H.)

6733
FUNDAMENTALS OF THE PHYSICAL RADIATION EFFECT ON SOLID BODIES. Rudolf Sizmann (Technische Hochschule, Munich). Nukleonik 1, 357-68(1959) Dec. (In German)
A summary is made of the present knowledge on the effects of radiation on solids. Consideration is given to primary effects, calculation of the number of Frenkel defects, displacement, displacement energy, ionization range, formation of foreign atoms, perturbation range, collision chain in crystal lattice, and reformation of lattice defects. 73 references. (J.S.R.)

5668
NUCLEAR RADIATION EFFECTS IN SOLIDS. H. Brooks (Harvard Univ., Cambridge, Mass.). Ann. Rev. Nuclear Sci. 6, 215-76(1956).
A review is given of the status of the theory of atomic displacements, with special emphasis on recent modifications and experimental tests of the theory. Data are included on radiation effects in graphite and uranium. Reviews are also included on work with metals and alloys, valence semiconductors, other valence crystals, and ionic crystals. 210 references. (B.J.H.)

7534 AERE-C/R-1575(1), AERE-C/R-1575(2) and AERE-C/R-1575(3)
Gt. Brit. Atomic Energy Research Establishment, Harwell, Berks, England.
SELECTED ABSTRACTS OF ATOMIC ENERGY PROJECT UNCLASSIFIED REPORT LITERATURE IN THE FIELD OF RADIATION CHEMISTRY and BIBLIOGRAPHY OF THE PUBLISHED LITERATURE. (Papers Noted up to March 1956). PART 1. THEORY, INTERPRETATIONS, WATER AND AQUEOUS INORGANIC SYSTEMS. PART 2. ORGANIC COMPOUNDS (INCLUDING POLYMERISATION REACTIONS). PART 3. GASEOUS SYSTEMS (EXCLUDING ORGANIC COMPOUNDS). R. W. Clarke, comp. (Papers Noted up to Dec. 1955 (Pt. 1). Jan. 1956. 98p.; Papers Noted up to Mar. 1956 (Pt. 2). Mar. 1956. 226p.; and (Pt. 3). Mar. 1956. 60p.).
These three parts were issued separately, but are cataloged as a unit.

6023
EFFECTS OF RADIATION ON PHYSICAL PROPERTIES AND STRUCTURE OF SOLID BODIES. A. I. Zakharov. Uspekhi Fiz. Nauk 57, 525-76(1955) Dec. (In Russian).
A review of theoretical concepts and up-to-date experimental data of radiation effects on solids are given. (R.V.J.)

2186
THE EFFECTS OF IRRADIATION ON THE PHYSICAL PROPERTIES OF SOLIDS. A. H. Cottrell (Atomic Energy Research Establishment, Harwell, Berks, England). Brit. J. Appl. Phys., Suppl. No. 5, 43-53(1956).
Ionizing radiations dissipate most of their energy in solids by exciting electrons. Severe radiation damage is produced in organic substances by this process, but in metals the free electron structure is immune to ionization damage. Atoms are displaced by collisions with fast particles and the point defects so created alter the electrical and thermal properties of solids. These point defects interact strongly with dislocations so that the mechanical properties of metals are sensitive to heavy-particle bombardment. In semiconductors the displaced atoms can trap electrons and holes, thereby producing large changes in electrical properties. Prolonged bombardments produce drastic effects; some minerals become non-crystalline, uranium crystals change shape, some metals become brittle, and rare gas atoms created by transmutations may produce swelling and disintegration at high temperatures. (auth)

16810
RADIATION EFFECTS IN SOLIDS. R. Smoluchowski (Carnegie Inst. of Tech., Pittsburgh). Nuovo cimento (10) 7, Suppl. No. 2, 523-43(1958).
The theory of radiation effects in solids is summarized, and the various phenomena are illustrated with examples. The effects produced by radiation are electronic excitation, elastic collisions, displacements, in-

RADIATION EFFECTS 6.15

SURVEYS AND THEORY 6.15.11

THEORY 6.15.11.03

elastic collisions, and transmutations. The effects on alkali halides, alpha brass, semiconductors, graphite, and uranium are specifically discussed. (J.S.R.)

12528

ON THE THEORY OF RADIATION CHEMISTRY. L. S. Polak and A. Ya. Temkin (Inst. of Petroleum Chemical Synthesis, Academy of Sciences, U.S.S.R.). Doklady Akad. Nauk S.S.S.R. 125, 584-7(1959) Mar. 21. (In Russian)

Interactions of tracks and the method of approximation calculation of free radicals and finite products of γ and β radiolysis were studied. (R.V.J.)

ON THE AVERAGE ENERGIES OF MOLECULAR IONS. D. P. Stevenson (Shell Development Co., Emeryville, Calif.). Radiation Research 10, 610-21(1959) June.

The average excitation energy $\bar{e}$ with which molecule ions are formed by electron impact-induced ionization in relation to the average energy expended in the formation of molecule ions is considered. Under the assumption of an essential applicability of a quasi-equilibrium rate theory to mass spectra, $\bar{e}$ can be related to two observable mass spectral qualities. The validity of the quasi-equilibrium theory is also independently checked by using the data to calculate an average energy required to form a molecule ion by methods which are not dependent on the theory. Comparison of the average excitation energies with which ions are formed as calculated from the equations indicate excellent agreement between the different methods. (J.R.D.)

3179 TID-8005

Technical Information Service, AEC.

THE INITIATION OF CHEMICAL REACTIONS BY GAMMA AND OTHER IONIZING RADIATIONS. David S. Ballantine, Brookhaven National Lab. Feb. 1956. 6p. $0.15(OTS).

One of Its monograph series "The Industrial Atom."

The interactions of γ radiation with matter to produce ion pairs, radicals, or excited molecules are discussed. The radiation-induced reactions, chain-type (ploymerization) and mass-action (irradiation of benzene) are reviewed. (J.E.D.)

13210 TID-10033

Knolls Atomic Power Lab., Schenectady, N. Y.

THE THERMAL SPIKE PICTURE OF RADIATION DAMAGE. Harvey Brooks. Aug. 4, 1950. Decl. Mar. 12, 1957. 50p. $0.40(OTS).

The influence of fission products and neutron recoils on metals is discussed in terms of filamentary of spherical heated regions left in the wake of the fast particle, which spread according to the laws of heat conduction. It is shown that the electrons play a negligible role in the process and that only the part of the thermal conductivity which is due to the lattice should be used in the calculation of the energy spread. Rates of reaction induced by radiation are calculated on the assumption that the elementary processes occur according to a frequency law of the form: $\nu_n T^n e^{-\epsilon/kT}$ and that the effective frequency of such processes in the presence of radiation is obtained by averaging over the temperature distribution in the thermal spikes. Various refinements are considered, such as the influence of the variation of thermal conductivity with temperature. Rates of motion of phase boundaries and effective diffusion constants in the presence of radiation are also considered.

2187

EFFECT OF ELECTRON IRRADIATION ON YOUNG'S MODULUS. H. Dieckamp and A. Sosin (Atomics International, Canoga Park, Calif.). J. Appl. Phys. 27, 1416-18 (1956) Dec.

Young's modulus and internal friction measurements are reported on high-purity Cu following electron bombardment. The modulus is observed to rise rapidly with electron flux reaching an early saturation followed by a slow decrease. The rapid saturation is attributed to dislocation pinning. The rate of saturation is temperature dependent in the range from −195°C to about 0°C suggesting defect migration at low temperatures. The slow decrease is attributed to a "bulk effect" due to interstitial-vacancy pairs. (auth)

7543

A SEARCH FOR THE CHEMICAL EFFECTS OF INTERNAL CONVERSION FOLLOWING RADIATIVE NEUTRON CAPTURE. A. G. Maddock and M. M. de Maine (Univ. Chemical Lab., Cambridge, England). Can. J. Chem. 34, 441-4(1956) Apr. 5.

The possibility that neutron capture would lead to oxidation, because of internal conversion of the capture radiation, has been investigated in solid compounds of the lower valence states of Sb, As, Ce, Cr and Tl. Except in the hydrated compounds, negative results have been obtained. It is possible that oxidation is followed by electron-transfer exchange in the irradiated solids. (auth)

RADIATION EFFECTS 6.15

SURVEYS AND THEORY 6.15.11

THEORY 6.15.11.03

7053
ACTIONS CHIMIQUES ET BIOLOGIQUES DES RADIATIONS (Chemical and Biological Effects of Radiations). M. Haissinsky, ed. Paris, Masson and Co., 1955. 252p.

A discussion is given on the physical aspects of radiobiology including topics such as modes of interaction between radiation and matter, measurement and determination of doses, localization of energy loss along the particle trajectory, and individual energy transfers between the ionizing particle and the absorbing medium. Radiochemistry is also fully discussed including topics such as the development of the chemistry of radiations of aqueous solutions, the effect of ionizing radiations on H_2O, the decomposition of oxygenated H_2O solutions, the radiochemical transformations of iron salt solutions, oxidation of mineral compounds, reductions induced by ionizing radiations, and the oxidation of organic compounds. Modern trends in radiation biochemistry are also discussed. (B.J.H.)

11469 BNL-3275
Brookhaven National Lab., Upton, N. Y.
DEFECTS IN SOLIDS AND CURRENT CONCEPTS OF RADIATION EFFECTS. G. J. Dienes. [Mar. 1957]. 91p. $13.80(ph OTS); $4.80(mf OTS).

Calculations and measurements of displaced atoms produced by nuclear radiation are given and discussed. (T.R.H.)

9157 NARF-57-17T
Convair, Fort Worth, Tex.
SEPARATION OF NUCLEAR EFFECTS IN A MIXED RADIATION FIELD. AN EXPERIMENTAL DESIGN FOR MATERIALS AND COMPONENT DAMAGE STUDIES. D. W. Gaylor and J. B. Wattier. Feb. 2, 1957. 33p. Project 6(1-9964). Contract AF33(600)-32054. (MR-N-153).

An experimental design is presented for separating the effects of fast neutron dose rate, gamma dose rate, integrated fast neutron dose, and integrated gamma dose on materials and electronic components exposed to mixed radiation fields. The experimental design consists of two parts: an exploratory experiment and a descriptive experiment. The purpose of the exploratory experiment is to determine which of the four radiation factors are significant for each material and component investigated. The purpose of the descriptive experiment is to determine the functional relationship between damage and these significant factors. This sequential procedure reduces the number of irradiations that would otherwise be required by about half. The procedures given can be extended to study the interactions of a nuclear environment with non-nuclear environments such as temperature, vibration, and pressure. The validity of the results depends upon the ability to measure dose rate or integrated dose on a relative scale and upon the validity of the assumption that the change in the functional response of a material or component is negligible for small spectrum changes (auth)

EFFECTS OF RADIATION ON MECHANICAL PROPERTIES OF SOLIDS. M. J. Makin (Atomic Energy Research Establishment, Harwell, Berks, Eng.). p.500-30 in "Progress in Nuclear Energy. Series V. Metallurgy and Fuels. Volume 2."

This article attempts to review present knowledge on the effect of irradiation on the mechanical properties of solids. The major part of the discussion is on metals since, because of their technological importance, the effect of irradiation on their mechanical properties has been studied more thoroughly than that on other classes of solid. Ionic crystals are discussed in some detail, however, and brief mention is made of the effects on polymers. The experimental evidence reviewed includes the effect on creep, hardness, tensile properties (yield strength, etc.), ductile brittle transition temperature, elastic constants, internal friction, and annealing behavior. The various explanations which have been put forward to account for the changes on irradiation are also discussed. (auth)

5785
RADIATIONS AND MATTER. André Berthelot. Translation of "Rayonnements de Particules Atomiques, Electrons et Photons." London, Leonard Hill [Books] Limited, 1958. 185p.

The book deals with the characteristics of various types of radiations and their behavior as they pass through matter. Topics treated include the theory of elastic collision, particles in motion, the path, ionization, deceleration, variation of charge, and Coulomb interactions of moving particles, fission fragment slowing down, electrons, and photons. (W.D.M.)

RADIATION EFFECTS 6.15

SURVEYS AND THEORY 6.15.11

THEORY 6.15.11.03

20586 ✓ NARF-59-28T
Convair, Fort Worth, Tex.
MECHANISMS OF RADIATION DAMAGE. A Series of Lectures. June 15, 1959. 353p. Project 6(1-9964). Contract AF33(600)-32054. (MR-N-232).

A summary of information contained in a series of 15 lectures on damage in irradiated materials is presented. A discussion is given on the specification of reactor flux spectra. Emphasis is placed on the importance of accurate flux specifications in reliable predictions of rates of production in neutron-induced defects in materials. Atomic displacements produced in solids by neutron bombardment are discussed. Displacement mechanisms, such as thermal spikes, displacement spikes, replacement collision, and the Silsbee effect, are considered. An interpretation of slip in crystals on the basis of dislocation theory is given. Kinds of slip, such as easy glide and parabolic work hardening, are discussed. Theoretical explanations of hardening techniques are given. Stresses around temperature spikes, generation of dislocation rings by radiation, persistence of rings, and the possibility of radiation-induced disordering in alloys are discussed. Structure, nomenclature, chemical and physical properties of organic compounds are discussed with emphasis on compounds of interest to radiation damage study and the aircraft industry. Preparation of polymers, resins, structure of plastics, polymerization mechanisms, nomenclature, and application aspects of the more important polymers are examined. A general discussion of radiation studies made in the past few years at various facilities concerning irradiation of organics is presented. A discussion of radiation damage mechanisms in polymers is given. Damage mechanisms, such as cross linking, gas evolution, end-linking, free-radical mechanisms, ionization processes, rearrangements, and chain scission, are considered. A discussion of silanes, silicates and siloxanes, is followed by a treatment of the chemistry of the silicon-carbon bond. General characteristics, molecular forces, structure, physical properties, and behavior of polymer systems are discussed. Rheological methods of correlating radiation-induced changes in physical properties of elastomers with mechanisms occurring on the molecular level are discussed. Various types of anti-rad materials are discussed and their applicability in organic materials is considered. General rules are given regarding the stability of organic compounds and some predictions of radiation stability in organic systems are made. (auth)

SOLVENT EXTRACTION 6.16

See also, this section,

ULTRASONICS (6.20),

and,

under FISSION PRODUCT, POISON, AND RADIOISOTOPE REMOVAL,

SOLVENT EXTRACTION (2.44),

and,

under FISSIONABLE MATERIAL--RECOVERY,

RECOVERY OF IRRADIATED TH AND U --Solvent Extraction (3.02.17),

RECOVERY OF PU--Solvent Extraction (3.03.14).

COSTS 6.16.01

10486
GENERAL ECONOMICS OF CHEMICAL REPROCESSING USING SOLVENT EXTRACTION PROCESSING. Presented at Nuclear Engineering and Science Conference, held at Chicago, March 17 to 21, 1958. Preprint 22, Session 37. F. L. Culler, Jr. (Oak Ridge National Lab., Tenn.). New York, American Institute of Chemical Engineers, 1958. 27p.

The competitive requirement of producing electricity at 8 mills/kwh in the United States places limitations on the over-all recycle costs of irradiated fuel and fertile material. The cost for the recycle of fuel, including de-

SOLVENT EXTRACTION 6.16

COSTS 6.16.01

cay cooling, radiochemical separations, metallurgical reprocessing, fuel element fabrication, and waste disposal, probably cannot exceed 0.75/kwh of electricity for reactors that cost $250/kw of installed capacity. To achieve radiochemical separations for costs much less than 0.75 mill/kwh of electricity, assuming that 4000 megawatt days of heat are extracted per ton of natural uranium fuel or its equivalent, the radiochemical processing plants must be large and capable of processing the output of many reactors; the economic size of a chemical plant may be of such a size that it will require the irradiated fuel and blanket material from twenty 500 MW electrical reactors. The throw-away case for fuel has been studied for natural, partially enriched, and highly enriched uranium as well as thorium and U^{233}. Based on buy-back prices published by the Atomic Energy Commission, the residual value of fissionable and fertile material in irradiated fuel is almost always greater than the cost of recycle. Reprocessing will always be required. The concept of the central chemical plant makes transportation of irradiated fuel an economic necessity. Costs of transporting irradiated fuel by rail for distances as great as 1000 miles are not restrictive for most reactor types. (auth)

10086 ORNL-1963
Oak Ridge National Lab., Tenn.
DESIGN OF AN AMINE EXTRACTION DEMONSTRATION PLANT. B. B. Klima, H. M. McLeod, Jr., A. D. Ryon, and R. R. Wiethaup—K. O. Johnsson, ed. Sept. 8, 1955. Decl. Jan. 10, 1956. 44p. Contract W-7405-eng-26.

The design of an amine extraction demonstration plant, along with installation costs, recovery costs, and operating costs are presented. (T.R.H.)

1302
COSTS ON NEW PROCESSES FOR SOLVENT EXTRACTION OF URANIUM. K. Glenn Shaw and Ray S. Long (Dow Chemical Co., Pittsburg, Calif.). Chem. Eng. 64, No. 11, 251-6(1957) Nov.

Operation and cost information on solvent extraction of U by hydrometallurgical operations is given. Solvents, equipment, principles, and cost factors are included. (W.L.H.)

EQUIPMENT 6.16.02

See also, this section,
RADIATION EFFECTS--Glass (6.15.04),
Metal (6.15.07),
and,
under PLANTS AND EQUIPMENT;
TRANSFER OF FLUIDS (5.16).

CONTACTORS (GENERAL) 6.16.02.01

CONTACTORS AND AUXILIARIES. J. K. Davidson. May 1, 1957. p.848-68 of SYMPOSIUM ON THE REPROCESSING OF IRRADIATED FUELS HELD AT BRUSSELS, BELGIUM, MAY 20-5, 1957. 1165p. (TID-7534(Bks. 1-3); KAPL-1808)

Pulse column, pulser pumps, and mixer-settlers are described in detail. 22 references.

2477
IMPROVEMENTS IN OR RELATING TO PACKINGS FOR LIQUID-LIQUID CONTACTING COLUMNS. Philip Michael Cherubino Lacey (to United Kingdom Atomic Energy Authority). British Patent 823,378. Nov. 11, 1959.

A packing element for liquid-liquid extraction columns is offered. It consists of spheres of 0.625 in. diam arranged in a plane in point contact with each other in a square or hexagonal pattern. A stack of such spheres would present a face-centered cubic lattice. The element can be injection molded of polythene. A column can be so designed that these elements can be stacked in it with half-spheres in contact with the column, thus avoiding edge effects. The sub-elements may also be shapes other than spherical to give different column characteristics. (T.R.H.)

5249 CRCE-790
Atomic Energy of Canada Ltd., Chalk River, Ont.
THE DEVELOPMENT OF A LARGE SCALE AIR LIFT

SOLVENT EXTRACTION 6.16

EQUIPMENT 6.16.02

CONTACTORS (GENERAL) 6.16.02.01

CONTACTOR. W. G. Mathers, L. C. Cornett, and E. E. Winter. Sept. 1959. 50p. (AECL-913). AECL.

A three-stage air lift contactor with a capacity suitable for the processing of 100 tons of uranium per year was designed, built, and tested. A method of interface control in a multistage contactor was tested, and measurements of the air required for proper physical operation under various conditions and of aqueous entrainment in the solvent phase were made. Estimates of required settler volumes and solvent overflow Weir widths are included. (J.R.D.)

12701 KAPL-1808
Knolls Atomic Power Lab., Schenectady, N. Y.
BELGIAN SYMPOSIUM ON CHEMICAL PROCESSING, I SESSION: ENGINEERING OF RADIOCHEMICAL PLANTS CONTACTORS AND AUXILIARIES. J. K. Davidson. June 27, 1957. 49p. Contract W-31-109-Eng-52. $0.35(OTS).

Several variations of the pulse column and mixer-settler contactors for liquid-liquid extraction in radiochemical facilities and pilot plants are described. Included are a $^3/_4$-in. pulse column, a 1-in. vertical pulse column, the profile pulse column, and the concatenated pulse column. The mixer-settler systems include the pilot plant "Midi" mixer-settler, the laboratory "Mini" mixer-settler, a critically safe mixer-settler, and a continuous solvent wash unit. Details of necessary auxiliaries are discussed, and reference is made to designs and construction. (auth)

MECHANICAL CONTACTORS FOR LIQUID-LIQUID EXTRACTION. Part 2. J. D. Thorton (Atomic Energy Research Establishment, Harwell, Berks, England). Nuclear Eng. 1, 204-9(1956) Aug.

Constructional details are given of various types of experimental contactors which have been used at Harwell. The calculations for the diameter and height of the column-type contactors are summarized. (auth)

4309
A CONTINUOUS ETHER EXTRACTOR. K. J. Jensen and R. W. Bane (Argonne National Lab., Lemont, Ill.). Analyst 82, 67-70(1957) Jan.

An apparatus is described and a procedure given for ether extraction of U from HNO_3 solutions. Determinations of U in U–Al solutions were made which, for 18.7 mg U/ml solution, had a standard deviation of 0.22 mg. (T.R.H.)

CRITICALLY SAFE EQUIPMENT FOR AQUEOUS SEPARATIONS PROCESS. T. J. Colven. 13p. (A/CONF.15/P/518)

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

The operating characteristics of critically-safe "slab" mixer-settlers and continuous solvent washers are described. The effectiveness of some experimental units of each type was evaluated for a number of tributyl phosphate-kerosene and $UO_2NO_3-HNO_3-Al(NO_3)_3$ systems. The units described are prototypes of equipment that could be used in a full scale solvent extraction plant. The mixer-settlers are of the "pump-mix" type and are shaped in the form of slabs, three inches high. When positioned in an operating area so that the large faces of the slab are at least six feet away from solid reflectors, the slabs are critically safe for concentrations of fissionable materials as high as 400 grams per liter of solution. The critically-safe solvent washers are cylinders, 7 inches in diameter and 12 feet high. A continuous washer was evaluated with 6 and 30% TBP in kerosene solutions. The effect of mixing speed, recirculation rate, cylinder height, and solution properties on the entrainment of a Na_2CO_3 wash in the solvent was determined at processing rates up to 0.5 gpm. A unitized equipment layout in which continuous washers are coupled directly to two mixer-settlers in such a way as to provide a closed solvent circuit is presented to illustrate a practical solvent extraction cycle with a minimum of equipment and solvent inventory.

144
EQUIPMENT FOR NUCLEAR FUEL REPROCESSING. T. J. Colven, Jr. Oct. 1957. 20p. (DP-239)

Design information and preliminary operating characteristics are given for a critically safe mixer-settler, two continuous solvent washing units, and a number of steam jet syphons that have been tested on HNO_3-TBP systems.

12428 CEI-40
National Research Council of Canada. Atomic Energy Project, Chalk River, Ont.
A SURVEY OF LIQUID-LIQUID CONTACTING EQUIPMENT. S. H. Russell. Feb. 15, 1952. 17p.

SOLVENT EXTRACTION 6.16

EQUIPMENT 6.16.02

CONTACTORS (GENERAL) 6.16.02.01

A literature survey of liquid-liquid extraction equipment presently in use is presented. The apparatus is considered from the point of view of its usefulness and capabilities when applied to primary separation processes. The major classification is determined by whether gravity or centrifugal force is used to separate the phases. Extractors are considered separately from the standpoints of design, operation, and maintenance. (J.R.D.)

7479 ✓ CF-51-10-71
[American Cyanamid Co., New York.]
SPECIAL PROCESS EQUIPMENT. E. R. Edmondson. Oct. 12, 1951. Decl. Feb. 13, 1957. 52p. $9.30(ph OTS); $3.60(mf OTS).

The report on the special process equipment includes, the solvent extraction columns, samplers, slug chargers, filters, agitators, pumps, jet syphons, valves, tube fittings, cranes, and service waste monitoring and metering equipment. (J.E.D.)

1301
AUTOMATIC SOLVENT EXTRACTION APPARATUS AND METHOD FOR URANIUM. C. A. Kienberger (Union Carbide Nuclear Co., Oak Ridge, Tenn.). Anal. Chem. 29, 1721-2(1957) Nov.

A simple and inexpensive extractor of unique design is described which can be constructed by the average chemist-glassblower. (L.T.W.)

LONGITUDINAL DISPERSION IN SOLVENT-EXTRACTION COLUMNS: PECLET NUMBERS FOR ORDERED AND RANDOM PACKINGS. Gabriel L. Jacques and Theodore Vermuelen. Nov. 1957. 152p. (UCRL-8029)

Dispersion phenomena in packed beds in both axial and radial directions were studied to provide basic data for extraction-tower design. Nine different beds were used, involving regular and random arrangements of spheres, and random arrangements of Raschig rings and Intalox saddles. For one-phase flow a wide range of Reynolds numbers was used (5 to 2000), covering regions of laminar, transition, and turbulent flow. For the axial Peclet number, different constant values were found in the turbulent range and in the laminar range. The existence of a fairly sharp transition region was observed. Radial-dispersion results showed similar behavior. The axial Peclet number was found to vary inversely with bed porosity, while the radial Peclet number was almost proportional to porosity. For two-phase flow, measurements were made up to the flooding point, which occurred at Reynolds numbers near 100. The axial Peclet number for both continuous and discontinuous phases is a function of the Reynolds numbers for the two individual phases. For each phase, the laminar value of the axial Peclet number obtained for one-phase flow is an upper limit. Values as low as one-fifth of this limiting value were found at relatively large flow rates of the second phase. The radial Peclet number was measured only for the continuous phase of two-phase systems; it was found to decrease in the same direction, but by a smaller percentage.

2456 DP-411
Du Pont de Nemours (E. I.) & Co. Savannah River Lab., Aiken, S. C.
APPARATUS FOR SOLVENT EXTRACTION OF MILLILITER QUANTITIES OF HIGHLY RADIOACTIVE SOLUTIONS. Richard M. Wallace and Henry Pollock. Sept. 1959. 10p. Contract AT(07-2)-1. OTS.

A description is given of apparatus designed for studying the solvent extraction of small quantities of highly radioactive solutions. The equipment is inexpensive and is easily assembled in a laboratory hood. (auth)

COUNTERCURRENT EXTRACTORS 6.16.02.02

6230 AERE-C/R-933(Del.)
Gt. Brit. Atomic Energy Research Establishment, Harwell, Berks, England.
COUNTER-CURRENT LIQUID-LIQUID EXTRACTION IN ANNULUS COLUMNS WITH SHEARING MOTION. R. Spence and R. J. W. Streeton. Sept. 22, 1952. Decl. with deletions Dec. 18, 1956. 45p.

The rotary extractor has been investigated both from the point of view of mechanism of operation and from the point of view of utility for the extraction of U. It has been shown that there is a hyperbolic relationship between h/d and d/R_1 (where R_1 is the radius of the rotor, d is the width of the annulus, and h is the height of a single vortex). The height of equivalent theoretical stage (H.E.T.S.)

SOLVENT EXTRACTION 6.16

EQUIPMENT 6.16.02

Countercurrent Extractors 6.16.02.02

varies as the cube root of the cross sectional area (hd) or the vortices and combination of these two relationships gives an expression for the H.E.T.S. in terms of R_1 and d, viz: $H = \text{const.}\ (d^4 + R^2/3)^{1/4}$. The expression holds over the whole of the range of sizes studied (R_1 = 0.075–6.66 cm.; d = 0.05–1.6 cm.). The rate of transfer of $UO_2(NO_3)_2$ across the interface between drops and continuous phase in the butex-aqueous system has been found to depend on the transport of the solvated $UO_2(NO_3)_2$ molecule through a solvent film at the interface. The rate of transfer in the case of a single drop in the rotary column is only about twice that which is found for a drop falling freely through a static continuous phase. (auth)

SOLVENT EXTRACTION APPARATUS USING JET MIXERS. J. H. Yeager, et al. (to U. S. Atomic Energy Commission). U. S. Patent No. 2,759,801, August 21, 1956.

An apparatus was developed for counter-current liquid extractions between liquids of different densities. It is specially designed for the extraction of an aqueous $UO_2(NO_3)_2$ solution by diethyl ether. The apparatus is a column with decanting chambers attached to both top and bottom. There is a heavy fluid inlet and light fluid outlet connected to the upper decanting chamber, and a light fluid inlet and heavy fluid outlet in the lower decanting chamber. Spaced along the length of the column are several recirculating assemblies, each consisting of a pump, tubing and a jet nozzle to afford thorough mechanical mixing of the two fluids. (auth)

A COUNTERCURRENT LIQUID-LIQUID EXTRACTOR. Harley A. Wilhelm and Raymond A. Foos (Iowa State Coll., Ames). Ind. Eng. Chem. 51, 633-6 (1959) May.

Liquid-liquid extraction has proved very useful for inorganic purifications in the atomic energy program. A convenient small extractor that would simulate countercurrent vertical column extractors was desired for testing extraction conditions set up on new systems from distribution and other data of single-stage tests and for preparing macro amounts of purified products. The equipment has been used in developing extraction systems for purifying niobium, tantalum, zirconium, hafnium, and yttrium. A batch of more than 100 pounds of hafnium containing less than 30 ppm of zirconium was prepared with the extractor described. This extractor is useful in studying behavior of components of a system during a multistage countercurrent extraction. Actual production can be achieved. (auth)

20945

THE APPLICATION OF PULSATING COLUMNS FOR SEPARATING URANIUM, DEGRADATION PRODUCTS AND PLUTONIUM BY THE COUNTER-CURRENT EXTRACTION METHOD. V. Dizdar. Tehnika (Belgrade) 13, No. 2, (1958). (Translated from Referat. Zhur. Khim. No. 1, 1959, Abstract No. 354.)

The process for separating uranium, the degradation products, and Pu by means of continuous countercurrent extraction in pulsating columns is described. (auth)

MAINTENANCE 6.16.02.03

See also, this section,

RADIATION EFFECTS (6.15),

and,

under PLANTS AND EQUIPMENT, CORROSION (5.05).

6977

PREVENTION OF SCALE FORMATION IN URANIUM SOLVENT EXTRACTOR. J. W. Delaplaine (to U. S. Atomic Energy Commission). U. S. Patent 2,812,232. Nov. 5, 1957.

A method for preventing the formation of scale in uranium solvent extraction apparatus is presented. The scale, consisting chiefly of precipitated silica and the sulfates of calcium and lead, may be prevented by a combination of measures, chiefly by prior heating and agitation to crystallize and remove silica, and by a maintenance of uranyl nitrate concentration in the feed and extractant above certain levels to increase the solubility of the calcium and lead sulfates.

SOLVENT EXTRACTION 6.16

EQUIPMENT 6.16.02.0

MIXER-SETTLERS 6.16.02.04

1589

SCALE-UP OF MIXER-SETTLERS. A. D. Ryon, F. L. Daley, and R. S. Lowrie (Oak Ridge National Lab., Tenn.). Chem. Eng. Progr. 55, No. 10, 70-5(1959) Oct.

The rate of uranium extraction in the Dapex process is proportional to the cube root of power input for both batch and continuous flow if the extraction in the mixer only is measured. Mixers can be scaled-up by geometric similitude at constant power input per unit mixer volume, demonstrated in 6-, 12-, 20-, and 36-in. diam. mixers, representing a volumetric scale-up ratio of 200 to 1. The flow capacity of settlers is characterized by a band of dispersion at the interface, the thickness of which is a measure of the approach to flooding. Settlers can be scaled-up based on flow rate per unit cross-sectional area, demonstrated in settlers 6 in. to 16 ft. diam., representing an area scale-up ratio of 1000 to 1. Control of mixing to form water-in-oil dispersions is recommended to minimize solvent entrainment and emulsions with solutions containing colloidal material which stabilize oil-in-water emulsions. (auth)

10128 CCC-1024-TR-195

Callery Chemical Co., Penna.

ANALYSIS OF POWER REQUIREMENTS AND DISPERSION ACTION OF MIXING IMPELLERS. D. K. Eads—P. M. Maginnity, ed. Aug. 2, 1956. 24p. Contract [NOa(s)-52-1023-c].

An analysis has been made of the related problems of mixing and dispersion, in operations performed by means of radial turbines, "Dispersators", and form drag impellers. By the application of a modified form of dimensional analysis, expressions were derived for the power number, the Reynolds number, the Weber number, and a new group which has been called the time number—in such a way that these numbers take into account impeller geometry. A correlation of power requirements for flat six-bladed radial turbines was obtained. Tentative values for these dimensionless ratios (except the time number) were established for the first two impeller types. No data were available for the time number, but it is suggested that its use should prove valuable in certain types of dispersion and reaction problems. (auth)

MIXER-SETTLER. M. E. Whatley (to U. S. Atomic Energy Commission). U. S. Patent No. 2,754,179, July 10, 1956.

A chemical apparatus was designed to facilitate the extraction of metal values by two immiscible liquids. The apparatus consists of a cascade of settling chambers, each connected to the other by a mixing chamber. A dense aqueous solution containing the metal value is intimately mixed with a less dense immiscible solvent such as TBP by an impeller, at which time some of the metal value is extracted by the TBP, and the mixture is pumped into a setting tank where a liquid interface is re-established by gravity, then the liquids are remixed and recycled through successive stages of the cascade until the desired degree of extraction has been attained. The apparatus provides better flow control of each liquid separately and improved mixing of the liquids, resulting in a greater degree of extraction. (auth)

11685 DP-255

Du Pont de Nemours (E. I.) & Co. Savannah River Lab., Augusta, Ga.

TURBINE CONTACTOR FOR SOLVENT EXTRACTION. G. Starr Nichols. Dec. 1958. 24p. Contract AT(07-2)-1. $0.75(OTS).

Test of a liquid-liquid contactor of commercial size showed that its performance was predictable on the basis of data previously obtained with a laboratory contactor having one fiftieth the volume. The criterion for composite performance of the mixer and settler was the heat conductance achieved at a given dispersion depth. (auth)

OPPOSED IMPELLER MIXER - SETTLER. J. A. Dukes and F. Parish (to U. K. Atomic Energy Authority). British Application 03406/56. Atom No. 5, 23(1957) Mar.

It is usual to achieve flow through a mixer-settler plant by gravity fall or with interstage pumping (e.g. air lift, screw, or centrifugal pumping). The level mixer-settler plant is operated without the need for pumping by virtue of the fact that homogeneous mixing is maintained throughout the volume of the mixers. This is achieved by use of a double paddle mixer, one paddle being above the mixer outlet port and one below. The paddle operates in such a way that the lower paddle tends to lift the heavier phase while the upper paddle tends to depress the lighter phase.

SOLVENT EXTRACTION 6.16

EQUIPMENT 6.16.02

MIXER-SETTLERS 6.16.02.04

EXTRACTION APPARATUS. A. E. Ballard and H. R. Brigham (to U. S. Atomic Energy Commission). U. S. Patent 2,858,196. Oct. 28, 1958.

An apparatus whereby relatively volatile solvents may be contacted with volatile or non-volatile material without certain attendant hazards is described. A suitable apparatus for handling relatively volatile liquids may be constructed comprising a tank, and a closure covering the tank and adapted to be securely attached to an external support. The closure is provided with a rigidly mounted motor-driven agitator. This agitator is connected from the driving motor into the interior of the tank through a gland adapted to be cooled with inert gas thereby eliminating possible hazard due to frictional heat. The closure is arranged so that the tank may be removed from it without materially disturbing the closure which, as described, carries the motor driven agitator and other parts.

8734 ✓

LABORATORY MIXER-SETTLERS. W. D. Jamrack (United Kingdom Atomic Energy Authority, Springfields, Eng.); D. H. Logsdall (Atomic Energy Research Establishment, Harwell, Berks, Eng.); and G. D. C. Short (United Kingdom Atomic Energy Authority, Windscale, Cumb., Eng.). pp. 332-54 in "Progress in Nuclear Energy. Series III. Process Chemistry. Volume 2."

The place of the laboratory mixer-settler in obtaining data for the chemical processing of nuclear materials is described. The development of laboratory mixer-settler equipment is traced from simple equilibrating tubes to remotely controlled mechanical mixer-settlers for continuous operation. Different types of mixer-settler are described in detail, their operating and design characteristics discussed and their advantages and disadvantages enumerated. (auth)

A RECENT MODEL OF THE MINIATURE MIXER-SETTLER. H. W. Alter and J. W. Codding. Dec. 15, 1954. Decl. Apr. 8, 1957. (KAPL-1246)

A modification of the miniature mixer-settler is described. The bank is fabricated from stainless steel and fluorothene, and it can be used for elevated temperature operation. Changes in stage design increase phase separation and minimize back-mixing. Described also is a feed system, composed of Zenith Gear Pumps. Pictures and production drawings are included.

4153 AECD-4067

Knolls Atomic Power Lab., Schenectady, N. Y.

A MINIATURE MIXER-SETTLER. H. W. Alter, B. V. Coplan, E. L. Zebroski. Dec. 21, 1950. Decl. with deletions Jan. 5, 1956. 40p. Contract W-31-109-Eng-52. $6.30(ph OTS); $3.00(mf OTS).

A minature mixer-settler has been developed that fulfills the requirements for a laboratory extraction unit and overcomes the disadvantages of the batch countercurrent units commonly used. The construction and operation of the unit are reviewed, and design drawings are given. (D.E.B.)

Du Pont de Nemours (E. I.) & Co. Savannah River Lab., Augusta, Ga.

TURBINE CONTACTOR FOR SOLVENT EXTRACTION. G. Starr Nichols. Dec. 1958. 24p. Contract AT(07-2)-1. $0.75(OTS).

Test of a liquid-liquid contactor of commercial size showed that its performance was predictable on the basis of data previously obtained with a laboratory contactor having one fiftieth the volume. The criterion for composite performance of the mixer and settler was the heat conductance achieved at a given dispersion depth.

4583

FLUID CONTACTOR APPARATUS. R. Spence and Robert J. W. Streeton (to U. S. Atomic Energy Commission). U. S. Patent 2,742,348. Apr. 17, 1956.

The fluid contactor apparatus comprises a cylindrical column mounted co-axially and adapted to rotate within a cylindrical vessel, for the purpose of extracting a solute from an aqueous solution by means of an organic solvent. The column is particularly designed to control the vortex pattern so as to reduce the height of the vortices while, at the same time, the width of the annular radius in the radial direction between the vessel and column is less than half the radius of the column. A plurality of thin annular fins are spaced apart along the rotor approximately twice the radial dimension of the column such that two contra-rotating substantially circular vortices are contained within each pair of fins as the column is rotated.

SOLVENT EXTRACTION 6.16

EQUIPMENT 6.16.02

PULSED COLUMNS 6.16.02.05

A POLYETHYLENE BOX-TYPE MIXER-SETTLER EXTRACTOR. Lester Knapp, Roman Schoenherr, John Barghusen, and Morton Smutz. Jan. 1958. 23p. (ISC-998)

Some distinguishing characteristics of the polyethylene box-type mixer-settler are: a built-in interface controller adjustable during operation; both phases enter the mixing chamber along the impeller axis; the impeller pumps the phases through the extractor; compactness—no interstage piping; unlimited number of stages; no back-mixing; and wide range of impeller speeds. A 300-hr run with rare-earth nitrate—TBP system, 10 times as viscous as water, was quite satisfactory.

9256

SOME OPERATING CHARACTERISTICS OF THE PUMP-MIX MIXER SETTLER. J. H. Holmes and A. C. Schafer (Knolls Atomic Power Lab., Schenectady, N. Y.). Chem. Eng. Progr. 52, 201-4(1956) May.

The design principles of the pump-mix mixer settler are described briefly and some of its operating characteristics are presented. Advantages of the mixer settler include high efficiency, reliability of operation, simplicity of control, flexibility, ease of maintenance, and minimum investment. (C.W.H.)

PULSED MIXER-SETTLER SOLVENT EXTRACTION CONTACTORS. W. S. Figg (to U. S. Atomic Energy Commission). U. S. Patent 2,847,283. Aug. 12, 1958.

A mixer-settler extractor is described for contacting immiscible liquids having different specific gravities in order to withdraw one or more components from one liquid with the aid of the other liquid. The extractor consists of a hollow column, a rotary drive shaft extending through the column with a number of impellers spaced thereon, an equal number of separator plate sets each consisting of one fluorothene and one stainless steel plate with peripheral recesses and flow slots mounted on the column, and a pulse generator. This apparatus is particularly useful in solvent extraction processes for recovering plutonium from aqueous acidic solutions of irradiated uranium.

PULSED COLUMNS 6.16.02.05

5283

LAB DEVELOPS TABLE-TOP NUCLEAR FUEL SEPARATOR. Etienne Guerin. Research/Develop. 11, No. 1, 25-7(1960) Jan.

A flowsheet for a table-size nuclear fuel separator (the Asea Column), capable of refining five tons of uranium a year, is given. A pulse pump initiates a movement which is transferred through the series-coupled tubes containing perforated bottom plates by way of the organic overflow, with no mixing of the aqueous streams. An auxiliary circuit is provided for level control and crude removal in one operation. Advantages of the column are discussed. (C.J.G.)

APPARATUS FOR LIQUID PHASE EXTRACTION. T. E. Hicks, H. R. Lehman, and B. Rubin (to U. S. Atomic Energy Commission). U. S. Patent 2,852,349. Sept. 16, 1958.

A pulse extraction device adapted for inverted density operation is described. It comprises a tubular column having upper and lower enlarged terminal portions, and a constricted central section containing fluid dispersal packing. Pulsing means are coupled to the upper portion of the column. The inlet for the less dense phase is located above the inlet for the denser phase and both are positioned so that liquids enter the constricted packing-filled central section. The apparatus also includes an interfacing level control, and means for sensing the level of the interface actuate apparatus for controlling the rate of flow of input or discharge. The outlet for the less dense phase is located in the upper packing free portion of the column and that of the denser phase in the lower portion.

6516 A/CONF.15/P/1173

ETUDE DES COLONNES A PULSATIONS A L'AIDE DU SYSTEME NITRATE D'URANYLE—ACIDE NITRIQUE—EAU—TRIBUTYLPHOSPHATE. (Study of Pulsed Columns with the System. Uranyl Nitrate—Nitric Acid—Water—Tributylphosphate.) J. Durandet, D. Defives, B. Choffe, and Y. L. Gladel (Inst. Français du Pétrole, France). 28p.

The performance of a pulsed column with perforated plates was studied with the aid of a uranyl nitrate—

SOLVENT EXTRACTION 6.16

EQUIPMENT 6.16.02

PULSED COLUMNS 6.16.02.05

nitric acid—water—tributyl phosphate system. The extraction of uranium from an aqueous acidic solution by an organic solvent and the extraction of uranium from organic solutions by water were the two cases investigated. The variation of the efficiency and the capacity of the pulsed column was determined as a function of the pulse amplitude and frequency, of the total flow rate, of the diameter of the holes, and of the choice of dispersed phase. The results showed that for a given amplitude and total flow rate the efficiency has a maximum with an increase in frequency. (J.S.R.)

ENGINEERING

8100

SOLVENT EXTRACTION EQUIPMENT. L. L. Burger (to U. S. Atomic Energy Commission). U. S. Patent No. 2,743,170, Apr. 24, 1956.

Modifications in a perforated-plate pulsed column are outlined. A vertical column is provided with perforated plates, inlet, outlet and bleeder lines at both top and bottom of the column, together with lines, storage tanks, pressure tanks and pumps for both liquids. The basic improvement of the system is the valve timing arrangement whereby the pulse is formed that facilitates the mixing. The bottom inlet, top outlet and bleeder for the lighter liquid, open and close at approximately the same time, likewise the top inlet and bottom outlet and bleeder for the heavier liquid. The rapid surging flow of each liquid, providing the needed pulse, results from timing each outlet and bleeder valve to close simultaneously with or slightly before each inlet valve. (auth)

7530 ✓ IDO-14312

Phillips Petroleum Co. Atomic Energy Div., Idaho Falls, Idaho.

PULSE COLUMN STUDIES USING STR AND SIR FEED SOLUTIONS. S. L. Friederichs, D. E. Griffin, G. E. Lohse, and C. E. Stoops. Sept. 27, 1954. Decl. Feb. 28, 1957. 42p. Contract AT(10-1)-205. $6.30(ph OTS); $3 00 (mf OTS).

Pilot plant studies were made to determine operating characteristics of pulsed columns proposed as the solvent extraction contactors for the recovery of uranium from spent STR and SIR feed assemblies. Twenty-five STR "A" Column runs, six SIR "A" Column runs, one "B" Column run, and eight "C" Column runs were made. Variation of pulse conditions resulted in HTU values for the STR "A" Column ranging from 2.2 to 11.6 ft. Reduction of the aqueous-to-organic flow ratio from the original flowsheet value of 4.5/1 to 1.4/1 resulted in HTU values as low as 1.5 ft. The HTU values for the SIR "A" Column ranged from 1.0 to 1.6 ft. The HTU values for the "C" Column ranged from 1.5 to 1.9 ft. The maximum flooding volume velocity was found to be 1300 gal/(hr)(sq.ft.) for the "A" Column on both the STR and SIR flowsheets. The maximum "B" Column flooding point was found to be 800 gal/(hr)(sq.ft.) The "C" Column flooding points were found to be dependent upon nitric acid concentration in the organic phase. The maximum flooding point varied from 800 gal/(hr)(sq.ft.) for 0.002 molar acid feed to 1100 gal/(hr)(sq.ft.) for 1.0 molar acid feed. (auth)

6998

LIQUID-LIQUID EXTRACTION COLUMNS. J. D. Thornton (to U. S. Atomic Energy Commission). U. S. Patent 2,818,324. Dec. 31, 1957.

This patent relates to liquid-liquid extraction columns having a means for pulsing the liquid in the column to give it an oscillatory up and down movement, and consists of a packed column, an inlet pipe for the dispersed liquid phase and an outlet pipe for the continuous liquid phase located in the direct communication with the liquid in the lower part of said column, an inlet pipe for the continuous liquid phase and an outlet pipe for the dispersed liquid phase located in direct communication with the liquid in the upper part of said column, a tube having one end communicating with liquid in the lower part of said column and having its upper end located above the level of said outlet pipe for the dispersed phase, and a piston and cylinder connected to the upper end of said tube for applying a pulsating pneumatic pressure to the surface of the liquid in said tube so that said surface rises and falls in said tube.

14330 ✓

DEVELOPMENTS IN HORIZONTAL PULSED CONTACTORS FOR LIQUID-LIQUID EXTRACTION PROCESSES. D. H. Logsdail (Atomic Energy Research Establishment, Harwell, Berks, Eng.) and J. D. Thornton (University of Durham, Eng.). Reactor Technol. 1, 15-24(1959) Apr.

SOLVENT EXTRACTION 6.16

EQUIPMENT 6.16.02

Pulsed Columns 6.16.02.05

An air-pulsed horizontal mixer-settler is described along with a horizontal pulsed-plate column which uses a single air pulse line regardless of the number of stages. Experimental setups and methods of operation are described and mass transfer data are given and discussed. It is concluded that the air-pulsed mixer-settler and the horizontal pulse column perform comparably with conventional equipment. Air-pulsing gives an advantage in corrosive and radioactive processing. The horizontal pulse column offers advantages over other horizontal contactors in ease of construction, maintenance, and stability of operation, and cascades are more compact, saving on shielding costs. (T.R.H.)

THE CONCATENATED PULSE COLUMN. A. C. Jealous and E. Lieberman (Oak Ridge National Lab., Tenn.). Chem. Eng. Progr. 52, 366-70(1956) Sept.

The concatenated pulse column is described in detail and its operation and engineering properties discussed. (T.R.H.)

THE VALVE ACTUATED PULSE COLUMN. 2. A STUDY OF THE TEMPERATURE EFFECT AND DESIGN VARIABLES. L. L. Burger and L. H. Clark. Feb. 16, 1953. Decl. Feb. 28, 1957. 23p. (HW-26459)

The valve-actuated pulse column is employed to give phase separation on each half cycle in the countercurrent flow cycle and provides independent control over each step in the cycle. The dependence on temperature of operation, on pulse frequency and amplitude, and on plate design and spacing has been studied for extraction and stripping of U with TBP in a hydrocarbon diluent. It is found that the efficiency increases quite significantly as the temperature is raised regardless of the direction of transfer and this increased efficiency is accompanied by greater operational freedom. A decrease in hole size, decrease in plate separation and increase in frequency all lead to higher efficiency in agreement with the concept that small drops continually formed coalesced and reformed, lead to maximum interphase transfer. The high extraction efficiency of the column is probably aided further by the relatively sharp pulse produced.

20984 CF-59-6-119
Oak Ridge National Lab., Tenn.
THEORETICAL STUDY OF SINGLE-TRANSFER LINE CONCATENATED PULSE COLUMN SYSTEMS. H. F. Johnson. June 19, 1959. 22p. Contract [W-7405-eng-26]. $4.80(ph), $2.70(mf) OTS.

Calculations indicate that single-transfer line concatenated pulse column systems can be operated with static pressures that are not excessive if a sufficient number of vessels are employed in the system. The required number of vessels can be attained by using a series of short columns or by using holdup pots in conjunction with a limited number of columns. General equations for calculating pressure drops and power requirements are presented. (auth)

20872 AECU-4329
Massachusetts Inst. of Tech., Oak Ridge, Tenn. Engineering Practice School.
AN ORIFICE PLATE PULSE COLUMN FOR LIQUID-LIQUID EXTRACTION (thesis). D. C. O'Brien. Aug. 23, 1954. 70p. For Carbide and Carbon Chemicals Co. K-25 Plant. Contract W-7405-eng-26, Subcontract 70. (KT-170). $10.80(ph), $3.90(mf) OTS.

A study was made of the performance of an orifice plate pulse column which is essentially a spray column containing internal constrictions in the form of orifices. The chemical system studied was acetic acid-water-hexone, and the variables considered were pulse frequency, throughput, phase continuity, and direction of solute transfer. For comparison purposes, several runs were made with a conventional spray column. Curves are presented showing the effect of the column variables on the column efficiency (HTU). The trends observed are explained as being due principally to the competing effects of the area for mass transfer and back-mixing. Photographs of a typical column section show qualitatively the effect of the important column variables. (auth)

21948 ISC-1095
Ames Lab., Ames, Iowa.
PULSE COLUMN DESIGN. Lawrence E. Burkhart and R. W. Fahien. Nov. 1958. 118p. Contract W-7405-eng-82. $2.75(OTS).

A stagewise approach was used in a theoretical analysis of pulse columns. In the analysis the column was arbitrarily divided into discrete stages comprising that

SOLVENT EXTRACTION 6.16

EQUIPMENT 6.16.02

Pulsed Columns 6.16.02.05

part of the column between two adjacent perforated plates. The operation of the pulse column was described mathematically using material balance equations, and a design method was derived which used two stage lines and two operating lines, one set for the pulse generator upstroke and one set for the downstroke. Assuming equilibrium contact, the effect of recycle in a pulse column was shown to cause a large decrease in the separation obtained as the pulse frequency was increased. Hold-up studies were made using isoamyl alcohol–water, and methyl isobutyl ketone–water. The hold-up per cycle of the dispersed phase for both systems was found to be equal to the interstage flow per cycle of the dispersed phase. Hold-up behavior at high frequencies was different for the two systems. Extraction runs were also made using the system methyl isobutyl ketone–acetic acid–water. The effects of recycle were found to result in a drop in column separation efficiency with increased pulse frequency. It was found to be theoretically possible for a column to operate in a pinched-in region even though this is not apparent from an examination of the superficial flow ratio of the two streams being fed to the column. Techniques for sampling interstage flow streams in a column operating in the mixer-settler region are described. (J.R.D.)

PULSED-COLUMN AUXILIARIES 6.16.02.06

7966 HW-36836
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
PUREX PULSE GENERATOR OPERATION. P. B. McCarthy. May 11, 1955. Changed from OFFICIAL USE ONLY May 16, 1957. 11p. Contract [W-31-109-Eng-52]. $3.30(ph OTS); $2.40(mf OTS).

A large (Size 2) Purex pulse generator was installed in the 321 Building Tank Farm to pulse solutions in the prototype Purex HA Column. Flow sheet considerations indicated a need for information on leakage rates with various size weep holes in the pulse generator piston. In addition to leakage tests, experiments were conducted to determine the air required under the piston to hold the column contents away from the pulse generator piston thereby preventing leakage during shut down and to determine the time required to bleed the air trapped in the pulse leg when the column is first filled. (auth)

1260 HW-52051
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
A HYDRAULIC POWER UNIT FOR SOLVENT EXTRACTION COLUMN PULSE GENERATORS. V. P. Kelly. Aug. 23, 1957. 13p. Contract W-31-109-eng-52. $3.30(ph OTS); $2.40(mf OTS).

A power unit comprised of a reciprocating hydraulic cylinder controlled by hydraulic, mechanical, and electrical elements was developed to drive pulse generators for solvent extraction columns in the Purex Hot Semiworks. Design bases included remote control of continuously-variable pulse amplitudes between $\frac{1}{2}$ and $1\frac{1}{2}$ inches, and pulse frequency up to 120 cycles/min. Each of nine power units operated 30 to 60 million cycles without serious difficulty, although minor modifications and corrections were made during scheduled shutdowns of the Semiworks. Operational irregularities, such as erratic or drifting amplitudes, occurred with sufficient frequency to indicate deficiencies in the original design. The triangular pulse

11524
APPARATUS FOR PRODUCING PULSES IN FLUIDS.
L. Hack (to U. K. Atomic Energy Authority). British Patent 772,544. Nuclear Eng. 2, 350(1957) Aug.

A glandless apparatus with only a single moving part having a large clearance fit (to avoid seizure by solid particles in the fluid) in its sealed chamber as part of the pipe line. The moving part is a (soft iron) plunger oscillated between soft-iron stop rings by an external electromagnetic system of four symmetrically arranged laminated cores with a-c windings. A pulsing rate of up to 200 pulses/min is obtainable, e.g., in countercurrent packed extraction columns. (auth)

1259 HW-51884
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
PLUG–PISTON PULSE GENERATOR. V. P. Kelly. Aug. 12, 1957. 11p. Contract [W-31-109-Eng-52]. $1.80(ph OTS); $1.80(mf OTS).

SOLVENT EXTRACTION 6.16

EQUIPMENT 6.16.02

PULSED-COLUMN AUXILIARIES 6.16.02.06

A graphite plug-piston assembly with a stainless steel cylinder and a piston rod sealed by a lower throttle bushing and upper Teflon chevron packing was operated 25 million cycles pulsing liquid in 3- and 4-inch-diameter process columns. During operation to an annulus between the rod seals, no measurable piston leakage was observed. Without the air purge, or with the air purge between the piston and throttle bushing, piston leakage rates up to one gal/hr occurred. The CSGBF graphite piston wore four mils (diametrally) during the first half of the test, with no change thereafter. The wear rate of Polypenco K-51 throttle bushing was one-third that of pile graphite. A hardened 17-4 PH stainless steel piston rod and cylinder showed no wear. (auth)

4602

REDISTRIBUTOR FOR LIQUID–LIQUID EXTRACTION COLUMNS. J. G. Bradley (to U. S. Atomic Energy Commission). U. S. Patent 2,811,423. Oct. 29, 1957.

An improved baffle plate construction to intimately mix immiscible liquid solvents for solvent extraction processes in a liquid-liquid pulse column is described. To prevent the light and heavy liquids from forming separate continuous homogeneous vertical channels through sections of the column, a baffle having radially placed rectangular louvers with deflection plates opening upon alternate sides of the baffle is placed in the column, normal to the axis. This improvement substantially completely reduces stripping losses due to poor mixing.

14683 A/CONF.15/P/515

General Electric Co. Hanford Atomic Products Operation, Richland, Wash.

IMPROVED PULSED SOLVENT EXTRACTION COLUMN CARTRIDGE. R. G. Geier. 26p. $0.50(OTS).

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

It has been demonstrated that improved efficiencies and/or throughputs can be obtained in pulse columns if deviations from equally-spaced, identical stainless steel sieve plates are incorporated into the design of the column internals. Some of the techniques found advantageous in uranium tributyl phosphate in diluent systems include the utilization of: nozzle plates, sieve plates of different geometry and spacing, and sieve plates fabricated from different materials of construction. Test results are delineated and compared with those from other studies. The influence of plate geometry on the effects produced by mixing metal and plastic sieve plates is discussed. (auth)

A NEW APPROACH TO PULSE COLUMN PIPING. R. C. Hollingshead and J. Oliver Ludlow. Oct. 17, 1955. 4p. (HW-39560)

A pulse column using a pulse generator with two interconnecting lines containing check valves is described. The pulse generator accelerates alternately the fluid in each of the pipes. The need for a remote flange is eliminated, smaller pipes are used, and remote pulse generators will not be necessary.

Oak Ridge National Lab., Tenn.

FLOODING CHARACTERISTICS OF ¼-in. FLUORO-THENE RASCHIG RINGS. R. L. Boles and S. H. Stainker. Apr. 27, 1959. 14p. Contract [W-7405-eng-26]. $3.30(ph), $2.40(mf) OTS.

The flooding characteristics of fluorothene Raschig rings at low liquid rates with the existing literature data on carbon rings are compared. Visual observations of flooding in the air–water system were correlated with pressure drop using a 3-in. i.d. glass column having an 18-in. long fluorothene Raschig ring packed section. A comparison of extrapolated literature data with these experimental data indicates the gas flow rates required to flood fluorothene packing are slightly higher than for carbon packing. At a liquid flow rate of 537 lbs/hr-ft^2, fluorothene rings flooded at a gas velocity of 842 lbs/hr-ft^2 whereas carbon rings are reported to flood at a gas velocity of approximately 755 lbs/hr-ft^2. (auth)

10979 HW-30588

General Electric Co. Hanford Atomic Products Operation, Richland, Wash.

TEFLON BELLOWS PULSE GENERATORS FOR SOLVENT EXTRACTION PULSE COLUMNS. P. B. McCarthy. Jan. 1, 1954. Decl. Jan. 11, 1955. 11p. Contract W-31-109-Eng-52. $3.30(ph), $2.40(mf) OTS.

SOLVENT EXTRACTION 6.16

EQUIPMENT 6.16.02

PULSED-COLUMN AUXILIARIES 6.16.02.06

A Teflon bellows-type pulse generator is described which consists of two 3-in. nominal diameter Teflon bellows mounted on either end of a flanged spool piece and rigidly connected internally by a push rod so that the two bellows move in tandem. The ends of the two bellows are closed by means of blind flanges. The spool piece is designed for insertion in a 6-in. diameter nozzle on a solvent extraction pulse column. The double bellows arrangement constitutes a safety feature to prevent loss of the column contents in the event of failure of the inner bellows in contact with column solution. Failure of the inner bellows may be detected by a conductivity probe mounted in the air space inside of the double bellows assembly. Reciprocating motion is imparted to the bellows by means of an electrically driven crank arm rigidly connected through a cross head and push rod to the face of the external bellows flange. The push rod is guided by means of linear ball bushings. Frequency variation over a range of 30 to 100 cycles/min. was obtained by use of a Thymotrol-controlled electric motor to drive the crank arm. Variable stroke adjustment (0 to 1-in. range) was possible by adjustment of linkages on the crank arm. A load compensating spring was found desirable to counteract the thrust on the push rod resulting from the static pressure at the bottom of the solvent extraction column. Without the spring, accelerated wear of the bearing on the crank arm occurred. The pulse generator operated uneventfully for 1776 hours (6.61×10^6 cycles) at a frequency of 62 cycles/min. and a bellows travel of 1-in. (equivalent to a displacement of 1.6 in. in a 3-in. diam. column). (auth)

RADIATION DAMAGE TO SOLVENTS 6.16.03

See also, this section,

RADIATION EFFECTS--Organic Fluids (6.15.08).

DILUENTS 6.16.03.01

3653 AECU-3932
Georgia Inst. of Tech., Atlanta. Engineering Experiment Station.
RADIATION CHEMISTRY OF ORGANIC SUBSTANCES. Annual Report No. 1 [for] January 1, 1957 to December 31, 1957. Raymond G. Wymer. Mar. 31, 1958. 48p. For Oak Ridge National Lab. Project No. A-323. Contract W-7405-eng-26, Subcontract No. 1082. $9.30(ph OTS); $3.60(mf OTS).

Work is reported on solvent radiation damage and permissible irradiation levels for solvents used in processing power reactor fuels. Amsco 125-82 was selected for use in the initial studies. It is a diluent typical of the types commonly used for extractants employed in fuel processing and raw material processing. It has also been used as a heat exchange fluid in equipment exposed to high radiation fields. Amsco 125-82 was fractionated into its pure components so that these components could be investigated individually. (W.L.H.)

13667✓ HW-34502(Rev.)
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
INVESTIGATION OF SOLVENT DEGRADATION PRODUCTS IN RECYCLED URANIUM RECOVERY PLANT SOLVENT. R. H. Moore. Apr. 28, 1955. Decl. Feb. 27, 1957. 19p. Contract W-31-109-eng-52. $0.30(OTS).

Flowsheets are presented for the separation and identification of degradation products found in the recycled plant solvent (20% TBP in Shell diluent). It is shown that the impurities arising from the diluent are the principal cause of deterioration in solvent quality. (C.W.H.)

13628 ✓ CF-56-5-56
Oak Ridge National Lab., Tenn.
A PRELIMINARY STUDY OF ULTRAVIOLET SPECTRA FOR DETECTION OF SOLVENT DAMAGE BY RADIATION. J. R. Flanary, A. H. Kibbey, and V. L. Tweedie. May 7, 1956. Decl. Mar. 13, 1957. 22p. Contract W-7405-eng-26. $4.80(ph OTS); $2.70(mf OTS).

SOLVENT EXTRACTION 6.16

RADIATION DAMAGE TO SOLVENTS 6.16.03

Diluents 6.16.03.01

A preliminary study was made of the use of ultraviolet spectra for detection of radiation-induced chemical change in Special Naphtha No. 1, a refined kerosene recommended as diluent for tributyl phosphate in highly radioactive solvent-extraction processes. The ultraviolet spectrum was more sensitive to chemical change than the usual chemical tests. Unsaturation produced by radiation was indicated by ultraviolet spectra and by iodine number and chromyl chloride reduction; however, the data were insufficient to establish correlations of spectra and chemical tests. The maximum irradiation level was 595 β watt-hr/liter of solvent. Results of batch extraction and stripping tests with irradiated Special Naphtha No. 1 and tributyl phosphate are presented. (auth)

ETHERS 6.16.03.02

2804
A SURVEY OF THE RADIATION CHEMISTRY OF SOME ALIPHATIC ETHERS. Amos S. Newton (Univ. of California, Berkeley). J. Phys. Chem. 61, 1485-9(1957) Nov.

The products resulting from the irradiation of seven liquid aliphatic ethers with helium ions have been determined. Correlation between the ethers and comparison with previous alcohol radiolysis data show the alkyl—oxygen bond to be most susceptible to rupture. The total G-value for reduction equivalent of 7 to 10 is in the same range as previous results with alcohols. A postulated rearrangement of the ethers to an alkene and an alcohol is shown to follow the number of hydrogens on carbon atoms beta to the oxygen. Possible mechanisms for the formation of certain other products are discussed. (auth)

6541 A/CONF.15/P/2293
RADIOLIZ I RADIATSIONNOE OKISLENIE ORGANICHESKIKH SOEDINENII. (Radiolysis and Radiation Oxidation of Organic Compounds.) N. A. Bakh, V. I. Medvedovskii, and V. V. Saraeva (U.S.S.R.). 19p.

Data are presented on the radiation effects on extraction of plutonium by simple ethers from aqueous solutions and radiation-induced transformations in pure ethers and aqueous solutions. (R.V.J.)

2805
SOME ASPECTS OF THE RADIOLYSIS OF ISOPROPYL ETHER. Amos S. Newton (Univ. of California, Berkeley). J. Phys. Chem. 61, 1490-6(1957) Nov.

Data are presented on the effects of variations in total energy input (total dose), temperature, type of radiation, added iodine, and added acetaldehyde on the radiolysis products of isopropyl ether. The changes in product yield with changes in these variables have been qualitatively interpreted in terms of a mechanism involving both radical reactions and molecular disproportionations. (auth)

TBP 6.16.03.03

GAMMA RADIOLYSIS OF TRIBUTYL PHOSPHATE AND TRIBUTYL PHOSPHATE DILUENT SYSTEMS. L. L. Burger and E. D. McClanahan, Jr. Sept. 1, 1957. 20p. (HW-52943)

The liquid products from gamma radiolysis of TBP (tributyl phosphate) include DBP (dibutyl phosphate), butyl alcohol, MBP (monobutyl phosphate), butyl ether, phosphoric acid, hydrocarbons, and polymers. DBP is produced in the largest yield, to the extent of 0.14 g/whr of gamma energy absorbed by pure TBP. This corresponds to a G value of 1.8. Dilution with a paraffin hydrocarbon does not change the radiolysis yield of DBP. Saturation with water reduces the yield of DBP about 30%. Dilution with benzene reduces the yields while dilution with CCl_4 greatly increases the TBP composition. In the latter case a post-irradiation effect is observed. The chloride yield from TBP-CCl_4 mixtures is constant at about 0.07 g/whr absorbed by the CCl_4 for mixtures containing from 25 to 100% CCl_4. Phosgene and C_2Cl_6 are also produced. The presence of water or O_2 does not effect the chloride yield. The presence of U and HNO_3 in the TBP systems reduces the yields of both DBP and chloride. 8 references.

13362 CF-59-1-90
Oak Ridge National Lab., Tenn.
SOME EFFECTS OF RADIATION ON SOLVENT EXTRACTION PROCESSES. Wallace Davis, Jr. and Robert M. Wagner. Jan. 26, 1959. 14p. Contract [W-7405-eng-26]. $3.30 (ph), $2.40 (mf) OTS.

SOLVENT EXTRACTION 6.16

RADIATION DAMAGE TO SOLVENTS 6.16.03

TBP 6.16.03.03

For presentation at the A.C.S. Convention, Boston, Apr. 1959.

The yield of total acid, G_{acid}, in the radiolysis of tributyl phosphate-Amsco 125-82 solutions is 2.7 times the electron fraction of TBP, or approximately 2.7 times the weight fraction of TBP, per 100 ev of energy absorbed by the solution. Dibutyl phosphoric acid, DBPA, constitutes about 85% of the acid. Radiolysis of TBP also results in the conversion of about 0.9 molecules of TBP to a polymer per 100 ev of energy absorbed. Uranium extraction-stripping tests with an 8 stage spinner column have shown that one mole of uranium is retained in the organic phase during the stripping operation per mole of DBPA added prior to the extraction operation. This is at least twice as much uranium as would be expected on the basis of a compound of the composition $UO_2(DBP)_2$. On the basis of a tentative molecular weight of 843 g/mole, the polymer retains in the organic phase during stripping operations about 1.2 moles of uranium per mole of polymer added prior to extraction. In addition to polymer and acids, condensed phase radiolysis products include olefins, whole yield, expressed as $G_{c=c}$, or number of double bonds formed per 100 ev of energy absorbed, decreases from ca 4 to 1 as the TBP concentration increases from 4.5 to 100 wt. %. Among newer extractants being examined for use in radiochemical reprocessing, diethyl carbonate, DEC, has been tested for uranium retention and fission product decontamination properties. After irradiation to the 400 watt-hr/liter level, the uranium retention by DEC on stripping is decreased, rather than increased. Although γ decontamination was adversely affected, β decontamination was essentially unaffected by irradiation to this level. (auth)

11347 ✓ CF-58-6-91
Oak Ridge National Lab., Tenn.
STATUS OF SUBCONTRACT WORK ON EFFECTS OF RADIATION ON SOLVENT EXTRACTION PROCESSES. W. Davis, Jr. June 18, 1958. 8p. Contract [W-7405-eng-26]. $1.80(ph OTS); $1.80(mf OTS).

The results of the Stanford Research Institute studies of the effects of radiation on solvent extraction processes are summarized, and a desirable direction for future work is indicated. Studies performed to date have shown that: (1) nearly all of the irradiation-induced increase in time required for TBP–Amsco–aqueous phase separation is due to formation of a polymer, of unknown chemical structure; (2) uranium retention by the TBP–Amsco phase during stripping operations is due approximately 65% to dibutyl phosphate and 20% to the polymer; (3) each 100 ev of energy absorbed forms about 0.5 hydrocarbon double bonds, with which fission product iodine could readily react; (4) dibutyl phenylphosphonate is about six times as resistant, and dibutyl butylphosphonate is twice as resistant, to radiation damage as is tributyl phosphate. (auth)

7130 ✓ ORNL-2287
Oak Ridge National Lab., Tenn.
RADIATION DAMAGE TO TBP AND DILUENTS; COVERING PERIOD OCTOBER–DECEMBER 1956. J. H. Goode. May 24, 1957. 17p. Contract W-7405-Eng-26. $0.20(OTS).

Polymerization or other interaction between the radiation damage products of a hydrocarbon diluent and TBP solvent, which is intensified by the presence of an aqueous phase and/or heat, results in the formation of emulsifiers, uranium complexing agents, and other deleterious materials in irradiated solvent. Vacuum distillation satisfactorily separates undamaged solvent from the higher boiling degradation products. (auth)

5243 ✓
RADIOLYSIS OF TRI-N-BUTYL PHOSPHATE. T. F. Williams and R. W. Wilkinson (A.E.R.E., Harwell, Berks). Nature 179, 540(1957) March 9.

7176
THE RADIOLYSIS OF TRIBUTYL PHOSPHATE. John G. Burr (Oak Ridge National Lab., Tenn.). Radiation Research 8, 214-21(1958) Mar.

11359 ✓ DP-250
Du Pont de Nemours (E. I.) & Co. Savannah River Lab., Augusta, Ga.
THE FORMATION AND EFFECTS OF DIBUTYL PHOSPHATE IN SOLVENT EXTRACTION. E. Kenneth Dukes. Nov. 1957. 22p. Contract AT(07-2)-1. $0.75(OTS).

SOLVENT EXTRACTION 6.16

RADIATION DAMAGE TO SOLVENTS 6.16.03

TBP 6.16.03.03

Data are presented for the rates of formation of di-n-butyl phosphate (DBP) from tri-n-butyl phosphate. The effects of DBP on the extraction of zirconium from nitric acid and uranyl nitrate are reported. (auth)

5245 ✓ CF-57-4-9
Oak Ridge National Lab., Tenn.
ACID FORMATION IN THE RADIOLYSIS OF PHOSPHORUS ESTERS. W. H. Baldwin. Apr. 3, 1957. 5p. Contract [W-7405-eng-26]. $1.80(ph OTS); $1.80(mf OTS).
Samples of TBP were exposed to a Co^{60} source to determine what effect diluents, water, and oxygen have on the radiolytic decomposition of TBP and acid formation. (auth)

16538 ✓ NP-7643
Polish Academy of Sciences. Inst. of Nuclear Research, Warsaw.
THE HOT ATOM CHEMISTRY OF ORGANIC PHOSPHORUS COMPOUNDS. PART 1. THE PRODUCTS OF NEUTRON IRRADIATION OF TRIBUTYL PHOSPHATE. Report No. 71/V. I. G. Campbell, A. Poczynajło, and A. Sidua. Mar. 1959. 26p.
A study was made of the chemical effects of neutron capture in tributyl phosphate. A procedure is given for fractionation of the inorganic P^{32} and preliminary fractionation of the P^{32} in organic combination. Retention of P^{32} in organic combination was studied as a function of neutron dose rate. Inorganic P^{32} is present predominantly as orthophosphate in final products, a small percentage of the total activity also being present as orthophosphite. (auth)

8733 ✓
RADIATION EFFECTS IN SOLVENT EXTRACTION PROCESSES. T. Rigg (United Kingdom Atomic Energy Authority, Windscale, Cumb., Eng.) and W. Wild (Atomic Energy Research Establishment, Harwell, Berks, Eng.). pp. 320-31 in "Progress in Nuclear Energy. Series III. Process Chemistry. Volume 2."
The yields of gaseous products from the irradiation of solvents are given together with the yields of acid phosphates from the irradiation of tributyl and other trialkyl phosphates. For tributyl phosphate, the influence on these products of dilution with kerosene, water, nitric acid, and uranyl nitrate are described and results on the retention of plutonium, uranium, and fission products, applicable to extraction processes are given. (auth)

20583 AECU-4300
Stanford Research Inst., Menlo Park, Calif.
RADIATION STABILITY OF ORGANIC LIQUIDS. Semi-Annual Report No. 5 for Period January 1 to June 30, 1959. R. M. Wagner and L. H. Towle. July 3, 1959. 26p. SRI Project No. SD-2080. For Oak Ridge National Lab. Contract W-7405-eng-26, Subcontract 1081. $4.80(ph), $2.70(mf) OTS.
The research conducted during this period was concerned with the degree of protection conferred upon TBP by certain organic additives in the Amsco diluent, the nature and degree of damage to TBP when exposed simultaneously to both 12 N nitric acid and radiation (100 or 400 whr/liter), irradiation of di-sec-butyl phenylphosphonate and dilutions of this compound in o-xylene–Amsco, studies of the efficiency of acidic species extraction from TBP with various caustic solutions, development of an acceptable method for the codetermination of nitric acid and DBP in TBP–Amsco systems, and the irradiation of N-benzyl-1-nonyl decyl amine and N-benzyl-1-undecyl dodecyl amine to 400 whr/liter levels. These six groups of studies are summarized in detail and the salient results are noted for each group. (auth)

OTHER SOLVENTS 6.16.03.04

16206
DIFFUSION AND HOT RADICAL KINETICS IN THE PHOTOLYSIS OF METHYL IODIDE IN CYCLOHEXANE. Roswell F. Pottie, William H. Hamill, and Russell R. Williams, Jr. (Univ. of Notre Dame, Ind.). J. Am. Chem. Soc. 80, 4224-30(1958) Aug. 20.
Solutions of methyl iodide in cyclohexane were photolyzed at 2537 A. Products and their quantum yields were: methane 0.190, cyclohexyl iodide 0.085, cyclohexene 0.057, iodine 0.048, hydrogen iodide 0.009. Methane is attributed to reaction of hot methyl radicals with solvent and to reaction of thermal methyl radicals with hydrogen iodide. Cyclohexene is attributed to a diffusion

SOLVENT EXTRACTION 6.16

RADIATION DAMAGE TO SOLVENTS 6.16.03

OTHER SOLVENTS 6.16.03.04

controlled reaction between cyclohexyl radicals and iodine atoms which remain in proximity after the hot reaction. Hydrogen iodide is twice as effective as iodine in reacting with methyl radicals. Recombination of methyl radical–iodine atom pairs following dissociation varies exponentially with the square root of hydrogen iodide concentration, indicating a diffusion-controlled process. (auth)

7175

RADIATION PROTECTION IN CYCLOHEXANE. Milton Burton, J. Chang, S. Lipsky, and M. P. Reddy (Univ. of Notre Dame, Ind.). Radiation Research 8, 203-13(1958) Mar.

4349

CHEMICAL EFFECTS OF NUCLEAR RADIATIONS ON 8-HYDROXYQUINOLINE IN AQUEOUS SOLUTION. S. Kertes, M. Lederer and M. Lefort. J. chim. phys. 53, 959-62(1956) Nov.–Dec. (In French)

A study was made of the effect of γ radiation on aqueous 0.1N HCl solutions of 8-hydroxyquinoline in order to determine whether this compound remains useful for the analysis and separation of fission products or other highly radioactive elements. The 8-hydroxyquinoline in aqueous HCl solution is extremely radioresistant and can be used in the presence of tens of curies of radioelements. Among the radiolysis products isolated by paper chromatography, some of them conserve the complex properties and have the neighboring structure of the exit oxine. For the others, formed in the course of a prolonged degradation, the cycle containing the nitrogen has been destroyed, but a benzene nucleus remains. (tr-auth)

7129

EFFECT OF GAMMA-RAYS ON DITHIZONE IN SOME ORGANIC SOLVENTS. J. Wilkinson and H. J. M. Fitches (Univ. of Durham). Nature 179, 863-4(1957) Apr. 27.

RADIATION STABILITY OF ORGANOPHOSPHORUS COMPOUNDS. R. M. Wagner, E. M. Kinderman, and L. H. Towle (Stanford Research Inst., Menlo Park, Calif.). Ind. Eng. Chem. 51, 45-6(1959) Jan.

Radiation damage to organophosphorus compounds used as extractants for uranium and plutonium can markedly influence process efficiency. Small changes in structure are important. (auth)

16278

THE DECOMPOSITION OF AQUEOUS SOLUTIONS OF HYDROXYLAMINE BY γ RAYS. Marc Lefort and Xavier Tarrago. Compt. rend. 247, 454-6(1958) July 28. (In French)

The γ rays of radiocobalt decompose hydroxylamine into ammonia, nitrogen, and hyponitrous acid. In the presence of oxygen some nitrates are formed. Measurement of the initial yields of the products formed permits the determination of the yield in free radicals: $G_H + G_{OH}$ are the result of the radiolysis of water. (tr-auth)

123 AEC-tr-3059

THE INFLUENCE OF GAMMA RAYS ON THE OXIDATION OF CETANE. M. A. Proskurnin, Yu. L. Khmel'nitskii, E. V. Barelko, A. T. Slepneva, and I. I. Melekhonova. Translated by V. Beak from Doklady Akad. Nauk S.S.S.R. 112, 886-9(1957). 7p.

RADIOLYSIS OF SIMPLE KETONES. P. Ausloos and J. F. Paulson (Univ. of Rochester, N. Y.). J. Am. Chem. Soc. 80, 5117-21(1958) Oct. 5.

The liquid and vapor phase radiolysis of acetone, methyl ethyl ketone, and diethyl ketone has been investigated briefly. The effect of temperature and scavengers on the G-values of the volatile products has been interpreted on the basis of known radical processes. In the case of acetone it has been shown that at least 85% of the methyl radicals are thermalized before abstracting a hydrogen atom from acetone. In the liquid phase recombination products were found to be present in appreciable amounts and were not removed by iodine or DPPH at concentrations up to 2.5×10^{-2} mole/1. (auth)

SOLVENT EXTRACTION 6.16

RADIATION DAMAGE TO SOLVENTS 6.16.03

Other Solvents 6.16.03.04

√ **20584** CF-59-1-90(Rev.)
Oak Ridge National Lab., Tenn.
SOME EFFECTS OF RADIATION ON SOLVENT EXTRACTION PROCESSES. Wallace Davis, Jr., and Robert M. Wagner. Jan. 26, 1959. 14p. Contract [W-7405-eng-26]. $3.30(ph), $2.40(mf) OTS.

Presented at the 135th National Meeting of the A.C.S., Boston, April 5-10, 1959.

The yield of total acid, G_{acid}, in the radiolysis of tributyl phosphate–Amsco 125-82 solutions is 2.7 times the electron fraction of TBP, or approximately 2.7 times the weight fraction of TBP, per 100 ev of energy absorbed by the solution. Dibutyl phosphoric acid, DBPA, constitutes about 85% of the acid. Radiolysis of TBP also results in the conversion of about 0.9 molecules of TBP to a polymer per 100 ev of energy absorbed. Uranium extraction-stripping tests with an 8 stage spinner column have shown that one mole of uranium is retained in the organic phase during the stripping operation per mole of DBPA added prior to the extraction operation. This is at least twice as much uranium as would be expected on the basis of a compound of the composition $UO_2(DBP)_2$. On the basis of a tentative molecular weight of 843 g/mole, the polymer retains in the organic phase during stripping operations about 1.2 moles of uranium per mole of polymer added prior to extraction. In addition to polymer and acids, condensed phase radiolysis products include olefins, whose yield, expressed as $G_{c=c}$, or number of double bonds formed per 100 ev of energy absorbed, decreases from ca 4 to 1 as the TBP concentration increases from 4.5 to 100 wt. %. Among newer extractants being examined for use in radiochemical reprocessing, diethyl carbonate, DEC, has been tested for uranium retention and fission product decontamination properties. After irradiation to the 400 watt-hr/liter level, the uranium retention by DEC on stripping is decreased, rather than increased. Although γ-decontamination was adversely affected, β-decontamination was essentially unaffected by irradiation to this level. (auth)

1554 ✓
OXIDATION OF CHLOROBENZENE IN AQUEOUS SOLUTIONS BY THE ACTION OF IRRADIATION. N. P. Krushinskaya and M. A. Proskurnin (L. Ya. Karpov Inst. of Physical Chemistry, Moscow). Zhur. Fiz. Khim. 33, 1954-61(1959) Sept. (In Russian).

The radiolytic oxidation of chlorobenzene in aqueous solutions was studied in vacuum and on continuous passage of oxygen. Under vacuum conditions analysis of the gaseous products showed the presence of oxygen and the complete absence of hydrogen. It was found that in addition to phenol and the isomers of chlorophenol non-phenolic types of compounds representing hydrogenated derivatives of diphenyl are formed. The presence of molecular oxygen leads to a threefold increase in the yield of phenols at the expense of the hydrogenated condensation products. An important part in this process was shown to be played by the radiation hydrolysis of chlorobenzene. On the basis of the data obtained the mechanism of hydroxylation of chlorobenzene was investigated. (auth)

REAGENT ADJUSTMENT AND RECOVERY 6.16.04

See also, this section,

RADIATION EFFECTS--Aqueous Solutions (6.15.01),
Organic Fluids (6.15.08).

CHLORIDES 6.16.04.01

CHLORIDE REMOVAL FROM METAL WASTE SOLUTIONS. L. H. Clark. Oct. 10, 1950. Decl. Feb. 19, 1957. 13p. (HW-19105)

Chloride ion in the RAW waste stream from the TBP metal recovery process would excessively corrode the stainless steel in a HNO_3 recovery step. The conditions under which peroxysulfate ($K_2S_2O_8$) will oxidize quantitatively the Cl^- to Cl_2, which may be removed by air sparging, are presented in detail. A preliminary survey of other oxidants was made. The results of this survey are tabulated, as are the per cent decomposition of $S_2O_8^{-2}$ by synthetic RAW, and formation of ClO_3 and ClO_4 during Cl^- removal.

SOLVENT EXTRACTION 6.16

REAGENT ADJUSTMENT AND RECOVERY 6.16.04

ENTRAINMENT 6.16.04.02

1575 ✓ MCW-1437
Mallinckrodt Chemical Works. Uranium Div., Weldon Spring, Mo.
DE-ENTRAINMENT OF ORGANIC EXTRACT BY ELECTROSTATIC PRECIPITATION. W. C. Philoon and J. T. Krieg. Nov. 6, 1959. 9p. Contract W-14-108-eng-8. OTS.

A pilot-scale electrostatic precipitator was evaluated in the Weldon Spring uranium refinery as a de-entrainment device for purifying organic extract. During the 4-week test period the treater required no maintenance and very little operator attention. Optimum performance occurred when the extract had at least one stage of washing prior to electrostatic treatment. Feed to the precipitator contained as much as 10% entrained material. With good temperature control, effluents containing as little as 0.001% entrained material were obtained. (auth)

13079 HW-46701(Del.)
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
BIBLIOGRAPHY AND ABSTRACTION OF DEENTRAINMENT LITERATURE. R. H. Bond and J. H. Murray. Nov. 13, 1956. Decl. with deletions June 3, 1958. 49p. Contract W-31-109-Eng-52. $7.80(ph OTS); $3.30 (mf OTS).

An abstract bibliography on deentrainment consisting of 81 references to periodical and research report literature is presented. It was prepared for Hanford personnel engaged in work requiring solution of deentrainment problems. (T.R.H.)

14669 ✓ A/CONF.15/P/497
Dow Chemical Co. Western Div., Pittsburg, Calif.
ENTRAINMENT STUDIES ON SOLVENT EXTRACTION OF URANIUM FROM HEAVY SLURRIES. D. A. Ellis, R. S. Long, and J. B. Byrne. 7p. $0.50(OTS).

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

A study was made of entrainment and of the design of contacting equipment to minimize losses. Solvent extraction processes can be operated in mixer-settler systems, pump mixers, internal mixer-settlers, pulse columns, or centrifugal contactors. Since the internal mixer-settler gives the lowest entrainment of solvent in heavy slurries, a laboratory unit of this type was used in making entrainment studies. Part of the solvent entrained in a slurry can be recovered by dilution with water. Addition of a surface-active agent to slurry-containing, entrained solvent has a deleterious effect, but addition of agents to slurry before contact with solvent is effective in reducing the subsequent entrainment. In general, entrainment is decreased with hydrophilic agents but is increased with hydrophobic agents. This effect is probably related to charges in solid-liquid contact angles. The more effective agents found were all organic sulfonates. (auth)

6592 DOW-145
Dow Chemical Co. Western Div., Pittsburg, Calif.
PROGRESS REPORT [FOR] MAY–JUNE 1956. Research Dept. R. H. Bailes. July 1, 1956. 23p. Contract AT-30-1-GEN-236. $4.80(ph OTS); $2.70(mf OTS).

Investigations of the possibility of production of high grade uranium products directly from the solvent extraction process streams, of methods for decreasing solvent entrainment in extraction of heavy slurries, and of the preparation and testing of new types of solvents are presented. (auth)

4386 ✓ IDO-14486
Phillips Petroleum Co. Atomic Energy Div., Idaho Falls, Idaho.
THE DETERMINATION OF EXCESSIVE EMULSIFICATION BY COALESCENCE BEHAVIOR MEASUREMENTS. O. W. Parrett. Nov. 25, 1959. 24p. Contract AT(10-1)-205. OTS.

The development of a remotely operated device for determining the coalescence times of plant process streams suspected of containing surfactants such as silicic compounds and fission product zirconium compounds is described. A general correlation between the coalescence times of pilot plant extraction column aluminum nitrate feeds and 3.25 percent tributyl phosphate extractant streams and the observations of column behavior of these streams is demonstrated. The application of the coalescence test to plant streams is given. (auth)

SOLVENT EXTRACTION 6.15

REAGENT ADJUSTMENT AND RECOVERY 6.16.04

ENTRAINMENT 6.16.04.02

PROCESS FOR DECONTAMINATING LIQUID FLUOROCARBONS OF OIL SUSPENDED THEREIN. H. Schneider and B. J. Massey (to U. S. Atomic Energy Commission). U. S. Patent 2,860,830. Dec. 9, 1958.

Decontamination of a bulk of liquid fluorocarbon contaminated with oil and water can be accomplished by passing the contaminated material through a bed of either silica gel, activated alumina or activated carbon to absorb the contaminants.

GENERAL 6.16.04.03

MECHANISMS OF PROTECTION IN RADIOLYSIS OF ORGANIC SYSTEMS. Milton Burton and S. Lipsky (Univ. of Notre Dame, Ind.). J. Phys. Chem. 61, 1461-5(1957) Nov.

The addition of relatively small amounts of certain substances to organic systems markedly reduces yields of radiolysis products. Several mechanisms can be invoked to explain this "protective" effect. These can be broadly classified into two types: first, a scavenger effect of the additives on free radicals produced from primarily excited and ionized solvent molecules; second, more pertinent to our present interest, an effect of additive on the precursors of the first chemical decomposition.

2459 IDO-14489
Phillips Petroleum Co. Atomic Energy Div., Idaho Falls, Idaho.
CHARACTERIZATION OF SURFACTANTS IN ALUMINUM-URANIUM FUEL REPROCESSING SOLUTIONS. R. D. Cannon. Oct. 20, 1959. 21p. Contract AT(10-1)-205. OTS.

Surface active materials in aluminum nitrate-nitric acid fuel reprocessing solutions were characterized. Polymerized silica, zirconium-modified silica and soluble dibutyl phosphate species were found to contribute to stable emulsion formation. These surfactants were reduced in effectiveness by added acid. (auth)

THE PERFORMANCE OF A SMALL HELICAL TUBE HEAT EXCHANGER. C. V. Chester. Dec. 19, 1957. 16p. (CF-57-9-33)

A high-speed solvent extraction contactor is desirable for use in radiochemical processing plants to reduce radiation damage to solvent. Such a contactor is being developed, using steam jets for mixing and pumping and hydroclones for phase separation. A cooler may be necessary after each steam jet to control the temperature of the liquids in the cascade. Minimum holdup, low pressure drop, and dependability are prime requisites of the cooler. A helical tube heat exchanger was designed, constructed, and extensively tested for this service. The holdup was 242 cm^3, average heat transfer area was 0.145 sq. meters, and the pressure drop in the tube was given by: ΔP (in. Hg) = $0.81\ G_t^{1.65}$, where G_t is tube side flow rate, l/min.

8838 HW-19852
[Hanford Works, Richland, Wash.]
NEUTRALIZATION OF ACIDIC DISTILLATES WITH LIMESTONE. H. L. Brandt and R. E. Burns. Dec. 27, 1950. Changed from OFFICIAL USE ONLY Feb. 20, 1956. 7p. Contract [W-31-109-Eng-52]. $1.80(ph OTS); $1.80 (mf OTS).

Laboratory investigations are presented for the suitability of some commercially available limestones for neutralization of slightly acidic distillates. Observations were made of flow rates, solution acidities, and temperatures. (W.L.H.)

HNO_3 6.16.04.04

15137
NITRIC ACID RECOVERY FROM WASTE SOLUTIONS. A. S. Wilson (to U. S. Atomic Energy Commission). U. S. Patent 2,882,129. Apr. 14, 1959.

The recovery of nitric acid from aqueous nitrate solutions containing fission products as impurities is described. It is desirable to subject such solutions to concentration by evaporation since nitric acid is regenerated thereby. A difficulty, however, is that the highly radioactive fission product ruthenium is volatilized together with the nitric acid. It has been found that by adding nitrous acid, ruthenium volatilization is suppressed and reduced to a negligible degree so that the distillate obtained is practically free of ruthenium.

REAGENT ADJUSTMENT AND RECOVERY 6.16.04

HNO_3 6.16.04.04

REMOVAL OF CHLORIDE FROM NITRIC ACID BY OXIDATION WITH PERMANGANATE, G.P. Lang et al., Nov. 23, 1959. (MCW-1436).

2460 ✓ MCW-1436
Mallinckrodt Chemical Works. Uranium Div., Weldon Spring, Mo.
REMOVAL OF CHLORIDE FROM NITRIC ACID BY OXIDATION WITH PERMANGANATE. G. P. Lang, S. W. Weidman, W. P. Armstrong, G. L. Martin, and W. G. Bradford. Nov. 23, 1959. 28p. Contract W-14-108-eng-8. OTS.

Lab. scale only.

Nitric acid is commonly recovered from uranium refinery raffinates by distillation and subsequent rectification. The presence of chloride in these raffinates presents a serious corrosion problem during nitric acid recovery because of the considerable build-up of this ion during the rectification step and its corrosive effect on stainless steel process equipment. Laboratory investigation of this problem resulted in a method of control whereby chloride can be removed by oxidation to chlorine with potassium permanganate. Studies included the effect of temperature, hydrogen ion concentration, air sparge rate, stainless steel surface, and tri-n-butyl phosphate on the reaction of permanganate with chloride in nitric acid solutions. The use of $NaNO_2$ following treatment with permanganate was recommended to dissolve MnO_2 formed by side reactions and to reduce excess permanganate, eliminating the need for filtration of the treated acid. (auth)

TBP a serious interference.

NITRIC ACID RECOVERY FROM RAFFINATE BY EVAPORATION AND FRACTIONAL DISTILLATION. D. S. Arnold, A. Whitman, and F. J. Podlipec (National Lead Co. of Ohio, Fernald). Chem. Eng. Progr. 52, 362-5(1956) Sept.

A twelve-plate fractionating column has been found to exhibit efficiencies of 33 to 54% with HNO_3 vapor feed from a raffinate evaporator. (T.R.H.)

6680 ✓ CRDC-614
Atomic Energy of Canada Ltd. Chalk River Project, Chalk River, Ont.
ELECTRODIALYSIS OF FISSION PRODUCT SOLUTIONS. R. W. Durham and P. D. Goulden. Mar. 1957. 19p. (AECL-437).

The recovery of HNO_3 by means of electrodialysis through anion exchange membranes has been investigated using TBP process waste fission product solutions. A continuous process using three cells has been devised. When operating under steady state conditions, the catholyte in the last cell has a pH of about 10 and a precipitate containing 80 to 90% of the original $\beta\gamma$ activity is formed. The supernatant solutions contains mainly cesium with some strontium and ruthenium. The recovered nitric acid contains about 3% of the initial $\beta\gamma$ activity. (auth)

REDUCTION OF ACIDITY OF NITRIC ACID SOLUTIONS BY USE OF FORMALDEHYDE. T. V. Healy (to U. S. Atomic Energy Commission). U. S. Patent 2,835,555. May 20, 1958.

A continuous method is described of concentrating by evaporation and reducing the nitrate ion content of an aqueous solution of metallic salts containing nitric acid not in excess of 8N. It consists of heating the solution and then passing formaldehyde into the heated solution to bring about decomposition of the nitric acid. The evolved gases containing NO are contacted countercurrently with an aqueous metal salt solution containing nitric acid in excess of 8N so as to bring about decomposition of the nitric acid and lower the normality to at least 8N, whereupon it is passed into the body of heated solution.

8228
PROCESS FOR REDUCING ACIDITY OF NITRIC ACID SOLUTIONS CONTAINING METAL SALTS. T. V. Healy (to U. K. Atomic Energy Authority). British Patent 784,609. Nuclear Eng. 3, 186(1958) Apr.

In the processing of aqueous nitric acid solutions of chemical substances (fission products), wherein reduction of the bulk of the solutions is carried out by evaporation, the concentration of the nitric acid may become undesirably high. It has been found that in a solution in which the nitric acid concentration does not exceed 8N the following reaction takes place with formaldehyde: $-3\ CH_2O$ +

SOLVENT EXTRACTION 6.16

REAGENT ADJUSTMENT AND RECOVERY 6.16.04

HNO_3 6.16.04.04

$4\ HNO_3 \rightarrow 4\ NO + 3\ CO_2 + 5\ H_2O$, and nitric oxide is capable of decomposing nitric acid of concentration above 8N to produce nitrogen dioxide and water. Thus, the process of reducing the acidity is first carried out with a heated solution not exceeding 8N which is treated with formaldehyde, yielding nitric oxide which is passed into contact with a further solution of acidity above 8N to reduce this acidity.

8228
PROCESS FOR REDUCING ACIDITY OF NITRIC ACID SOLUTIONS CONTAINING METAL SALTS. T. V. Healy (to U. K. Atomic Energy Authority). British Patent 784,609. Nuclear Eng. 3, 186(1958) Apr.

In the processing of aqueous nitric acid solutions of chemical substances (fission products), wherein reduction of the bulk of the solutions is carried out by evaporation, the concentration of the nitric acid may become undesirably high. It has been found that in a solution in which the nitric acid concentration does not exceed 8N the following reaction takes place with formaldehyde: $-3\ CH_2O + 4\ HNO_3 \rightarrow 4\ NO + 3\ CO_2 + 5\ H_2O$, and nitric oxide is capable of decomposing nitric acid of concentration above 8N to produce nitrogen dioxide and water. Thus, the process of reducing the acidity is first carried out with a heated solution not exceeding 8N which is treated with formaldehyde, yielding nitric oxide which is passed into contact with a further solution of acidity above 8N to reduce this acidity.

9510 ✓ NLCO-721
National Lead Co. of Ohio. Cincinnati.
ELECTROLYTIC OXIDATION OF CHLORIDES IN NITRIC ACID. Wendell S. Miller, Edway R. Johnson, and Robert L. Doyle. June 25, 1958. 34p. Contract AT(30-1)-1156. $1.25(OTS).

An electric process for removing small amounts of chlorides from nitric acid was developed. Process variables were investigated to determine the feasibility of continuous flow cell. (auth)

2744 AD-129193
THE KINETICS AND MECHANISM OF NITRIC OXIDE DECOMPOSITION. A. I. Rozlovskii. Translated by Theodor Ranov from Zhur. Fiz. Khim. 30, 1349-55(1956). 14p.

KINETICS OF THERMAL DECOMPOSITION OF LIQUID NITRIC ACID. C. W. Tait, J. A. Happe, R. W. Sprague, and H. F. Cordes (U. S. Naval Ordnance Test Station, China Lake, Calif.). J. Am. Chem. Soc. 78, 2670-3(1956) June 20.

The decomposition of liquid HNO_3 at temperatures of 65 and 70°C was studied by observing the rate of evolution of O_2 from the system at atmospheric pressure. The rate data were interpreted in terms of a unimolecular decomposition of molecular N_2O_5, which results from the self-dissociation of HNO_3 into N_2O_5, NO_2^+, NO_3^-, and H_2O. Estimates of the H_2O from the dissociation obtained from this kinetic study agree with estimates obtained by other experimental techniques. Calculations based upon the parameters for the gas-phase decomposition of N_2O_5 indicate that the liquid-phase decomposition of HNO_3 can be explained if 3.2% of the total N_2O_5 from the self-dissociation is in the molecular form at 65° and 5.4% is in the molecular form at 70°C. (auth)

15073 ✓ AEC-tr-3735
NITRIC ACID OXIDATION KINETICS. [PART] II. E. Abel. Translated by K. S. Bevis (Savannah River Lab.) from Monatsh. Chem. 89, 79-82(1958). 4p. $1.80(ph), $1.80(mf) JCL.

A discussion is presented of the mechanism and kinetics of oxidation by nitric acid in the presence not only of nitrous acid but also nitric oxide. (auth)

22085
KINETICS AND MECHANISM OF NITROGEN OXIDATION UNDER ELECTRON IMPACT. M. T. Dmitriev and S. Ia. Pshezhetskii (L. Ia. Karpov Inst. of Physical Chemistry). p. 23-8 of "Primary Acts in Radiation Chemical Processes."

Data are presented on the mechanisms and kinetics of nitrogen oxidation under electron impact. Emphasis is placed on the action of fast electrons at atmospheric pressure. The amount of nitrogen oxides formed was measured in relation to irradiation time, composition of gas mixture, pressure, temperature, thickness of irradiated gas layer, beam strength, and electron energy. Data are tabulated. (C.H.)

22046
NITRIC ACID RECOVERY FROM WASTE SOLUTIONS. A. S. Wilson (to U. S. Atomic Energy Commission). U. S. Patent 2,882,129. Apr. 14, 1959.

SOLVENT EXTRACTION 6.16

REAGENT ADJUSTMENT AND RECOVERY 6.16.04

HNO_3 6.16.04.04

The recovery of nitric acid from aqueous nitrate solutions containing fission products as impurities is described. It is desirable to subject such solutions to concentration by evaporation since nitric acid is regenerated thereby. A difficulty, however, is that the highly radioactive fission product ruthenium is volatilized together with the nitric acid. It has been found that by adding nitrous acid, ruthenium volatilization is suppressed and reduced to a negligible degree so that the distillate obtained is practically free of ruthenium.

6232

PROTON RESONANCE SHIFTS IN NITRIC ACID SOLUTIONS OF ALUMINUM NITRATE. Robert C. Axtmann, Woodfin E. Shuler, and Bruce B. Murray (E. I. du Pont de Nemours and Co., Aiken, S. C.). J. Phys. Chem. 64, 57-61 (1960) Jan.

Proton magnetic resonance spectra were used to measure the dissociation of nitric acid between 25 and 80°C in solutions that were 0 to 1.5 M in aluminum nitrate. The salt repressed the dissociation of the acid markedly. At temperatures above room temperature, the salt was even more effective at diminishing the dissociation of the acid. The results at room temperature agree fairly well with a simple theory that combines a common ion effect due to the added nitrate ion and the assumption that water bound in the hydrated aluminum ion is unavailable for reaction with molecular nitric acid. (auth)

6482 A/CONF.15/P/2022

STUDIES IN THE RADIATION CHEMISTRY OF AQUEOUS SOLUTIONS. M. A. Proskurnin and Y. M. Kolotyrkin (L. Y. Karpov Inst. of Physical Chemistry, U.S.S.R.). 37p.

It is generally considered that the yields of radiation-chemical reactions in aqueous solutions are determined by the initial formation of approximately 4 pairs of radicals (H + OH) per 100 ev. New data are presented which indicate that it is possible to involve short-lived particles in reactions (radicals, ions, and excited molecules) whose role should not be overlooked and whose reaction capacity proves to be considerable. Studying the reaction of oxidation of Fe^{2+} in a vacuum and in an atmosphere of N_2O is a compelling reason for giving up the generally accepted Krenz–Dewhurst's scheme. T. Rigg's, G. Stein's, and J. Weiss' assumption regarding the oxidizing action of H_2^+ has not been confirmed in experiments on oxidation of Sn^{2+} and Ti^{3+} ions in a vacuum. The above contradictions are eliminated by the new assumptions. (auth)

SOLVENT EVALUATION 6.16.04.05

22011

MINIMUM CONSUMPTION OF FRESH SOLVENT IN MULTIPLE EXTRACTION. M. Vender (Inst. of Chemical Tech., Prague). Collection Czechoslov. Chem. Communs. 24, 2089-95(1959) July.

A procedure is shown for the calculation of the minimum consumption of fresh solvent in multiple extraction for the case of a ternary system with a region of limited miscibility. The proposed procedure can be used to calculate, with given initial and final concentrations of the raffinate and a given number of extraction stages, the feeds of fresh solvent in the various stages in order that the over-all consumption of solvent be minimal. It can also be used, with a given initial concentration of the raffinate, a given number of extraction stages, and a given over-all consumption of fresh solvent, to determine the partial feeds so as to attain the lowest concentration of the extracted constituent in the raffinate after the extraction. The use of the procedure is illustrated by an example. (auth)

5947 DP-178

Du Pont de Nemours (E. I.) & Co. Savannah River Lab., Augusta, Ga.

A TURBIDIMETER FOR IN-PROCESS ANALYSIS OF ORGANIC CONTAMINATION. Donald W. Colvin. Oct. 1956. 15p. Contract AT(07-2)-1. $0.20(OTS).

A turbidimeter is described which measures organic solvent content in aqueous-organic mixtures up to 1% organic. The probe may be placed in a remote process line. (T.R.H.)

A TEST FOR SOLVENT QUALITY. Thomas P. Garrett, Jr. Aug. 1957. 16p. (DP-237)

A method was devised to give a measure of the degradation products that appear in the solvent used in the extrac-

REAGENT ADJUSTMENT AND RECOVERY 6.16.04

SOLVENT EVALUATION 6.16.04.05

tion process for the recovery of U and Pu from irradiated material. The method is referred to as the Zr Index Test. The value or "Z" number obtained from the test makes it possible to predict the performance of the solvent in the extraction process. The method involves the equilibration of a portion of the solvent with a Zr tracer solution of known concentration and activity. The equilibrated solvent is washed three times with HNO_3 and an aliquot is mounted and counted. The activity remaining in the solvent and the known concentration and activity of the tracer are used to calculate the amount of Zr retained by the solvent in moles per billion liters. This value is the "Z" number.

5196
GENERALIZED ACIDITY IN RADIOCHEMICAL SEPARATIONS. Ralph A. Horne and Charles D. Coryell (Massachusetts Inst. of Tech., Cambridge) and Lionel S. Goldring (Nuclear Development Corp. of America, White Plains, N. Y.). Ann. Rev. Nuclear Sci. 6, 163-78(1956).
84 references.

9623 FMPC-427
National Lead Co. of Ohio, Cincinnati.
EXTRACTION COLUMN RAFFINATE CONTROL. B. G. Ryle, E. J. Beer, and J. F. Blum. May 1954. Decl. Feb. 19, 1957. 21p. Contract AT(30-1)-1156. $0.30(OTS).
An Ohmart density measurement system has been evaluated in the FMPC pilot plant as an instrumental method of preventing U losses in the extraction column raffinate stream. It has been established that the system is capable of detecting the presence of low U concentrations in a multicomponent, two-phase system as produced in the tributyl phosphate-nitric acid-uranium extraction process. (auth)

14224 IDO-14460
Phillips Petroleum Co. Atomic Energy Div., Idaho Falls, Idaho.
SPECTROPHOTOMETRIC STUDY OF THE TERNARY SYSTEM, ALUMINUM NITRATE–NITRIC ACID–WATER. APPLICATION TO ANALYSIS. Robert W. Henry and Glenn L. Booman. Mar. 31, 1959. 10p. Contract AT(10-1)-205. $1.80(ph), $1.80(mf) OTS.
The ternary system aluminum nitrate–nitric acid–water was studied spectrophotometrically. Acid and aluminum concentrations are determinable by measuring absorbances at 295 mμ and 2.667μ. This study appears worthy of evaluation as the basis of an in-line measuring system. (auth)

6249 AERE-C/M-66(Del.)
Gt. Brit. Atomic Energy Research Establishment, Harwell, Berks, England.
THE DETERMINATION OF SMALL CONCENTRATIONS OF NITRITE IN AQUEOUS AND BUTEX SYSTEMS. A. L. Mills, K. Saddington, and K. D. B. Johnson. Apr. 25, 1950. Decl. with deletions Dec. 18, 1956. 3p.
A number of methods for the determination of small amounts of nitrite in aqueous and solvent media, in the absence or presence of uranyl nitrate have been examined and are discussed. Only the Griess-Ilosvay method has proved satisfactory for the purpose. Various modifications of the method must be made when uranyl nitrate is present in the systems and these modifications are described. (auth)

NEW AND IMPROVED ANALYSES FOR TRI-N-BUTYL PHOSPHATE. Robert J. Allen and Michael A. DeSesa (National Lead Co., Winchester, Mass.). Nucleonics 15, No. 10, 88, 90, 92-4, 96, 98(1957) Oct.
The acid-saturation, decomposition, and uranium-saturation methods for the indirect determination of TBP were evaluated. The decomposition method is based on a conversion of the TBP to phosphoric acid followed by spectrophotometric or volumetric determination of the phosphate. The acid-saturation method consists of contacting the organic solvent containing TBP with a nitric acid solution and determining the amount of acid extracted into the organic phase. The uranium-saturation method consists of contacting the organic phase with a uranyl nitrate solution, and the amount of uranium extracted is determined fluorometrically or spectrophotometrically. New and improved versions of the saturation method are reported. The various reported versions of the decomposition method were not satisfactory either because of the excessive time and care required to obtain a complete conversion of TBP to phosphate or because of incomplete and variable decomposition of TBP. (L.T.W.)

SOLVENT EXTRACTION 6.16

REAGENT ADJUSTMENT AND RECOVERY 6.16.04

Solvent Evaluation 6.16.04.05

16253
ASSAY OF TRIBUTYL PHOSPHATE IN ORGANIC SOLVENTS. U. A. Sant and H. Sankar Das (Atomic Energy Establishment, Bombay). Anal. Chim. Acta 19, 202-3 (1958) Aug.

Tributyl phosphate may be determined in organic diluents such as kerosene by low-temperature alkali fusion to inorganic orthophosphate followed by titration with bismuthyl perchlorate. (T.R.H.)

DEGRADATION PRODUCTS OF TRIBUTYL PHOSPHATE. Robert A. Ewing, Samuel J. Kiehl, Jr., and Arthur E. Bearse. Feb. 17, 1956. Decl. Apr. 2, 1957. 54p. (BMI-1073)

A method for determination of dibutyl phosphate (DBP) in solvent streams containing tributyl phosphate (TBP) is outlined. The method is based on analysis for total U or total PO_4^{3-} after removal of U and PO_4^{3-} in excess of that present as uranyl dibutyl phosphate. Results on plant solvent samples are presented. Difficulties with precipitation of uranous dibutyl phosphate, when fresh solvents were employed, are discussed. Small amounts of nonphosphate U-complexing agents were found in plant solvents. Diluent degradation is postulated as the source of these components. Emulsion-formation tendencies appeared to correlate better with concentrations of these contaminants than with DBP content.

6498 A/CONF.15/P/31
THE SIGNIFICANCE OF CERTAIN COMPLEXES OF RUTHENIUM, NIOBIUM, ZIRCONIUM AND URANIUM IN PLANT PROCESSES. P. G. M. Brown, J. M. Fletcher, C. J. Hardy, J. Kennedy, D. Scargill, A. G. Wain, and J. L. Woodhead (Atomic Energy Research Establishment, Harwell, Berks, Eng.). 31p.

Solutions of fuels in nitric acid may contain a variety of ruthenium species, and methods for their identification and estimation in such solutions are presented. On the basis of laboratory investigations, the significance of the various species in solvent extraction processes (e.g., with dibutyl carbitol or tributyl phosphate as the solvent) and in related processes such as effluent treatment by precipitation, or by evaporation, are assessed and discussed. The complexing of uranium, zirconium, and niobium by tributyl phosphate and its degradation products arising from radiolysis or from hydrolysis has been examined and implications drawn as to the manner in which the formation of such complexes can affect zirconium and niobium decontamination factors in TBP separation processes. (auth)

TBP 6.16.04.06

12405 TID-10156
New Brunswick Lab., AEC, N. J.
QUARTERLY PROGRESS REPORT FOR THE PERIOD ENDING JUNE 30, 1952. C. J. Rodden. Dec. 24, 1952. Decl. Mar. 6, 1957. 144p. $0.80(OTS).

A study is made of the separation of Th from ThF_4 sludge, merits of carbonate leaching methods for recovery of U from by-product materials, separation of TBP from kerosene using 75% methanol, and removal of Ra from the filtrate of the ThF_4 precipitation using MgF_2, $CaCO_3$, and $BaSO_4$. Disproportionation of UF_6 up to 900°C, protective oil coating of machined U slugs and evaluation of oil coatings by accelerated corrosion tests, analysis of Th metal, flame photometry, and spectrochemical analysis of rare earths. An apparatus for the hydrofluorination of UO_2 is described. (J.E.D.)

13053 NLCO-744
National Lead Co. of Ohio, Cincinnati.
TBP-KEROSENE SOLVENT DEGRADATION: LITERATURE SEARCH. Robert H. Ellerhorst and Robert K. Klopfenstein. Feb. 11, 1958. 23p. Contract AT(30-1)-1156. $1.00(OTS).

A search of the classified and unclassified literature concerning the degradation of a tributyl phosphate (TBP)-kerosene solvent has been conducted. Chemical Abstracts, Nuclear Science Abstracts, Abstracts of Classified Reports, and the card file of the National Lead Company of Ohio's Library were included in the search. Pertinent information is presented, in abstract form, as it applies to extractant (TBP) degradation, diluent (kerosene) degradation, and solvent clean-up techniques. (auth)

SOLVENTS 6.16.05

See also, under FISSIONABLE MATERIALS--RECOVERY,

SOLVENT EXTRACTION--Synergistic Reagents (3.02.17.08)

8752 ✓
SOLVENTS FOR EXTRACTION PROCESSES. pp. 560-1 in "Progress in Nuclear Energy. Series III. Process Chemistry. Volume 2."
Tables are presented of solvents which have been chosen for operations in a full-scale production plant or have been considered for such use. (W.L.H.)

8751 ✓
STRUCTURES AND SOURCES OF EXTRACTION REAGENTS. K. B. Brown (Oak Ridge National Lab., Tenn.). pp. 557-9 in "Progress in Nuclear Energy. Series III. Process Chemistry. Volume 2."

AMINES 6.16.05.01

See also, under FISSIONABLE MATERIAL--RECOVERY,

SOLVENT EXTRACTION--Sulfate Systems (3.02.17.06).

7443 ✓ AECU-4631
Massachusetts Inst. of Tech., Cambridge.
EQUILIBRIUM EXTRACTION CHARACTERISTICS OF ALKYL AMINES AND NUCLEAR FUELS METALS IN NITRATE SYSTEMS. Quarterly Progress Report No. V for the Period July 1–September 30, 1959. Edward A. Mason and Victor C. Vaughen. Dec. 4, 1959. 22p. For [Oak Ridge National Lab.] under Contract W-7405-eng-26, Subcontract 1327. OTS.

Extraction of HNO_3 by trilauryl amine was studied by equilibrating equal volumes of aqueous and organic phases at 25°C. At HNO_3 concentrations of 2 to 10 N the acid in the organic phase in excess of that equivalent to the amine concentration was proportional to the concentration of HNO_3 in the equilibrated aqueous phase. Other workers report similar results with nitric acid and tri-n-octyl amine in benzene. Zirconium extractions carried out at 10g Zr/l with 0.35 M TLA nitrate in toluene showed a fourth power dependence of E_A^0Zr on HNO_3 (aq) over the range 2 to 10N. Maximum distribution ratios calculated from samarium scouting experiments using amines in kerosene were about 5×10^{-3} for Primene JMT, 10^{-4} for TLA, 10^{-5} for S-24, and less than zero for DTDA. Distribution ratios in Ru extractions ranged from E_A^0 Ru = 0.12 for 0.35M TLA shaken with an initially new 2N HNO_3 solution for twenty four hours, to E_A^0 Ru = 0.0018 for 0.1M TLA shaken with new 8N HNO_3 solution for fifteen minutes. Data on Zr and Ru standardization in TLA solution for spectrophotometric analyses are included. (For preceding period see AECU-4351.) (J.R.D.)

1580 ✓ CF-59-9-85
Oak Ridge National Lab., Tenn.
CHEMICAL TECHNOLOGY DIVISION, CHEMICAL DEVELOPMENT SECTION C, MONTHLY PROGRESS REPORT, SEPTEMBER 1959. K. B. Brown, K. A. Allen, C. A. Blake, C. F. Coleman, D. J. Crouse, B. Weaver, and R. P. Wischow. Oct. 20, 1959. 48p. Contract [W-7405-eng-26]. OTS.

The recovery of U from amines by the high nitrate–water stripping method is described. The amine extraction of $Pu(NO_3)_3$ from $Al(NO_3)_3$ solutions containing HNO_3 is discussed. The extraction of $Pu(NO_3)_4$ from HNO_3 with phenylphosphonate esters was studied. The effect of salt type on rare earth extraction from nitrate solutions by neutral organophosphorus reagents was studied. Neptunium was recovered nearly completely from laboratory salvage by extraction with a primary amine from H_2SO_4. The recovery of fission product Sr from a synthetic Purex 1-WW solution was studied. (W.L.H.)

21938 AECU-4351
Massachusetts Inst. of Tech., Cambridge.
EQUILIBRIUM EXTRACTION CHARACTERISTICS OF ALKYL AMINES AND NUCLEAR FUELS METALS IN NITRATE SYSTEMS. Quarterly Progress Report IV for the Period April 1–June 30, 1959. Edward A. Mason and Victor C. Vaughen. June 30, 1959. 42p. For [Oak Ridge National Lab]. Contract W-7405-eng-26, Subcontract 1327. $7.80(ph), $3.30(mf) OTS.

Work has progressed on the solvent extraction of zirconium, molybdenum, and samarium and has been initiated on ruthenium. For the first three metals,

SOLVENT EXTRACTION 6.16

SOLVENTS 6.16.05

AMINES 6.16.05.01

studies have been made on the following variables; amine concentration in the organic phase, metal concentration in the aqueous phase, nitric acid concentration in the aqueous phase, effect of an added nitrate on the extractions carried out in 2N HNO_3, diluent type (toluene and Amsco Odorless Solvent), and amine type and branching (tertiary-trilauryl amine, secondary–low branching di (tridecyl)amine, secondary – high branching – Amine S-24, and primary – Primene JMT). (auth)

15922 ✓ AECU-4239
Massachusetts Inst. of Tech., Cambridge.
EQUILIBRIUM EXTRACTION CHARACTERISTICS OF ALKYL AMINES AND NUCLEAR FUELS METALS IN NITRATE SYSTEMS. Progress Report for Period February 1, 1959–March 31, 1959. (Quarterly Report III). Edward A. Mason and Victor C. Vaughen. Mar. 31, 1959. 16p. For [Oak Ridge National Lab.]. Contract W-7405-eng-26, Subcontract No. 1327. $3.30(ph), $2.40 (mf) OTS.

In studies of equilibrium extraction characteristics of alkyl amines and fuels a program was initiated for investigation of Zr, Ru, Mo, Sm, Nb, Tc, and Ce in that order. Temporary delays are reported in the Zr and Ru programs, and interim work on Mo was started. U extraction studies, undertaken to provide a check on the procedures used for the other metals, were completed. Extraction agitation methods were also studied, and it appears that equally good contacting can be achieved by shaking or stirring. Also, preliminary results on the extraction behavior of Zr, Ru, and Mo are reported. (J.R.D.)

12706 ✓ WIN-61
National Lead Co., Inc. Raw Materials Development Lab., Winchester, Mass.
TECHNIQUES FOR THE EVALUATION OF AMINES AS URANIUM EXTRACTANTS AND PROPERTIES OF SOME SATISFACTORY AMINES. Henry G. Petrow, Oscar A. Nietzel, John C. Apidianakis, Robert W. Lindstrom, and Charles F. Lubinger. Aug. 26, 1957. 48p. Contract AT(49-6)-924. $7.80(ph OTS); $3.30(mf OTS).

The techniques used at the Winchester Laboratory to evaluate commercially available amines for use as U extractants from sulfate leach liquors are presented. The techniques include initial loss and steady state solubility loss to 0.5M H_2SO_4, U extraction and stripping properties, U capacity, solvent entrainment loss, and amine solubility loss to a leach liquor. The techniques are designed to yield maximum information with a minimum of analytical services. Data are presented for those amines which are considered as satisfactory for use as U extractants. The amines include Amine 9D-178, Amine S-24, triisooctylamine, trilaurylamine, Amine RC-3749, Amine D-205, Amine 7F-802, and N-benzylheptadecylamine. Data are also presented demonstrating that by the judicious choice of amine extractant, leach liquor potential, stripping reagent, and precipitation technique, a U concentrate can be produced from a variety of leach liquors which will meet concentrate specifications. (auth)

11118 ✓
INVESTIGATIONS IN NON-AQUEOUS SOLVENTS. PART II. SOLUBILITY AND CONDUCTANCES OF SOME RARE EARTH METAL AND THORIUM ACETATES IN ETHYLENEDIAMINE. T. Muniyappan and B. Anjaneyalu (Presidency Coll., Madras). Proc. Indian Acad. Sci. A45, 412-17 (1957) June.

In investigations preliminary to the study of the electrolysis of certain rare earth metal and thorium salts in anhydrous basic solvents, it was found that anhydrous acetates of La^{3+}, Ce^{3+} and Th^{4+} are moderately soluble in anhydrous ethylenediamine. The resulting solutions show the conductance behavior of weak electrolytes. Cerium^{3+} acetate solution in ethylenediamine undergoes oxidation in air to cerium^{4+}. (auth)

151 ORNL-2380
Oak Ridge National Lab., Tenn.
PROGRESS REPORT ON RAW MATERIALS FOR JUNE 1957. Chemical Technology Div. K. B. Brown, C. F. Coleman, D. J. Crouse, and A. D. Ryon. Oct. 17, 1957. 36p. Contract W-7405-eng-26. $1.00(OTS).

Some new organonitrogen compounds are described, and U extraction power in standard tests and reagent loss by distribution to an aqueous solution is reported. Preliminary attempts to adapt the Amex Process to treatment of ore leach slurries resulted in excessive volume losses of the organic phase and preferential loss of amine from the diluent. Uranium extraction isotherms were determined

SOLVENT EXTRACTION 6.16

SOLVENTS 6.16.05

AMINES 6.16.05.01

for two new tertiary amine samples. The stage efficiency of a baffled tank mixer for extraction of U by di(2-ethylhexyl) phosphoric acid in 97% kerosene – 3% TBP from a solution of typical composition increased with increased impeller speed at fixed residence time, ranging from 14% at 150 rpm to 92% at 1170 rpm in 4.8 min. Measurement of light scattering by amine species in benzene solution is described. (For preceding period see ORNL-2366.) (W.L.H.)

16954

AGGREGATION OF SOME OF THE AMINE EXTRACTANT SPECIES IN BENZENE. Kenneth A. Allen (Oak Ridge National Lab., Tenn.). J. Phys. Chem. 62, 1119-23(1958) Sept.

In light scattering measurements at 546 mμ the normal sulfates of tri-n-octylamine, di-1-isobutyl-3,5-dimethylhexylamine, methyldi-n-octylamine, 1,3-ethylpentyl-4-ethyloctylamine, and di-n-decylamine showed aggregation numbers of 1, 4, 9, 11, and 38, respectively, in benzene solution. Tri-n-octylamine bisulfate is dimeric. The linear dependences on concentration of the 90°/0° scattering ratios indicated that these solutes were monodisperse. The uranyl sulfate complexes of these amine sulfates were all monomeric except that of the primary amine, which appeared to be a dimer. The monomeric natures of tri-n-octylamine sulfate and of the di-n-decylamine sulfate–uranyl sulfate complex were confirmed by isopiestic and viscosimetric measurements. (auth)

214 ORNL-1959

Oak Ridge National Lab., Tenn.

AMINE EXTRACT PROCESSES FOR URANIUM RECOVERY FROM SULFATE LIQUORS. VOL. I. D. J. Crouse and K. B. Brown. Sept. 30, 1955. 84p. Contract W-7405-eng-26.

Bench scale studies have been made of processes for recovery of U from ore leach liquors by solvent extracting with long chain amines in an organic diluent. Several process schemes have been evaluated which show considerable promise both from the standpoint of operation and chemical costs. (auth)

4120 ORNL-2388

Oak Ridge National Lab., Tenn.

PROGRESS REPORT ON RAW MATERIALS FOR JULY 1957. Chemical Technology Div. K. B. Brown, C. F. Coleman, D. J. Crouse, and A. D. Ryon. Jan. 9, 1958. 25p. Contract W-7405-eng-26. $0.75(OTS).

Systematic Studies. Three new amine samples, tris(tridecyl)-, N-(1-undecyldodecyl)dodecyl-, and N-(1-nonyldecyl)-dodecylamine, showed good uranium extraction performance and low losses to aqueous liquors. Tests with a new batch of trilaurylamine continued to show the potential utility of this reagent. The purity of the new sample was lower than for previous batches, however, and the phase separations during extraction were slower. Tests with tri(n-octyl)phosphine oxide and 2-phenylvinyl(dioctyl)-phosphine oxide showed the latter to be the poorer extractant. In further tests of synergistic reagent systems for uranium extraction, diamyl amylphosphonate (DAAP) was shown to be a promising reagent for process use. The compound is potentially available in commercial quantities at a price lower than that of dihexyl hexylphosphonate (DHHP). Uranium extraction performance and losses to aqueous liquors are similar to those for DHHP. Process Development. The coefficients for extraction of uranium with tri-n-octylamine in kerosene–alcohol diluent were approximately equivalent with capryl alcohol, mixed decanols, or tridecanol in concentrations of 0.04 to 0.20 M alcohol. The coefficients decreased markedly when the alcohol concentration was increased to 0.3 and 0.5 M. With 0.10 M nonylphenol or 0.04 dinonylphenol added to the solvent, coefficients were similar in magnitude to those obtained with 0.04 to 0.20 M alcohol modifier. Addition of nonylphenol or dinonylphenol in concentrations as high as 0.2 M, however, severely depressed uranium extraction. The effect of small concentrations of chloride and nitrate on thorium recovery by the Amex process from uranium-barren Blind River liquors was evaluated. The chloride or nitrate enters the liquor during ion-exchange recovery of uranium. The presence of chloride depressed thorium extraction but not sufficiently to interfere seriously with process performance. Nitrate interference, on the other hand, was severe, indicating that special provisions would be necessary for treating uranium-barren raffinates where nitrate elution is used in the ion-exchange process. Nitrate was extracted effectively, suggesting the possibility of nitrate recovery from the liquor prior to thorium extraction. In addition, a process for simultaneous recovery of thorium and nitrate

SOLVENT EXTRACTION 6.16

SOLVENTS 6.16.05

AMINES 6.16.05.01

by the Amex process appears economically attractive and is being given further study. Engineering Studies. The rate of uranium extraction from two synthetic sulfuric acid leach liquors by the Dapex solvent was proportional to the cube root of power input in mixers of three different sizes. The rate was nearly independent of the particular combinations of uranium concentration, phase ratio, and solvent strength. Scale-up of mixers by constant power input per unit mixer volume was demonstrated to be slightly conservative for 6-, 12-, and 20-in. baffled tank mixers which are geometrically similar. (auth)

5567 ✓ ORNL-2099
Oak Ridge National Lab., Tenn.
URANIUM EXTRACTION WITH ORGANONITROGEN COMPOUNDS. Progress Report. D. J. Crouse, K. B. Brown, W. D. Arnold, J. G. Moore, and R. S. Lowrie. May 14, 1956. 68p. Contract W-7405-eng-26. $0.45(OTS).

A detailed study, including experimental data, on U extraction with long chain amines is presented. (For preceding period see ORNL-1734.) (auth)

SOLVENT EXTRACTION WITH ALKYL AMINES. C. F. Coleman, K. B. Brown, J. G. Moore, and D. J. Crouse (Oak Ridge National Lab., Tenn.). Ind. Eng. Chem. 50, 1756-62(1958) Dec.

The chemistry of extraction by organic solutions of amines and their salts is described. Several hundred organonitrogen compounds were tested for their extraction performance. Out of the compounds tested, simple amines of molecular weights in the range of 250 to 600 generally gave a more favorable extraction performance. Some of the reagents are commercial mixtures of compounds in isomeric or homologous series. This includes the iso-octyl-, tridecyl-, and trialkylmethyl groups. Extractions by the three classes of amines are similar, and also parallel to sorptions by the amine or alkylammonium groups in anion exchange resins. It is concluded that the liquid-form extractants make amine extraction more versatile than resin sorption. (J.H.M.)

14678 ✓ A/CONF.15/P/510
Oak Ridge National Lab., Tenn.
AMINE SALTS AS SOLVENT EXTRACTION REAGENTS FOR URANIUM AND OTHER METALS. C. F. Coleman, K. B. Brown, J. G. Moore, and K. A. Allen. 21p. $0.50 (OTS).

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

Extractions with organic solutions of high molecular weight alkylammonium salts can be described in terms of ion exchange equilibria, in close parallel to sorption by anion exchange resins. At the same time there are significant differences between the liquid and the resinous extractants, important to process, analytical, and physicochemical applications. A major advantage of the amine solutions, in addition to certain inherent advantages of liquid over solid and of disperse over polymerized reagents, lies in the wide variations of extraction power and selectivity available through choice of reagent structure and diluent. The versatility of this controlled extraction behavior is described in terms of extraction of acids, extractions of uranium(VI) from sulfate solutions, selectivity (i.e., relative extractions of various metal ions) from sulfate solutions, and extractions of several types of metal ions from different aqueous systems. (auth)

RECOVERY OF URANIUM VALUES. Keith B. Brown, D. J. Crouse, Jr., and J. G. Moore (to U. S. Atomic Energy Commission). U. S. Patent 2,877,250. Mar. 10, 1959.

A liquid-liquid extraction method is presented for recovering uranium values from an aqueous acidic solution by means of certain high molecular weight amine in the amine classes of primary, secondary, heterocyclic secondary, tertiary, or heterocyclic tertiary. The uranium bearing aqueous acidic solution is contacted with the selected amine dissolved in a nonpolar water-immiscible organic solvent such as kerosene. The uranium which is substantially completely extracted by the organic phase may be stripped therefrom by water, and recovered from the aqueous phase by treatment into ammonia to precipitate ammonium diuranate.

SOLVENT EXTRACTION 6.16

SOLVENTS 6.16.05

AMINES 6.16.05.01

1300
LONG-CHAIN AMINES. VERSATILE ACID EXTRACTANTS. Fletcher L. Moore (Oak Ridge National Lab., Tenn.). Anal. Chem. 29, 1660-2(1957) Nov.

A new anionic liquid-liquid extraction technique uses solutions of long-chain amines in chloroform to extract acids on complex metal acids from aqueous solutions. Extractions of some anionic complexes of Po, Pu, Zr, and Pa are demonstrated. (auth)

19887
SOLVENT EXTRACTION WITH AMINES. I. THE SYSTEM Th–HNO_3–TRIOCTYLAMINE. D. J. Carswell (New South Wales Univ. of Tech., Kensington, Sydney) and J. J. Lawrence (A.A.E.C. Research Establishment, Southerland, Australia). J. Inorg. and Nuclear Chem. 11, 69-74(1959) June.

The extraction of nitric acid and thorium from nitric acid solutions into tri-n-octylamine and tri-iso-octylamine is reported. The results confirm the existence of an anionic thorium complex $Th(NO_3)_6^{2-}$, and a mechanism is suggested for the extraction of this complex. (auth)

19757 ORNL-2771
Oak Ridge National Lab., Tenn.
EMULSION STABILIZATION BY SILICIC ACID. K. A. Allen and W. J. McDowell. Aug. 31, 1959. 16p. Contract W-7405-eng-26. $0.50(OTS).

With a given amine (vs. an aqueous phase containing silicic acid) the stability of the emulsion formed was dependent on the anion present in the order $SO_4^{=}$ (most stable) > Cl^- > ClO_4^- (least stable). Emulsion stability increased with silicon concentration and with the age of the silicic acid solution. Other organic solutes that also had electron-donating tendencies (e.g., oxygen atoms) showed smaller but real emulsion-forming abilities when contacted with silicic acid liquors. Organic additives with active hydrogen atoms decreased phase separation times of a typical silica liquor-amine sulfate solution dispersion by factors of 2 or more, presumably by competing with the silicic acid hydrogen atoms for bonding sites on the amine sulfate oxygens. It is suggested that silicic acid stabilizes oil-in-water type emulsions by hydrogen bonding to electron-donating groups adsorbed on the surfaces of the organic droplets and by forming typical silicic acid networks in the aqueous phase. (auth)

DIBUTYL CARBITOL 6.16.05.02

16240 TID-7555(p.240-55)
Oak Ridge National Lab., Tenn.
SOLVENT EXTRACTION. J. C. White. p.240-55 [of] FIRST CONFERENCE—ANALYTICAL CHEMISTRY IN NUCLEAR REACTOR TECHNOLOGY, NOVEMBER 4-6, 1957, GATLINBURG, TENNESSEE. 16p.

Organo-nitrogen and organo-phosphorus compounds used as analytical reagents are discussed. Experimental data are cited to show the utility of amines in extracting mineral acids, organic acids, Po, PuO_2^{2+}, Zr, Pa, Th, U, rare earths, and fission products using such compounds as tri-N-benzylamine, methyl-di-N-octylamine, and tri(iso-octyl) amine. The physical properties and extractability of various trialkyl phosphine oxides are tabulated, graphed, and discussed. (T.R.H.)

10738 AECD-4003
Tennessee Eastman Corp., Oak Ridge, Tenn.
REACTIVITY OF DIBUTYL CARBITOL. C. D. Susano. Apr. 1, 1947. Decl. Jan. 5, 1956. 24p. Contract W-7401-eng-23. (H-1.740.8(Paper 11)). $4.80(ph OTS); $2.70(mf OTS).

The nature and degree of reactivity of dibutyl carbitol–nitric acid mixtures are described. It has been observed that mixtures of dibutyl carbitol and HNO_3 containing NO or reducing agents are susceptible to varying degrees of reactivity depending on the concentration of the HNO_3. Indications are that the reaction of HNO_3 and dibutyl carbitol proceeds in two steps, fission and hydrolysis, and oxidation. Products of the reaction are CO_2, NO and monobutyl cellosolve, oxalic acid, and small amounts of organic acids. The presence of monobutyl carbitol, up to 20% in dibutyl carbitol enhances the distribution coefficient of dibutyl carbitol for U from aqueous, HNO_3 solutions. (W.L.H.)

SOLVENT EXTRACTION 6.16

SOLVENTS 6.16.05

DIBUTYL CARBITOL 6.16.05.02

11291 AERE-C/R-2221
United Kingdom Atomic Energy Authority. Research Group. Atomic Energy Research Establishment, Harwell, Berks, England.
SOME FISSION PRODUCT PARTITION DATA FOR BUTEX (DIBUTYL CARBITOL). H. A. C. McKay, K. Alcock, and D. Scargill. 1958. Date of MS. June 16, 1953. Decl. with amendment Sept. 10, 1956. 6p. $0.32(BIS).

Declassified version of AERE-C/R-1212.

The partition of traces of Y and Ce between water and butex was determined in the presence of HNO_3, using Y^{91} and Ce^{144} tracers. The partition of Zr between water and butex in the presence of HNO_3, using Zr^{95} as a carrier-free tracer, has also been determined. (W.L.H.)

8279 AERE-C/R-800
Gt. Brit. Atomic Energy Research Establishment, Harwell, Berks, England.
SOLVENT EXTRACTION OF Pu(VI) NITRATE FROM HNO_3 AND NH_4NO_3. T. V. Healy and A. W. Gardner. Feb. 15, 1952. Decl. June 28, 1956. 56p.

The absorption spectra of $Pu(NO_3)_4$ in dibutyl carbitol has been studied over a large range of acidity. Partition coefficients of $Pu(NO_3)_4$ has been obtained for the systems dibutyl carbitol/aqueous HNO_3 over the acidity range 0 to 9 normal and for the system dibutyl carbitol/aqueous HNO_3, NH_4NO_3 from 0 to 2N acid. The change over from the neutral dinitrate complex to the stable plutonyl trinitrate $HPuO_2(NO_3)_3$ in dibutyl carbitol has been demonstrated spectrophotometrically as well as by partition studies. This trinitratoplutonyl complex is responsible for the very marked enhancement of Pu^{4+} extraction into the organic solvent. As an example, Kp_u for the system, dibutyl carbitol and 8 molar NH_4NO_3 of 0.1 normal acidity (all dinitrate in the solvent) is 1.7 whereas on raising the acidity to 1.0 normal (all trinitrate in the solvent) Kp_u is 24. Similar studies carried out on a mixed solvent, 97% dibutyl carbitol plus 3% tributyl phosphate, showed a general decrease in the amount of the trinitrate complex at the higher acidities with an eventual production of what might be a dinitrate–TBP complex at very low acidities. While the partition of the trinitrate complex was higher than that of the neutral dinitrate (6 and 19 compared with the above figures of 1.7 and 24) the general level of partition at the lower acidities was much higher than in pure dibutyl carbitol. (auth)

Primary Separation Plant at Windscale Works, H.G.DAVEY. Nuclear Power v 1 n 2 June 1956 p 53-61. Details of British method of extracting plutonium from irradiated fuel elements; after removal from piles, irradiated fuel rods are first stored under water for several weeks; they are then dissolved in nitric acid and solution passed through three towers from which streams of fission products, plutonium and uranium are obtained by solvent extraction with dibutyl carbitol.

SEPARATION PROCEDURES

11119 AERE-C/R-518
Gt. Brit. Atomic Energy Research Establishment, Harwell, England.
THE EXTRACTION SOLVENT. PART 3. INFRA-RED DETERMINATION OF HYDROXYLIC IMPURITIES. J. Gaunt and G. M. Meaburn. Aug. 1950. Decl. Apr. 2, 1957. 18p.

The investigation of the O-H fundamental vibration bands of butyl alcohol, monobutyl carbitol, and monobutyl cellosolve in carbon tetrachloride and dibutyl carbitol solution is described. The position of the absorption bands indicates a high degree of association between the O-H group and the C-O-C linkage, which renders impossible the characterization of the hydroxylic impurity in crude Butex. The effect of water on the position and intensity of the absorption bands is also discussed. A method of analysis for monobutyl carbitol and water in dibutyl carbitol solution has been worked out and applied to the analysis of crude Butex, on the assumption that monobutyl carbitol is the chief hydroxylic impurity in this material. (auth)

SOLVENT EXTRACTION 6.16

SOLVENTS 6.16.05

ETHERS 6.16.05.03

9737 A-2329(Pt. II)
Brown Univ., Providence. Metcalf Research Lab.
INVESTIGATIONS RELATING TO THE CHEMISTRY OF URANIUM. PART II. INVESTIGATIONS RELATING TO THE USE OF POLYETHERS AS EXTRACTION AGENTS. [Report for the Period] May 1, 1944–March 15, 1946. E. Charles Evers and Charles A. Kraus. May 1, 1946. Decl. Dec. 8, 1955. 289p. (BT-68(Pt. II)). $45.00(ph OTS); $11.10(mf OTS).

A compilation is given of the results of numerous investigations relating to the separation of U^{6+} from aqueous solution by extraction with polyethers. Polyethers investigated are: dibutyl "Cellosolve," $C_4H_9OC_2H_4OC_4H_9$; dibutyl "Carbitol," $C_4H_9OC_2H_4OC_2H_4OC_4H_9$; dibutoxytriethylene glycol, $C_4H_9(OC_2H_4)_3OC_4H_9$; dibutoxytetraethylene glycol, $C_4H_9(OC_2H_4)_4OC_4H_9$. Diethyl ether and methylisobutyl ketone were also studied. The polyethers were specially prepared by Carbide and Carbon Chemicals Corporation and most of them are now available commercially. (auth)

10501
THE PROPERTIES OF INORGANIC COMPOUNDS IN ORGANIC SOLVENTS. I. THE HYDROLYSIS AND POLYMERIZATION OF QUADRIVALENT PLUTONIUM NITRATE SPECIES IN DIETHYLENE GLYCOL DIBUTYL ETHER. D. G. Tuck (United Kingdom Atomic Energy Authority, Industrial Group, Windscale Works, Cumberland, Eng.). J. Inorg. & Nuclear Chem. 6, 252-6(1958).

Color changes in weak acid solutions of plutonium tetranitrate in diethylene glycol dibutyl ether have been shown to be due to hydrolysis, and subsequent formation of a polymeric form of Pu^{4+}, which shows many correspondences with the colloid formed in aqueous solution. The polymer appears to be fairly stable in this solvent at room temperature but on heating the solution, a precipitate is formed; x-ray analysis shows that this is a hydrated plutonium dioxide. A possible reaction scheme for the formation of the polymer is discussed. (auth)

10832 √ AERE-C/R-2029
United Kingdom Atomic Energy Authority. Research Group. Atomic Energy Research Establishment, Harwell, Berks, England.
THERMODYNAMICS OF THE SYSTEMS HNO_3-H_2O-ETHERS. E. Glueckauf and B. Davies. Aug. 1956. 27p.

In the ternary system HNO_3-H_2O-Ether a great number of interactions between the components can be detected from an analysis of the activity and heat-of-mixing data. Solubility of H_2O in ethers alone at low concentrations of water is an endothermic process, but for ethers which can take up much water there is a co-operative effect between the dissolved water molecules, which renders the solution of water exothermic at higher water concentrations. Solution of HNO_3 in pure ether is strongly exothermic (~7,200 cal/mol) (apparently independently of the type of ether) and a 1:1 complex is formed by hydrogen bonding, which shows practically no dissociation except for ethers of high water solubility. The water uptake by water-free HNO_3-ether mixtures is an exothermic process, the heat release varying greatly with the affinity of the ether for water, from a few hundred cal/mol H_2O for Bu_2O to 4,000 for dibutylcarbitol. In equilibrium with aqueous solutions of nitric acid, the HNO_3 uptake by the ether is at first proportional to the HNO_3 activity, and there is an additional water uptake by the ether, at first proportional to the HNO_3 uptake. This varies from 0.5 H_2O per HNO_3 for n-di-hexyl ether to 8 H_2O per HNO_3 for penta ether. The affinity of the ether for HNO_3 rises in the same order as the uptake of excess water, and the above-mentioned heat effects suggest that it is the ternary Ether$-HNO_3-H_2O$ interaction rather than the binary Ether$-HNO_3$ interaction which determines the extraction affinity of the ether for HNO_3. All the ether solutions of nitric acid show, from their activity curves, that the dissolved nitric acid-ether complex is subject to a solute-solute attraction in the ether phase. In the case of poly-ethers, which contain more than 1 oxygen atom per molecule, when the molal ratio HNO_3: Ether = 1:1 is approached, the other ether oxygens also take part in binding HNO_3, but with less heat release than the first oxygen atom. In the case of mono-ethers, more than one molecule of nitric acid per molecule of ether can be extracted into the ether, provided the ether is sufficiently stable

SOLVENT EXTRACTION 6.16

SOLVENTS 6.16.05

KETONES 6.16.05.04

See also, this section,

SPECIAL PROCESSES--Redox (6.17.19).

2331 MonT-408
Clinton Labs., Oak Ridge, Tenn.
THE EXTRACTION OF URANYL NITRATE BY METHYL ISOBUTYL KETONE. A THERMODYNAMIC STUDY. H. M. Feder. Oct. 13, 1947. Decl. Dec. 6, 1955. 52p. Contract W-35-058-Eng-71. $9.30(ph OTS); $3.60(mf OTS).

The partition coefficient for un-ionized HNO_3 between hexone and H_2O is 3.85 ± 0.20. The dissociation constant of nitric acid in water-saturated hexone is $(2.5 \pm 0.5) \times 10^{-4}$. The hydrolysis and ionization of $UO_2(NO_3)_2$ in H_2O and the form of $UO_2(NO_3)_2$ in hexone are discussed, and its activity coefficients obtained. The equilibrium constant for the formation of un-ionized $UO_2(NO_3)_2$ in hexone from ionic $UO_2(NO_3)_2$ in H_2O is 0.19 ± 0.01. The distribution of $UO_2(NO_3)_2$ between hexone and H_2O containing HNO_3 and $Al(NO_3)_3$ is predicted and shown to be in reasonable agreement with experimental data. (auth)

12371 ✓ HW-19303
Hanford Works, Richland, Wash.
PHYSICAL PROPERTIES OF THE SYSTEM URANYL NITRATE–ALUMINUM NITRATE–NITRIC ACID–WATER–HEXONE. PART VI. DISTRIBUTION COEFFICIENTS OF PLUTONIUM (IV) NITRATE. C. Groot, I. M. Rehn, and C. M. Slansky. Dec. 1, 1950. Decl. Mar. 28, 1957. 16p. Contract W-31-109-eng-52. $0.30(OTS).

7120 AERE-CE/R-2408
United Kingdom Atomic Energy Authority. Research Group. Atomic Energy Research Establishment, Harwell, Berks, England.
PROPERTIES OF HEXONE IN RELATION TO ITS USE FOR URANYL NITRATE EXTRACTION. D. Bradley. 1958. Date of MS. Dec. 1953. Decl. Apr. 1957. 31p. $0.81(BIS).

Declassified version of AERE-CE/M-78.

This paper summarizes the properties of hexone, both chemical and physical, with particular emphasis on the partition of uranyl nitrate between hexone and water. (auth)

8806 KAPL-8
Knolls Atomic Power Lab., Schenectady, N. Y.
4-METHYL-2-PENTANONE (HEXONE). Charles A. Burkhard. Nov. 17, 1947. Changed from OFFICIAL USE ONLY Feb. 20, 1956. 36p. Contract [W-31-109-eng-52]. $6.30(ph OTS); $3.00(mf OTS).

An investigation was made to determine the impurities present in hexone, to determine the reaction products of hexone with HNO_3 and $K_2Cr_2O_7$ mixture, to prepare a pure sample of hexone, and to determine some of the physical properties of purified hexone. (auth)

10833 ✓ MonN-319
Clinton Labs., Oak Ridge, Tenn.
VARIABLES AFFECTING SOLVENT EXTRACTION AND A SUMMARY OF TWO CYCLE HEXONE EXTRACTIONS EMPLOYING IRRADIATED U-235. Problem Assignment Number TX5-11. Progress report [for] period Dec. 13, 1946 to May 1, 1947. F. R. Bruce. July 3, 1947. Decl. Mar. 2, 1957. 29p. Contract W-35-058-Eng-71. $7.80 (ph OTS); $3.30(mf OTS).

The effects of the following variables on the hexone extraction of uranium were investigated: temperature, and U, HNO_3, and salting-agent concentrations. Ruthenium was the main contaminant in the product resulting from two-cycle hexone extractions of $Al-U^{235}$ alloys. (C.W.H.)

144 AERE-C/R-542
Gt. Brit. Atomic Energy Research Establishment, Harwell, Berks, England.
THE INFLUENCE OF SULPHAMATE ON THE PARTITION OF NITRIC ACID BETWEEN WATER AND BUTEX. E. Busk and H. A. C. McKay. June 1950. Decl. with Amendment Nov. 1956. 14p.

The effect of (a) ammonium sulphamate and (b) sulphamic acid on the partition coefficient of nitric acid between butex and water has been determined, both in the absence and in the presence of 8N ammonium nitrate. A large part of the effect can be explained in terms of incomplete ionization of sulphamic acid, and pH titration curves of this acid indicate a dissociation constant of about the right magnitude. With sulphamic acid itself, however, there is an anomaly, and the partition data appear to conflict with the pH measurements. The suggestion is made that the amino-group in sulphamic acid may be very weakly basic.

SOLVENT EXTRACTION 6.16

SOLVENTS 6.16.05

KETONES 6.16.05.04

1331

ABSORPTION SPECTRA OF THE COMPLEXES OF URANIUM (VI) WITH SOME β-DIKETONES. H. I. Feinstein (U. S. Geological Survey, Washington). Microchem. J. 1, 237-44(1957).

The absorption spectra of the complexes of U^{6+} with four β-diketones were determined under various conditions of pH, concentration of uranium, and alcohol concentration. Under optimum conditions, the maximum molar absorptivity (31,200) is obtained using 2-furoyltrifluoroacetone. This compares with about 4,000 and 19,000 for the thiocyanate and dibenzoylmethane complexes, respectively. (auth)

A SYSTEMATIC STUDY OF SOLVENT EXTRACTION WITH ACETYLACETONE (thesis). Anoop Krishen. 1957. 140p. (NYO-6498)

Using Acetylacetone as solvent and chelating agent, the extraction behavior of ten metals was studied. It was possible to interpret the results from a theoretical point of view and, in some cases, to calculate the successive formation constants of the acetylacetonates. The versatility of separations was enhanced when extractions were carried out in the presence of a competing complexing agent. The effect of EDTA, fluoride and tartrate was studied for a number of metals. Schemes for separation of U from Bi, Cu from Al, and Hf from Zr were devised and tested. U was separated in the presence of EDTA and Hf with tartrate. In the case of Cu, separation from Al was achieved by the use of the continuous countercurrent distribution technique. The need for precise constants for various compounds was brought out and it was noted that when such values were available, better agreement of calculated and experimental values of extraction could be expected. Qualitatively, the following factors were found to affect the action of a masking agent: (a) stability of the complex formed, (b) ionization constant of the agent, and (c) the concentration of the agent. It was observed that the $pH_{1/2}$ value was raised in the presence of a masking agent, while the slope of the extraction curve was lowered.

ACETYLACETONE AS ANALYTICAL EXTRACTION REAGENT. CALCULATION OF SUCCESSIVE FORMATION CONSTANTS FROM EXTRACTION DATA. Anoop Krishen and Henry Freiser (Univ. of Pittsburgh). Anal. Chem. 31, 923-6(1959) May.

Acetylacetone has been employed as both a chelating agent and solvent in solvent extraction studies. To determine the formation constants, the extraction behavior of seven acetylacetonates was studied. Allowing for the presence of charged intermediates, the successive formation constants for nine acetylacetonates were calculated. These show close agreement with values obtained potentiometrically. In the case of copper(II) acetylacetonate, theoretical calculation of the extraction curve over the pH range 0 to 5 has produced a curve that is duplicated in an experimentally obtained curve. (auth)

STUDIES ON THE EXTRACTION OF METAL COMPLEXES. XXVII. THE DISTRIBUTION OF SOME ACTINIDES AND FISSION PRODUCTS BETWEEN METHYL ISOBUTYL KETONE AND AQUEOUS SOLUTIONS OF HNO_3 AND $Ca(NO_3)_2$. Jan Rydberg and Brita Bernstrom (Research Inst. of National Defence, Sundbyberg, Sweden). Acta Chem. Scand. 11, No. 1, 86-97(1957).

The distribution ratios of U^{6+}, Pu^{6+}, Pu^{4+}, Th^{4+}, Zr^{4+}, La^{3+}, Ca^{2+}, Na^{+} and HNO_3 between methyl isobutyl ketone and aqueous solutions of varying HNO_3 and $Ca(NO_3)_2$ concentrations have been determined and are given. Some possible methods of separating U and Pu from certain fission products are discussed. (auth)

2481

URANIUM RECOVERY PROCESS. H. H. Hyman and J. L. Dreher (to U. S. Atomic Energy Commission). U. S. Patent 2,893,824. July 7, 1959.

The recovery of uranium from the acidic aqueous metal waste solutions resulting from the bismuth phosphate carrier precipitation of plutonium from solutions of neutron irradiated uranium is described. The waste solutions consist of phosphoric acid, sulfuric acid, and uranium as a uranyl salt, together with salts of the fission products normally associated with neutron irradiated uranium. Generally, the process of the invention involves the partial

SOLVENT EXTRACTION 6.16

SOLVENTS 6.16.05

KETONES 6.16.05.04

neutralization of the waste solution with sodium hydroxide, followed by conversion of the solution to a pH 11 by mixing therewith sufficient sodium carbonate. The resultant carbonate-complexed waste is contacted with a titanated silica gel and the adsorbent separated from the aqueous medium. The aqueous solution is then mixed with sufficient acetic acid to bring the pH of the aqueous medium to between 4 and 5, whereby sodium uranyl acetate is precipitated. The precipitate is dissolved in nitric acid and the resulting solution preferably provided with salting out agents. Uranyl nitrate is recovered from the solution by extraction with an ether such as diethyl ether.

7468

SOLVENT FOR EXTRACTING ACTINIDE SALTS. L. Kaplan (to U. S. Atomic Energy Commission). U. S. Patent 2,910,442. Oct. 27, 1959.

A mixture of hexone and 2-hexylpyridine can be used for the selective extraction of actinide values.

ORGANOPHOSPHOROUS 6.16.05.05

See also, this section,

TBP (6.16.05.08),

and,

under FISSIONABLE MATERIAL--RECOVERY,

SOLVENT EXTRACTION--Phosphate Systems (3.02.17.05).

TRIALKYL PHOSPHATES AND DIALKYL ALKYLPHOSPHONATES IN URANIUM AND THORIUM EXTRACTION. T. H. Siddall, III (E. I. du Pont de Nemours & Co., Aiken, S. C.). Ind. Eng. Chem. 51, 41-4(1959) Jan.

The ability of organic compounds that contain the phosphoryl group to extract lanthanides, actinides, and zirconium has been reported. However, most of the work reported has dealt with tri-n-butyl phosphate (TBP) and not with efforts to improve selectivity by altering the substituents on the phosphorus atom. The objective of the work presented was to examine the potential of several trialkyl phosphates and dialkyl alkylphosphonates for extracting uranium and thorium from nitric acid solutions and for separating them from zirconium. (auth)

133

STUDIES ON MONO- AND DI-n-BUTYLPHOSPHORIC ACIDS. II. THE SOLUBILITY AND DISTRIBUTION OF MONO- AND DI-n-BUTYLPHOSPHORIC ACIDS IN AQUEOUS–ORGANIC SOLVENT SYSTEMS. C. J. Hardy and D. Scargill (Atomic Energy Research Establishment, Harwell, Berks, Eng.). J. Inorg. & Nuclear Chem. 11, 128-43(1959) Sept.

The solubilities of mono- and di-n-butylphosphoric acid (H_2MBP and HDBP, respectively) at 25°C in water, aqueous nitric acid, odorless kerosene and benzene were measured using P^{32}-labeled acids. The net distribution coefficient (D_{HDBP}) for HDBP was measured as a function of the concentration of HDBP for; a number of organic solvents in contact with 1 M HNO_3, chloroform in contact with 0.1 and 1 M perchloric acid, and the kerosene–HNO_3 system from 0 to 15 M HNO_3. In TBP–kerosene–HNO_3 systems, D_{HDBP} and D_{H_2MBP} were measured as functions of the concentrations of HDBP, H_2MBP, TBP, and HNO_3. TBP was shown to associate with both HDBP and H_2MBP. The data can be applied to the calculation of equilibrium distribution coefficients for HDBP and H_2MBP in TBP–HNO_3 systems over a range of [TBP] from 0.1 to 100% v/v in the diluent, of [HNO_3] from 0 to 15 M, and of [HDBP] and [H_2MBP] either separately or together in the range 10^{-6} to 0.05 M. The solubility and distribution data for HDBP are qualitatively interpreted in terms of a series of equations for the dissociation of HDBP, the dimerization of HDBP in both aqueous and organic phases, the association of HDBP with TBP and with HNO_3, the association of TBP with HNO_3; and the distribution of the HDBP monomer and dimer, and the HDBP·HNO_3 species, between the phases. The dimerization constants of HDBP in various organic solvents, the distribution constants of the monomer and the dimer in aqueous–organic solvent systems, and the association constant between TBP and HDBP in kerosene were calculated. (auth)

SOLVENT EXTRACTION 6.16

SOLVENTS 6.16.05

ORGANOPHOSPHOROUS 6.16.05.05

THE EXTRACTION OF NITRATES BY PHOSPHORYLATED REAGENTS. I. THE RELATIVE EXTRACTION RATES AND SOLVATING MECHANISMS FOR URANYL NITRATE. T. V. Healy and J. Kennedy (Atomic Energy Research Establishment, Harwell, Berks, Eng.). J. Inorg. & Nuclear Chem. 10, 128-36(1959) Apr.

The partitioning of uranyl nitrate between a number of non-ionic phosphorylated reagents and dilute nitric acid was measured. Experiments at varying concentrations of the extractants (S) in an inert diluent such as benzene indicate that in most cases the disolvates $UO_2(NO_3)_2$ 2S are formed. Non-ionic reagents containing the diphosphoryl group =P(O)−Y−(O)P=, (where Y is oxygen, methylene, or ethylene) also give disolvates when the latter are in large excess of the uranium, but when saturated with uranyl nitrate the solvates $[UO_2(NO_3)_2S]_n$, which are probably polymeric, predominate. The implications of these results are discussed. Electronegative or base weakening substituents on the phosphoryl group decrease the partition coefficient, and that obtained with triphenyl phosphate is almost six orders of magnitude lower than the partition obtained with tributyl phosphine oxide. Preliminary experiments with di-n-butyl phosphoric acid, indicate that this reagent extracts uranium by a chelating mechanism accompanied by hydrogen ion liberation from solutions low in nitric acid (< 3.0 M), and by a solvating mechanism similar to that with non-ionic reagents at higher aqueous acid concentrations. The structure of the uranyl dibutyl phosphate complex is discussed. (auth)

15106 ✓

THE EXTRACTION OF NITRATES BY PHOSPHORYLATED REAGENTS. II. THE RELATIVE EXTRACTION RATES OF URANYL, COBALTOUS, AND SODIUM NITRATES WITH PHOSPHATE ESTERS. T. V. Healy, J. Kennedy, and G. M. Waind (Atomic Energy Research Establishment, Harwell, Berks, Eng.). J. Inorg. & Nuclear Chem. 10, 137-47(1959) Apr.

The partition of uranyl, cobaltous, and sodium nitrate between non-ionic phosphate esters and water was measured, and the deviations from ideality which occur at high nitrate concentrations in the organic phase were used to obtain solvation numbers for the extracted salts. With tri-n-butyl phosphate (TBP), the extracted species are the di-solvates, $UO_2(NO_3)_2 \cdot 2TBP$, and the tri-solvates $Co(NO_3)_2 \cdot 3TBP$ and $NaNO_3 \cdot 3TBP$. Tri-solvates are also formed between uranyl nitrate and weak phosphate ester extractants such as diphenyl butyl and triphenyl phosphates. (auth)

19999

EXTRACTION OF NITRIC ACID BY DERIVATIVES OF BUTYLPHOSPHINIC ACIDS. A. V. Nikolaev, S. M. Shubina, and N. M. Sinitsyn (Kurnakov Inst. of General and Inorganic Chemistry, Academy of Sciences, U.S.S.R.). Doklady Akad. Nauk S.S.S.R. 127, 578-80 (1959) July 21. (In Russian)

Data are presented on the extraction properties of butylphosphoric compounds of tributylphosphate $(C_4H_9O)_3$ PO, dibutyl ether butylphosphine acid $(C_4H_9O)_2$ C_4H_9 PO, butyl ether dibutylphosphine acid (C_4H_9O) $(C_4H_9)_2$ PO, and tributylphosphinoxide $(C_4H_9)_3$ PO. The nitric acid distribution relation to the concentration of the extractive reagent and the uranyl nitrate in the solution was investigated. (R.V.J.)

14705 ✓ A/CONF.15/P/1550

Oak Ridge National Lab., Tenn.

SOLVENT EXTRACTION OF URANIUM AND OTHER METALS BY ACIDIC AND NEUTRAL ORGANOPHOSPHORUS COMPOUNDS. C. A. Blake, Jr., C. F. Baes, Jr., K. B. Brown, C. F. Coleman, and J. C. White. 22p. $0.50(OTS).

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

The properties of a number of new organophosphorus reagents are summarized, and their positions in a new solvent extraction technology are discussed. In contrast to the familiar tributyl phosphate (TBP), the applications of which have been limited to highly salted nitrate solutions, several other neutral and acidic or organophosphorus compounds have demonstrated the ability to extract uranium from a wide variety of

10484

URANIUM AND PLUTONIUM EXTRACTION BY ORGANOPHOSPHORUS COMPOUNDS. L. L. Burger

SOLVENT EXTRACTION 6.16

SOLVENTS 6.16.05

ORGANOPHOSPHOROUS 6.16.05.05

(General Electric Co., Richland, Wash.). J. Phys. Chem. 62, 590-3(1958) May.

Organic phosphates, phosphonates, phosphinates, and phosphine oxides are compared as solvents for uranium and plutonium nitrates. Distribution coefficients for U(VI) and Pu(IV) and (VI) indicate a large difference in solvent strength among these groups of compounds. The introduction of an electronegative group greatly reduces the solvent strength. The P—O stretching frequency can be correlated with solvent strength for these compounds. Some solid complexes of the organophosphorus compounds with uranyl nitrate have been isolated. (auth)

5762 HW-44888
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
THE ESTERS OF THE ACIDS OF PHOSPHORUS AS SOLVENTS. L. L. Burger. Jan. 3, 1957. 25p. Contract W-31-109-Eng-52. $4.80(ph OTS); $2.70(mf OTS).

A series of alkyl and aryl esters of phosphoric and substituted phosphonic and phosphinic acids as well as some substituted phosphine oxides have been prepared. Their physical properties and extraction behavior for U and Pu have been determined. These organo-phosphorus compounds offer an extremely wide range of physical properties, including solvent strength. An increase in solubility and viscosity, a decrease in density, and a decrease in the stretching frequency of the phosphoryl bond are noted on passing from phosphate to phosphonate to phosphinate to phosphine oxide. The extraction ability of these compounds for U increases in the order phosphate < phosphonate < phosphinate < phosphine oxide. A similar trend was noted for extraction of $Pu(NO_3)_4$ and $Pu(NO_3)_6$ by phosphates, phosphonates, and phosphinates. Electronegative substituents such as chlorine strongly depress the solvent strength of these compounds. Solid complexes formed between several of the compounds and $UO_2(NO_3)_2$ were isolated and identified. (auth)

11655 ✓ CF-54-12-6
Oak Ridge National Lab., Tenn.
STATUS OF THE RESEARCH PROGRAM FOR URANIUM CHEMISTRY OF RAW MATERIALS. K. B. Brown. Dec. 2, 1954. Decl. Mar. 14, 1957. 21p. Contract [W-7405-eng-26]. $4.80(ph OTS); $2.70(mf OTS).

Results are reported from an evaluation of organophosphorus reagents and some other types of organic compounds as extractants for U from acid liquors. (C.H.)

ORGANO-PHOSPHORUS COMPOUNDS FOR SOLVENT EXTRACTION. C. E. Higgins, W. H. Baldwin, and J. M. Ruth. Aug. 14, 1952. Decl. Mar. 11, 1957. 17p. (ORNL-1338)

The influence of the structure of phosphate esters on the distribution coefficient of U between solutions of these esters in CCl_4 and aqueous nitrate solutions is studied. The distribution coefficients of esters of secondary alcohols are compared with those of primary alcohols. The extraction efficiencies of alkyl and phenyl esters are compared, and the effect of negative groups on extraction is studied. The relationship of the number of C-P bonds on extraction is studied, and the distribution coefficients of tributyl phosphine oxide with $UO_2(NO_3)_2$, UO_2SO_4, UO_2Cl_2, $Th(NO_3)_4$, $Pu(NO_3)_4$, and fission products in nitrate solution are investigated.

11072 ✓
POSSIBLE HYDROGEN BONDING IN CERTAIN INTERACTIONS OF ORGANIC PHOSPHORUS COMPOUNDS. D. F. Peppard, J. R. Ferraro, and G. W. Mason (Argonne National Lab., Lemont, Ill.). J. Inorg. and Nuclear Chem. 4, 371-2 (1957).

OTHERS 6.16.05.06

SOLVENT EXTRACTION OF HEAVY METAL CHELATES WITH FURFURAL. F. K. Cole and L. H. Brown (Quaker Oats Co., Barrington, Ill.). Ind. Eng. Chem. 51, 58-9(1959) Jan.

Furfural is an effective selective solvent for the separation of uranium–thorium, zirconium–hafnium, and niobium–tantalum chelate mixtures. A possible thorium–rare earth separation has been pointed out. Furfural is relatively stable to aqueous nitric acid. All this points the way toward future work on the development of commercial methods for the separation of metals using inexpensive and readily available furfural as an extraction solvent. (auth)

SOLVENT EXTRACTION 6916

SOLVENTS 6.16.05

OTHERS 6.16.05.06

U.S. Patent 2,874,176

Separating Metallic Cations with Perfluorocarboxylic Acids.
G.F. Mills and H.B. Whetsel
Describes a versatile solvent extraction process as well as some unusual precipitations. For example, strontium is extractable, and cesium precipitatable.

17041
THE EXTRACTION OF URANIUM AS ANILIN-URANYL-TRIACETATE. V. M. Vdovenko and L. N. Lazarev. Zhur. Neorg. Khim. 3, 155-9(1958). (In Russian)

The possibility of extracting uranyl acetate with aniline was demonstrated. It was shown that aniline uranyl acetate was formed in the organic phase. This compound was separated and its chemical properties were determined. (J.S.R.)

1201 AECU-3879
Michigan. Univ., Ann Arbor.
TTA EXTRACTION CURVES. E. Sheperd and W. W. Meinke. Oct. 24, 1958. 5p. Project No. 7. Contract AT(11-1)-70. $1.80(ph OTS); $1.80(mf OTS).

Curves are given of pH versus percent extraction for Ac, Be, Bi, Ce, Dy, Eu, Gd, Hf, Ho, In, Fe, La, Pb, Nd, Np, Pu, Pr, Pm, Ra, Sm, Sc, Tl, Tm, Th, U, V, and Y by thenoyl trifluoroacetone in benzene. (T.R.H.)

2833
THE SOLVENT EXTRACTION BEHAVIOR OF INORGANIC COMPOUNDS. IV. VARIATION OF THE DISTRIBUTION QUOTIENT WITH CHLORIDE, HYDROGEN ION HELD CONSTANT. R. M. Diamond (Cornell Univ., Ithaca, N. Y.). J. Phys. Chem. 61, 1522-31(1957) Nov.

The variation in the distribution quotient for tracer indium(III) distributing between the oxygenated solvents, diethyl, butyl ethyl, dibutyl and β,β'-dichlorodiethyl ether, methyl isobutyl and diisobutyl ketone, dibutyl phthalate, methyl benzoate, methyl salicylate, 2-ethylhexanol and decanol, and aqueous solutions of hydrochloric acid, in which the concentration of HCl was varied while keeping the total ionic strength constant with $HClO_4$ or HNO_3, was studied. The results are compared with the behavior expected from expressions derived for D and d log D/d log [HCl]. It is shown that certain aspects of the variation in D are indeed indicated by the derived expressions, particularly a "common ion" effect when the ion $InCl_4^-$ is the principal organic phase indium-containing species and an increase in D when ion associations containing $InCl_4^-$ are possible in the organic phase. The remaining major effect is interpreted on the basis of a previous suggestion that the availability, or non-availability, of water in the aqueous phase for solvating ionic species is an important factor in the extraction process. These considerations are not restricted to the indium(III) chloride system, but appear to hold generally for those metal halides which extract into oxygenated organic solvents as the anions of strong acids. (auth)

7503
THE EFFECT OF THE SOLVENT ON THE POLAROGRAPHIC REDUCTION OF CATIONS. Douglas B. Bruss and Thomas De Vries (Purdue Univ., Lafayette, Ind.). J. Am. Chem. Soc. 78, 733-6(1956) Feb. 20.

The effect of the solvent on the reduction of metal ions was investigated at a dropping mercury electrode with a silver-silver chloride electrode as reference. Nine cations were studied in water, ethylene glycol, formamide, dimethylformamide, pyridine, and the normal alcohols through pentanol and isopropyl alcohol. The half-wave potentials were found to vary in a regular manner with the dielectric constant of the solvent, usually becoming more positive as the dielectric constant decreased. A correlation was found between the shift in half-wave potential per dielectric constant unit and the quotient of the magnetic susceptibility of the ion divided by the ionic radius. The variation of the

SOLVENT EXTRACTION 6.16

SOLVENTS 6.16.05

Others 6.16.05.06

diffusion current with the square root of the fluidity of the solvent demonstrated that, for the majority of the ions, viscosity was not the only factor affecting the diffusion coefficients. (auth)

10036 ✓ ORNL-2346
Oak Ridge National Lab., Tenn.
PROGRESS REPORT ON RAW MATERIALS FOR APRIL 1957. K. B. Brown, C. F. Coleman, D. J. Crouse, and A. D. Ryon. July 22, 1957. 27p. Contract W-7405-eng-26. $0.25(OTS).

Systematic Studies. The enhanced U extraction coefficients obtained with synergistic combinations of di(2-ethylhexyl)phosphoric acid (D2EHPA) and trialkylphosphate, dialkyl alkylphosphonate, or trialkylphosphine oxide reach maxima at characteristic concentrations of the neutral components which appear to be essentially independent of the D2EHPA concentration. The coefficients vary with D2EHPA concentration and with pH and sulfate concentration of the aqueous solution in the same way as in extractions with D2EHPA alone. In brief examination of the extractions of some metals other than U by amines, alkylphosphoric acids, and phosphine oxides, strong extraction of Th was found with D2EHPA, and potential separations were indicated between rare earths, Hf–Zr and Co–Ni. Process Development Studies. Aqueous slurries of several different commercial magnesias stripped U effectively from tri-n-octylamine in kerosene plus 3% alcohol. Stripping of Amine 9D-178 in kerosene was more rapid at 38°C than 28°C although stripping results were favorable at both temperatures. The presence of Mo in the amine extracts decreased the rate of U stripping. Molybdenum was stripped more slowly than U. Engineering Studies. Studies were made of the mixing requirements for stripping U from the Dapex type solvent with Na_2CO_3. Stage efficiency was greater than 90% with a residence time of 0.38 min and a power input of 0.01 hp/gal. (For preceding period see ORNL-2306.) (auth)

TBP – DILUENTS 6.16.05.07

PHYSICAL PROPERTIES OF TRIBUTYLPHOSPHATE-DILUENT SOLUTIONS. W. F. Johnson and R. L. Dillon. Sept. 1, 1953. Decl. Feb. 7, 1957. 25p. (HW-29086)

In connection with the study of aqueous-tributylphosphate (TBP) systems, the study of the interactions of various diluents with the TBP has been extended. The diluents used were mineral oil, Amsco 125-90W, dodecane, n-hexane, cyclohexane, benzene, n-octyl bromide, carbon tetrachloride, chloroform, and pentachloroethane. The solubility of TBP in water from solutions of these diluents and the solubility of water in the organic phase was determined. For the paraffin hydrocarbons as diluents, the solubility of TBP in water was found to increase with increasing molal volume of the diluent. No evidence for the formation of a TBP water complex of simple mole ratio was found. The partial molal volume of TBP in all the diluents was constant up to a volume fraction of 0.75 and equal to the molal volume of TBP within ±0.5 per cent. No simple relationship could be found which related the viscosity to the concentration for TBP in the diluents dodecane, n-hexane, cyclohexane, and pentachloroethane whether the solutions were dry or water-saturated. The self-diffusion coefficient of TBP was found to be 2.29×10^{-6} cm^2/sec. For dodecane and pentachloroethane solutions the product $D\eta$ was found to be essentially constant.

14123 ✓ NLCO-769
National Lead Co. of Ohio, Cincinnati.
STUDIES OF THE BEHAVIOR OF THE TBP-KEROSENE SOLVENT IN URANIUM REFINING: DEGRADATION OF THE KEROSENE DILUENT. Robert K. Klopfenstein. Aug. 12, 1958. 49p. Contract AT(30-1)-1156. $1.50(OTS).

Extensive studies have been conducted of kerosene-type diluents for the tri-n-butyl phosphate (TBP)-kerosene solvent used in uranium extraction. Wide variations have been shown in the paraffinic, isoparaffinic, aromatic, and olefinic hydrocarbon contents of these diluents. Such diluents have been shown to be unstable in the presence of high nitric acid concentrations, and in the presence of small amounts of nitrous acid. This instability is primarily a function of the non-paraffinic hydrocarbon content of the diluent and of the nitrous acid concentration of the contacting medium. Degradation of a diluent has been shown to discolor a diluent, increase its density and viscosity, and influence uranium distribution during re-extraction operations. Solvent treatment with Na_2CO_3, followed by H_2O and nitric acid washes, has been shown to be only partially effective in removing diluent degradation products. The diluent degradation products have been characterized.

SOLVENTS 6.16.05

TBP - Diluents 6.16.05.07

chemically and spectrally, as carboxylic acids, organic nitrates, organic nitro compounds, and nitroso compounds. (auth)

EFFECT OF DILUENTS ON THE EXTRACTION OF URANYL NITRATE WITH TRIBUTYL PHOSPHATE. Zdenko I. Dizdar, Jelena K. Rajnvajn, and Olga S. Gal. Bull. Inst. Nuclear Sci. "Boris Kidrich" (Belgrade) 8, 59-66(1958) Mar.

At lower concentrations of uranium (≤ 150 g/l) the extraction of uranyl nitrate with tributyl phosphate (TBP) depends very much on the nature of diluents used. The greatest extraction ability is shown by TBP diluted with carbon tetrachloride, than with xylene, kerosene, hexane, dibutyl ether, diethyl ether, and isopropyl ether. At greater uranium concentrations these differences disappear. (auth)

COMPARISON OF DILUENTS FOR TRIBUTYL PHOSPHATE. L. L. Burger. Oct. 6, 1950. 22p. Decl. Feb. 20, 1957. (HW-19065)

A study of the composition and physical properties of a number of hydrocarbon diluents with some generalizations regarding relations between these properties is summarized. Tabular data on the following are included: physical properties of pure diluents; physical properties of typical pure hydrocarbons; flash points of TBP-diluent mixtures; properties of diluent-TBP mixtures saturated with H_2O at 25°C; and comparison of Stoke's factors for metal recovery systems and for purex systems. Curves for ASTM distillation of hydrocarbon diluents of various kinds, viscosity-flash point relation for hydrocarbon mixtures, and viscosity of organic phase containing $UO_2(NO_3)_2$ at 25°C are included. 18 references.

TBP - GENERAL 6.16.05.08

See also, under FISSIONABLE MATERIAL--RECOVERY,

RECOVERY OF IRRADIATED TH AND U --Solvent Extraction--Sulfate Systems (3.02.17.06),

RECOVERY OF PLUTONIUM--Solvent Extraction--TBP (3.03.14.01).

See also, for possible explosive reaction of TBP and HNO_3, or TBP and UNH, under, Hazards and Protection, History (Incidents) — Explosions, 4.02.04.02,

THE CHEMISTRY OF TRIBUTYL PHOSPHATE: A REVIEW. L. L. Burger. Oct. 27, 1955. Decl. Feb. 24, 1958. 75p. (HW-40910)

The preparation, purification, and chemical properties of TBP have been reviewed with emphasis on the hydrolytic reactions. TBP is chemically a very stable compound as evidenced by its thermal stability and resistance to oxidation. The most important reactions are hydrolytic which cleave the butyl or butoxy group and normally produce butyl alcohol together with dibutyl and monobutyl phosphate (DBP and MBP, respectively), and eventually H_3PO_4. Hydrolysis occurs in either the organic phase or the aqueous phase and is first order with respect to the ester. Although the rate in the aqueous phase is much faster than in the organic phase, the solubility is so low in aqueous solutions that the organic phase reactions become more important. Acid hydrolysis depends on both the nature of the acid and the concentration. The order with respect to acid concentration is close to one but often less than one. Hydrolysis is catalyzed by both acids and bases. In the latter case, the reaction occurs only in the aqueous phase and normally stops with the formation of DBP. The hydrolysis rate increases greatly as the temperature is raised and an activation energy of the order of 20 kcal is often found. The rates observed in the presence of 5 M acid at 60 and 70°C may be high enough to cause some concern in solvent extraction technology, since the product, DBP, has undesirable properties. Impurities produced during manufacture or by thermal degradation during purification such as the pyrophosphates; if present, would yield the same objectionable products as TBP hydrolysis, but at a faster rate. Included in the survey is a selected tabulation of physical properties of TBP.

SOLVENT EXTRACTION 6.16

SOLVENTS 6.16.05

TBP - General 6.16.05.08

A RATIONALE FOR THE RECOVERY OF IRRADIATED URANIUM AND THORIUM BY SOLVENT EXTRACTION. T. H. Siddall, III. 24p. (A/CONF.15/P/521)

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

Some of the calculations which can be made to predict the proper operating conditions for multistage contractors in which tri-n-butyl phosphate is the extractant are given. The calculations are made from the results of equilibrations in single stages in the laboratory. Tri-n-butyl phosphate is compared with other trialkyl phosphates and with certain dialkyl alkylphosphonates. The basis of the comparison is the method of calculation that was developed.

587 AERE-Lib/Trans-792
ON THE THERMODYNAMICS OF EXTRACTION WITH TRIBUTYLPHOSPHATE. A. M. Rozen and L. P. Khorkhorina. Translated by V. Beak (U.K.A.E.A. Atomic Energy Research Establishment) from Zhur. Neorg. Khim. 2, 1956-69(1957). 22p.

A thermodynamic interpretation of the data on the separate and combined distribution of nitric acid and uranyl nitrate between an aqueous solution and TBP with a diluent is presented. The data from literature concerning the distribution of a number of actinide elements in tracer concentrations are also examined, and the main laws of the extraction equilibria in which TBP takes part are discussed. (J.R.D.)

6368
RECOVERY OF METAL VALUES FROM AQUEOUS SOLUTIONS BY SOLVENT EXTRACTION. R. L. Moore (to U. S. Atomic Energy Commission). U. S. Patent 2,902,454. Sept. 1, 1959.

An organic solvent mixture is described for extracting actinides from aqueous solutions; the solvent mixture consists of from 10 to 25% by volume of tributyl phosphate and the remainder a chlorine-fluorine-substituted saturated hydrocarbon having two carbon atoms in the molecule.

7560 Y-838
Carbide and Carbon Chemicals Co. Y-12 Plant, Oak Ridge, Tenn.
CRITICAL LITERATURE SURVEY OF TRIBUTYL PHOSPHATE AS A URANIUM EXTRACTANT. W. B. Wright, Jr. Jan. 14, 1952. Decl. Feb. 11, 1957. 27p. Contract W-7405-eng-26. $4.80(ph OTS); $2.70(mf OTS).

Important data and discussions obtained from ten primary references pertaining to the solvent extraction of U with TBP are collected in this survey. Topics covered are: physical and chemical properties of pure and diluted TBP, organic diluent for TBP, the mechanism of U extraction by TBP, factors influencing the extraction, interferences and other ions extracted, recovery of U from the organic phase, and purification of TBP for re-use. (auth)

8750
TRI-n-BUTYL ORTHOPHOSPHATE (TBP) AS AN EXTRACTING SOLVENT. H. A. C. McKay and T. V. Healy (Atomic Energy Research Establishment, Harwell, Berks, Eng.). pp. 546-56 in "Progress in Nuclear Energy. Series III. Process Chemistry. Volume 2."

The physical properties and solubilities of various inorganic compounds in tri-n-butyl orthophosphate are presented. (W.L.H.)

11570 KLX-1211
Kellex Corp., New York.
SOLVENT EVALUATION FOR LIQUID-LIQUID EXTRACTION (23-F). Closing Report. Jan. 15, 1951. Decl. Mar. 28, 1957. 19p. Contract AT(30-1)-848. $0.30(OTS).

Comparison of diethyl ether, TBP-kerosene, hexone, dibutyl carbitol, and pentaether as alternative solvents for U extraction in the MCW refining process was made using a Scheibel column for extraction and a packed column for scrubbing and stripping. TBP-kerosene, 15 and 30%, gives high U extraction efficiencies, produces a product of the highest purity among the solvents tested, and has good operability from a physical and mechanical view point. (auth)

579 ORNL-887
Oak Ridge National Lab., Tenn.
THE PREPARATION OF TRI-n-BUTYL PHOSPHATE P^{32}.

SOLVENT EXTRACTION 6.16

SOLVENTS 6.16.05

TBP - General 6.16.05.08

W. H. Baldwin and C. E. Higgins. Feb. 5, 1951. Decl. Oct. 4, 1955. 5p. Contract W-7405-eng-26.

The preparation of esters by the metathetical reaction between the silver salt of an acid and an alkyl halide, though usually considered a time-consuming and expensive method, is useful in specific applications. Since radioactive phosphorus, P^{32}, is readily available in high specific activity as orthophosphoric acid, the most direct method of synthesis of tri-n-butyl phosphate-P^{32} seemed to be through the reaction of silver phosphate with excess n-butyl bromide. For synthetic purposes it appears satisfactory to precipitate the phosphate quantitatively with a slight excess of silver without sacrificing the yield of ester. (auth)

7426 ✓ HW-20936
Hanford Works, Richland, Wash.
THE SOLUBILITY OF TRIBUTYL PHOSPHATE IN AQUEOUS SOLUTIONS. Leland L. Burger and R. C. Forsman. Apr. 2, 1951. Decl. Mar. 2, 1957. 17p. Contract W-31-109-Eng-52. $3.30(ph OTS); $2.40(mf OTS).

Solubilities of pure tributyl phosphate and of TBP in an inert diluent were determined in water and in solutions of nitric acid of various concentrations. Measurements were also made in solutions typical of various process streams and in more concentrated uranyl nitrate solutions. In the case of the TBP-diluent-water system the equilibrium water content in the organic phase was also determined. Data are tabulated. (C.H.)

EXTRACTION OF CHLORIDES FROM HYDROCHLORIC ACID SOLUTIONS WITH TRIBUTYL PHOSPHATE. Ivan J. Gal and Aleksandar Ruvarac. Bull. Inst. Nuclear Sci. "Boris Kidrich" (Belgrade) 8, 67-74(1958) Mar.

The partition of the chlorides of H^+, Fe^{3+}, Fe^{2+}, UO_2^{2+}, Cd^{2+}, Ni^{2+}, Co^{2+}, Sr^{2+}, Zr^{4+}, Ce^{3+}, RuO^{2+} (possibly a mixture of tri- and tetravalent ruthenium) as well as of VO_3^- between 30% (v/v) tributyl phosphate in dibutyl ether and aqueous solutions of different HCl concentration was determined. The dependence of the extraction on the TBP concentration in the organic phase for some of these elements was also investigated. Possibilities of mutual separations are discussed and formulas for some of the complexes extracted are proposed. (auth)

TBP - INTERFERENCES 6.16.05.09

8732 ✓
THE DECOMPOSITION REACTIONS OF TRIBUTYL PHOSPHATE AND ITS DILUENTS AND THEIR EFFECT ON URANIUM RECOVERY PROCESSES. L. L. Burger (General Electric Co., Richland, Wash.). pp. 307-19 in "Progress in Nuclear Energy. Series III. Process Chemistry. Volume 2."

The properties of tributyl phosphate and its common contaminants are discussed. Tributyl phosphate decomposition occurs mainly by hydrolysis. Rate constants for the reaction are given as a function of temperature and phase composition. Impurities which are important from a process standpoint may also be introduced by radiation-induced decomposition of tributyl phosphate and by chemical and radiation decomposition of the diluents that are usually employed with it. (auth)

5293 ✓ NP-7238
Polish Academy of Sciences. Inst. of Nuclear Research, Warsaw.
EFFECTS OF CHLORIDES AND SULPHATES ON EXTRACTION OF URANIUM AND IRON BY TRI-N-BUTYL PHOSPHATE. Report No. 53/IV. W. Korpak and C. Deptuka. Dec. 1958. 17p.

Experiments were performed to determine the effects of chlorides and sulfates on the extraction of $UO_2(NO_3)_2$. The effects of chlorides on the extraction of Fe^{3+} in the presence of $UO_2(NO_3)_2$ were investigated. Sulfates were found to reduce the yield of U from nitrate leaching solutions. No perceptible effects of chlorides on U extraction were revealed, but they increased the content of Fe in the organic phase. (W.L.H.)

4386 IDO-14486
Phillips Petroleum Co. Atomic Energy Div., Idaho Falls, Idaho.
THE DETERMINATION OF EXCESSIVE EMULSIFICATION BY COALESCENCE BEHAVIOR MEASUREMENTS. O. W. Parrett. Nov. 25, 1959. 24p. Contract AT(10-1)-205. OTS.

The development of a remotely operated device for determining the coalescence times of plant process streams suspected of containing surfactants such as silicic com-

SOLVENT EXTRACTION 6.16

SOLVENTS 6.16.05

TBP - Temperature Effects 6.16.05.11

pounds and fission product zirconium compounds is described. A general correlation between the coalescence times of pilot plant extraction column aluminum nitrate feeds and 3.25 percent tributyl phosphate extractant streams and the observations of column behavior of these streams is demonstrated. The application of the coalescence test to plant streams is given. (auth)

6291 AERE-CE/R-968
Gt. Brit. Atomic Energy Research Establishment, Harwell, Berks, England.
TRI-n-BUTYL PHOSPHATE STUDIES. PARTS I, II, III. J. Kennedy and S. S. Grimley. Oct. 6, 1952. Decl. Nov. 30, 1956. 30p.
The chief impurities in commercial TBP have been identified and estimated. The isolation of mono- and di-n-butyl phosphates for characterization and estimation purposes is described. A method is given for their identification based on the melting points of their s-benzyl thiouronium salts and is applied to the commercial TBP medium. Other impurities are dealt with quantitatively and a rapid method for the prufication of the commercial solvent for extraction purposes is presented. (auth)

THE FORMATION AND EFFECTS OF DIBUTYL PHOSPHATE IN SOLVENT EXTRACTION. E. Kenneth Dukes. Nov. 1957. 22p. (DP-250)
Data are presented for the rates of formation of di-n-butyl phosphate (DBP) from tri-n-butyl phosphate. The effects of DBP on the extraction of Zr from HNO_3 and $UO_2(NO_3)_2$ are reported.

TBP - NITRATE SYSTEMS 6.16.05.10

See also, under FISSIONABLE MATERIAL--RECOVERY,

SOLVENT EXTRACTION--Nitrate Systems--TBP (3.02.17.04.06).

1588
THE INTERNAL CIRCULATION OF EXTRACTABLE MATERIALS IN TRIBUTYL. A. M. Rozen. Atomnaya Energ. 7, 277-81(1959) Sept. (In Russian)
The internal circulation of extractable materials was studied by plotting HNO_3 γ-x diagrams with individual curves for uranyl nitrate acid. Distributions of the concentrate along the column and the extraction with flushing and reduction partition are determined. An additional circulation by flushing places extraction at its maximum; the flux phase ratio $n_o = V/L_o$ is increased in comparison to extraction without flushing $n_o = f(n : n)$. The U and Pu reduction partition column was studied at large flux ratios (n>10). A sharp use in acid concentration purges uranium, inducing internal circulation of uranyl nitrate. (R.V.J.)

5268
NITRIC ACID, WATER AND TRIBUTYLPHOSPHATE EQUILIBRIUM SYSTEM. L. Damiani and V. Fattore (SICEDISON, Porto Marghera, Italy). Energia nucleare (Milan) 6, 793-8(1959) Dec. (In English)
An investigation was made of the HNO_3 and H_2O extraction mechanism using TBP. From stoichiometric data and a consideration of the polar bonds, the following extraction formulae are postulated in organic phase: $TBP \cdot H_2O \cdot HNO_3$, $TBP \cdot HNO_3$, $TBP \cdot H_2O \cdot 2HNO_3$. The fields of existence, curves, and equilibrium constants for these are also given. The experimental results obtained are discussed. (auth)

17005
SOLVENT EXTRACTION STUDIES. PART II. THE SYSTEM NITRIC ACID—WATER—Tri-n-BUTYL PHOSPHATE. D. G. Tuck (Univ. of Manchester, Eng.). J. Chem. Soc., 2783-9(1958) Aug.
The extraction of nitric acid from aqueous solution by tri-n-butyl phosphate has been investigated. From the volume changes it has been shown that up to ~3.6 M-acid in the organic phase the extraction is due to the formation of Bu_3PO_4,HNO_3 by addition at the strongly polar P → O group. When this donor oxygen is saturated, further addition takes place at the less basic oxygen atom of a P—O—C bond; in this case, the acid is shown to be extracted as H_2O,HNO_3. The experimental results are discussed in relation to mass-action equations derived for these two types of equilibrium. (auth)

SOLVENT EXTRACTION 6.16

SOLVENTS 6.16.05

TBP – NITRATE SYSTEMS 6.16.05.10

9227

THE EXTRACTION OF NITRATES BY TRI-n-BUTYL PHOSPHATE (TBP). PART 2. THE NATURE OF THE TBP PHASE. T. V. Healy and H. A. C. McKay (Atomic Energy Research Establishment, Harwell, Berks, England). Trans. Faraday Soc. 52, 633-42(1956) May.

Evidence is presented pointing to the conclusion that nitrates are extracted by TBP in the neutral, unionized form, always solvated by a definite number of TBP molecules, e.g. $Ce(NO_3)_3 . 3TBP$, $UO_2(NO_3)_2 . 2TBP$; the compounds formed are sometimes hydrated, sometimes almost unhydrated. The evidence is much fuller for uranyl than for any other nitrate, but all the data support this general conclusion. (auth)

THE EXTRACTION OF NITRATES BY TRI-n-BUTYL PHOSPHATE (TBP). PART 3. EXTRACTION AT TRACE CONCENTRATIONS. E. Hesford and H. A. C. McKay (Atomic Energy Research Establishment, Harwell, Berks, Eng.). Trans. Faraday Soc. 54, 573-86(1958) Apr.

Metal nitrates apparently exist in TBP predominantly in a single molecular form $M(NO_3)_p . qTBP$. A theory of TBP extractions has been developed, based on this observation, and has been applied to experiments of three main types: (1) The TBP concentration is varied by adding an inert diluent. The results enable us to determine the solvation number q. (2) The nitric acid concentration is varied. In certain cases the measured partition coefficients then enable us to calculate the thermodynamic equilibrium constant and the activity coefficient of a trace of the extractable nitrate in a nitric acid medium. Reasonable functions are obtained for the latter quantity. (3) An inextractable nitrate, e.g. sodium nitrate, is added as a salting-out agent. Our theory enables us to estimate the resulting rise in partition coefficient. Separation factors (partition coefficient ratios) for pairs of similar elements have also been considered. (auth)

COMPLEXES BETWEEN TRIBUTYL PHOSPHATE AND INORGANIC NITRATES. T. V. Healy and H. A. C. McKay (Atomic Energy Research Establishment, Harwell, Berks, England). Rec. trav. chim. 75, 730-6(1956) June.

Many inorganic nitrates dissolve freely in tributyl phosphate (TBP), forming complexes with the solvent. TBP saturated with uranyl nitrate, for example, corresponds exactly to the composition $UO_2(NO_3)_2 . 2TBP$ and has a sharp melting-point of -6°C; no other complex involving uranyl nitrate and TBP appears to be of any significance. There is only slight ionization in the TBP phase. A useful general method of determining the number of TBP molecules attached to the inorganic nitrate is by partition experiments between TBP and water. The aqueous phase is kept constant in composition while an inert diluent, e.g. benzene or kerosene, is added to the TBP; the partition of a trace of the inorganic nitrate in question is then measured at a series of dilutions. At high dilutions a limiting law of the form: Partition coefficient ~ (TBP Concentration)^n is obtained in which the exponent n gives the number of TBP molecules in the complex. Formulae have been established for different complexes. (auth)

17001 NLCO-749

National Lead Co. of Ohio, Cincinnati.

THE TRIBUTYL PHOSPHATE–NITRIC ACID COMPLEX AND ITS ROLE IN URANIUM EXTRACTION. Thomas J. Collopy. Apr. 23, 1958. 15p. Contract AT(30-1)-1156. $0.75(OTS).

The distribution of nitric acid between tributyl phosphate and water at 25°C is shown for equilibrium aqueous nitric acid concentrations of 0 to 12 molar. Results are presented for uranium extraction from sulfate, chloride, fluoride, and phosphate systems with TBP and with TBP that contained an equimolar complex with nitric acid. (auth)

6303

TRI-n-BUTYL PHOSPHATE AS AN EXTRACTING SOLVENT FOR INORGANIC NITRATES—I. K. Alcock, F. C. Bedford, W. H. Hardwick, and H. A. C. McKay (Atomic Energy Research Establishment, Harwell, Didcot, Berks). J. Inorg. and Nuclear Chem. 4, 100-5(1957) Mar.

The extraction of tracer Zr by 19% v/v (0.69 M) tri-n-butyl phosphate (TBP) in kerosene has been studied from aqueous solutions consisting of (i) nitric acid, (ii) nitric acid plus an inextractable nitrate (sodium nitrate), and (iii) nitric acid plus an extractable nitrate (uranyl nitrate). The partition coefficients increase steeply with rising acidity, they are little affected by substitution of sodium nitrate for nitric acid, and they fall considerably on addition of uranyl nitrate to the system. (auth)

SOLVENT EXTRACTION 6.16

SOLVENTS 6.16.05

TBP - Nitrate Systems 6.16.05.10

8627 NP-7237
Polish Academy of Sciences, Inst. of Nuclear Research, Warsaw.
THE TRI-N-BUTYL PHOSPHATE (TBP)-DILUENT-HNO_3-H_2O SYSTEM. Report No. 51/IV. W. Korpak and C. Depiuła. Dec. 1958. 13p.

The distribution of nitric acid between water and a 5% solution of TBP was investigated in relation to the concentration of nitric acid in the aqueous phase. The experiments revealed the oxonic character of extraction of nitric acid with TBP, and the existence of four complexes is postulated. TBP dissolves in 95% nitric acid to form one phase. This is the basis for an approximate quantitative determination of TBP in mepasine. (auth)

STUDIES ON THE EXTRACTION OF METAL COMPLEXES. XXVIII. THE DISTRIBUTION OF SOME ACTINIDES AND FISSION PRODUCTS BETWEEN TRIBUTYL PHOSPHATE (TBP) AND AQUEOUS SOLUTIONS OF HNO_3 AND $Ca(NO_3)_2$. Brita Bernström and Jan Rydberg (Research Inst. of National Defence, Sundbyberg, Sweden). Acta Chem. Scand. 11, 1173-82(1957).

The distribution ratios of U^{6+}, Pu^{6+}, Pu^{4+}, Th^{4+}, Ru, Zr^{4+} Nb^{5+}, La^{3+}, Sr^{2+}, and Ca^{2+} between tributyl phosphate (TBP) and aqueous solutions of varying concentrations of HNO_3 have been determined at room temperature for different (constant) concentrations of $Ca(NO_3)_2$ in the aqueous phase. The extraction curves are discussed with respect to the different complexes formed and to the possibility of using the investigated systems for separating Pu and U from the fission products. (auth)

THE SOLUBILITIES OF SOME METAL NITRATE SALTS IN TRI-n-BUTYL PHOSPHATE. Wesley W. Wendlandt and John M. Bryant (Texas Tech., Lubbock). J. Phys. Chem. 60, 1145-6(1956) Aug.

A study of the solubilities of a series of metal nitrate salts was made in pure tri-n-butyl phosphate, to investigate other possible metal ions which may be separated by liquid-liquid extraction. (W. L. H.)

7481 CF-52-2-113
Oak Ridge National Lab., Tenn.
THE PRIMARY EXTRACTION MECHANISMS OF NITRIC ACID AND THORIUM NITRATE EXTRACTION BY TBP. Quarterly Report [for] Period November 10, 1951-February 10, 1952. Chemical Technology Div. J. E. Savolainen. Feb. 13, 1952. Decl. Feb. 16, 1957. 18p. Contract [W-7405-eng-26]. $3.30(ph OTS); $2.40(mf OTS).

Results obtained on the simultaneous extraction of HNO_3 and $Th(NO_3)_4$ from aqueous solutions by TBP in a paraffin hydrocarbon diluent indicate that $Th(NO_3)_4 \cdot 4TBP$ is the primary form in which Th is extracted. The extraction of HNO_3 by 10 and 12.5% TBP in hexane has been found to show inflections of the distribution coefficient curve in those regions where the HNO_3/TBP ratio approaches 0.25, 0.5, and 0.75 and probable inflections at 0.125 and 0.875. This behavior is not noticed on extraction with 7.5% TBP. It is postulated that with high $Th(NO_3)_4$ concentration in TBP a polymerization occurs, resulting in the formation of a compound having the limiting composition of $Th(NO_3)_4 \cdot 2TBP$. This postulate may account for TBP/Th ratios of less than 4 in the organic phase and also for the formation of two organic phases at high Th concentrations and the dispersing effect of an aromatic diluent on these two organic phase systems. (auth)

TBP - TEMPERATURE EFFECTS 6.16.05.11

6191 CF-60-1-99
Oak Ridge National Lab., Tenn.
DIFFERENTIAL THERMAL ANALYSIS. QUALITATIVE STUDY OF NITRATE-BUTYL PHOSPHATE SYSTEMS. W. H. Baldwin. Jan. 25, 1960. 12p. Contract [W-7405-eng-26]. OTS.

An evaluation of differential thermal analysis for the study of nitrate-organic reactions was given. In tributyl phosphate saturated with nitric acid an exothermic reaction was initiated near 150°C. Nitrate salts were found to be involved in exothermic reactions at higher temperatures usually after an endothermic reaction at lower temperatures. (auth)

14686 A/CONF.15/P/519
Du Pont de Nemours (E. I.) & Co. Savannah River Lab., Augusta, Ga.
TEMPERATURE EFFECTS ON TBP SOLVENT EXTRACTION PROCESSES. D. G. Karraker. 11p. $0.50(OTS).

SOLVENT EXTRACTION 6.16

SOLVENTS 6.16.05

TBP - Interferences 6.16.05.09

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

TBP extracts uranium (VI) and plutonium (IV) well, and most of the fission products poorly. Ruthenium, zirconium, and niobium are the most difficult fission products to separate, and the radioactivity of the end products is determined by the extent to which these fission products are eliminated. The general behavior of these fission products indicates that slow changes among their species restrict their separation. An increase in temperature accelerates changes among these species, and results in an increased fission product separation for TBP processes. A combination of adsorption processes for removal of residual fission products with the improved, high temperature, Purex process achieves decontamination factors greater than 5×10^6 for the uranium product and greater than 5×10^7 for the plutonium product. This performance equals the decontamination achieved in the normal Purex process (two or three solvent extraction cycles), with a considerable reduction in the required equipment. The decontamination of both cycles of the 25 process was improved by operation at 70°C. The ruthenium decontamination of the first cycle was a factor of 100 greater at 70°C than at 30°C, and the decontamination for zirconium-niobium (combined) was improved by a factor of ten. The improvement in the second cycle decontamination varied with first cycle performance, and apparently was mainly limited by the activity present in the feed. Samples of the organic phase from the extraction-scrub contactor of the first cycle showed that the same amount of activity was extracted at both temperatures; but the extracted activity was removed much faster at higher temperatures. The radiolysis of TBP by the radioactivity of the feed solution was observed in experimental work on the 25 process. The results indicated that the radiolytic product, dibutylphosphate, impairs decontamination. These results were obtained with miniature solvent extraction equipment, operated continuously for extended periods, and processing feed solutions prepared from irradiated material. (auth)

6545 A/CONF.15/P/2466
SOME EXPERIMENTS ON THE SOLVENT EXTRACTION OF SLURRIES. Rasit Tolum (Turkey). 6p.

The breakdown rate of emulsions as a function of temperature and pH of the system was investigated. Synthetic slurries consisting of finely-ground quartz and an aqueous solution either acidified with HCl or alkalinized with NaOH were used. The organic phase consisted of TBP and di-ethylhexyl phosphoric acid in kerosene. It was found that temperature increase improves phase disengagement and aids in the destruction of the unstable emulsion. Also, an increase of hydrogen ion concentration improves the action of the solvent-slurry extraction. The principles of flotation and of emulsion formation in slurries are discussed. (J.R.D.)

SURVEYS AND THEORY 6.16.05

FLOODING AND FLOWRATE 6.16.06.01

22062
FLOODING CHARACTERISTICS AND SEPARATION EFFICIENCIES OF PULSED SIEVE-PLATE EXTRACTION COLUMNS. Experimental Data Applied to Design of Extraction Columns. L. D. Smoot, B. W. Mar, and A. L. Babb (Univ. of Washington, Seattle). Ind. Eng. Chem. 51, 1005-10(1959) Sept.

Generalized flooding and mass transfer correlations were developed from experimental data for pulsed sieve-plate extraction columns. The flooding correlation is based on 665 data points and permits the prediction of total flooding velocities with an average deviation of 20% from the experimental values for systems in which water is the continuous phase. The mass transfer correlation is based on 285 data points and permits the prediction of over-all transfer unit heights with an average deviation of 16.4% from the experimental values. This correlation applies to systems in which mass transfer occurs from the dispersed phase to the aqueous continuous phase and where the major resistance to transfer is in the dispersed phase. Nomographs facilitate design calculations. (auth)

17216
MASS TRANSFER AT LOW FLOW RATES IN A PACKED COLUMN. V. P. Dorweiler and R. W. Fahien (Iowa State Coll., Ames). A.I.Ch.E. Journal 5, 139-144 (1959) June.

SOLVENT EXTRACTION 6.16

SURVEYS AND THEORY 6.16.06

FLOODING AND FLOWRATE 6.16.06.01

Mass transfer in packed columns has been investigated for a variety of column and packing sizes but at flow rates restricted to fully developed turbulent conditions. The present work was undertaken to investigate mass transfer at flow-rate conditions in the transition and laminar regions. A dual treatment of experimental data required a knowledge of the variation of concentration and velocity with radial position. A tracer-injection technique was employed which consisted in the introduction of a tracer gas into the center of a bulk gas stream and the measurement of the tracer-gas concentration at various radial positions downstream. The velocity distribution for the packed column was determined by means of a five-loop, circular, hot-wire anemometer. The test column was a vertical 4-in. pipe, packed with ¼-in. spherical, ceramic catalyst-support pellets. Mass transfer diffusivity and Peclet number were determined from two solutions of the differential-diffusion equation applied in previous investigations. An analytical solution in terms of Bessel functions was used to calculate values of average diffusivity and Peclet number and a seminumerical solution in terms of homogeneous linear-difference equations to calculate values of point diffusivity and Peclet number. Variation of diffusivity and Peclet number with radial position is shown, average diffusivity and Peclet number being correlated with Reynolds number. The interaction of molecular and eddy mass transfer mechanisms with decreasing mass velocity is illustrated by defining a molecular and an eddy Peclet number and correlating with Reynolds number. Eddy diffusivity is correlated as a function of local flow conditions. (auth)

13893

MIXING OF FLUIDS FLOWING THROUGH BEDS OF PACKED SOLIDS. Earl A. Ebach and Robert R. White (Univ. of Michigan, Ann Arbor). A.I.Ch.E. Journal 4, 161-9(1958) June.

An extension of the known data and the results of an investigation of the axial mixing of liquids flowing through fixed beds of solids are presented. The variables which were investigated were fluid velocity, particle diameter, particle shape, and liquid viscosity (J.R.D.)

5929

SOLVENT EXTRACTION SYSTEM FOR ENRICHED URANIUM. Joe Dykstra, B. H. Thompson, and R. J. Clouse (Union Carbide Nuclear Co., Oak Ridge, Tenn.). Ind. Eng. Chem. 50, 161-5(1958) Feb.

Test data show that the most efficient extractive column operation occurs 10 to 20 r.p.m. below flood for the solvent extraction of enriched U. (W.L.H.)

OPERATION OF A CENTER-FED CONTINUOUS-FLOW THERMOGRAVITATIONAL COLUMN WITH UNEQUAL FLOW RATES IN THE TWO COLUMN SECTIONS. COMPARISON OF THEORY AND EXPERIMENT. John E. Powers and C. R. Wilke (Univ. of California, Berkeley). J. Chem. Phys. 27, 1000-1(1957) Nov.

Equations of Jones and Furry are shown to agree well with experimental data over a range of ratios of rectifying section flow to stripping section flow from 0.3 to 5. On the assumption of an effective feed location which yields maximum separation a tentative modification of the Jones-Furry equation is developed which appears preferable at smaller values of the flow rate. (auth)

2304 HW-52927

General Electric Co. Hanford Atomic Products Operation, Richland, Wash.

LITERATURE SURVEY OF FLOW PATTERNS ASSOCIATED WITH TWO-PHASE FLOW. T. W. Ambrose. Oct. 8, 1957. 11p. Contract [W-31-109-Eng-52]. $3.30(ph OTS); $2.40(mf OTS).

2274

LIQUID-LIQUID EXTRACTION. PART XII. FLOODING RATES AND PERFORMANCE DATA FOR A ROTARY DISC CONTACTOR. D. H. Logsdail, J. D. Thornton, and H. R. C. Pratt (Atomic Energy Research Establishment, Harwell, Berks). Trans. Inst. Chem. Engrs. (London) 35, 301-15(1957) Oct.

Flood-point data have been obtained for a 3 in. diameter rotary disc contactor using a number of solvent–water systems. The variables studied were the rotor speed, compartment height, and the disc and stator diameters. In the presence of an undistributed solute, it was found that the total throughput increased by 100 to 200% when solute transfer took place from the solvent to the aqueous phase, whereas in the reverse direction of transfer the throughput was reduced by approximately 10% compared with the solute-free system. In the absence of solute, data have

SOLVENT EXTRACTION 6.16

SURVEYS AND THEORY 6.16.06

FLOODING AND FLOWRATE 6.16.06.01

been interpreted in terms of the characteristic droplet velocities and these have been correlated with the column geometry and the physical properties. Mass transfer data have been obtained for both directions of transfer using two solvents, i.e. toluene and butyl acetate, with acetone as the solute. It has been shown that the over-all values of the mass transfer coefficient or H.T.U. cannot be split into the individual "film" values, and a new correlation has been obtained in which allowance is made for variations in interfacial area with column geometry and rotor speed. (auth)

2275

LIQUID-LIQUID EXTRACTION. PART XIII. THE EFFECT OF PULSE WAVE-FORM AND PLATE GEOMETRY ON THE PERFORMANCE AND THROUGHPUT OF A PULSED COLUMN. J. D. Thornton (Atomic Energy Research Establishment, Harwell, Didcot, Berks). Trans. Inst. Chem. Engrs. (London) 35, 316-30(1957) Oct.

Flood-point data have been determined for six solvent—water systems using a 3 in. diameter pulsed plate-column. The variables investigated were the plate spacing, plate geometry (i.e. hole-size and percentage free area), and the pulse wave-form. In the latter case, sinusoidal, saw-tooth, and semi-square wave-forms were selected for study. In the absence of an undistributed solute, the data were interpreted in terms of the characteristic velocity which was correlated with the column variables, the physical properties of the system and the pulse characteristics. Mass transfer data were obtained for both directions of transfer using the systems water—toluene and water—butyl acetate with acetone as solute. Overall (H.T.U.) values for a given system were correlated in terms of variations in the interfacial area. (auth)

2276

LIQUID-LIQUID EXTRACTION. PART XIV. THE EFFECT OF COLUMN DIAMETER UPON THE PERFORMANCE AND THROUGHPUT OF PULSED PLATE COLUMNS. D. H. Logsdail and J. D. Thornton (Atomic Energy Research Establishment, Harwell, Berks). Trans. Inst. Chem. Engrs. (London) 35, 331-42(1957) Oct.

Flood-point data have been determined for 6 in. and 12 in. diam. pulsed columns using the systems water—toluene and water—white spirit. Mass transfer measurements, together with a limited number of hold-up measurements, have also been carried out with 6 in., 9 in., and 12 in. diam. columns using the system water—toluene with acetone as solute transferring from the solvent to the aqueous phase. The data were correlated by means of the expressions developed previously for a 3 in. diam. column and it was found that whereas the limiting throughput scaled up geometrically with column diameter, the over-all H.T.U. was proportional to exp. $(d_c/2)$. The application of these results to the design of pulsed plate columns is discussed in detail. (auth)

1256 AERE-CE/R-2201

Gt. Brit. Atomic Energy Research Establishment, Harwell, Berks, England.

FLOOD-POINT DATA FOR "SPRAYPAK" COLUMN PACKINGS AT HIGH LIQUID RATES. F. R. Dell. May 1957. 13p.

The flood-point data for SPRAYPAK given in earlier reports have been extended to high liquid rates, using the air—water system. It has been found that the earlier correlation did not express accurately the effects of course height and mesh strand width on the flood point under these conditions, and for practical design purposes a separate modified correlation is given for high liquid-gas ratios. (auth)

5273

LIQUID EXTRACTION. Robert E. Treybal (New York Univ., New York). Ind. Eng. Chem. 49, 514-25(1957) March.

A literature review is presented on the industrial aspects of liquid extraction. Analytical chemistry applications are excluded. 341 references. (T.R.H.)

7087 CCC-1024-TR-222

Callery Chemical Co., Penna.

RESIDENCE TIMES AND CONVERSIONS IN CONTINUOUS-FLOW, STIRRED-VESSEL REACTOR SYSTEMS. D. K. Eads — L. S. Stone and P. M. Maginnity, eds. Jan. 22. 1957. 22p.

By regarding a continuous reactor as a means of taking an infinite sequence of micro reactors to reaction conditions, keeping them there for a certain length of time

SOLVENT EXTRACTION 6.16

SURVEYS AND THEORY 6.16.06

FLOODING AND FLOWRATE 6.16.06.01

and then returning them to non-reacting conditions, an equation was developed for the design of continuous-flow, multi-vessel, reactor systems. This equation involves the residence-time characteristics of such a system, and an expression for these characteristics was developed on the basis of the usual assumption of instantaneous mixing in every stage. One analytical and two graphical design procedures were developed from these equations. Also, a rather crude but useful procedure for approximate reactor design was developed to permit direct orienting of design and thus eliminate much of the tedious trial-and-error calculation that is normally required. (auth)

REPROCESSING OF REACTOR FUEL ELEMENTS AND CLADDING MATERIALS BY SOLVENT EXTRACTION. F. L. Culler. Khim. yadernogo goryuchego, 258-308(1956). (Translated from Referat. Zhur. Khim. No. 3, 1957, abstract no. 8766).

The following questions are discussed: (1) nuclear fuels and the requirements of the regeneration process; among the topics covered are the types of nuclear fuels used and flow sheets for the chemical treatment processes, the cooling periods required for the decomposition of the radioactive contaminants, the contamination by the fission products, and the purification of the fissionable material and of the material to be used in the preparation of secondary nuclear fuels from the daughter products formed during neutron irradiation; (2) the technical processing of the fuel elements, with flow sheets describing the recovery of the fissionable materials and the materials used in the preparation of secondary fuel elements by solvent extraction; (3)

2699 ✓ AECU-3939
Massachusetts Inst. of Tech., Oak Ridge, Tenn. Engineering Practice School.
VISUAL OBSERVATION OF LIQUID-LIQUID SPRAY COLUMN PERFORMANCE. M. H. Fontana and R. W. Wolff. Nov. 27, 1956. 31p. For [Oak Ridge Gaseous Diffusion Plant. Contract W-7405-eng-26, Subcontract 70]. (KT-250). $6.30(ph OTS); $3.00(mf OTS).

The degree and type of mixing in a two-phase spray column can be studied visually. In this investigation various types of spray column packings and bafflings were observed by this method. The effect of back mixing was also determined by means of the residence time technique. The main criteria to be observed in this study were: The relative size of the droplets of the discontinuous phase; size, shape, and behavior of eddies of the continuous phase; column holdup of the discontinuous

6767
A SYSTEM FOR COUNTING VARIABLES IN SEPARATION PROCESSES. Mooson Kwauk (Hydrocarbon Research, Inc., New York). A.I.Ch.E. Journal 2, 240-8(1956) June.

In the proposed system for counting variables in separation processes the processes are resolved into their simpler component classes, e.g., theoretical plates, heat exchangers, reboilers, distillation columns, etc., and a distinction is made between those variables which are inherent in the systems and those which may be specified for design. Results are presented for the most commonly occurring component classes, and all possible process relations existing among these classes are expressed by a set of generalized equations. The procedure of counting variables is therefore reduced to composition from variables for the component classes by use of the generalized equations. (auth)

/ **6598**
FLOODING CHARACTERISTICS OF A PULSE EXTRACTION COLUMN. R. B. Edwards and G. H. Beyer (Iowa State Coll., Ames.). A.I.Ch.E. Journal 2, 148-52(1956) June.

An investigation using the hexone–water system was made of flooding in a 1-in.-diam. ten-plate pulse column. An analysis of column operation led to the derivation of an equation for predicting conditions of inadequate pulsation and for establishing the amount of liquid recycled under any operating conditions. (auth)

7584
PREDICTION OF IDEAL-STAGE REQUIREMENTS IN COMPLEX LIQUID-LIQUID EXTRACTION SYSTEMS. H. C. Peterson and G. H. Beyer (Iowa State Coll., Ames). A. I. Ch. E. Journal 2, 38-41(1956) Mar.

A procedure for obtaining equilibrium data and predicting ideal-stage requirements in a complex liquid-liquid extraction system is presented. Preliminary equilibrium data are obtained from a simulated column run involving a series of batch contacts operated in such a manner as to approach

SOLVENT EXTRACTION 6916

SURVEYS AND THEORY 6.16.06

FLOODING AND FLOWRATE 6.16.06.01

steady state countercurrent conditions. The flow ratios and stage requirements for continuous operation are then estimated by trial-and-error by use of a modified McCabe-Thiele method. The procedure lends itself particularly well to those systems with interdependent distribution of the two components. Data for the separation of Hf from Zr are presented to show the utility of the method. (auth)

3558 AECU-3927
Massachusetts Inst. of Tech., Oak Ridge, Tenn. Engineering Practice School.
FLOW PATTERNS IN VERTICALLY PARTITIONED SPRAY COLUMNS. James R. Kennedy and Domenic B. Vassallo. Jan. 20, 1956. 10p. For Carbide and Carbon Chemicals Co. K-25 Plant. Contract W-7405-eng-26, Subcontract 70. (KT-223). $1.80(ph OTS); $1.80(mf OTS).

A study of the flow patterns existing inside a vertically-partitioned glass spray column was made. A water-kerosene system was employed in which the water was the continuous phase, and the kerosene, which was dyed, was the discontinuous phase. Observations and colored motion pictures of the system flow patterns were made with an unbaffled column and with the column containing vertical plate partitions and vertical glass tables. It was concluded that the phase distribution in this type column is unstable, and channeling of the continuous and discontinuous phases occurs regardless of continuous phase distributor used. (J.R.D.)

4557
PERFORMANCE OF A PULSED SPRAY COLUMN. C. J. Billerbeck, Jack Farquhar, III, R. C. Reid, J. C. Bresee, and A. S. Hoffman (Massachusetts Inst. of Tech. Engineering Practice School, Oak Ridge, Tenn.). Ind. Eng. Chem. 48, 183-7(1956)

A study of the effect of pulse frequency and throughput on the operation of a simple pulsed spray column was made. The system employed was water–acetic acid–methyl isobutyl ketone which has been used for liquid-liquid extraction studies. (auth)

2341 HW-20305
[Hanford Works, Richland, Wash.]
DESIGN CONSIDERATIONS FOR A PULSE COLUMN SYSTEM. V. R. Cooper and C. Groot. Mar. 9, 1950. Changed from OFFICIAL USE ONLY July 6, 1956. 9p. Contract [W-31-109-Eng-52]. $1.80(ph OTS); $1.80(mf OTS).

The hydraulic forces in the countercurrent flow, vertical type extractor pulse column were evaluated and their effects on pulse column design were determined. (F.S.)

PROCESSES 6.16.06.02

See also, under PLANTS AND EQUIPMENT, INSTRUMENTATION AND CONTROL --Transfer of Fluids (5.11.08).

14323
SALTING EFFECTS IN THE SOLVENT EXTRACTION BEHAVIOR OF INORGANIC COMPOUNDS. R. M. Diamond (Cornell Univ., Ithaca, N. Y.). J. Phys. Chem. 63, 659-67(1959) May.

The variation in the distribution ratio for tracer indium(III) distributing between an oxygenated organic solvent and aqueous solutions of hydrochloric acid in which the concentration of HCl was varied while keeping the initial ionic strength constant with various chloride salts was studied as a model system for the extraction behavior of ionic species under these conditions. It is seen that the extraction of acid species plays a somewhat unique role due to the special ability of the hydronium ion to hydrogen-bond water molecules in first shell solvation and to enhance further coördination with the solvent molecules. In such acid extractions, the coördinating ability of the organic solvent, determined by its basicity and the steric availability of the donor atom, and not the solvent's dielectric constant, is of paramount importance. In contrast, the extraction of large, relatively unhydrated salt species, such as $N(C_2H_5)_4^+InCl_4^-$, depends primarily on the dielectric constant of the organic solvent and not on its coördinating ability. With solvents of high dielectric constant, i.e., nitrobenzene, the extraction of ionic species increases with increasing size of the ions. With solvents of low dielectric constant but good coördinating ability, such as diethyl ether, the extraction of hydrated cations

SOLVENT EXTRACTION 6.16

SURVEYS AND THEORY 6.16.06

PROCESSES 6.16.06.02

decreases with increasing crystallographic size, exactly the reverse order. The variations in the extraction of the indium tracer with replacement of the aqueous HCl by the different salt chlorides are explained on the basis of (1) the creation of new extracting species involving an added ion and the ion of interest; (2) the creation of new extracting species not involving the ion of interest, but repressing the latter's extraction; (3) a change in the relative proportion of the extractable species in the aqueous phase; (4) a change in the nature of the aqueous phase with changing water activity and dielectric constant. (auth)

14385 CF-58-11-91
Oak Ridge National Lab., Tenn.
RECENT DEVELOPMENTS IN FEED PREPARATION AND SOLVENT EXTRACTION. F. R. Bruce, R. E. Blanco, and J. C. Bresee. Mar. 20, 1959. 67p. Contract [W-7405-eng-26]. $10.80(ph), $3.90(mf) OTS.

For presentation at the 5th Nuclear Congress, Cleveland, Apr. 7, 1959.

Increasing emphasis has been placed recently on the application of solvent extraction to the recovery of uranium and plutonium from spent power reactor fuels. Zircaloy-2 jackets were removed from PWR blanket-type fuels by dissolution with the Zirflex Process, and the UO_2 cores were dissolved in 10 M HNO_3. Zirflex treatment of prototype samples irradiated to 2500 Mwd/ton resulted in satisfactory dissolution rates and losses to the dejacketing solution generally less than 0.2% for U and Pu. U–Zr alloy fuels were dissolved in 6 M NH_4F and adjusted for solvent extraction by the addition of $Al(NO_3)_3$ and HNO_3. In an alternative procedure, fluoride was recycled by metathesis and precipitation. Stainless steel jackets were removed from Consolidated Edison-type fuels by dissolution in 6 M H_2SO_4 (Sulfex Process), and the ThO_2–UO_2 core was dissolved in 13 M HNO_3–0.04 M F–0.04 M Al. Dejacketing losses in unirradiated samples were about 0.02%. Use of the ORNL Reference Darex flowsheet for APPR processing resulted in solvent extraction feed containing 30 ppm chloride. Mechanical equipment was designed to declad SRE fuel, and chop and leach techniques are being developed to treat stainless and Zr clad ceramic fuel. A solvent extraction flowsheet was developed for Foreign Research Reactor Fuels, (Al–20% enriched U alloy) using a revised tributyl phosphate extraction system for the separation of Pu from U. Feasibility tests were carried out on the coupling of Redox solvent extraction with Darex and Niflex head-end treatments. New solvents for the reprocessing of power reactor fuels are being studied. Among these, the amines show an order of magnitude greater radiation stability than does tributyl phosphate. A primary amine was proposed for the recovery of U and Pu from Sulfex decladding wastes.

19374
ION EXCHANGE PROCESS FOR THE RECOVERY AND PURIFICATION OF MATERIALS. R. S. Long and R. H. Bailes (to U. S. Atomic Energy Commission). U. S. Patent 2,830,874. Apr. 15, 1958.

A process for the recovery of certain metallic ions from aqueous solutions by ion exchange techniques is described. It is applicable to elements such as vanadium, chromium, manganese, and the like, which are capable of forming lower valent cations soluble in aqueous solutions and which also form higher valent anions soluble in aqueous acidic solutions. For example, small amounts of vanadium occurring in phosphoric acid prepared from phosphate rock may be recovered by reducing the vanadium to a trivalent cation adsorbing the vanadium in a cationic exchange resin, then treating the resin with a suitable oxidizing agent to convert the adsorbed vanadium to a higher valent state, and finally eluting the vanadium as an anion from the resin by means of an aqueous acidic solution.

13837
EXTRACTION AS A METHOD OF SEPARATION AND INVESTIGATION OF RADIOACTIVE ELEMENTS. V. M. Vdovenko. Zhur. Neorg. Khim. 3, 145-54(1958). (In Russian)

A review is presented on the extraction methods used in radiochemistry. 34 references. (J.S.R.)

14735 A/CONF.15/P/602
Mallinckrodt Chemical Works, St. Louis.
THE NEWEST UNITED STATES URANIUM PROCESSING PLANT. Harold E. Thayer. 24p. $0.50(OTS).

SOLVENT EXTRACTION 6.16

SURVEYS AND THEORY 6.16.06

PROCESSES 6.16.06.02

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

The refining operation is carried out in pumper-decanter extraction units, designed to operate with a TBP-hexane solvent system, which permits the attainment of very high through-put per square foot of cross section. Washing of the extract and re-extraction into water is accomplished in pulse columns with teflon-coated perforated plates, which also have high through-put. Both saturation control and a second extraction cycle using diethyl ether are available, providing optional means of attaining the ultimate in purity of product. The production of green salt is carried out in the tubular stirred bed reactors which have proven so successful in the past. Improved production rates are achieved, however, through the use of fluid bed equipment for the reduction step. The UO_2 intermediate is suitable for diversion to power reactor fuel element usage. The UF_4 product is suitable for conversion to UF_6 or to metal. (auth)

1307

THE SOLVENT EXTRACTION IN NUCLEAR TECHNOLOGY. R. De Leone. Energia nucleare (Milan) 4, 391-8 (1957) Oct. (In Italian)

A general survey on the solubility of inorganic salts in organic solvents is given and the solubility of transition metals salts in oxygenated solvents is emphasized. The features of uranyl and thorium nitrates purification, of the U-Th and Zr-Hf separations and of U recovery from leach liquors, by solvent extraction, are reported. (auth)

17940

BATCH FRACTIONAL LIQUID-LIQUID EXTRACTIONAL PROCESS. D. W. Fosbury (to U. K. Atomic Energy Authority). British Application 3563/56. Atom No. 5, 23-4(1957) Mar.

The components of a mixture to be separated are dissolved in a suitable solvent (the first solvent) the solution is brought into intimate contact continuously with a substantially immiscible solvent (the second solvent) which will preferentially extract one or more component from the solution, the extract is contacted with a phase obtained by stripping from the second solvent with the first solvent the component or components still held by the second solvent after contacting with the said phase and, when a steady state has been reached in the system, a portion of the said phase is continuously withdrawn as product. By appropriate selection of the second solvent all the components of a mixture may be separated consecutively.

FACTORS AFFECTING THE SOLUTION OF INORGANIC SALTS IN ORGANIC SOLVENTS. Leonard I. Katzin (Argonne National Lab., Lemont, Ill.). J. Inorg. and Nuclear Chem. 4, No. 3-4, 187-204(1957).

Inorganic salts are appreciably soluble in organic media, principally of the oxygenated forms (alcohols, ketones, ethers, esters), when they are capable of forming neutral molecules in solution. This property is largely confined to the transition elements. Those of the first long series, and in the trans-radium region, accomplish this without forming undebatably covalent compounds; other subgroup elements which are solvent-soluble show a stronger tendency toward covalent bond formation, and may show solubility in chlorinated hydrocarbons and benzenoid solvents in which the first group mentioned are not usually soluble. The salts of the first three groups of the periodic table generally are not organic-soluble, because their co-ordinative power is relatively low, and their solid lattice energies are high. They tend strongly to approach the point-charge characterization on which the electrostatic approach to aqueous solution chemistry is based. (auth)

6606

PROCESS ENGINEERING AT THE HANFORD SEPARATION PLANTS. Charles A. Rohrmann (Hanford Atomic Products Operation, Richland). Nucleonics 14, No. 6, 66-8(1956) June.

Plants for the separation of U, Pu, and fission products from spent reactor fuel by continuous solvent extraction processes and the problems that must be solved in remotely controlled, radioactive corrosive systems are discussed. (F.S.)

SOLVENT EXTRACTION 6.16

SURVEYS AND THEORY 6.16.06

Processes 6.16.06.02

7591
LIQUID-LIQUID EXTRACTION PROCEDURES IN INORGANIC ANALYSIS. T. S. West (Department of Chemistry, University of Birmingham). Metallurgia 53, 132-4(1956) March.
Liquid-liquid extraction techniques in inorganic analysis, with particular reference to metallurgical analysis, are surveyed. The first article in this series appeared in Metallurgia 53, 96(1956).

2336 CF-54-11-185
Oak Ridge National Lab., Tenn.
DESIGN OF A PILOT PLANT TO STUDY LIQUID-LIQUID EXTRACTION OF URANIUM FROM ACIDIC SULFATE LEACH LIQUORS. B. B. Klima, J. F. Manneschmidt, and J. R. Buchanan. Nov. 30, 1954. Decl. Feb. 20, 1956. 51p. Contract W-7405-eng-26. $9.30(ph OTS); $3.60(mf OTS).
The design of a pilot plant for the Amine Extraction Process is given. Criteria, material balances, equipment, and calculations are included. (T.R.H.)

STAGE EFFICIENCY 6.16.06.03

22063
LONGITUDINAL MIXING IN A PULSED SIEVE-PLATE EXTRACTION COLUMN. B. W. Mar and A. L. Babb (Univ. of Washington, Seattle). Ind. Eng. Chem. 51, 1011-14(1959) Sept.
The effects of plate geometry, pulsing conditions, liquid properties, and liquid flow rates on longitudinal mixing in a pulsed sieve-plate extraction column 2 inches in diameter were correlated. Experimental data for the hexane-water, benzene-water, and carbon tetrachloride-water systems were obtained using steady-state and delta injection techniques. Empirical correlation of the data permits estimation of continuous phase Peclet numbers with an average deviation of 17% from the experimental values. A nomograph facilititates the calculation. Back-mixing can be decreased by use of higher continuous-phase flow rates, smaller plate spacings, and smaller holes in the plates. (auth)

22064
PERFORATED PLATE COLUMN STUDIES BY THE BOX METHOD OF EXPERIMENTATION. Donald A. Dechman and Matthew Van Winkle (Univ. of Texas, Austin). Ind. Eng. Chem. 51, 1015-18(1959) Sept.
This study verifies the applicability of the Box method of experimentation for correlating efficiency and pressure drop with operating and design variables of a perforated plate column. The independent variables were reflux ratio, vapor mass velocity, weir height, and hole diameter. A second-order polynomial equation with all possible second-order interactions included was selected. The constants were evaluated by a least squares fit of 246 data points and with 28 data points selected by the Box design technique. The lack of fit variance and confidence limits were established for each equation. The Box correlations for efficiency and pressure drop agreed with experimental data in the range of its design. Comparison of the two equations for efficiency showed the Box-designed equation to be satisfactory. (auth)

15066 UCRL-8658
California. Univ., Berkeley. Lawrence Radiation Lab.
LONGITUDINAL DISPERSION IN PACKED EXTRACTION COLUMNS. Gabriel L. Jacques, John E. Cotter, and Theodore Vermeulen. Apr. 1959. 71p. Contract W-7405-eng-48. $2.50(OTS).
Longitudinal dispersion (axial mixing) was studied for counter-current flow of liquids through packed beds, using six different combinations of packing type and size and bed arrangements. The Péclet number, used as a dimensionless measure of the longitudinal dispersion coefficient, was measured in both the continuous and the discontinuous phase of the systems kerosene in water, water in kerosene, and water in mineral oil. The observed values ranged from 100 to 10% of the Péclet number measured for the same packing in single-phase laminar flow. Péclet numbers for each phase were correlated as a function of the two flow rates. An effect of packing diameter and sphericity on the continuous-phase Péclet number was also found. Different mechanisms were indicated for dispersion in the discontinuous phase, depending upon whether or not that phase wets the packing. (auth)

SOLVENT EXTRACTION 6.16

SURVEYS AND THEORY 6.16.06

STAGE EFFICIENCY 6.16.06.03

5248 ✓ CF-59-12-45
Oak Ridge National Lab., Tenn.
DESCRIPTION OF A PROPOSED PROGRAM: DYNAMIC ANALYSIS OF RADIOCHEMICAL PLANT COMPONENTS. M. E. Whatley. Dec. 15, 1959. 8p. Contract [W-7405-eng-26]. OTS.

The mathematical models which are now available to describe the separation operations being used in the radiochemical separations plants are inadequate either for use in computer controlled plants or for adequate understanding of the fundamentals of their operation. A study is proposed to analyze the solvent extraction operation in order to produce a method of calculating steady state contactor performance for optimizing new flow sheets, and to describe the transient effects in the operation in order to better understand the limitations of the steady state model, to establish quantitative criteria for control, and to provide models for future application to computer controlled plants. (auth)

13038 ✓ ANL-5874
Argonne National Lab., Lemont, Ill.
THE EFFECT OF PULSATION ON LIQUID-LIQUID MASS-TRANSFER RESISTANCES. Manuel R. Eugenio. July 1958. 119p. Contract W-31-109-eng-38. $2.50 (OTS).

A study of individual mass-transfer resistances in liquid-liquid extraction and the effects on these of such variables as flow rate is presented. A partially miscible system was used in the determination of individual film mass-transfer resistances in a pulsed column and to investigate the effect of pulsed column variables, such as pulse frequency, pulse amplitude, and flow rates, on the individual resistances. (W.L.H.)

13828 ✓
ESTIMATION OF THE STAGE EFFICIENCY OF SIMPLE, AGITATED VESSELS USED IN MIXER-SETTLER EXTRACTORS. Robert E. Treybal (New York Univ.). A.I.Ch.E. Journal 4, 202-7(1958) June.

A method of calculation is presented by which estimates may be made of the stage efficiency of continuously operated, agitated, baffled vessels used in mixer-settler extractors. The calculations are limited to cases where the agitating impeller is a flat-blade turbine, and do not include estimates of the entrance and exit effects. The method has been tested with all the available experimental data, which include three different sizes of vessels, systems, and impeller sizes, and a variety of operating conditions including speeds of agitation, rates of flow, and ratios of contacted liquids. (auth)

14688 A/CONF.15/P/521
Du Pont de Nemours (E. I.) & Co. [Savannah River Lab., Augusta, Ga.].
A RATIONALE FOR THE RECOVERY OF IRRADIATED URANIUM AND THORIUM BY SOLVENT EXTRACTION T. H. Siddall, III. 24p. $0.50(OTS).

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

Some of the calculations which can be made to predict the proper operating conditions for multistage contractors in which tri-n-butyl phosphate is the extractant are given. The calculations are made from the results of equilibrations in single stages in the laboratory. Tri-n-butyl phosphate is compared with other trialkyl phosphates and with certain dialkyl alkylphosphonates. The basis of the comparison is the method of calculation that was developed. (A.C.)

A RATIONALE FOR THE RECOVERY OF IRRADIATED URANIUM AND THORIUM BY SOLVENT EXTRACTION. T. H. Siddall, III. 24p. (A/CONF.15/P/521)

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

Some of the calculations which can be made to predict the proper operating conditions for multistage contractors in which tri-n-butyl phosphate is the extractant are given. The calculations are made from the results of equilibrations in single stages in the laboratory. Tri-n-butyl phosphate is compared with other trialkyl phosphates and with certain dialkyl alkylphosphonates. The basis of the comparison is the method of calculation that was developed.

4759 UCRL-8029
California. Univ., Berkeley. Radiation Lab.
LONGITUDINAL DISPERSION IN SOLVENT-EXTRACTION COLUMNS: PECLET NUMBERS FOR ORDERED AND

SOLVENT EXTRACTION 6916

SURVEYS AND THEORY 6.16.06

STAGE EFFICIENCY 6.16.06.03

RANDOM PACKINGS. Gabriel L. Jacques and Theodore Vermuelen. Nov. 1957. 152p. Contract W-7405-eng-48. $24.30(ph OTS); $7.50(mf OTS).

Dispersion phenomena in packed beds in both axial and radial directions were studied to provide basic data for extraction-tower design Nine different beds were used, involving regular and random arrangements of spheres, and random arrangements of Raschig rings and Intalox saddles. For one-phase flow a wide range of Reynolds numbers was used (5 to 2000), covering regions of laminar, transition, and turbulent flow. For the axial Peclet number, different constant values were found in the turbulent range and in the laminar range. The existence of a fairly sharp transition region was observed. Radial-dispersion results showed similar behavior. The axial Peclet number was found to vary inversely with bed porosity, while the radial Peclet number was almost proportional to porosity. For two-phase flow, measurements were made up to the flooding point, which occurred at Reynolds numbers near 100. The axial Peclet number for both continuous and discontinuous phases is a function of the Reynolds numbers for the two individual phases. For each phase, the laminar value of the axial Peclet number obotained for one-phase flow is an upper limit. Values as low as one-fifth of this limiting value were found at relatively large flow rates of the second phase. The radial Peclet number was measured only for the continuous phase of two-phase systems; it was found to decrease in the same direction, but by a smaller percentage. (auth)

1952 ✓ ISC-900
Ames Lab., Ames, Iowa.
PREDICTION OF CONTINUOUS EXTRACTOR PERFORMANCE FROM SIMULATED EXTRACTION COLUMN DATA. Morton Smutz. [Sept. 1957.] 7p. Contract [W-7405-eng-82.] $1.80(ph OTS); $1.80(mf OTS).

A discussion of solvent extraction is presented with emphasis on solvent selection. It is pointed out that calculation methods for determining the operating conditions required for a given separation are well known; however, equilibrium data must be included. After sufficient equilibrium data have been obtained to approximate the flow ratios and the number of ideal stages required for a given separation, simulated column runs are made. The use of simulated column runs can provide information on the potentiality of the process and production rate in a particular type of extractor. By comparing results from one system with another, it is possible to estimate probable limiting production rates in a particular extractor, thus avoiding expensive pilot plant operations. (J.R.D.)

1311
LIQUID-LIQUID EXTRACTION. PART IX. A FURTHER STUDY OF INDIVIDUAL FILM COEFFICIENTS FOR A PACKED COLUMN. R. Gayler and H. R. C. Pratt (Atomic Energy Research Establishment, Harwell, Berks). Trans. Inst. Chem. Engrs. (London) 35, 267-72(1957) Aug.

The coefficients for the continuous (but not the dispersed) phase are reduced by longitudinal mixing, and a driving force correction factor must be introduced into the mass transfer expression for this phase to allow for the resulting reduction in driving force. (auth)

1312
LIQUID-LIQUID EXTRACTION. PART X. OVERALL MASS TRANSFER COEFFICIENTS ON AN AREA BASIS FOR THE EXTRACTION OF ACETONE IN PACKED COLUMNS. R. Gayler and H. R. C. Pratt (Atomic Energy Research Establishment, Harwell, Berks). Trans. Inst. Chem. Engrs. (London) 35, 273-91(1957) Aug.

1313
LIQUID-LIQUID EXTRACTION. PART XI. MASS TRANSFER DATA FOR THE EXTRACTION OF URANYL NITRATE IN A PACKED COLUMN. L. E. Smith, J. D. Thornton, and H. R. C. Pratt (Atomic Energy Research Establishment, Harwell, Berks). Trans. Inst. Chem. Engrs. (London) 35, 292-300(1957) Aug.

Mass transfer and hold-up data are presented for the transfer of uranyl nitrate between water and methyl isobutyl ketone in a 4 in. diam. column packed with 8.0 ft. of ½ in. Raschig rings. Four series of runs were carried out; in three the transfer was from the aqueous into the solvent phase using a range of aqueous phase inlet concentrations, and in one the transfer was in the opposite direction. (auth)

SOLVENT EXTRACTION 6.16

SURVEYS AND THEORY 6.16.06

STAGE EFFICIENCY 6.16.06.03

10584 ANL-5741
Argonne National Lab., Lemont, Ill.
MASS TRANSFER IN A CONTINUOUS-FLOW MIXING VESSEL. Virgil G. Trice, Jr. July 1957. 43p. Contract W-31-109-eng-38. $0.30(OTS).

Individual continuous-phase film coefficients have been measured on an area basis for the transfer of mass between partially miscible liquids in a continuous-flow mixing vessel. For the systems studied (isobutanol dispersed in water and nitrobenzene dispersed in ethylene glycol) the mass transfer coefficient, in agreement with the penetration theory, was found to vary as the square root of the molecular diffusivity. The applicability of a previously suggested relationship was demonstrated by the correlation of the experimental data in terms of the Sherwood, Schmidt, and Reynolds numbers. The relationship obtained was in fundamental agreement with correlations developed by investigators using other types of liquid-liquid contacting apparatus. (auth)

EVALUATION OF AN EXPERIMENTAL FRENCH WET SCRUBBER "SOLIVORE." Edward Kristal, Richard Dennis, and Leslie Silverman. Mar. 29, 1957. 57p. (NYO-4612)

Performance tests were conducted on an experimental model of a French wet scrubber called the "Solivore." This unit (600 cfm capacity) consists of 4 similar collection stages in series, each containing two 1½ hp spray generators rated at 9 gpm and 7 psi, and a special Venturi tube. A distinctive feature of this device is the spray generator which uses a rotating mechanical interrupter to disintegrate small liquid jets. Weight collection efficiencies were obtained with several aerosols for inlet dust loadings ranging from 0.02 to 2 grains/ft^3, water rates of 6 to 12 gpm per spray generator and for one, two, and three stage operation. Efficiencies determined for a single stage varied from 99% for resuspended fly ash to 22% for iron

12679
SOLVENT EXTRACTION IN ANALYTICAL CHEMISTRY. George H. Morrison and Henry Freiser. New York, John Wiley & Sons, Inc., 1957. 277p.

1823 ORNL-2214
Oak Ridge National Lab., Tenn.
PROCESS TEST SECTION PROGRESS REPORT FOR AUGUST 1956. A. D. Ryon and K. O. Johnsson. Dec. 12, 1956. 13p. Contract W-7405-eng-26. $0.20(OTS).

A study of extraction rates and stripping with a Dapex-type liquor, and a high uranium, high vanadium liquor is reported. The equipment used was a 6-in. mixer cell with a 3-in. 4 flat-blade turbine impeller. The organic extractant used was di-(2-ethylhexyl) phosphoric acid and TBP in kerosene. (T.R.H.)

13582 ANL-5602
Argonne National Lab., Lemont, Ill.
CHEMICAL ENGINEERING DIVISION QUARTERLY REPORT [FOR] APRIL, MAY, JUNE 1956. Aug. 1, 1956. Decl. Feb. 18, 1957. 134p. Contract W-31-109-eng-38. $0.75(OTS).

Light scattering correlations were developed for 25% dispersions and several runs were made to investigate mass transfer in a continuous-flow mixing chamber using the system isobutanol–water. Fluoride volatilization separation processes were investigated. The production of UF_4 from ore concentrates by hydrogenation and hydrofluorination by fluidized-bed techniques are reported. The work on fluidization process development included the production of green salt from $UO_2(NO_3)_2$ solution, from solvent extraction refineries, and the production of UF_6 by direct fluorination of U ore concentrates. Studies were continued on reactor chemistry, chemical metallurgical separation processes, determination of U by x-ray spectrophotometric method, and radioactive waste processing. (J.E.D.)

LIQUID-LIQUID EXTRACTION PROCEDURES IN INORGANIC ANALYSIS. A REVIEW OF PRACTICAL APPLICATIONS WITH PARTICULAR REFERENCE TO METALLURGICAL ANALYSIS. T. S. West (Univ. of Birmingham, England). Metallurgia 54, 47-51(1956) July.

The growing complexity of modern materials has led to difficulties in their chemical analysis resulting from the increasing multiplicity of other substances accompanying the constituent to be determined. The development of new separation methods has made possible the continued use of classical analytical procedures in the face of increasing competition from physical methods, and in the present series of articles the author surveys liquid-liquid solvent

SOLVENT EXTRACTION 6.16

SURVEYS AND THEORY 6.16.06

STAGE EFFICIENCY 6.16.06.03

extraction techniques in inorganic analysis, with particular reference to metallurgical analysis. In the sixth of the series, the author discusses the extraction of Group VIa, VIIa and the transition metals. (auth)

8338 ✓ ISC-860
Ames Lab., Ames, Iowa.
EXTRACTION EFFICIENCY OF A PULSE COLUMN OF VARIED GEOMETRY. Lawrence E. Burkhart and R. W. Fahien. June 1956. 48p. Contract W-7405-eng-82. $1.25(OTS).

An investigation was made of the effects of plate spacing, hole diameter, percent free plate area, pulse amplitude, and pulse frequency on the extraction efficiency of a 1-in. diameter pulse column. The hexone–acetic acid–water system was used. Two levels of each factor were incorporated in an experimental design based on statistical methods. The diameter of the holes in the perforated plates of a pulse column produced the greatest effect on extraction efficiency of the factors studied. Percent free area produced only a small effect which was dependent on the degree of pulsation applied to the column. Extraction efficiency varied inversely with pulse amplitude and pulse frequency. The recycle or back mixing, which exists in the pulse column was postulated to have a profound effect upon extraction efficiency. (auth)

7588 ✓
CONCENTRATION EFFECTS ON SOLVENT EXTRACTION COEFFICIENTS OF SOME TRIVALENT METAL HALIDES. Jerome Saldick (Brookhaven National Laboratory, Upton, N. Y.). J. Phys. Chem. 60, 500-1(1956) April.

The effects of an extractable acid on the solvent extraction of trivalent metal halides in strong acid solutions by an organic solvent are discussed. The derived relationships indicate that the extraction coefficient $E_{org/aq}$ is a function of the acidity in the organic phase. (C.W.H.)

4124 NP-tr-22
RECTIFICATION OF LOW CONCENTRATION BINARY MIXTURES. K. Pohl. Translated by W. A. Olscher from Chem -Ing.-Tech. 28, 562-4(1956). 10p.

A numerical process is given for calculating reflux ratio, theoretical plates, and plate concentrations vs. temperature in distillation of binary mixtures with a concentration so low that the equilibrium curve is a straight line. (T.R.H.)

10762 CF-55-11-30
Oak Ridge National Lab., Tenn.
A NEW CALCULATION METHOD FOR EVALUATING SOLVENT EXTRACTION CONTACTOR PERFORMANCE. M. E. Whatley. Nov. 3, 1955. 11p. Contract W-7405-eng-26. $3.30(ph OTS); $2.40(mf OTS).

A new method of calculating the performance of solvent extraction contractors is presented in its conceptual form. This method is similar to the conventional HETS and HTU methods developed from the concept of a differential height of column being equivalent to a mixer settler stage with differential efficiency. Some of its implications and advantages are discussed. (auth)

LIQUID-LIQUID EXTRACTION—THEORY AND LABORATORY EXPERIMENTS. L. Alders. Amsterdam-Houston-London-New York, Elsevier Publishing Co., 1955. 206p.

California. Univ., Livermore. Radiation Lab.
EFFECT OF FLUID PROPERTIES ON MASS TRANSFER IN THE GAS PHASE. Edward J. Lynch and C. R. Wilke. Apr. 2, 1953. 63p. (UCRL-2057 (Rev.)). *Am. Inst. Chem. Engr. J.* 1, 9-19(1955).

The role of gas film resistance was studied by determining the effect of fluid properties on the rate of mass transfer in a packed tower. The HTU, heights of a transfer unit, was compared to the Reynolds numbers at constant liquid rate and with the Schmidt number for flow perpendicular to single cylinders. A bibliography of 51 references is included.

9641 NYO-1176
Catalytic Construction Co., Philadelphia.
MULTICOMPONENT EXTRACTION CALCULATION PROCEDURE. LONG RANGE SURVEY PROGRAM. Job No. 3900. F. W. Winn. Jan. 1, 1953. Decl. Mar. 1, 1957. 27p. Contract AT(30-1)-1311. $4.80(ph OTS); $2.70(mf OTS).

Detailed descriptions are given of the calculation procedure for use in evaluating proposed extraction systems

SOLVENT EXTRACTION 6.16

SURVEYS AND THEORY 6.16.06

Stage Efficiency 6.16.06.03

prior to pilot-plant demonstrations. The procedure is applicable to extraction, stripping, and compound columns and is also used to compute the composition of the two phases on all trays in the column. The procedure is illustrated by application to a Th purification problem in a compound column. (J.A.G.)

12429 CEI-48
Atomic Energy of Canada Ltd. Chalk River, Project, Chalk River, Ont.
ENTRAINMENT AND EFFICIENCY STUDIES IN A SMALL DIAMETER BUBBLECAP PLATE COLUMN.
J. Chrones. July 2, 1952. 37p.

Efficiency and entrainment studies were made in a 4-inch diameter bubblecap plate column using a plate spacing of 4 inches. The plates contained one bubblecap each, and were designed so that they could be easily inserted in a single length of 4 inch OD stainless steel tubing. An entrainment of 10% was obtained for the water-steam system at a column vapor velocity of 1.9 fps or a vapor mass rate of 255 pounds of steam per hour per square foot. This entrainment value is generally accepted as the maximum allowable without impairing the plate efficiency appreciably. This low entrainment may be partly due to the small column diameter with the walls acting as entrainment separators. The Murphree vapor plate efficiency, as measured for alcohol-water, was found to depend on the slope of the equilibrium line and the mass velocity factor $V\rho_v^{0.5}$. An empirical equation was developed relating these factors. Fow low values of the slope of the equilibrium line (2 or less it was noted, that for practical purposes, the actual point efficiency on the plates was equal to the measured Murphree vapor plate efficiency regardless of the degree of mixing on the plate. (auth)

6516 ✓ A/CONF.15/P/1173
ETUDE DES COLONNES A PULSATIONS A L'AIDE DU SYSTEME NITRATE D'URANYLE–ACIDE NITRIQUE–EAU–TRIBUTYLPHOSPHATE. (Study of Pulsed Columns with the System. Uranyl Nitrate–Nitric Acid–Water–Tributylphosphate.) J. Durandet, D. Defives, B. Choffe, and Y. L. Gladel (Inst. Français du Pétrole, France). 28p.

The performance of a pulsed column with perforated plates was studied with the aid of a uranyl nitrate–nitric acid–water–tributyl phosphate system. The extraction of uranium from an aqueous acidic solution by an organic solvent and the extraction of uranium from organic solutions by water were the two cases investigated. The variation of the efficiency and the capacity of the pulsed column was determined as a function of the pulse amplitude and frequency, of the total flow rate, of the diameter of the holes, and of the choice of dispersed phase. The results showed that for a given amplitude and total flow rate the efficiency has a maximum with an increase in frequency. (J.S.R.)

INTERFACE (AND CONTROL) 6.16.06.04

See also, under FISSIONABLE MATERIAL--RECOVERY, SOLVENT EXTRACTION--Nitrate Systems--General (3.02.17.04.03).

1426 ✓ A-1084
Mallinckrodt Chemical Works, St. Louis.
CHANGES IN EXTRACTORS TO PREVENT EMULSIONS.
H. V. Farr, C. D. Harrington, J. A. Kyger, R. R. Midlam, W. C. Rosenbaum, H. E. Thayer, H. L. Wibbels, and H. Yeager. May 25, 1945. Decl. Sept. 30, 1959. 34p.
OTS.

A laboratory-scale extractor was built and used to study the breaking of emulsions. A random experiment led to the discovery that by pumping ether layer and bottom of extractor simultaneously in the proper ratio (during recycling) the emulsion is broken up. The production extractors were then modified. The experiments and resultant modifications are described. (T.R.H.)

12358 ✓ UCRL-5214
California. Univ., Livermore. Radiation Lab.
STABILITY OF THE INTERFACE BETWEEN TWO IMMISCIBLE LIQUIDS OF UNEQUAL VISCOSITY IN A POROUS SOLID. Ray E. Kidder. May 7, 1958. 7p.
Contract W-7405-eng-48. $0.50(OTS).

The stability of the interface between two immiscible fluids moving at a uniform rate through a porous medium is investigated. The conditions for stability are derived, and the rate of growth of instabilities of speci-

SOLVENT EXTRACTION 6.16

SURVEYS AND THEORY 6.16.06

INTERFACE (AND CONTROL) 6.16.06.04

fied wavelength is obtained. The expression for the rate of growth of instability is similar in form to that obtained by Taylor for the case of uniformly accelerated incompressible liquids, but the growth rate is found to be inversely proportional to the wavelength rather than inversely proportional to the square root of the wavelength. (auth)

A REMOTABLE FLOAT-TYPE LIQUID INTERFACE CONTROLLER. K. J. Hahn and H. M. Jones. Feb. 27, 1958. 9p. (HW-55166)

Float-type interface controllers have performed well and appear to have eliminated some of the problems associated with previously used interface control systems. Present plans are to install float-type instruments in the bottom disengagement section of extraction columns at the Purex Plant.

14700 A/CONF.15/P/832
Oak Ridge National Lab., Tenn.
THE USE OF RADIOISOTOPES FOR THE MEASUREMENT OF INTERFACIAL AREA. J. C. Bresee and C. V. Chester. 12p. $0.50(OTS).

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

A method for direct determination of an interfacial area by measuring the rate of reaction between a radioisotope in one phase and a radiation-sensitive substance in the second phase was developed. It is based on the fact that, if the particle emitted by the radioisotope has a short range, the reaction is restricted to the vicinity of the interface. If the minimum dimensions of the second phase are large compared with the range of the particle, the reaction rate is directly proportional to the interfacial area. To test the radioisotope method experimentally, Po^{210} was the most satisfactory isotope. The experimental system was an aqueous solution of polonium-210 nitrate in contact with a liquid fluorocarbon. The fluorine atoms underwent an α,n reaction at the interface, and boron trifluoride tubes external to the contactor detected neutrons and therefore measured the interfacial area. The proposed method has the advantage of being direct, adaptable to remote operations, and capable of being applied to opaque systems. It has the limitations of being applicable only to two phase systems with extreme immiscibility, requiring materials of construction upon which radioisotope selective deposition or plating will not occur, and imposing stringent requirements on the containment of radioactivity. (auth)

1303
GAMMA DENSITY CONTROLS EXTRACTION COLUMN. Bernard G. Ryle (National Lead Co. of Ohio, Cincinnati). Chem. Eng. Progr. 53, 551-5(1957) Nov.

Gamma absorption density gauging can monitor both the organic extract and the aqueous raffinate and can control column feed rates to maintain normal steady-state operation in a liquid-liquid uranium extraction process. (auth)

12700 HW-46065
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
TECHNICAL INFORMATION FOR APPLICATION OF DIFFERENTIAL PRESSURE TRANSMITTERS IN PULSE COLUMN BOTTOM INTERFACE CONTROL SYSTEMS. A. E. Smith. Oct. 8, 1956. 7p. Contract [W-31-109-Eng-52]. $1.80(ph OTS); $1.80(mf OTS).

The information in this report is based on experience with a similar unit installed on experimental pulse columns in the 321 Building. These installations were made to demonstrate the feasibility of the system. In one case, the installation on the 2A Prototype Column, the differential pressure cell (D/P cell) bottom interface control system was monitored by a capacitance-type instrument. The other installations were made on glass columns and were monitored visually. (auth)

AN INTERFACIAL BARRIER OBSERVED DURING LIQUID-LIQUID EXTRACTION OF URANYL NITRATE. J. B. Lewis (Atomic Energy Research Establishment, Harwell, Berks, England). Nature 178, 274-5(1956) Aug. 4.

4169 ✓ CF-52-5-142
Oak Ridge National Lab., Tenn.
INSTRUMENTATION FOR EXTRACTION COLUMN INTERFACE CONTROL. P. E. Brown. May 16, 1952.

SOLVENT EXTRACTION 6.16

SURVEYS AND THEORY 6.16.06

INTERFACE (AND CONTROL) 6.16.06.04

Decl. Dec. 20, 1955. 12p. Contract [W-7405-eng-26]. $3.30(ph OTS); $2.40(mf OTS).

During a visit to ORNL a group from Chalk River observed an extraction column interface controller. Specific questions concerning the controller are answered, and recommendations and discussion of various factors important to satisfactory column interface control are included. (C.H.)

6355

DISPERSION SEPARATION. (to United Kingdom Atomic Energy Authority). British Patent 825,923. Dec. 23, 1959.

A device is described which will separate immiscible phases in a dispersion, for example furfural and heptane. The dispersion is stirred under pressure in a vessel which has a porous membrane through which one phase flows to another container. The choice of porous membrane and pressure varies with the materials to be separated and the desired degree of separation. An example is given in which fritted glass of pore size 35 to 85 μ gave 100% separation of ether and water at 29.8 mm Hg and a rate of 10 cc/min. (T.R.H.)

1864 CF-47-10-89

Clinton Labs., Oak Ridge, Tenn.

PHASE SEPARATION BY ELECTRICAL CONDUCTIVITY (ELECTRODE DESIGN). Final Report for the Period October 15, 1946–November 18, 1946 on Semi-Works Problem No. TX5-11-11. Clyde D. Watson. [1947]. Decl. Feb. 16, 1957. 11p. $3.30(ph OTS); $2.40(mf OTS).

An electrode was designed for use in the separation of immiscible liquid phases by utilizing the difference in electrical conductivity of the two phases. This was successfully demonstrated, providing an emulsion was not present, for the organic-aqueous phases in the "23" and "25" batch solvent extraction processes. The electrode was constructed of stainless steel piping, packed and insulated with "Teflon" and no failures attributable to the electrode were encountered in test runs or in the actual production of "23" in Cell #4 of the Semi-Works. With the electrode 6" below the discharge side of a ¼" decant tube and at an aqueous decantation rate of 1.64 L/min., the

544 ✓

United Kingdom Atomic Energy Authority. Research Group. Atomic Energy Research Establishment, Harwell, Berks, England.

LIQUID-LIQUID EXTRACTION: A CRITICAL SURVEY OF THE LITERATURE DEALING WITH THE MECHANISM OF TRANSFER OF SOLUTES ACROSS PHASE BOUNDARIES WITH SPECIAL REFERENCE TO LIQUID-LIQUID INTERFACES. J. B. Lewis. Nov. 1950. 21p. (AERE-Ce/R-910).

The literature dealing with the theory of mass-transfer between two liquid phases is critically examined. The analogous cases of mass transfer in solid-liquid and in gas-liquid systems, and of heat transfer are also considered. A bibliography containing 145 references is appended.

ULTRASONICS, USE OF 6.16.07

9256 NYO-7936

Aeroprojects, Inc., West Chester, Penna.

INVESTIGATIONS TO DETERMINE THE FEASIBILITY OF ULTRASONIC LIQUID-LIQUID EXTRACTION. J. L. Straughn and W. B. Tarpley. Feb. 1957. 66p. Contract AT(30-1)-1798. $10.80(ph OTS); $3.90(mf OTS).

Ultrasonic emulsification followed by ultrasonic emulsion breaking as an approach to mixer-settler contactors for liquid-liquid extraction was explored with a number of liquid systems, including $UO_2(NO_3)_2-HNO_3$ and TBP in kerosene, and the laboratory feasibility of an ultrasonic multistage extractor was established. An ultrasonic mixer array was devised, permitting the production of dispersed droplets in the order of 2 μ diameter and demonstrating an extraction efficiency of 85 to 99% at flow rates as high as 100 ml/min with the above systems. Continuous-flow emulsion-breaker studies established the laboratory feasibility of this step, and successful emulsion breaking was achieved at flow rates up to 60 ml/min. Frequencies for

4363 NYO-7784

Aeroprojects, Inc., West Chester, Penna.

INVESTIGATIONS TO DETERMINE THE FEASIBILITY OF ULTRASONIC LIQUID-LIQUID EXTRACTION. Ninth

Progress Report Covering Period From December 1, 1956 to January 31, 1957. Jan. 1957. 11p. Contract AT(30-1)-1798. $1.80(ph OTS); $1.80(mf OTS).

SOLVENT EXTRACTION 6.16

ULTRASONICS, USE OF 6.16.07

A two-stage ultrasonic mixer-settler system was tested in the extraction of a $UO_2(NO_3)_2-Al(NO_3)_3-HNO_3$ feed solution with 6% TBP in kerosene. Over-all extraction efficiency was 85 to 88%, while a corresponding non-ultrasonic control run showed only 48% efficiency. (auth)

1822 NYO-7783
Aeroprojects, Inc., West Chester, Penna.
INVESTIGATION TO DETERMINE THE FEASIBILITY OF ULTRASONIC LIQUID-LIQUID EXTRACTION. Progress Report No. 8 Covering Period from October 1 to November 30, 1956. Nov. 1956. 15p. Contract AT(30-1)-1798. $3.30(ph OTS); $2.40(mf OTS).

The two-stage mixer-settler system resulted in stage extraction efficiencies of approximately 99% under the influence of ultrasonic treatment as compared with 52% for the non-ultrasonic control. Individually, ultrasonic treatment resulted in extraction of 91% of the acid present in Stage 2 and 90.7% of the remainder in Stage 1 as opposed to 33.5 and 27.8% respectively for the control runs. Average approach to equilibrium for the ultrasonic tests was 98% compared with but 48% for the controls. Fabrication was completed on a newly designed standing-wave emulsion-breaking vessel which incorporates an adjustable reflector-to-radiator distance and angle to obtain the correct standing wave relationship. (auth)

185 NYO-7782
Aeroprojects, Inc., West Chester, Penna.
INVESTIGATIONS TO DETERMINE THE FEASIBILITY OF ULTRASONIC LIQUID-LIQUID EXTRACTION. Progress Report No. 7 Covering Period August 1 to September 30, 1956. Oct. 1956. 16p. Contract AT(30-1)-1798. $3.30(ph OTS); $2.40(mf OTS).

Analytical techniques for determination of U content in solutions containing $UO_2(NO_3)_2$ were established, and preliminary experiments in the transfer of $UO_2(NO_3)_2$ established the superiority of ultrasonic over non-ultrasonic extraction in the experimental array. Problems in the design and operation of ultrasonic emulsion-breaking equipment were partially overcome and successful accomplishment of continuous emulsion breaking appears imminent. Assembly of an ultrasonic multistage mixer-settler device was initiated and some of the experimental problems in small scale effective liquid transport from one stage to another were delineated. (For preceding period see NYO-7781.) (auth)

8283 NYO-7780
Aeroprojects, Inc., West Chester, Penna.
INVESTIGATIONS TO DETERMINE THE FEASIBILITY OF ULTRASONIC LIQUID-LIQUID EXTRACTION. Progress Report No. 5 Covering Period from April 1 to May 31, 1956. May 1956. 12p. Contract AT(30-1)-1798. $3.30 (ph OTS); $2.40(mf OTS).

Experiments at 270 kilocycles have established that it is possible by ultrasonic means to induce coalescence of a dispersed phase and breaking of an emulsion in a kerosene-water system even when these emulsions are stabilized with gelatin. (auth)

7571 NYO-7491
Aeroprojects Inc., West Chester, Penna.
INVESTIGATIONS TO DETERMINE THE FEASIBILITY OF ULTRASONIC LIQUID-LIQUID EXTRACTION. Progress Report No. 4 Covering Period from February 1, to March 31, 1956. Apr. 1956. 13p. Contract AT(30-1)-1798. $3.30(ph OTS); $2.40(mf OTS).

As a potentially applicable technique for the problem of transferring the heavy and light phases of a liquid-liquid system to subsequent stages in a multistage contactor design, the feasibility of utilizing the pumping action produced by ultrasonic energy was investigated. Such feasibility was established; water was raised to a height of 42 in. in a 1.5-mm bore glass column and to a height of 11 in. in a column of 4-mm bore. The approximate area of the ultrasonic coupler face and critical distance above the face at which such pumping action can be achieved were preliminarily determined. Subsequent development work with this ultrasonic pumping process will be required to achieve adequate flow rates. The effect on emulsification of phase layer thickness and relative position with regard to the ultrasonic coupler face were determined with a water and methyl-isobutyl ketone system. Good emulsification was obtained with thin water and ketone layers when a sound absorber was inserted at the liquid interface. As a result of this experimental work, a new emulsion vessel was devised for the treatment of thin phase layers; in preliminary tests, this vessel showed superior emulsification. Exploratory work previously carried out furnished much of the background data for the current design of a new emulsion-breaker vessel. This batch-type vessel incorporates a barium titanate transducer element operating in a 6-inch glass tube, which is equipped with an adjustable reflector plate. Preliminary tests at 300 kc and 800 kc were successful, and the wave pattern could be controlled by means of the adjustable reflector plate. (auth)

SOLVENT EXTRACTION 6.16

ULTRASONICS, USE OF 6.16.07

7570 NYO-7490
Aeroprojects, Inc., West Chester, Penna.
INVESTIGATIONS TO DETERMINE THE FEASIBILITY OF ULTRASONIC LIQUID-LIQUID EXTRACTION. Progress Report No. 3 Covering Period from December 1, 1955 to January 31, 1956. Feb. 1956. 17p. Contract AT(30-1)-1798. $3.30(ph OTS); $2.40(mf OTS).

With the initiation of Phase II of the ultrasonic liquid-liquid extraction program, research effort has dealt primarily with emulsifier vessel design and fabrication as a preliminary step toward construction of a multi-stage mixer-settler. Continued search for a suitable extraction system, which will approach as closely as possible the end-use application, has resulted in the testing of the acetic acid–water and methyl–isobutyl ketone system. Experimental work carried out at 25 kc with previously developed emulsification equipment resulted in extraction efficiencies of 91%. New treatment apparatus was designed to promote greater contact between the system phases and to insure uniform ultrasonic treatment of the entire volume of liquid utilized. This equipment was tested with various feeding configurations, and the optimum configuration with ultrasonic treatment produced extraction efficiencies significantly greater than the corresponding non-ultrasonic control system. (auth)

Aeroprojects, Inc., West Chester, Penna.
INVESTIGATIONS TO DETERMINE THE FEASIBILITY OF ULTRASONIC LIQUID-LIQUID EXTRACTION. Interim Report No. 1 Covering Period from June 1 to November 30, 1955. Dec. 1955. 59p. Contract AT(30-1)-1798. $9.30(ph OTS); $3.60(mf OTS).

Preliminary experiments established the feasibility of ultrasonic liquid-liquid extraction, based on the concept of ultrasonic emulsification of the immiscible phases in a cavitation field, followed by ultrasonic emulsion breaking in a standing-wave field. Operation of continuous-flow ultrasonic emulsification equipment at a flow rate of 60 ml/min successfully dispersed immiscible phases in the aqueous nitric acid-tributyl phosphate-kerosene system and the kerosene-acetic acid-water system, producing a droplet size of the dispersed phase of less than 2μ in diameter and effecting 90 ± 10% extraction efficiency with one pass through the ultrasonic field. The emulsion spontaneously separated into dilute oil-in-water and water-in-oil emulsions. Initial studies with batch-type emulsion-breaking equipment were moderately effective in breaking the dilute oil-in-water emulsion. The results indicate the possibility of utilizing ultrasonic emulsification equipment in the development of a multistage extraction system smaller than conventional systems, with no mechanically moving parts, with only readily expendable components exposed to a radioactive field, and incorporating emulsion-breaking equipment only at each end of the contactor system. (auth)

(SPECIAL) PROCESSES 6.17

See also, under

Fissionable Material - Recovery - Dissolation (General) 3.01.04,

and, under
Plants and Equipment - Materials (Construction) 5.14.

7514 CF-56-8-100
Oak Ridge National Lab., Tenn.
FEED MATERIALS PROCESSING STATUS REPORT FOR AUGUST 1956. R. E. Blanco and W. K. Eister. Aug. 15, 1956. Decl. Feb. 20, 1957. 18p. Contract [W-7405-eng-26]. $0.30(OTS).

Metallex Process. Preliminary experiments indicated that aqueous washing of the Th amalgam may be eliminated by dissolution of the $ThHg_x$ in the Hg phase and filtering while hot to separate it from undissolved impurities. Further work with antioxidants in an aqueous wash solution showed that they consistently lower the quality of the metal. X-ray-diffraction studies of Th amalgam showed that the species of $ThHg_x$ produced are dependent on time and temperature, but not on washing technics. Installation of the interim facility for larger-scale production of Th quasi amalgam by the dry salt flowsheet was completed.
Fluorox Process. The rate of oxidation of UF_4 by air

(SPECIAL) PROCESSES 6.17

increased rapidly as the temperature was increased from 700 to 800°C. In preliminary oxidation runs in the lined moving-bed reactor, in which graphite pellets were used as the source of the heat, the UF_4 pellets fused. An unlined fluidized bed was designed in which temperature control should be more precise. Excer Process. The high decontamination factors obtained in electrolysis of a solution of crude ore concentrate produced by solvent extraction indicated that in some cases the ion-exchange step may be omitted. (auth)

7513 ✓ CF-56-7-101
Oak Ridge National Lab., Tenn.
POWER REACTOR FUEL PROCESSING STATUS REPORT FOR JULY 1956. R. E. Blanco, W. K. Eister, and D. E. Ferguson. July 15, 1956. Decl. Feb. 14, 1957. 23p. Contract [W-7405-eng-26]. $0.30(OTS).

Fused Salt — Fluoride Volatility Process. Information on the corrosion of Ni, rate of dissolution of Zr–U alloy in fused $NaF-ZrF_4$, fluoridation of molten $NaF-ZrF_4-UF_4$, and decomposition of $UF_6 \cdot 3NaF$ is reported. Zircex Process. Data on hydrochlorination rates, rare earth separations, fuel element core leaching, heat transfer problems, and sublimed $ZrCl_4$ are included. Hermex Process. Dissolution of U in Mg amalgam and U in Bi amalgam are reported. General Dissolution Studies. Dissolution studies of Zircaloy-2 in aqueous $FeCl_3$ and $FeCl_3-NaCl$ and HCl were made. Cladding removal is included. Mechanical Dejacketing and Handling Studies. A mechanical decladder and a leaching-dissolving device are being investigated. Materials Evaluation. Results of tests of materials for resistance to HNO_3 are reported. (T.R.H.)

7512 ✓ CF-56-7-100
Oak Ridge National Lab., Tenn.
FEED MATERIALS PROCESSING STATUS REPORT FOR JULY 1956. R. E. Blanco and W. K. Eister. July 15, 1956. Decl. Feb. 14, 1957. 19p. Contract [W-7405-eng-26]. $0.30(OTS).

Metallex Process. The effect of a small addition of Mg to Th quasi-amalgam before and after reduction is reported along with the relation between free voidage of the product Th and its quality. Fluorox Process. The results of studies on C removal from UF_4 with H_2 and on surface areas of UO_2F_2 and UF_4 and on the isolation of an unknown compound are given. New equipment and modifications are reported. Excer Process. The application of the process to ore processing is being investigated and the results of some experiments and tests are reported. (T.R.H.)

AMERICIUM RECOVERY 6.17.01

Solvent extraction from metallurgical (spent fuel) waste in HNO_3 solution from which Pu has been extracted. HNO_3 is neutralized, Am is extracted with TBP and purified by ion exchange.

See also, this section,

SOLVENTS--TBP - General (6.16.05.08),

and,

under FISSION PRODUCT, POISON, AND RADIOISOTOPE REMOVAL,

TRANSURANICS--Americium (2.54.01).

Neptunium (2.54.05).

14692 A/CONF.15/P/537
Oak Ridge National Lab., Tenn.
AMERICIUM AND NEPTUNIUM RECOVERY PROCESSES. W. H. Lewis. 13p. $0.50(OTS).

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

Solvent extraction processes were developed on a pilot plant scale for recovering neptunium-237 and americium-241 from irradiated uranium and fuel cycle wastes. Neptunium was recovered from irradiated normal uranium, irradiated enriched uranium alloyed with aluminum, and fluorination ash produced from the conversion of recovered UO_3 to UF_6. Americium was separated from a fuel cycle waste containing large quantities of lanthanum and cerium. Final purification of both americium and neptunium from solvent extraction processes was achieved by ion exchange. A detailed description of these processes is presented. (J.R.D.)

BISMUTH PHOSPHATE 6.17.02

See also, this section,

PRECIPITATION (6.14),

and,

under FISSIONABLE MATERIAL--RECOVERY,

(SPECIAL) PROCESSES 6.17

BISMUTH PHOSPHATE 6.17.02

RECOVERY OF IRRADIATED TH AND U--Precipitation--Phosphate (3.02.15.07),

RECOVERY OF PLUTONIUM--Precipitation--Phosphate (3.03.12.05).

22167

IMPROVED PROCESS OF PLUTONIUM CARRIER PRECIPITATION. B. F. Faris (to U. S. Atomic Energy Commission). U. S. Patent 2,892,676. June 30, 1959.

This patent relates to an improvement in the bismuth phosphate process for separating and recovering plutonium from neutron irradiated uranium, resulting in improved decontamination even without the use of scavenging precipitates in the by-product precipitation step and subsequently more complete recovery of the plutonium in the product precipitation step. This improvement is achieved by addition of fluomolybdic acid, or a water soluble fluomolybdate, such as the ammonium, sodium, or potassium salt thereof, to the aqueous nitric acid solution containing tetravalent plutonium ions and contaminating fission products, so as to establish a fluomolybdate ion concentration of about 0.05 M. The solution is then treated to form the bismuth phosphate plutonium carrying precipitate.

13381

BISMUTH PHOSPHATE CARRIER PROCESS FOR Pu RECOVERY. T. G. Finzel (to U. S. Atomic Energy Commission). U. S. Patent 2,872,286. Feb. 3, 1959.

An improvement in the bismuth phosphate carrier precipitation process for recovering plutonium is described. It has been found that a more granular and more easily filterable carrier precipitate is formed if the addition of the bismuth and phosphate ions is effected by first adding 9/10 of the bismuth ions necessary, then slowly adding all of the source of the phosphate ions to be incorporated in the precipitate, while digesting at 75°C and afterwards incorporating the remainder of the total bismuth ions necessary.

BISMUTH PHOSPHATE PROCESS FOR THE SEPARATION OF PLUTONIUM FROM AQUEOUS SOLUTIONS. S. G. Thompson and G. T. Seaborg (to U. S. Atomic Energy Commission). U. S. Patent 2,785,951. March 19, 1957.

A method is given for separating Pu from fission products arising with it in the irradiated U. The method makes use of the fact that Pu^{6+} forms a soluble phosphate while lower valent plutonium forms an insoluble phosphate. The irradiated U is dissolved, Bi ion is added to serve as a carrier, and while the Pu is being maintained in its lower valent state, PO_4 is added, causing the precipitation of the Pu and any fission products which form insoluble phosphate. This precipitate is filtered out and then redissolved in an aqueous acid solution. The Pu is then oxidized to the higher valent state causing it to remain in solution when phosphate ion is added to precipitate out the phosphate insoluble fission products which accompanied the plutonium in the first precipitation. (auth)

12537 HW-48141

General Electric Co. Hanford Atomic Products Operation, Richland, Wash.

THE REMOVAL OF CESIUM-137 AND STRONTIUM-90 FROM SCAVENGED BISMUTH PHOSPHATE SOLUTIONS. J. R. McHenry. Feb. 7, 1957. 16p. Contract W-31-109-Eng-52. $3.30(ph), $2.40(mf) OTS.

The passage of a scavenged first-cycle waste solution through a soil representative of the Hanford project reduced the Sr^{90} content of at least five column volumes of effluent solution below the detection limits for the analytical procedures employed. This reduction was effected equally well in synthetic and process bismuth phosphate waste solutions. The removal of Cs^{137} from a first-cycle waste solution, synthetic or process, by passage through a column of a representative Hanford project soil was less efficient than for Sr^{90}. A breakthrough of Cs^{137} was obtained generally within one to four column volumes of effluent. The concentration of Cs^{137} in the influent solutions usually was of the same order of magnitude as the Handbook 52 MPC for drinking water. Consequently the relatively high C/C_0 values for Cs^{137} in the effluents represented concentrations of less than one-tenth the MPC value. The volume of scavenged first cycle waste solution which may be disposed safely to ground is dependent on the Cs^{137} content of the waste in question. Three, and perhaps five, column volumes

(SPECIAL) PROCESSES 6.17

BISMUTH PHOSPHATE 6.17.02

of the scavenged bismuth phosphate waste solution can be disposed to ground. To date all available field data corroborate laboratory estimates of the removal of Cs^{137} and Sr^{90} from scavenged bismuth phosphate waste solutions by soil. (auth)

187 TID-10142
Hanford Atomic Products Operation, Richland, Wash.
DECONTAMINATION OF BISMUTH PHOSPHATE PROCESS FIRST CYCLE WASTE SUPERNATANTS. R. E. Burns, H. S. Gile, and C. R. McMullen. Sept. 10, 1953. Decl. Oct. 2, 1956. 17p. Contract [W-31-109-Eng-52]. $0.30 (OTS).

First decontamination cycle wastes from the Hanford Atomic Products Operations Bismuth Phosphate Process are made alkaline and stored in large underground tanks. Settling of solids produced on making the wastes alkaline effects considerable decontamination but not enough that very large volumes of the supernatants can be discharged to ground. The effectiveness of tannic acid, Ca_2PO_4, CaC_2O_4, $Fe_4[FeCN_6]_3$, and Fe_2FeCN_6 as carrier precipitates, and CaC_2O_4, Attaclay, and Attapulgus clay as solid adsorbents for the decontamination of supernatants from aged first cycle wastes has been studied. Plutonium, fission-product, and gross salt content of supernatants from several waste tanks are given along with decontamination factors for Pu and fission products obtained when these supernatants were treated under varying conditions with the adsorbents and carrier precipitates noted above. (auth)

9622 CN-3274
Hanford Engineer Works, Richland, Wash.
SUMMARY OF RESULTS OF INVESTIGATIONS ON THE INFLUENCE OF HYDRAZINE IN THE BISMUTH PHOSPHATE EXTRACTION STEP. D. R. Miller. June 11, 1945. Decl. Feb. 16, 1957. 5p. (SE-PC-45). $1.80(ph OTS); $1.80(mf OTS).

This report reissued under auspices of Contract W-7405-eng-37.

Results of an investigation on the influence of hydrazine in the bismuth phosphate extraction step are presented. (W.L.H.)

10764 CN-828
[Chicago. Univ.] Metallurgical Lab.
TECHNOLOGY—CHEMICAL ENGINEERING; REPORT FOR MONTH ENDING JULY 26, 1943. Decl. Jan. 18, 1956. 15p. Contract [W-7401-eng-37]. $3.30(ph OTS); $2.40(mf OTS).

Laboratory-scale studies on the Bismuth Phosphate Process were continued with emphasis on the dissolution and extraction steps. Steam jets were used to transfer process solutions and slurries. (C.W.H.)

BUTEX 6.17.03

See also, this section,

SOLVENTS--Dibutyl Carbitol (6.16.05.02),

and,

under FISSIONABLE MATERIAL--RECOVERY,

RECOVERY OF PLUTONIUM--Solvent Extraction (3.03.14).

THE DEVELOPMENT OF THE BUTEX PROCESS FOR THE INDUSTRIAL SEPARATION OF PLUTONIUM FROM NUCLEAR REACTOR FUELS. C. M. Nicholls (Atomic Energy Research Establishment, Harwell, Berks, Eng.). J. Brit. Nuclear Energy Conf. 3, 279-89(1958) Oct.

The chemical engineering work done in Canada in developing the Butex Process is described. (auth)

7395 PGR-71(W)
United Kingdom Atomic Energy Authority. Production Group. Chemical Services Dept., Windscale, Sellafield, England.
ANALYTICAL METHOD FOR THE INSPECTION OF BUTEX (BIS BB, BUTOXY ETHYL ETHER). 1959. 25p. BIS.

The acetaldehyde equivalent, acidity, chlorine, and hydroxy- and unsaturated compounds are determined titrimetrically, and the boron and total peroxides absorptiometrically. Water, distillation range, specific gravity, viscosity and sulfated residue are determined by conventional techniques. (auth)

(SPECIAL) PROCESSES 6.17

DAREX 6.17.04

A head-end treatment for processing stainless-steel containing fuels: dissolution in $HCl-HNO_3$, removal of chloride by distillation, solvent extraction recovery of uranium.

See also, this section,

DISTILLATION AND CONDENSATION (6.06),

SOLVENTS--TBP - General (6.16.05.08),

and,

under FISSIONABLE MATERIAL--RECOVERY,

DISSOLUTION (GENERAL) (3.01.04),

STAINLESS STEEL FUEL ELEMENTS (3.01.09),

SOLVENT EXTRACTION--Nitrate Systems (3.02.17.04).

6319 CF-59-11-26
Oak Ridge National Lab., Tenn.
TERMINAL REPORT—DESIGN OF A DAREX DISSOLUTION FACILITY IN ORNL BLDG. 3019 FOR ENRICHED POWER REACTOR FUEL ELEMENTS. Roy C. Robertson. Nov. 30, 1959. 101p. OTS.

The design drawings and specifications for a Darex pilot plant to be constructed at ORNL were about 75% complete when the project was terminated in September 1959. As a feed preparation step, this plant was designed to dissolve enriched, stainless-steel-clad, power reactor fuel elements in dilute aqua regia. The pilot plant design included a dissolver column, evaporator, clarifier, scrubbers, storage vessels, and condensers, for a total of about 31 major pieces of equipment, of which about 16 were to be constructed of titanium and most of the remainder of Type 304L stainless steel. These were arranged into nine units inside the cell and in three major service areas outside the cell. The unitized concept for arrangement of the cell equipment reduced the required number of high-grade field welds significantly. About 50 drawings were essentially completed and five specifications were written. This terminal report emphasizes the phases of the mechanical design having general application to other Darex facilities. (auth)

19978 AECU-4304
Georgia Inst. of Tech., Atlanta. Engineering Experiment Station.
A STUDY OF EQUILIBRIA INVOLVED BETWEEN DAREX OFF-GASES AND SOLUTIONS. Annual Report No. 1 [for] December 1, 1958 through July 31, 1959. Clyde Orr, Jr., Margaret C. Kordecki, J. K. Gladden, and Clyde T. Young. 39p. Project No. A-427. For Oak Ridge National Lab. Contract W-7405-eng-26, Subcontract No. 1373. $6.30(ph), $3.00(mf) OTS.

In the Darex process, aqua regia is used to dissolve the materials of spent reactor fuel elements. For economic reasons, it is desirable to recover the off-gases from the dissolving process, especially the chlorine, in acid solutions that can be recycled in the system. This project was undertaken to develop a method of analyzing the off-gases and to determine their solubility in various concentrations of hydrochloric acid, nitric acid, and aqua regia at several temperatures. Work was concentrated on the development of a precise method for the quantitative analysis of gaseous mixtures containing some or all of the following gases:

NO, N_2O, $NO_2(N_2O_4)$, NOCl, HCl, Cl_2, and water vapor. The initial emphasis was placed on gas chromatography, but no combination of columns and conditions was found by which accurate results for all these gases could be obtained. Analyses by spectrophotometric methods appeared at first to be unsatisfactory because of the masking by NO_2 and Cl_2 in the visible and the ultraviolet range, the absorption by water in the infrared region, and the absence of absorption by Cl_2 in the infrared region. Experiments have now shown, however, that water vapor does not entirely mask the other components. A complete analysis can be accomplished if, after the gaseous mixture has been scanned with an infrared spectrophotometer, the separation of chlorine is effected by dissolving the mixture in CCl_4 and shaking it with water. Only the chlorine remains in the CCl_4 layer and can be analyzed spectrophotometrically in the ultraviolet or visible region. Preliminary experiments of this type have shown how the gases from aqua regia at atmospheric pressure vary with time. Samples taken 20 minutes after mixing showed the presence of HCl in the gas phase and gave an indication of some water

(SPECIAL) PROCESSES 6.17

DAREX 6.17.04

vapor. Samples taken after 70, 120, and 205 minutes did not reveal HCl but gave progressively larger NOCl peaks and showed a yellow color, indicating the presence of chlorine. After 48 hours the sample peaks were essentially the same as those from the 205-minute sample. (auth)

19979 AECU-4319
Georgia Inst. of Tech., Atlanta. Engineering Experiment Station.
A STUDY OF EQUILIBRIA INVOLVED BETWEEN DAREX OFF-GASES AND SOLUTIONS. A LITERATURE SURVEY ON THE SOLUBILITY OF ARGON, CHLORINE, HYDROGEN CHLORIDE, NITRIC OXIDE, NITROGEN, NITROGEN DIOXIDE, NITROUS OXIDE AND OXYGEN IN WATER, NITRIC ACID, HYDROCHLORIC ACID AND AQUA REGIA. Special Report No. 1. Thomas W. Wilson. June 1, 1959. 107p. For Oak Ridge National Lab. Contract W-7405-eng-26, Subcontract 1973. $16.80(ph), $5.70(mf) OTS.

A literature survey on the solubilities of Ar, Cl, H, chlorides, nitrogen oxides, N, and O in various liquids such as H_2O, HNO_3, aqueous HCl, and aqua regia was conducted. The findings are tabulated. References reviewed include Applied Science and Technology Index, 1958, Chemical Abstracts, 1937 to Feb. 1959, Engineering Index, 1958, and The Industrial Arts Index, 1955 to 1957. 117 references. (J.R.D.)

257 IDO-14483
Phillips Petroleum Co. Atomic Energy Div., Idaho Falls, Idaho.
EFFECT OF DISSOLVED STAINLESS STEEL COMPONENTS ON VAPOR-LIQUID EQUILIBRIA IN AQUA-REGIA. Bernice E. Paige. July 1, 1959. 20p. Contract AT(10-1)-205. OTS.

Vapor-liquid equilibrium values were determined for chloride and nitric acid in nitric acid-hydrochloric acid-ferric nitrate-water systems. It was found that chloride vapor-liquid equilibrium values increased with increasing hydrogen ion concentration, but were more than an order of magnitude lower in the presence of 1M ferric ion for solutions of greater than 6M total acid. Nitric acid vapor-liquid equilibrium values increased with increasing hydrogen ion and were further increased by the addition of ferric nitrate. A study was also made of the decomposition gases which escaped from the equilibrium still during the first 1½ hour period of operation. (auth)

11054 ✓ CF-57-7-32
Oak Ridge National Lab., Tenn.
PRELIMINARY AQUA-REGIA DISSOLUTION STUDIES. K. J. Fritz. July 10, 1957. 19p. Contract [W-7405-eng-26]. $3.30(ph OTS); $2.40(mf OTS).

Batch and continuous aqua-regia dissolution of 304 and 304-L stainless steels and APPR plate were studied. Dissolution techniques are described and preliminary results are presented. A short survey of the known chemistry of aqua-regia is also presented. (auth)

RECOVERY OF ENRICHED URANIUM FROM URANIUM DIOXIDE-STAINLESS STEEL FUEL ELEMENTS BY SOLVENT EXTRACTION. J. R. Flanary and J. H. Goode (Oak Ridge National Lab., Tenn.). Ind. Eng. Chem. 51, 23-6(1959) Jan.

Laboratory studies of the batch Darex dissolution process, as applied to prototype APPR fuel, followed by continuous solvent extraction have yielded chemical flowsheets which provide excellent uranium recovery and decontamination from fission products and inert impurities. The advantages of the process include rapid dissolution of stainless steel-containing fuels, hydrogen-free off-gas, and relatively high throughput. The principal disadvantages are corrosion and the carry-through of inert metals with limited solubility to the radioactive waste storage system. (auth)

18658 ✓ ORNL-2753
Oak Ridge National Lab., Tenn.
DAREX PROCESSING OF APPR FUEL: EFFECT OF ACIDITY AND GAS SPARGING ON RATE OF CHLORIDE REMOVAL FROM DISSOLVER PRODUCT DURING REFLUXING. B. C. Finney and F. G. Kitts. Aug. 7, 1959. 15p. Contract W-7405-eng-26. $0.50(OTS).

The rate of chloride removal varied directly with HNO_3 concentration in an APPR-type Darex dissolver product containing 100 g/liter metal loading, 0.58 M initial chloride, and initial HNO_3 concentrations of 8, 9 10,

DAREX 6.17.04

12, and 14 M. The removal rate with 8 and 9 M HNO_3 was very low. After 6 hr refluxing, the chloride content decreased to 0.50 and 0.36 M, respectively. After refluxing for the same time with 10 to 14 M HNO_3, the product contained 0.064 to 0.0007 M (2270 to 25 ppm) chloride. The effect of air sparging was approximately equivalent to refluxing without sparging at a HNO_3 concentration 2 M higher. After 6 hr sparging and refluxing the chloride content varied from 0.034 to < 0.00014 M (1200 to < 5 ppm) for initial HNO_3 concentrations from 8 to 14 M. (auth)

9689 HW-59033
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
REMOVAL OF CHLORIDE ION FROM DAREX PROCESS DISSOLVER SOLUTION. J. E. Mendel and W. W. Schulz. Jan. 12, 1959. 22p. Contract W-31-109-Eng-52. $0.75(OTS).

The chloride ion concentration of Darex Process dissolver solutions can be reduced readily to 0.1 g/l or less by simple batch boildown with addition of nitric acid. Efficiency of chloride removal is improved by preliminary concentration of the Darex solution: twofold concentration appears optimum. Utilization of nitric acid during boildown is improved at low boiloff rates but longer time cycles are required for adequate chloride removal. Boildown efficiency was not significantly improved by air sparging. Solvent extraction of Darex dissolver solutions with TBP-diluent solutions is a chemically feasible alternative to the batch boildown procedure for chloride separation and removal. With 30 per cent TBP-hydrocarbon diluent as extractant, chloride distribution ratios were in the range 0.05 to 0.1. Dual scrubbing—six scrubs with 3 M HNO_3, 0.07 M UNH and three scrubs with water at an organic/aqueous volume ratio of 5/1—reduced the chloride concentration of organic phases derived from Darex solutions 0.19 to 1.9 M in HCl to 0.005 g/l. (auth)

10470 CF-58-2-135
Oak Ridge National Lab., Tenn.
DAREX PROCESS: BATCH DISSOLUTION AND CHLORIDE REMOVAL STEPS FOR YANKEE ATOMIC FUEL. J. H. Goode and J. R. Flanary. Feb. 21, 1958. 17p. Contract [W-7405-eng-26]. $3.30(ph OTS); $2.40(mf OTS).

In laboratory scale experiments dilution of a Darex dissolver product from the integral dissolution of Yankee Atomic fuel specimens with at least an equal volume of 15.8M HNO_3, followed by distillation, produced a solvent extraction feed containing less than 50 ppm of chloride. Properly designed chloride stripping and nitric acid rectification columns are capable of conserving acids for recycle to the dissolver and chloride removal still. In initial experiments about 87% of the nitric acid and 36% of the hydrochloric acid were recovered. (auth)

11168 CF-59-1-137
Oak Ridge National Lab., Tenn.
REVIEW OF THE MAXIMUM ALLOWABLE CHLORIDE CONCENTRATION IN THE PROPOSED DAREX PILOT PLANT PRODUCT. J. M. Holmes. Jan. 15, 1959. 7p. $1.80(ph), $1.80(mf) OTS.

A review of data on the effects of chlorides in nitric acid solutions upon stainless steel indicates that the proposed maximum chloride specification of 350 ppm chloride in the Darex Pilot Plant product is reasonable. (auth)

10980 HW-58600
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
PRELIMINARY DAREX FLOWSHEET. J. J. Shefcik and A. M. Platt. Dec. 18, 1958. 8p. Contract W-31-109-Eng-52. $1.80(ph), $1.80(mf) OTS.

The preliminary Darex flowsheet for a Yankee fuel provides a two-step process for the total dissolution of stainless steel clad uranium fuels in a mixture of hydrochloric and nitric acids (aqua regia) and the removal of excess chloride from the dissolver solution by a batch volatilization procedure. Four major pieces of equipment are indicated, a dissolver-evaporator, a down draft condenser, a condensate receiver, and a caustic scrubber. The first three pieces of equipment are exposed to aqua regia and must be constructed of a corrosion resistant material such as titanium. Metallic copper is added (in contact with the fuels) as an activator to prevent passivation effects. Under these conditions, the dissolution of cladding proceeds smoothly. (auth)

(SPECIAL) PROCESSES 6.17

DAREX 6.17.04

8783 ✓ CF-58-11-82
Oak Ridge National Lab., Tenn.
THE DAREX PROCESS: PROGRESS SINCE THE 1958 GENEVA CONFERENCE. F. G. Kitts, B. C. Finney, and J. H. Goode. Nov. 28, 1958. 24p. Contract W-7405-eng-26. $4.80(ph), $2.70(mf) OTS.

For presentation at the American Chemical Society's Nuclear Technology Symposium, Boston, April 5-10, 1959.

Darex Process is a head-end process for stainless steel-containing fuels consisting basically of three steps: (1) dissolution in dilute nitric–hydrochloric acid, (2) chloride removal, and (3) solvent extraction feed adjustment. The process may be carried out continuously, semi-continuously, or batchwise using nitric acid concentrations from 61 to 95 wt. %. Apparatus for continuous operation including instrumentation for the controlled recycle of concentrated nitric acid was assembled and operated to process APPR fuel elements. Reflux flowsheets were developed on a 1 to 2 liter scale for both batchwise and semi-continuous operation using either 95 or 61 wt. % nitric acid to volatilize and/or decompose chloride. These four flowsheets, employing a period of refluxing with high nitric acid concentrations, accomplished chloride removal to less than 350 ppm by the batchwise addition of only the amount of nitric acid consumed per batch. A batchwise Recycle flowsheet was also developed which uses an excess of 61% nitric acid, added continuously, with a 61% nitric acid recycle in addition to the aqua regia recycled to the dissolution step. A 6-in. ID continuous dissolver 9 ft high and a 4 in. ID chloride removal and feed adjustment tank were constructed and operated to verify flowsheet and equipment performance. (auth)

2832 ✓ CF-58-11-106
Oak Ridge National Lab., Tenn.
DEVELOPMENT OF BATCHWISE AND SEMI-CONTINUOUS DAREX FLOWSHEETS USING 61 WT PER CENT HNO_3. F. G. Kitts and B. C. Finney. Nov. 19, 1958. 8p. Contract W-7405-eng-26. $1.80(ph OTS); $1.80 (mf OTS).

Both batch-wise and semi-continuous Darex flowsheets were developed on a 1.4-liter scale using 61 wt. % nitric acid to volatilize and/or decompose chloride. The 61% acid is favored over higher concentrations for safety and availability. The batch flowsheet features simplicity and ease of operation; either batch or continuous dissolution can be used. The semi-continuous scheme, which assumes continuous dissolution, offers reduced cycle times but more complex operation. Both flowsheets accomplish chloride removal to <350 ppm by adding batch-wise only the amount of nitric acid required for solvent extraction feed adjustment and regeneration of dissolvent. (auth)

3677 CF-58-9-83
Oak Ridge National Lab., Tenn.
CHLORINE REMOVAL FROM THE DAREX OFF-GAS. T. A. Gens. Sept. 17, 1958. 10p. Contract [W-7405-eng-26]. $1.80(ph OTS); $1.80(mf OTS).

A 2 M NaOH solution is a satisfactory scrubber for the Darex off-gas if sufficient nitrite is present to reduce all hypochlorite to chloride. If there is not enough nitrite produced in the scrubber solution by the reaction of nitrogen dioxide and nitrogen sesquioxide with sodium hydroxide, more reducing agent must be added. Both nitrite and sulfite rapidly reduce hypochlorite in basic solution. The scrubber solution can be used as long as it remains basic. (auth)

4811 BMI-1242
Battelle Memorial Inst., Columbus, Ohio.
EVALUATION OF CONTAINER MATERIALS FOR ZIRCEX AND DAREX NUCLEAR-FUEL-RECOVERY PROCESSES. Paul D. Miller, Charles L. Peterson, Earl L. White, and Frederick W. Fink. Dec. 11, 1957. 44p. Contract W-7405-eng-92. $1.25(OTS).

The selection of a suitable material for the processing equipment is most important in fuel-recovery processes. The problem arises because of the aggressive nature of the reagents that must be used for disintegrating or dissolving some spent fuel elements. In this investigation, corrosion tests were made under conditions simulating those that may occur in several proposed processes in order that candidate materials of construction could be evaluated. No material has been found which would allow both the hydrochlorination and dissolution steps of the vapor-phase Zircex process to be carried out in a single vessel. For a two-vessel process, several materials, such as Inconel, Haynes 25, Type S-816 alloy, Illium R, and

DAREX 6.17.04

the Hastelloys B and C, appear promising for construction of a hydrochlorinator; the dry residue would then be transferred to another vessel, perhaps of titanium or tantalum, for dissolution. In the alternate liquid-phase Zircex process, preliminary tests have given promising results with nickel and nickel-chromium alloys. However, the only metals studied, which withstand the corrosive boiling $AlCl_3 \cdot NH_4Cl$ plus gaseous HCl environment with but negligible weight losses have been platinum, gold, tungsten, and molybdenum. A variety of laboratory studies have shown that titanium will apparently withstand any condition likely to occur in the critical areas of the Darex process, including the dissolver and chloride-stripping column. It will undergo a severe attack in HCl vapor, but this is easily prevented by the presence of a small amount of nitric acid vapor or oxides of nitrogen. Tantalum is another promising material disclosed by these tests, but its use in the dissolver seems negated because embrittlement was detected under certain conditions. No signs of cracking or other attack have been observed on Type 347 stainless steel specimens exposed at room temperature with various levels of stress for over 6 months to Purex-type acid extraction systems containing chlorides far in excess of the concentrations expected during operation of either the Zircex or Darex head-end processes. (auth)

4554 CF-57-11-77
Oak Ridge National Lab., Tenn.
DAREX FLOWSHEET (BATCH OPERATIONS) PARTS V AND VI, SOLVENT EXTRACTION. J. R. Flanary and J. H. Goode. Nov. 5, 1957. Decl. Dec. 10, 1958. 6p. Contract W-7405-eng-26. $1.80(ph OTS); $1.80(mf OTS).

Parts V and VI, two cycles of solvent extraction with an intercycle evaporation, of the Darex flowsheet are presented. Part V, the first cycle, was tested with both unirradiated material and with irradiated specimens at activity levels up to approximately 10% of that expected for an actual process. Decontamination factors were obtained using the first cycle solvent extraction flowsheet in mini-mixer-settlers. Part VI represents an extension of the second solvent extraction cycle of the 25-TBP Process, as applied to enriched uranium–stainless steel fuels. Since the product of the first cycle is free of stainless steel contaminants, application of the second cycle of a fully-developed process was considered adequate. (auth)

A CONTINUOUS DAREX PROCESS FLOWSHEET. F. G. Kitts. Oct. 10, 1957. 15p. (CF-57-10-89)

A Darex flowsheet is presented wherein dissolution of stainless steel-type fuel elements in dilute aqua regia, stripping of chloride from spent dissolver solution, adjustment of stripper product composition and rectification of HNO_3-H_2O vapors are accomplished in a completely continuous relatively simple loop process. Product from the loop needs only dilution with water to be made suitable as feed for a solvent extraction processing plant. The proposed scheme differs from earlier concepts. A continuous, pot dissolver (well-mixed liquid phase) is utilized rather than a trickle type dissolver. A strip vapor slightly lower in HNO_3 concentration than the azeotrope is used. A feed adjustment tank serves not only to recover HNO_3-H_2O vapor, but also to dehydrate any silica present and to adjust the product composition such that upon water addition the material is ready for feed to solvent extraction. In most previous concepts, separate, batchwise feed adjustment was contemplated.

2831 CF-57-9-73
Oak Ridge National Lab., Tenn.
DAREX FLOWSHEET (BATCH OPERATIONS).
Walter E. Clark. Sept. 18, 1957. Decl. Nov. 28, 1958. 10p. Contract [W-7405-eng-26]. $3.30(ph OTS); $2.40 (mf OTS).

A complete Darex flowsheet is presented which represents an interim flowsheet since continuous operation will prove to be advantageous. (auth)

12365 CF-56-8-167
Oak Ridge National Lab., Tenn.
DAREX: HEP DESIGN AND RECOMMENDATIONS. C. E. Guthrie. Aug. 23, 1956. Decl. Mar. 19, 1957. 10p. Contract [W-7405-eng-26]. $3.30(ph OTS); $2.40(mf OTS).

The dilute aqua regia (Darex) system for processing stainless steel fuel elements consists of a continuous dissolution and chloride distillation system followed by a batch feed adjustment. The preliminary design is presented along with an outline of the development work required before a final design is possible. (auth)

(SPECIAL) PROCESSES 6.17

DEBOER 6.17.05

U alloy fuel elements are decontaminated from all long--lived fission products except Zr by formation of UI_4, volatilization of the iodide followed by its decomposition with deposition of U on a hot wire.

U is separated from Zr by selective condensation of the UI_4.

See also, this section,
DISTILLATION AND CONDENSATION (6.06),
and,
under FISSIONABLE MATERIAL--RECOVERY,
ELECTROCHEMICAL (3.02.08).

2825 KAPL-1788
Knolls Atomic Power Lab., Schenectady, N. Y.
REPROCESSING URANIUM BY THE DEBOER VOLATILE IODIDE PROCESS. W. L. Robb. May 1, 1957. 25p. Contract W-31-109-Eng-52. $0.75(OTS).

Experiments were conducted to demonstrate the use of the deBoer volatile iodide process as a method for decontaminating U metal or U alloy fuel elements. Decontamination from all long-lived radioactive fission products except Zr was obtained by forming UI_4, volatilizing the iodide, and then decomposing it to deposit U metal on a hot wire. By selective condensation of the UI_4, separation of U and Zr was produced. The ability to codeposit U and Zr as might be desired in reprocessing U–Zr fuel elements, was demonstrated. The deposition of molten U metal on a heated ZrC cylinder and the continuous removal of the U from the cylinder were also investigated. A design of a processing plant is described, and the problems inherent to its construction and operation are discussed. (auth)

Battelle Memorial Inst., Columbus, Ohio.
THE TRANSFER OF IMPURITIES IN ZIRCONIUM PREPARED BY THE DE BOER PROCESS. H. H. Bulkowski, L. C. Beale, J. J. Sebenick, I. E. Campbell, and B. W. Gonser. Mar. 6, 1951. Decl. Nov. 15, 1955. 15p. Contract AT(30-1)-771.

The transfer of Fe, Ti, Al, N, C, Si, Ni, and Cr from the feed stock to the crystal bar by the de Boer process was studied. The feed materials were prepared by making binary alloys of these elements with iodide-deposited Zr from the de Boer bulb. High-carbon sponge Zr and high-temperature vacuum-sintered sponge Zr also were used in the experiments described. Noncorrodible Zr also was used as feed stock to determine the effect of redeposition on the purity and corrodibility of the product. Several runs produced Zr of good corrosion resistance, and the purity of the product was essentially the same as that of the starting material. Experiments showed that the rate of deposition of Zr and the transfer of impurities were not affected by the iodine concentration. (auth)

DIBAN ION EXCHANGE 6.17.06

For decreasing the storage volume of highly radioactive waste containing $Al(NO_3)_3$. Al is converted to dibasic aluminum nitrate by destructive distillation and fission products are removed from the solution of this compound by ion exchange.

See also, this section,
DISTILLATION AND CONDENSATION (6.06),
and,
under FISSIONABLE MATERIAL--RECOVERY,
AL-U FUELS (3.01.01),
and,
under WASTE TREATMENT AND DISPOSAL,
AL-CONTAINING WASTES (8.05.01).

8339 ORNL-1984
Oak Ridge National Lab., Tenn.
DIBAN—ION EXCHANGE WASTE DISPOSAL SCHEME.

(SPECIAL) PROCESSES 6.17

DIBAN ION EXCHANGE 6.17.06

[PART] I. I. R. Higgins and R. G. Wymer. Nov. 28, 1955. 20p. Contract W-7405-eng-26. $0.20(OTS).

A scheme is proposed for decontaminating radioactive wastes whose chief ingredients are $Al(NO_3)_3$ and HNO_3. Ninety percent of the Nb and more than 95% of the Ru and Zr are removed by a $Fe(OH)_3-MnO_2$ scavenging precipitation. The $Al(NO_3)_3$ is converted to $Al(OH)_2NO_3$ by destructive distillation, and dissolved by a 12-hr digestion at 160°C. The Sr, Cs, and rare earths are removed by cation exchange in a continuous contactor. The decontaminated waste can be volume-reduced to about 6 M Al before disposal and then stored cheaply. The fission products can be eluted from the column, concentrated into a small volume of highly radioactive waste, and stored with all necessary precautions. (auth)

DRUHM 6.17.07

See also, under FISSIONABLE MATERIAL--RECOVERY,

REDUCTION (3.02.16).

Direct reduction of UF_6 to metal with Na vapor.

84 CF-59-8-118
Oak Ridge National Lab., Tenn.
EVALUATION OF LINER MATERIAL AND PRODUCT CONSOLIDATION IN THE DRUHM PROCESS. D. R. Edwards. Sept. 3, 1959. 10p. OTS.

Tests were made to evaluate magnesia and graphite as possible liner materials for the Druhm reaction ($UF_6 + 6Na \rightarrow U + 6NaF$) and to evaluate the effect of temperature on product consolidation and separation. In these tests, graphite held up better than magnesia; however, both were attacked by sodium. Appreciable uranium metal consolidation took place at temperatures greater than 1180°C. The results of a literature survey indicated that the most promising liner materials are magnesia, graphite, and graphite coated with a refractory material. (auth)

EXCER 6.17.08

A method for preparing metal-grade UF_4 from either pure or crude uranium salts by ion-exchange conversion of the uranium to uranyl fluoride and electrolytic reduction to UF_4.

See also, this section,

ION TRANSFER (MEMBRANES) (6.13),

ELECTROCHEMISTRY (6.07),

and,

under FISSION PRODUCT, POISON, AND RADIOISOTOPE REMOVAL,

ELECTROCHEMICAL (2.16),

and,

under FISSIONABLE MATERIAL--RECOVERY,

URANIUM (3.02.13.02),
PRECIPITATION (3.02.15),
REDUCTION (3.02.16).

4557 ORNL-2554
Oak Ridge National Lab., Tenn.
DEVELOPMENT OF THE EXCER PROCESS V: RECOVERY, PURIFICATION, AND IRON REDUCTION OF URANIUM USING A SULFATE ION EXCHANGE SYSTEM. R. R. Holcomb and I. R. Higgins. Jan. 27, 1959. 41p. Contract W-7405-eng-26. $1.25(OTS).

A uranium sulfate process flowsheet for the production of UF_4 from sulfate ore leach liquors is presented. The uranium is sorbed on an anion-exchange resin from 0.2 M sulfate ore leach and eluted with 2 M H_2SO_4. Iron powder reduces UO_2SO_4 to $U(SO_4)_2$ which is then sorbed on anion-exchange resin to separate it from the iron and eluted from the anion-exchange resin with 2 M H_2SO_4. The resulting $U(SO_4)_2$ is sorbed on a cation-exchange resin from the H_2SO_4 solution and eluted from the resin with 5 to 6 M HCl. The UCl_4 thus produced may be treated with stoichiometric amounts of HF in order to precipitate $UF_4 \cdot {}^3/_4H_2O$. (auth)

(SPECIAL) PROCESSES 6.17

EXCER 6.17.08

2834 ✓ ORNL-2490
Oak Ridge National Lab., Tenn.
DEVELOPMENT OF THE EXCER PROCESS. IV. CHLORIDE SYSTEM. I. R. Higgins, W. J. Neill, and L. E. McNeese. Dec. 3, 1958. 49p. Contract W-7405-eng-26. $1.50(OTS).

An Excer flowsheet for the recovery of uranium from a chloride system by anion exchange is presented. Ore concentrate is dissolved in HCl, purified by anion exchange, electrolytically reduced to uranium tetrachloride, and then precipitated as hydrated uranium tetrafluoride by the addition of HF. The hydrated uranium tetrafluoride is filtered, dried, pelletized, granulated, and then dehydrated in a fluidized-bed dehydrator at 450°C. In the anion exchange purification step decontamination factors of greater than 10^3 are realized for all impurities except trivalent iron, pentavalent vanadium, and molybdenum. These three impurities are re-

585 ORNL-2469
Oak Ridge National Lab., Tenn.
DEVELOPMENT OF THE EXCER PROCESS. III. PREPARATION OF URANIUM TETRAFLUORIDE FROM URANYL CHLORIDE BY IRON REDUCTION AND PRECIPITATION. W. J. Neill and I. R. Higgins. Oct. 22, 1958. 12p. Contract W-7405-eng-26. $0.50 (OTS).

A flowsheet, based on laboratory-scale experiments, is presented for preparation of uranium(IV) fluoride solution by reduction of uranyl chloride with metallic iron. The reduction was rapid, with 95% efficiency for 100% reduction. Precipitation of $UF_4 \cdot 3/4\ H_2O$ from this solution with HF separated the iron from the uranium by a factor of 10^3. (auth)

6532
PREPARING URANIUM TETRAFLUORIDE BY ION EXCHANGE AND ELECTROLYSIS. I. R. Higgins, J. T. Roberts, and C. W. Hancher (Oak Ridge National Lab., Tenn.) and J. A. Marinsky (Ionics Inc., Cambridge, Mass.). Ind. Eng. Chem. 50, 285-92(1958) Mar.

A detailed description of the Excer Process is given. The process is used for preparation of UF_4 from impure natural U in ore leach liquors, ore concentrates, or from reactor-irradiated U partially decontaminated by solvent extraction. (W.L.H.)

14676 ✓ A/CONF.15/P/506
Oak Ridge National Lab., Tenn.
THE EXCER PROCESS—AN AQUEOUS METHOD FOR PRODUCTION OF PURE URANIUM TETRAFLUORIDE FROM CRUDE URANIUM SOURCES. I. R. Higgins, W. J. Neill, and L. E. McNeese. 14p. $0.50(OTS).

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

An aqueous method of preparing pure UF_4 from ore leach liquors by ion exchange and electrical or chemical reduction is presented. The process may be adapted to a variety of crude uranium sources, such as concentrates and irradiated fuel or metal scrap solutions as well as ore leach liquors. The uranium is separated from the bulk of the anions by an anion exchange resin, and cation resins are used to eliminate all but the desired anionic species. The partially purified uranyl salt is reduced in a continuous electrolytic cell, or with iron, to uranous ion, and the hydrated green salt ($UF_4 \cdot 3/4\ H_2O$) is precipitated by addition of hydrofluoric acid. The wet green salt is dried to a free-flowing powder and is then ready for bomb reduction. (J.R.D.)

9665 ✓ ORNL-1979
Oak Ridge National Lab., Tenn.
DEVELOPMENT OF THE EXCER PROCESS. II. J. A. Marinsky. Apr. 17, 1956. Decl. Mar. 12, 1957. 35p. Contract W-7405-eng-26. $0.40(OTS).

Two revised Excer process flowsheets are presented for the production of UF_4 from $UO_2(NO_3)_2$ solution. In both, the U is sorbed on a cation-exchange resin and eluted with aqueous HF. In one, the resulting UO_2F_2 in dilute HF is mixed with dilute H_2SO_4 or HCl and electrolytically reduced in a cation-exchange-membrane cell, and the precipitated $UF_4 \cdot 0.75\ H_2O$ is dried and dehydrated to anhydrous UF_4. In the other, NH_4F is also added to the UO_2F_2 solution before the electrolytic reduction step, and the precipitate is $UF_4 \cdot 0.4\ NH_4F$. A flowsheet for preparing UF_4 from raw ore is also shown. The U is sorbed on an anion-exchange resin from a H_2SO_4 leach liquor and eluted with dilute HCl. Ammonium fluoride and HF are added to the UO_2Cl_2—HCl

1864 ✓ CF-55-11-130
Oak Ridge National Lab., Tenn.
THE ION EXCHANGE-EXCER PROCESS FOR THE PRODUCTION OF METAL GRADE UF_4 FROM CRUDE URANIUM SOURCES. I. R. Higgins. Nov. 29, 1955.

EXCER 6.17.08

Decl. Sept. 26, 1958. 8p. Contract [W-7405-eng-26]. $1.80(ph OTS); $1.80(mf OTS).

Discussions of the Higgins countercurrent exchanger for partially purifying crude product and an ion exchange conversion of crude uranium solutions to UO_2F_2 with subsequent electrolytic reduction to UF_4 are presented, with the emphasis on production of high-purity metal at the uranium ore mill site. The process may be divided step wise into anion exchange isolation, cation conversion, and electrolytic reduction. (J.R.D.)

7573 CF-54-12-169
Oak Ridge National Lab., Tenn.
EXCER: MOVING-BED REACTOR STUDIES. A LITERATURE SURVEY CONCERNING THE ONE-STEP PRODUCTION OF UF_4 FROM UO_3. J. E. Moore. Dec. 9, 1954. Decl. Feb. 16, 1957. 31p. Contract W-7405-eng-26.

7495 CF-54-2-130
Oak Ridge National Lab., Tenn.
MOVING BED–EXCER COST COMPARISON. F. E. Harrington, J. E. Moore, and J. T. Roberts. Feb. 17, 1954. Decl. Feb. 14, 1957. 24p. Contract [W-7405-eng-26]. $4.80(ph OTS); $2.70(mf OTS).

The results of a cost study made of the Moving Bed process and the Excer process on a comparable basis are given for a plant processing 22 tons of decontaminated U per day. The two processes are described and are illustrated by schematic diagrams. Detailed equipment costs are presented for both processes and are factored to obtain total capital investment. Operating costs, details, and summaries are included. (auth)

7494 CF-54-2-99
Oak Ridge National Lab., Tenn.
EXCER [PROCESS]: ANODE CORROSION IN AQUEOUS HYDROFLUORIC ACID ELECTROLYTES. J. H. Goode. Feb. 5, 1954. Decl. Feb. 14, 1957. 17p. Contract W-7405-eng-26. $3.30(ph OTS); $2.40(mf OTS).

A major problem in the electrolytic reduction step in the Excer Process for the conversion of $UO_2(NO_3)_2$ to UF_4 is the stability of the anodes required in the reduction cell. A survey of possible anode materials was made in an effort to find the most suitable electrode for use in the electrolytic stage of the process. Results of the survey indicated that a number of grades of graphite were suitable for use as consumable anodes, under flowsheet conditions of 3M HF and boiling temperature, if the fine degradation particles can be kept from contaminating the U product slurry by a suitable wrapping or other means. Of the metallic anodes tested, commercially pure Pt appeared to have the smallest weight losses, but under the flowsheet conditions the losses were too high to make its use economically attractive. (auth)

7900 CF-53-11-156
Oak Ridge National Lab., Tenn.
THE EXCER PROCESS FOR THE CONVERSION OF URANYL NITRATE TO URANIUM TETRAFLUORIDE. F. R. Bruce. Nov. 25, 1953. Decl. Feb. 21, 1957. 6p. Contract [W-7405-eng-26]. $0.25(OTS).

The Excer Process for the preparation of UF_4 from aqueous $UO_2(NO_3)_2$ is reviewed. Uranyl nitrate is converted to UO_2F_2 by ion exchange; then, UO_2F_2 is electrolytically reduced to hydrated UF_4. Advantages of the Excer Process include adequate fission-product decontamination, production of UF_4 at a lower cost, and better separation of U from ionic impurities; the main disadvantage results from the handling of aqueous HF and its associated corrosion problems. Cost estimates are included. (C.W.H.)

7493 CF-53-11-191
Oak Ridge National Lab., Tenn.
EXCER [PROCESS]: PRELIMINARY INVESTIGATION OF ELECTROLYTIC REDUCTION AND PRECIPITATION OF URANIUM FROM A NITRATE SYSTEM. E. D. Arnold and E. O. Nurmi. Nov. 20, 1953. Decl. Feb. 14, 1957. 17p. Contract W-7405-eng-26. $3.30(ph OTS); $2.40(mf OTS).

Several preliminary investigations indicated favorable results upon electrolyzing high (~ 50g/liter) uranium concentration solutions which yield black precipitates that remain black after washing. $NaNO_3$ and NH_4NO_3 systems were tried but NH_4NO_3 was found to be the most efficient. Cation membranes under favorable conditions produce the black precipitate but washing techniques will have to be improved to meet product specifications. (F.S.)

FLUOROX 6.17.09

Purified or crude uranium oxide simultaneously reduced and hydrofluorinated to UF_4 in a moving-bed reactor. The UF_4 is oxidized to UF_6 by oxygen. Energy for activation is about 850°C.

$$2UF_4 + O_2 \rightarrow UO_2F_2 + UF_6$$

See also, this section,

FLUIDIZED BEDS (6.08),

FLUORINATION (6.09),

and,

under PROCESS CHEMISTRY AND ENGINEERING,

URANIUM--Fluorides (1.43.04),

and,

under FISSIONABLE MATERIALS--RECOVERY,

FLUIDIZED BEDS (3.02.09),

FLUORINATION AND HYDROFLUORINATION (3.02.10),

OXIDATION (3.02.14),

REDUCTION (3.02.16),

and,

under HAZARDS AND PROTECTION,

PYROPHORICITY (4.01.10),

and,

under PLANTS AND EQUIPMENT,

HIGH-TEMPERATURE SYSTEMS--Vessels (5.10.04),

and,

under CHEMISTRY AND PHYSICS OF IMPORTANT ELEMENTS,

URANIUM--Fluorides--UF_6 (1.43.04.02).

16092 ✓ ORNL-2690
Oak Ridge National Lab., Tenn.
RECYCLE OF UO_2F_2 IN THE FLUOROX PROCESS: REACTION OF UO_2F_2 WITH HYDROGEN. L. M. Ferris and R. P. Gardner. July 9, 1959. 16p. Contract W-7405-eng-26. $0.75(OTS).

The reaction of UO_2F_2 with hydrogen at 700 to 850°C was briefly investigated since it could constitute a major step in the recycle of UO_2F_2 in the Fluorox process. The reaction in this temperature range proceeds according to the equation $UO_2F_2 + H_2 \longrightarrow UO_2 + 2\ HF$. Rates of the chemical reaction were not obtained, since, with the thermogravimetric technique used, the rate-controlling process was bed diffusion. However, there is no doubt that the rate of chemical reaction is sufficiently high for the desired application. No significant amount of UF_4 was formed by the back-reaction, $UO_2 + 4\ HF \longrightarrow UF_4 + 2\ H_2O$. (auth)

16756 CF-59-5-93
Oak Ridge National Lab., Tenn.
THE DEVELOPMENT OF A FLUIDIZED BED REACTOR FOR THE FLUOROX PROCESS: UNIT OPERATIONS MONTHLY STATUS REPORTS FOR THE PERIOD NOVEMBER 1958 THROUGH MAY 1959. J. C. Bresee, C. D. Scott, and R. W. Horton. May 26, 1959. 42p. Contract [W-7405-eng-26]. $7.80(ph), $3.30(mf) OTS.

Results of four experimental runs in the Fluorox fluidized bed reactor system are reported. The engineering feasibility of UF_6 production from UF_4 by use of dry air of O_2, $2UF_4 + O_2 = UF_6 + UO_2F_2$, in an Inconel fluidized bed reactor at 800 to 850°C was demonstrated in two experimental tests in which greater than 90% of the theoretical amount of UF_6 was collected or measured. Two runs made with crude UF_4 (produced from unpurified mill concentrate) as the feed material, showed that UF_6 could be produced at 700 to 725°C but corrosion on Inconel was prohibitive. (auth)

5878 GAT-L-434
Goodyear Atomic Corp., Portsmouth, Ohio.
HYDROFLUORINATION OF FLUOROX PELLETS. Gordon A. Rampy. Dec. 26, 1957. 6p. Contract [AT(33-2)-1]. $1.80(ph OTS); $1.80(mf OTS).

(SPECIAL) PROCESSES 6.17

FLUOROX 6.17.09

In order to obtain data needed to express mathematically the hydrofluorination-reduction step of the Fluorox Process, the changes in surface area and physical dimensions of the pellets as a result of reaction were determined. Data were also obtained on the chemical changes taking place during hydrofluorination. (W.L.H.)

9271 NLCO-675
National Lead Co. of Ohio, Cincinnati.
A KINETIC STUDY OF THE REACTION, $UO_2 + 4HF \rightarrow UF_4 + 2H_2O$. PART I. DEVELOPMENT OF THE RATE EQUATION. Richard E. DeMarco, DuWane C. Bonfer, and Robert C. Abbott. Apr. 18, 1957, 27p. Contract AT(30-1)-1156. $0.25(OTS).

A study has been made of the rate-controlling factors in the hydrofluorination of UO_2. A rate equation is proposed for the hydrofluorination reaction which is dependent on the UO_2 concentration and the rate of diffusion of HF through the UF_4 formed to the UO_2 interface. An Arrhenius-type equation expresses the dependence of the observed reaction rate on the hydrofluorination temperature. An activation energy of 8 kcal/mole was calculated, from this equation, for the hydrofluorination reaction. (auth)

185
A STUDY OF THE REACTION: $2UF_4 + O_2 \rightarrow UF_6 + UO_2F_2$. I. SIDE REACTIONS AND THERMODYNAMICS. L. M. Ferris (Oak Ridge NationalLab., Tenn.). J. Am. Chem. Soc. 79, 5419-21(1957) Oct. 20.

The reaction of UF_4 with dry O_2 has been investigated in the temperature range of 600 to 900°C. In addition to the expected products, UF_6 and UO_2F_2, UF_5 was isolated from the system, and is postulated to be the product of a side reaction between UF_6 and UF_4. Only slight decomposition of UO_2F_2 was observed in this temperature range in accordance with thermodynamic predictions. (auth)

7139 CF-56-12-128
Oak Ridge National Lab., Tenn.
CHEMICAL TECHNOLOGY DIVISION, UNIT OPERATIONS SECTION MONTHLY PROGRESS REPORT [FOR] DECEMBER 1956. J. C. Bresee, J. T. Long, E. O. Nurmi, and C. D. Watson. May 22, 1957. 81p. Contract [W-7405-eng-26]. $13.80(ph OTS); $4.80(mf OTS).

A fluidized bed dehydration run on Excer-produced $UF_4 \cdot {}^3/_4\, H_2O$ is reported. In Fluorox development, the reaction velocity constant for the reaction $UF_4 + O_2 \rightarrow UF_6 + UO_2F_2$ was expressed by $\ln K = 13.3 - 14.9\ (1000/T)$. Equipment installation and testing for homogeneous reactor fuel and blanket processing development is continued. Zircex Process experience with hydrochlorination of U–Zr alloys is described. Other power reactor fuel reprocessing studies reported include decladding, cutting, sawing, dissolver design, pulse column interfacial solids removal, and corrosion of Ti. Volatility process development continued with studies of F_2 disposal and gas-liquid contacting studies. (T.R.H.)

11053 CF-56-11-143
Oak Ridge National Lab., Tenn.
CHEMICAL TECHNOLOGY DIVISION; UNIT OPERATIONS SECTION MONTHLY PROGRESS REPORT [FOR] NOVEMBER 1956. W. K. Eister, J. C. Bresee, J. T. Long, E. O. Nurmi, and C. D. Watson. 111p. Contract [W-7405-eng-26]. $18.30(ph OTS); $6.00(mf OTS).

Chemical Engineering Research. Experiments with the three-stage pump hydroclone solvent extraction contactor show the stage efficiency with acetic acid-water-hexone to be nearly 100%. Excer. Uranium tetrafluoride with a tap density of 1.9 gm/cc can be briquetted to 3.6 gm/cc at 9 tsi. Fluorox. Three UF_4 oxidation runs in the fluidized bed reactor are described and discussed. Data are also given for three runs in a pellet-fed moving bed reactor. HR Fuel and Blanket Processing. Loop experiments with plating out of ZrO_2 from $Zr(SO_4)_2$–UO_2SO_4 solutions and flange testing are described. Other work in hydroclone development, pressurizer tests, blanket processing loops, and electrolytic production of H_2 and O_2 is reported. Power Reactor Fuel Reprocessing. Zircex reaction rates have been obtained and emissivities for Zircaloy-2 and U–Zr alloys up to 60 wt. % U have been calculated. Studies of decladding and disassembly of power reactor fuels continued. Other studies reported are corrosion studies of Ti and nickel alloys, pulse column interfacial crud removal, and waste studies. (For preceding period see CF-56-10-83.) (T.R.H.)

9666 ORNL-2227
Oak Ridge National Lab., Tenn.
FEED MATERIALS PROCESSING STATUS REPORT FOR NOVEMBER 1956. R. E. Blanco and J. C. Bresee. Feb.

FLUOROX 6.17.09

13, 1957. Decl. Feb. 28, 1957. 9p. Contract W-7405-eng-26. $0.25(OTS).

The Fluorox Process UF_6 scrubbing column was operated unsuccessfully and is being modified. Fluidization of UF_4 with N_2 at 600°C was accomplished. In Excer Process development electrolytic reduction cells were studied. The 12-in. Higgins Contactor was tested on V ore with 99.8% recovery. (T.R.H.)

10836 ✓✓ ORNL-2209
Oak Ridge National Lab., Tenn.
FEED MATERIALS PROCESSING STATUS REPORT FOR OCTOBER 1956. R. E. Blanco and W. K. Eister. Jan. 23, 1957. Decl. Mar. 14, 1957. 18p. Contract W-7405-eng-26. $0.30(OTS).

Metallex Process. Niobium was neither amalgamated nor dissolved by boiling in Hg at 356°C or by heating in fused $ZnCl_2$–KCl–NaCl matrix, containing $HgCl_2$, over Hg. In eight batch runs, made to produce Th quasi amalgam for metallurgical examination, a total of 2740 g of Th metal, as amalgam, was produced with an average yield of 84.5%. Fluorox Process. Oxidation at 600°C or higher of UF_4 produced by the Excer Process, which has a high specific surface area at 25°C, resulted in rapid sintering. Pelleting tests and two-stage reduction and hydrofluorination runs in moving-bed equipment with ore concentrate feed have been scheduled. In fluidization tests in which Excer UF_4 was

12995 ✓ CF-56-9-100
Oak Ridge National Lab., Tenn.
FEED MATERIALS PROCESSING STATUS REPORT FOR SEPTEMBER 1956. R. E. Blanco and W. K. Eister. Sept. 15, 1956. Decl. Feb. 20, 1957. 13p. Contract [W-7405-eng-26]. $0.25(OTS).

Hot filtration of Th quasi amalgam to remove reduction reaction by-products has not proved satisfactory. Less than 50% of the Th appeared in the filtrate. The quality of the sintered product was affected by the temperature differential between the metal sample and the furnace, by the holding temperature and pressure during retorting, and the holding time. Fluorox Process. The reduction-hydrofluorination moving-bed reactor was further modified. In the oxidation step, 3.5% C, added as graphite pellets, did not provide sufficient heat for the reaction. Excer Process. A new UF_4 preparation step is being studied in which UCl_4 is formed in the cell and UF_4 is precipitated in a separate step by adding HF. Ion-exchange Ore Processing. A 12-in.-diam Higgins continuous ion-exchange column has operated satisfactorily for 13 days with Dysart ore pulp at Grand Junction, Colo. (auth)

7511 ✓✓ CF-56-6-100
Oak Ridge National Lab., Tenn.
FEED MATERIALS PROCESSING STATUS REPORT FOR JUNE 1956. R. E. Blanco and W. K. Eister. June 15, 1956. Decl. Feb. 14, 1957. 22p. Contract [W-7405-eng-26]. $0.30(OTS).

Metallex Process. Methanol and petroleum ether from which dissolved O_2 has been removed have less oxidative effect upon Th amalgam than water with or without dissolved O_2. A rough correlation between the free voidage of sintered Th and its oxide content, and therefore the metal quality, is indicated. A procedure was developed for determining the total voidage and the atmosphere-connected voidage in sintered Th. Fluorox Process. The specific surface area of UF_4 appears to be constant over the temperature range 25 to 600°C. Above 600°C the specific surface area decreases as sintering begins. The major impurities found in UF_6 produced from African ore concentrate were Al, Ca, Cu, and Fe. The reduction-hydrofluorination and oxidation reactors were modified to improve their operability, and 2 runs were made with the improved oxidation reactor. Low-carbon UF_4, suitable for fluorination to UF_6 or reduction to metal, was prepared by burning the carbon in the UF_4 with O_2. Excer Process. Results of runs in the 2-in. diameter Higgins continuous ion-exchange contactor indicate that UO_2Cl_2 product may be withdrawn at concentrations as high as 140 g of U per liter and containing as low as 0.05M NaCl. Conductivity instruments provided excellent process control, which in turn allowed efficient usage of 5M NaCl eluant. This eliminated the need for recycle of the NaCl. The presence of Na or Ca did not affect the density of the electrolytic $UF_4 \cdot \frac{3}{4}H_2O$ product. An electrolytic cell with 64 in.^{2} inside area was operated 5 hr to produce $UF_4 \cdot \frac{3}{4}H_2O$. The resulting slurry of $UF_4 \cdot \frac{3}{4}H_2O$ appears acceptable as far as physical characteristics are concerned for making specification grade UF_4 after filtration and dehydration. A nickel ribbon flite dehydrator is being constructed. (For preceding period see CF-56-5-100.) (auth)

FLUREX 6.17.10

See also, this section,

ELECTROCHEMISTRY (6.07),

FLUORINATION (6.09),

ION TRANSFER (MEMBRANES) (6.13),

and,

under HAZARDS AND PROTECTION,

FLUORINE (4.01.05).

14733 A/CONF.15/P/534
General Electric Co. Hanford Atomic Products Operation, Richland, Wash. and Utah. Univ., Salt Lake City.
THE FLUREX PROCESS: A WET CHEMICAL METHOD FOR THE PRODUCTION OF URANIC FLUORIDE SALTS. W. W. Schulz, E. W. Neuvar, J. L. Carroll, and R. E. Burns. 17p. $0.50(OTS).
Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.
The basic element of the Flurex Process is a three compartment electrolytic cell having a center feed compartment separated from the anode compartment by an anion permeable membrane and from the cathode compartment by a cation permeable membrane. Ideally, uranyl nitrate enters the feed compartment from which nitrate ion passes through the anion membranes to the anolyte and uranyl ion through the cation membrane to

13223 HW-59780
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
EVALUATION OF MATERIALS FOR USE AS ANODES IN THE FLUREX PROCESS. W. W. Schulz and E. W. Neuvar. Feb. 15, 1959. 23p. Contract W-31-109-Eng-52. $0.75(OTS).
The anodic corrosion of several readily available materials in synthetic Flurex Process anolyte solutions of the composition 0.1 M NH_4NO_3, 0.0003 M NH_4F, pH 7-9 was measured to provide a basis for the selection of satisfactory anode materials. Of the materials tested, Pt and the stainless steels 304 L and 347 were selected for more extensive tests. Anodic corrosion rate measurements over the entire span of probable Flurex Process operating conditions showed that Pt and stainless steels 304 L and 347 are satisfactory anode materials for use in the process. Based on the anodic corrosion rates obtained, it is estimated that the cost of anode material dissolved per pound of U processed in the electrolytic cell would be about 0.4 cent for either SS 304 L or 347 and about 1.4 cents for Pt. The anodic corrosion of Pt in HNO_3 solutions was also investigated. (auth)

FUSED SALT – FLUORIDE VOLATILITY 6.17.11

Recovery and decontamination of uranium from irradiated fuel elements by volatilization of the fluoride. Metal is dissolved in molten mixture of fluorides in the presence of HF; the UF_4 thus formed is converted to UF_6 by F_2. The partially decontaminated volatile UF_6 is further decontaminated by passage through NaF beds.

See also, this section,

DISTILLATION AND CONDENSATION (6.06),

and,

under FISSIONABLE MATERIAL--RECOVERY,

FLUORINATION AND HYDROFLUORINATION (3.02.10),

FLUORIDE VOLATILITY (3.02.18.01),

and,

under HAZARDS AND PROTECTION,

FLUORINE (4.01.05),

and,

under PLANTS AND EQUIPMENT,

HIGH-TEMPERATURE SYSTEMS--Vessels (5.10.04).

(SPECIAL) PROCESSES 6.17

FUSED SALT – FLUORIDE VOLATILITY 6.17.11

GENERAL – FLOWSHEETS 6.17.11.01

10983 ORNL-2661
Oak Ridge National Lab., Tenn.
THE FUSED SALT-FLUORIDE VOLATILITY PROCESS FOR RECOVERING URANIUM. G. I. Cathers, M. R. Bennett, and R. L. Jolley. April 15, 1959. 33p. Contract W-7405-eng-26. $1.00(OTS).

A fluoride volatility process for recovering enriched uranium from UF_4–NaF–ZrF_4 melts, produced by the dissolution of reactor fuel elements in molten NaF–ZrF_4 through hydrofluorination, is described. The UF_4 is fluorinated to UF_6, which volatilizes, and is separated from volatile fission products in a NaF absorption-desorption cycle. In laboratory studies gross beta and gamma decontamination factors of 10^5 with a uranium loss of less than 0.1% were secured. The product UF_6 may be conveniently returned to UF_4 or metal for further use as reactor fuel. Chemical studies, supplementary to process development work, showed that impurities, possibly oxides, have a much greater effect in the fused salt-fluorination step than such factors as use of nitrogen with the fluorine, or the method of introducing the fluorine into the fluoride melt. The colloidal behavior of NiF_2, a slightly soluble corrosion product formed in the fluorination, indicated that this material would not interfere in molten salt handling if the fused salt was not allowed to stand without agitation for prolonged periods. The absorption of UF_6 in NaF was found to be due to the formation of a UF_6–NaF complex. The equilibrium between gaseous UF_6 and solid complex was established for the temperature range of 80 to 320°C. Decomposition of the UF_6–NaF complex to a UF_5–NaF complex does not lead to appreciable uranium loss in the process if specified process conditions are maintained. (auth)

2454 CF-59-9-2
Oak Ridge National Lab., Tenn.
URANIUM RECOVERY FROM SODIUM–ZIRCONIUM FLUORIDE SALT MIXTURES, VOLATILITY PILOT PLANT RUNS L-1 THROUGH L-9. C. L. Whitmarsh. Sept. 30, 1959. 75p. Contract [W-7405-eng-26]. OTS.

About 606 kg of sodium-zirconium fluoride salt mixture, similar to the ARE fuel but nonirradiated, was reprocessed in the Volatility Pilot Plant. Of the 72.28 kg of fully enriched uranium in the feed, 99.2% was recovered as uranium hexafluoride product, with 0.01% measured process losses. An additional 1% was reclaimed from sodium fluoride beds and clean-out of the system. The product was of sufficient purity to meet specifications for material designated for reduction to uranium metal. Plant operation was stopped for 20 days between Runs L-4 and L-5 to clean out the fluorinator. Precipitation of nickel fluoride had plugged all interior piping in that vessel. Development studies concerning decontamination and plutonium behavior were carried out. Fluoride salt from reactor loop experiments was added to Run L-7, with a resultant feed salt gross gamma activity of 5.1×10^6 cpm/g salt. Complete decontamination from fission products was achieved Results from three runs in which plutonium tetrafluoride was added to the feed salt indicated that greater than 99% of the plutonium would remain in the salt after fluorination. (auth)

8784 CF-59-1-115
Oak Ridge National Lab., Tenn.
DISSOLUTION OF ZIRCONIUM MATRIX FUELS IN MOLTEN FLUORIDE SALTS. R. W. Horton and M. E. Whatley. Jan. 30, 1959. 18p. Contract [W-7405-eng-26]. $3.30(ph), $2.40(mf) OTS.

For presentation at the American Chemical Society's Nuclear Technology Sumposium, Boston, April 5-10, 1959.

The application of the Volatility Process for reprocessing spent reactor fuels to zirconium fuel elements requires the conversion of the fuel to a fluoride form. This conversion can be accomplished by the dissolution of such fuels in a bath of molten fluoride salts with a hydrogen fluoride sparge. Studies with dummy zirconium fuel elements showed dissolution rates averaging 2 mg/sq cm-min. The effect of submergence, geometrical configuration, and HF impingement rate appeared more significant than the effect of temperature. Experiments with partially submerged elements have indicated that at temperatures in the neighborhood of 700°C the gas phase reaction rate can exceed the salt phase rate. The use of the process involves problems with corrosion, with entrainment of solids in the off gas, and with the recovery and recycle of the hydrogen fluoride. Tentative solutions to these problems have been studied. (auth)

(SPECIAL) PROCESSES 6.17

FUSED SALT – FLUORIDE VOLATILITY 6.17.11

GENERAL – FLOWSHEETS 6.17.11.01

RECOVERY OF URANIUM FROM HIGHLY IRRADIATED REACTOR FUEL BY A FUSED SALT—FLUORIDE VOLATILITY PROCESS. G. I. Cathers, W. H. Carr, R. B. Lindauer, R. P. Milford, and M. E. Whatley. 14p. (A/CONF.15/P/535)

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

A fused-salt fluoride volatility process is being developed at Oak Ridge National Laboratory for recovering U from either molten fluoride salt fuel or Zr–U heterogeneous reactor fuel. The basic step in the process is conversion of the U to UF_6 with F_2 in a fluoride salt melt with volatilization of the UF_6 from the melt. In the case of the Zr–U fuels the fuel element is dissolved in a fluoride salt melt with HF at 600 to 700°C, which with the molten fluoride fuels the UF_4 is a constituent of the melt. Volatile fission product fluorides are removed from the UF_6 in an absorption-desorption step with a pair of NaF beds, and the decontaminated UF_6 is collected in cold traps. Compared to aqueous processing, the fused salt fluoride volatility process has fewer chemical steps in the over-all U fuel cycle, an appreciably lower waste volume, and the probability of a more compact plant.

10974✓ CF-58-5-113
Oak Ridge National Lab., Tenn.
A SERIES OF SEVEN FLOWSHEET STUDIES WITH NONRADIVE SALT, VOLATILITY PILOT PLANT RUNS C-9 THROUGH C-15. C. L. Whitmarsh. May 12, 1958. 62p. Contract [W-7405-eng-26]. $10.80(ph), $3.90(mf) OTS.

A series of seven flowsheet demonstrations of the Volatility Process was performed using nonirradiated material. In this series (a) the Pilot Plant was proven to be operable but not sufficiently reliable for remote operation; (b) the feasibility of a bed of NaF pellets as a trap for Cr and Zr fluorides was demonstrated; and (c) the most desirable fluorine flow rate for fluorination of molten salt in the existing equipment was determined to be in the range 10 to 30 std. liters per min. (auth)

10820 ✓✓CF-56-5-101
Oak Ridge National Lab., Tenn.
POWER REACTOR FUEL PROCESSING STATUS REPORT FOR MAY 1956. R. E. Blanco and W. K. Eister. May 15, 1956. Decl. Feb. 15, 1957. 10p. Contract [W-7405-eng-26]. $0.25(OTS).

Fused Salt–Fluoride Volatility Process. Dissolution rates of a Zr-clad U–Zr fuel element in $NaF \cdot ZrF_4$ were affected by the rate of flow of HF through the melt and the position of the metal relative to the HF inlet. The solubility of NiF_2 in $NaF \cdot ZrF_4$ appeared to be less than 1 wt % at 600°C. At 1 wt % and higher concentrations, NiF_2 was a stable colloid. The complex $UF_6 \cdot 3NaF$ was decomposed when heated at 400°C in the presence of excess UF_6. The U content of the residue was about 22%. Decomposition was much slower at 300°C or in the presence of excess F_2. The residual U was effectively pentavalent, as a result of the release of F_2 during the heating. Molten KHF_2 effectively decontaminated metals that had been exposed to molten fluoride mixtures containing fission products. Zircex Process. The heat of reaction in the hydrochlorination of Zr caused a rapid temperature rise. The temperature was controlled by introducing controlled amounts of H_2 with a constant HCl gas flow. The Zircaloy-2 cladding was chemically removed from a U–Mo alloy in laboratory studies. More than 99% of the U was obtained in a water solution separated from the insoluble molybdic acid remaining after dissolution of the core alloy. Hermex Process. Uranium that had been irradiated to an nvt of 5×10^{11} and decayed for 10 days was decontaminated from gross γ and β activities by factors of 20 and 70, respectively, by dissolution in Hg and distillation of the mercury. Continuous distillation of the Hg was shown to be possible. Sodium Amalgam Reduction of UCl_4. Uranium chloride was reduced to UHg_4 by sodium amalgam with a yield of approximately 80.5%. (auth)

10973 CF-58-5-112
Oak Ridge National Lab., Tenn.
EQUIPMENT CHECKOUT PRIOR TO ARE PROCESSING, VOLATILITY PILOT PLANT RUN B-1. C. L. Whitmarsh. May 29, 1958. 14p. Contract [W-7405-eng-26]. $3.30(ph), $2.40(mf) OTS.

The head-end system for the ARE salt was tested with barren salt and shown to be operable. Transfer of the final batch of salt by gravity flow from the hold tank was shown to be feasible with a redesigned feed salt freeze valve. The CRP trap was also redesigned but the remote dumping mechanism did not function properly. (auth)

(SPECIAL) PROCESSES 6.17

FUSED SALT – FLUORIDE VOLATILITY 6.17.11

General - Flowsheets 6.17.22.01

10972 CF-58-5-62
Oak Ridge National Lab., Tenn.
VOLATILITY PILOT PLANT–WASTE SALT TRANSFER STUDIES. J. B. Ruch. May 21, 1958. 15p. Contract [W-7405-eng-26]. $3.30(ph), $2.40(mf) OTS.
The Volatility Pilot Plant waste salt transfer operation was simulated with water in clear tubing and glass equipment to demonstrate hydraulically the feasibility and desirability of transferring the salt by a siphoning method. Surge chambers placed in the vertical legs of the freeze valve loops were found necessary to produce a satisfactory seal. (auth)

DESIGN 6.17.11.02

5931
ENGINEERING DESIGN OF OAK RIDGE FLUORIDE VOLATILITY PILOT PLANT. Robert P. Milford (Oak Ridge National Lab., Tenn.). Ind. Eng. Chem. 50, 187-91 (1958) Feb.
The new facility will demonstrate U recovery from spent Zr-enriched U fuels by volatilization of UF_6 and absorption on NaF. (auth)

CORROSION AND MAINTENANCE 6.17.11.03

See also, under PLANTS AND EQUIPMENT,

CORROSION--By Fused Salts and Liquid Metals (5.05.03),

HIGH-TEMPERATURE SYSTEMS--Materials (5.10.02).

EQUIPMENT DECONTAMINATION METHODS FOR THE FUSED SALT–FLUORIDE VOLATILITY PROCESS. R. L. Jolley, R. D. Baybarz, D. O. Campbell, and G. L. Cathers. Sept. 10, 1958. 15p. (ORNL-2550)
The decontamination methods studied included barren salt flushes, 0.5 M $(NH_4)_2C_2O_4$ solution, 5 wt. % HNO_3—5 wt. % $Al(NO_3)_3$ solution, and 10 wt. % NaOH—2.5 wt. % H_2O_2—10 wt. % $Na_2C_4H_4O_6$ solution. Salt flushes were relatively ineffective for decontamination, their effect being mainly dilution of residual salt. $(NH_4)_2C_2O_4$ solutions at 90 to 100°C dissolved NaF–ZrF_4 (50-50 mole %) and removed much activity. The HNO_3–$Al(NO_3)_3$ solution was required to remove a metallic scale formed in inconel equipment as the result of contact with HF, F_2, and UF_6 at 600°C. The alkaline-peroxide-tartrate solution was particularly effective in decontaminating descaled reactors and equipment exposed to gaseous reactants and products of the fluoride volatility process. Some application of the principles involved in the decontamination procedures appears possible in other fused salt projects, for example, in molten salt reactor technology.

211 ORNL-2495
Oak Ridge National Lab., Tenn.
PROGRESS REPORT ON VOLATILITY PILOT PLANT CORROSION PROBLEMS TO APRIL 21, 1957. L. R. Trotter and E. E. Hoffman. Oct. 17, 1958. 41p. Contract W-7405-eng-26. $1.25(OTS).
Many complex corrosion problems have been encountered in the preliminary bench tests and early operation of the Fused-Fluoride Volatility Process. Several of the problems have been investigated and are fairly well understood, such as embrittlement of nickel by sulfur-containing fluoride salts and the heavy attack on even the most promising structural materials by fused fluoride salts in the presence of air contamination at elevated temperatures. The heavy attack encountered on nickel rods in the fluorination vessel during early test runs is not completely understood, although it is believed that improved handling techniques and maintenance of a gas-tight system will reduce the attack. This report merely serves to emphasize that in a complicated high-temperature system of this type there are many material and design problems which must be solved before reliable long-time operation can be assured. (auth)

7184 ANL-5662
Argonne National Lab., Lemont, Ill.
CORROSION OF MATERIALS IN THE PRESENCE OF FLUORINE AT ELEVATED TEMPERATURES. Martin J

(SPECIAL) PROCESSES 6.17

FUSED SALT - FLUORIDE VOLATILITY 6.17.11

Corrosion - Process 6.17.19.02

Steindler and Richard C. Vogel. Jan. 1957. 22p. Contract W-31-109-eng-38. $0.25(OTS).

In development of materials of construction for the Fluoride Volatility Process, corrosion tests in F_2 at high temperatures were run on Ni, Ni alloys, Copper, CaF_2, and Al_2O_3. These results are presented and compared with results of other investigators on Al, stainless steels, steel sheet, and Armco iron. (T.R.H.)

HF RECOVERY 6.17.11.04

10849 ✓ CF-58-12-55
Oak Ridge National Lab., Tenn.
VOLATILITY PILOT PLANT MODIFICATIONS—HYDROGEN FLUORIDE CONDENSERS, FV-2004, FV-2005. S. Mann. Dec. 8, 1958. 9p. Contract [W-7405-eng-26]. $1.80(ph), $1.80(mf) OTS.

Off-gas from the hydrofluorinator will be cooled and 94% of the hydrogen fluoride condensed by two heat exchangers connected in series. The first unit, water-cooled, discharges gas and condensed vapor at 50 to 65°F. The second unit, freon-cooled, discharges condensed vapor and noncondensable hydrogen and nitrogen at −60 to −75°F. This report concerns a design description of the condensers and a heat transfer evaluation. The condensers will be fabricated at ORNL. (auth)

THE PROBLEM OF SCRUBBING THE HYDROFLUORINATION OFF GAS WITH LIQUID HF. M. E. Whatley and L. E. McNeese. July 11, 1958. 17p. (CF-58-7-98)

Radioactive solids will be entrained by the off-gas from the hydrofluorination step of the Volatility Process. The Volatility Process is being developed as a method for recovering U from Zr matrix fuel elements. It is proposed to scrub these solids from the off-gas in a packed bed using refluxed liquid HF. A development program has been planned and a unit designed for the experimental studies.

VOLATILITY PILOT PLANT—DESIGN OF A NaF PACKED TOWER FOR REMOVING HF FROM FLUORINE. J. S. Watson. Mar. 7, 1957. 9p. (CF-57-3-43)

An adsorption column packed with $\frac{1}{8}$ in. NaF pellets was designed to reduce the HF content of the F_2 being used by the Volatility Pilot Plant from 5% to less than 0.01%. It will be a non-isothermal packed bed with the gas inlet heated to 100°C to avoid plugging and the exit cooled to 25°C for more complete HF removal.

CONSTRUCTION MATERIALS 6.17.11.05

16087 ✓ BMI-1348
Battelle Memorial Inst., Columbus, Ohio.
CONSTRUCTION MATERIALS FOR THE HYDROFLUORINATOR OF THE FLUORIDE-VOLATILITY PROCESS. Paul D. Miller, Charles L. Peterson, Oliver M. Stewart, Elmer F. Stephan, and Frederick W. Fink. June 3, 1959. 44p. Contract W-7405-eng-92. $1.50 (OTS).

Fuel elements clad with Zr or containing Zr as a diluent can be recovered by a fluoride-volatility process. The first step consists of hydrofluorination of the elements in a bath of molten fluoride salts using an HF sparge. In this case the two salt systems considered were $NaF-ZrF_4$ and NaF−LiF. Materials evaluated at Battelle for possible use in the construction of this hydrofluorinator include Inconel, "A" Nickel, copper, silver, Monel, Hastelloy B, Hastelloy W, INOR-1, and INOR-8. The metals were exposed to molten fluoride salts through which HF was bubbled continuously. The data indicate that the NaF−LiF systems are much more corrosive than the $NaF-ZrF_4$ system. The systems are most corrosive when the alkali fluoride component is high. An elevation in temperature increases the corrosion significantly as does an increase in the HF flow rate. Hydrogen in the HF flow stream retards the corrosion of the sodium−zirconium salts significantly, but appears to have less effect on the sodium−lithium systems. The areas at the interface of the liquid and vapor phases were most seriously damaged under the exposure conditions usually used. However, appreciable reduction in attack was experienced when zirconium was actually hydrofluorinated. INOR-8 was the most promising of the materials evaluated. (auth)

(SPECIAL) PROCESSES 6.17

HCl•ALCOHOL 6.17.12

A method for dissolving zirconium and its alloys in anhydrous alcohol-hydrochloric acid.

See also, this section,

RADIATION EFFECTS--Alcohols (6.15.08.01),

and,

under FISSIONABLE MATERIALS--RECOVERY,

DISSOLUTION (GENERAL) (3.01.04),

ZR-U FUEL ELEMENTS (3.01.12).

HERMEX 6.17.13

Decontamination of irradiated uranium or thorium metal, or direct recovery of uranium and thorium from scrap metal. Metal is dissolved in hot mercury and filtered and/or washed in acid to remove impurities. The amalgam is cooled and filtered and the U and Th recovered by heating to volatilize the Hg.

See also, Metallex Process.

See also, this section,

DISTILLATION AND CONDENSATION (6.06),

and,

under FISSIONABLE MATERIAL--RECOVERY,

DISTILLATION (3.02.07),

and,

under HAZARDS AND PROTECTION,

MERCURY (4.01.08).

19817 IGR-176(O/CA)
United Kingdom Atomic Energy Authority. Industrial Group. Capenhurst Works, Capenhurst, Ches., England.
RECOMMENDED LABORATORY PROCEDURE FOR THE PURIFICATION OF RECOVERED MERCURY.
June 12, 1959. 7p. $0.21(BIS).
Impurities are oxidized by prolonged compressed air blowing and removed by repeated water and nitric acid washes. (auth)

13986
THE SYSTEM MERCURY-THORIUM. R. F. Domagala, R. P. Elliott, and W. Rostoker (Illinois Inst. of Tech., Chicago). Trans. Met. Soc. AIME 212, 393-5(1958) June.
The phase equilibria of the Hg-Th system over the composition range 0 to 100% Th and temperatures up to 1000°C have been studied for a small-volume, closed system. The solubility of Th in liquid Hg is about 5% at 300°C and decreases sharply with decreasing temperature. Two intermediate phases occur, Hg_3Th and HgTh. The structures of these are hexagonal (nonideally close-packed) and face-centered cubic, respectively. The HgTh phase decomposes eutectoidally at 400 to 500°C. The solubility of Hg in solid thorium seems to be negligible. (auth)

17222
TRANSPORT AND CHEMICAL CONTROL IN THE DISSOLUTION OF METALS IN MERCURY. J. A. R. Bennet and J. B. Lewis (Atomic Energy Research Establishment, Harwell, Berks, Eng.). J. chim. phys. 55, 83-90(1958) Feb. (In French)
The rate of dissolution of cylinders of lead, tin, and zinc in mercury were studied and the results were compared with the dissolution of benzoic acid in water since it is known as being a process of controlled diffusion. While the dissolution of lead and tin is also a process of controlled diffusion, the dissolution of zinc is in a part a chemical process. In order to have conditions of reproducible agitation, the cylindrical samples, attached coaxially, were mounted in a revolving cylinder containing the mercury. Some experiments with cylindrical single crystals of zinc have shown that the different crystal faces have different speeds of dissolution. Qualitatively, the same phenomena were found for bismuth and cadmium. (tr-auth)

(SPECIAL) PROCESSES 6.17

HERMEX 6.17.13

10474 ORNL-2242
Oak Ridge National Lab., Tenn.
STATUS OF THE HERMEX PROCESS. O. C. Dean, E. Sturch, B. H. Morrison, and R. E. Blanco. Aug. 12, 1957. 34p. Contract W-7405-eng-26. $0.30(OTS).

A preliminary flowsheet is presented for the Hermex process, a method for decontaminating irradiated metals and for purifying scrap metals. As applied to uranium, the process consists in dissolving the metal in boiling mercury, recrystallizing and precipitating the metal or its mercuride by cooling the solution, filtering off the metal or alloy crystals, washing the amalgam with dilute acid to further remove contaminants, recovering the metal by volatilizing off the mercury in vacuum or inert gas stream, and melting the resulting sponge to massive metal. The feasibility of a continuous Hermex process for uranium was demonstrated on a 20- to 50-g/day basis, and larger-scale stainless steel equipment was constructed. (auth)

13682 ORNL-2210(Del.)
Oak Ridge National Lab., Tenn.
POWER REACTOR FUEL PROCESSING STATUS REPORT FOR OCTOBER 1956. R. E. Blanco, W. K. Eister, and D. E. Ferguson. Jan. 23, 1957. Decl. with deletions Mar. 14, 1957. 20p. Contract W-7405-eng-26. $3.30(ph OTS); $2.40(mf OTS).

Fused Salt – Fluoride Volatility Process. A decontamination factor of $> 10^6$ was achieved with a U loss of 0.14%. Studies on embrittlement of Ni are reported. Hermex Process. Dissolution studies of STR II prototype specimens in fused salt systems over Hg containing a $ZnCl_2$–KCl–NaCl matrix are reported. Crystal bar Zr and the STR II specimens dissolved rapidly and smoothly in $AlCl_3$–NaCl containing NH_4Cl or $HgCl_2$. Zircex Process. Hydrochlorination of U–Zr gave good U recovery, but not for U–Nb or U–Al. Data for design of larger equipment are reported. Darex Process. Uranium does not form volatile chloride complexes; the chloride may be distilled off in 3 stages. Tantalum withstands Darex dissolution conditions. General Processing Studies. The dissolution of Zr and Zircaloy-2 in aqueous nitrate-chloride and radiation effects on Amsco were studied. (T.R.H.)

12366 ✓ CF-56-8-200
Oak Ridge National Lab., Tenn.
A PROPOSED FUEL ELEMENT DESIGN TO FACILITATE REPROCESSING BY THE HERMEX METHOD. O. C. Dean. Aug. 31, 1956. Decl. Apr. 1, 1957. 5p. Contract [W-7405-eng-26]. $1.80(ph OTS); $1.80(mf OTS).

A fuel element design is reported which will facilitate reprocessing by the Hermex method. A can, positioned vertically in the reactor, is to be filled with small rods, wires, or pellets of U and the voids filled with molten Na. The ends of the cans should be so equipped that they can be remotely removed without destruction, in order to discharge the contents to a Hermex reprocessing vessel. The fuel element is to be surrounded by another tube which provides a channel for Na coolant. A gas space should be provided at the top of each element connected to a header to serve as a pressure relief and as a bleed for fission product gases. (L.T.W.)

7506 ✓ CF-56-1-151
Oak Ridge National Lab., Tenn.
THE HERMEX PROCESS FOR METAL DECONTAMINATION BY MERCURY PROCESSING. B. H. Morrison and R. E. Blanco. Jan. 25, 1956. Decl. Feb. 14, 1957. 19p. Contract [W-7405-eng-26]. $0.30(OTS).

The Hermex Process for the decontamination of irradiated metals and purifying scrap virgin metals is presented. The process as applied to irradiated U consists of the following steps: the irradiated U is continuously dissolved in boiling Hg at 365°C; the hot U–Hg solution from the dissolver vessel is withdrawn continuously leaving a slag layer containing ~87% of the fission products; the U–Hg solution is cooled to 25°C and concentrated by vacuum filtration; the U quasi-amalgam is washed with dilute HCl to remove the fission products; the Hg is volatilized from the quasi-amalgam; and the U melted to dense metal by slow heating to 1250°C in a single operation. (W.L.H.)

METALLEX 6.17.14

Preparing Th, U, and similar metals from their salts. E.g., thorium chloride is reduced with Na amalgam; the thorium amalgam formed is heated to volatilize the Hg.

(See also) Hermex Process.

See also, this section,

DISTILLATION AND CONDENSATION (6.06),

and,

under FISSIONABLE MATERIAL--RECOVERY,

DISTILLATION (3.02.07),

and,

under HAZARDS AND PROTECTION,

MERCURY (4.01.08).

THE PREPARATION OF THORIUM METAL BY SODIUM AMALGAM REDUCTION OF THORIUM CHLORIDE: THE METALLEX PROCESS. O. C. Dean and G. K. Ellis (Oak Ridge National Lab., Tenn.). Nuclear Sci. and Eng. 4, 509-21(1958) Oct.

A process, developed at Oak Ridge National Laboratory, produced thorium metal by the continuous reduction of anhydrous thorium tetrachloride with sodium amalgam on a scale up to 3.5 pounds per hour. The salt was vigorously agitated with an excess of sodium amalgam which was produced by the electrolysis of aqueous sodium hydroxide. The resulting slurry of thorium mercuride in mercury was washed free from impurities and reaction by-products with dilute HCl and water. A solid concentrate of the thorium mercuride was prepared by filter-pressing the dilute slurry. The remaining mercury was removed by vacuum-distillation, resulting in massive metal of about 0.8 of the theoretical thorium density. The metal was fabricated into rods by direct extrusion or by arc-melting followed by extrusion. (auth)

9442 BNL-483(p.102-8)
Oak Ridge National Lab., Tenn.
THORIUM PRODUCTION BY THE SODIUM AMALGAM REDUCTION OF THORIUM TETRACHLORIDE. O. C. Dean. p.102-8 [of] THORIUM-U^{233} SYMPOSIUM, SPONSORED BY THE UNITED STATES ATOMIC ENERGY COMMISSION AT BROOKHAVEN NATIONAL LABORATORY, JANUARY 9-10, 1958. 7p.

The reduction of thorium tetrachloride by sodium in a mercury medium has many advantages in producing reactor grade thorium economically. All steps in which corrosion of equipment is a factor are carried out at low temperature. All steps up to the elimination of mercury are continuous. The metal is protected from loss and contamination by a coating of mercury during separation from by-products and impurities. The removal of the processing medium (mercury) leaves a nonpyrophoric massive metal which can be safely processed to billets and rods. Two reduction procedures have been developed, either of which could be scaled up to pilot plant

5920 CF-57-10-46
Oak Ridge National Lab., Tenn.
COST ESTIMATE FOR PRODUCTION OF THORIUM TETRACHLORIDE AND THORIUM METAL BY THE METALLEX PROCESS. O. C. Dean, W. F. Schaffer, and J. C. Suddath. Oct. 14, 1957. 19p. Contract [W-7405-eng-26]. $3.30(ph OTS); $2.40(mf OTS).

The cost of producing thorium metal from thorium nitrate is estimated as $1.81 per pound by the PDA (propylene diamine) flowsheet and $1.96 per pound by the Dry Salt flowsheet. The initial plant investment for the PDA plant is higher ($7,018,725) than that for the Dry Salt plant ($6,235,971), but the annual operating cost, not including depreciation, is higher for the Dry Salt plant ($3,942,910 vs. $3,388,480 per year). The total annual operating costs for each plant, including 20% annual depreciation, are $4,792,224 for the PDA plant and $5,190,101 for the Dry Salt plant, with each producing 4 tons of thorium metal sinter per day (330 days' operation per year). (auth)

10836 ✓ ORNL-2209
Oak Ridge National Lab., Tenn.
FEED MATERIALS PROCESSING STATUS REPORT FOR OCTOBER 1956. R. E. Blanco and W. K. Eister. Jan. 23,

METALLEX 6.17.14

1957. Decl. Mar. 14, 1957. 18p. Contract W-7405-eng-26. $0.30(OTS).

Metallex Process. Niobium was neither amalgamated nor dissolved by boiling in Hg at 356°C or by heating in fused $ZnCl_2$–KCl–NaCl matrix, containing $HgCl_2$, over Hg. In eight batch runs, made to produce Th quasi amalgam for metallurgical examination, a total of 2740 g of Th metal, as amalgam, was produced with an average yield of 84.5%. Fluorox Process. Oxidation at 600°C or higher of UF_4 produced by the Excer Process, which has a high specific surface area at 25°C, resulted in rapid sintering. Pelleting tests and two-stage reduction and hydrofluorination runs in moving-bed equipment with ore concentrate feed have been scheduled. In fluidization tests in which Excer UF_4 was oxidized, 60 to 150 mesh particles were the most satisfactory size. Excer Process. The density of $UF_4 \cdot {}^3/_4H_2O$ was increased from about 1 g/cc to about 3 g/cc by pressing and grinding. The uranium decontamination by electrolysis was as good when UF_4 was precipitated after electrolysis by adding HF to the UCl_4 electrolytic product as it was when the HF was present in the electrolytic cell and reduction and precipitation were simultaneous. The slurry-Hg separation equipment of the electrolytic cell was redesigned. Results of dehydration runs showed that about 80% water removal is possible with present equipment. Ore Processing. In preliminary tests high-pressure carbonate leaching resulted in extraction of more U and less silica than low-pressure. (auth)

12439 NP-7418
Mine Safety Appliances Co., Callery, Penna.
REMOVAL OF MERCURY IN RESIDUAL SODIUM FROM A DRAINED SYSTEM USING A SODIUM FLUSH. Memo Report 103. E. F. Batutis, C. A. Palladino, and K. F. Barker. Jan. 17, 1956. 9p. Contract NObs-65426.

The use of a fresh sodium charge to dilute mercury in the residual sodium of a drained system is a practical method for eventually removing the mercury. This was confirmed by using a hold-up pocket filled with a sodium amalgam and circulating fresh sodium past the ½ in. pocket opening. The sodium amalgam in the bottom of the pocket was more effectively diluted by raising the system temperature to 600°F. The major portion of the mercury, in the pocket, mixed with the flush sodium circulating at 2.5 fps, at any temperature from 350 to 600°F in 15 to 30 minutes. (auth)

13653 CF-56-6-73(Del.)
Oak Ridge National Lab., Tenn.
CHEMICAL DEVELOPMENT STATUS REPORT FOR WEEK ENDING JUNE 1, 1956. R. E. Blanco and D. E. Ferguson. June 6, 1956. Decl. with deletions Mar. 12, 1957. 10p. Contract [W-7405-eng-26]. $1.80(ph OTS); $1.80(mf OTS).

Progress is reported on the development of Hermex and Metallex Processes, Thorex pilot plant operation, heterogeneous reactor fuel reprocessing, uranium tetrafluoride production by Excer Process, rare earth separation by ion exchange, homogeneous reactor fuel and blanket studies, and protactinium–uranium–thorium processing. (C.W.H.)

12992 CF-55-9-173
Oak Ridge National Lab., Tenn.
A HIGH-TEMPERATURE METALLEX STUDY. R. C. Reid. Sept. 9, 1955. Decl. Apr. 4, 1957. 41p. Contract [W-7405-eng-26]. $0.40(OTS).

The vapor phase, high-temperature Na reduction of $Th(Cl)_4$ was investigated and the results are presented. Mercury vapor acting as a carrier for sublimed $Th(Cl)_4$ was contacted with Na vapor, diluted with He. The products when cooled were noted to consist of nearly equal amounts of a solid powder phase and a phase which appeared to be clean Hg. Thorium, Na and chloride were found predominantly in the powder phase. Both phases were washed with 3N HCl and water. Thorium was distributed (a) as a filterable quasi in the Hg phase, (b) as dissolved Th in the acid wash, and (c) as a powder which was not wettable with mercury nor soluble in HCl. Analytical data indicate that a large fraction of the Th in the product was present as reduced metal. Corrosion of the equipment resulted in Fe, Ni, and Cr contamination of the product. The data also indicated that the vapor pressure of $ThCl_4$ in an impure feed salt was much lower than would have been anticipated from vapor pressure data of the pure salt. (auth)

12652 AERE-X/R-1503
General Electric Co., Ltd. Research Labs., Wembley, England.
THE EFFECT OF MERCURY ON THE CORROSION AND PROPERTIES OF VARIOUS MATERIALS. FINAL REPORT. PART 3. A SURVEY OF THE INTERACTIONS OF THE METALLIC ELEMENTS WITH STATIC LIQUID MERCURY AT ROOM TEMPERATURE AND 500°C. Report No. 11,727. J. F. Strachan and N. L. Harris. July 19, 1954. 45p.

METALLEX 6.17.14

A general survey of the interactions, saturated solubility, and weight loss of most metals in mercury is presented. Determinations were made at room temperature and at 500°C with annealed and unstressed samples. The complexity of Hg attack is noted, and various methods of overcoming ambiguities are discussed in relation to experimental technique. Data are tabulated. (J.R.D.)

METAL RECOVERY 6.17.15

Originally a solvent extraction process for recovering U and Pu from a nitric acid solution of tank farm waste, using TBP. The process has been modified for use with different types of feeds.

See also, this section,

SOLVENT EXTRACTION--Solvents
--TPB - Nitrate Systems (6.16.05.10),

and,

under FISSIONABLE MATERIAL--RECOVERY,

RECOVERY OF IRRADIATED TH AND U
--Solvent Extraction--Nitrate Systems
(3.02.17.04),

RECOVERY OF PLUTONIUM--Solvent Extraction
(3.03.14).

14691 A/CONF.15/P/536
Oak Ridge National Lab., Tenn.
OPERATING EXPERIENCE WITH TWO DIRECT-MAINTENANCE RADIOCHEMICAL PILOT PLANTS.
F. R. Bruce, E. M. Shank, R. E. Brooksbank, J. R. Parrott, and G. S. Sadowski. 51p. $0.50(OTS).

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

The Thorex plant has a capacity of 150 kg of thorium per day, yielding thorium and uranium-233 products suitable for reactor recycle. The Metal Recovery Facility has a capacity of 450 kg of uranium per day and has been used for the processing of a wide variety of irradiated and unirradiated plutonium and uranium. The Thorex Process is applicable to the recovery of uranium and thorium from heterogeneous reactor fuels and from the blanket of the aqueous homogeneous reactor. Operation of the Thorex and Metal Recovery facilities has given information on process performance, cost of construction and operation, and control of radiation exposure to personnel. (J.R.D.)

pH MONITORING OF METAL RECOVERY NEUTRALIZED WASTE. C. E. Michelson. Sept. 24, 1954. Decl. Feb. 27, 1957. 23p. (HW-33148)

In order to ensure adequate control of pH in the continuous neutralization of Metal Recovery Process wastes, a continuous recording pH meter has been successfully applied. The mechanical difficulties of a pH sensing system working in a relatively inaccessible location with a continuously operated jet sampler have been overcome using a replaceable unit with provisions for standardizing the pH meter and automatically acid flushing the cell and sampler. A unique feature of the unit is that the acid flush which is introduced at regular intervals provides, by means of the pH meter response, a positive indication on the recorder chart that both the sampler and the pH meter are functioning properly. The close pH control made possible by this installation has been shown to be adequate for the successful scavenging of radiocesium from process wastes and has also reduced the frequency of over-neutralization of the waste.

CHLORIDE REMOVAL FROM METAL WASTE SOLUTIONS. L. H. Clark. Oct. 10, 1950. Decl. Feb. 19, 1957. 13p. (HW-19105)

Chloride ion in the RAW waste stream from the TBP metal recovery process would excessively corrode the stainless steel in a HNO_3 recovery step. The conditions under which peroxysulfate ($K_2S_2O_8$) will oxidize quantitatively the Cl^- to Cl_2, which may be removed by air sparging, are presented in detail. A preliminary survey of other oxidants was made. The results of this survey are tabulated, as are the per cent decomposition of $S_2O_8^{2-}$ by synthetic RAW, formation of ClO_3 and ClO_4 during Cl^- removal.

(SPECIAL) PROCESSES 6.17

NEPTEX 6.17.16

Solvent extraction process for recovering uranium and neptunium from irradiated enriched uranium fuel elements. U and Np are extracted from nitric acid solution of the fuel by TBP, stripped together, and separated by ion exchange.

See also, this section,

SOLVENTS--TBP - Nitrate Systems (6.16.05.10),

and,

under FISSIONABLE MATERIAL--RECOVERY,

SOLVENT EXTRACTION--Nitrate Systems (3.02.17.04),

and,

under FISSION PRODUCT, POISON, AND RADIOISOTOPE REMOVAL,

TRANSURANICS--Neptunium (2.54.05).

12541 ✓ ORNL-2235
Oak Ridge National Lab., Tenn.
RECOVERY OF Np^{237} BY THE NEPTEX SOLVENT-EXTRACTION PROCESS. J. R. Flanary, J. H. Goode, R. G. Mansfield, and R. P. Wischow. Apr. 3, 1957. Decl. May 6, 1959. 39p. Contract W-7405-eng-26. $6.30 (ph), $3.00 (mf) OTS.

Flowsheets are presented for the solvent extraction recovery of uranium and Np^{237} from special highly irradiated MTR fuel elements and from nonvolatile fluoride residues from the Oak Ridge Gaseous Diffusion Plant. In both cases the material containing the neptunium and uranium is dissolved and digested to inactivate emulsion-forming impurities, the neptunium and uranium are extracted with tributyl phosphate solvent, using aluminum nitrate as the primary salting agent, scrubbed free of fission products and/or ionic contaminants, and finally separated and recovered by selective stripping with nitric acid. (auth)

PUREX 6.17.17

Separation of U and Pu from a nitric acid solution of irradiated natural aluminum-clad uranium fuel elements. U and Pu [as Pu(IV)] are extracted with TBP and stripped separately after the Pu has been reduced to Pu(III). Pu is purified by ion exchange.

Also, after other head-end treatments, for the reprocessing of fuels clad with metals other than aluminum.

See also, this section,

RADIATION EFFECTS--Aqueous Solutions (6.15.01),

SOLVENT EXTRACTION--Solvents-- TBP - Nitrate Systems (6.16.05.10),

and,

under FISSIONABLE MATERIAL--RECOVERY,

SOLVENT EXTRACTION--Nitrate Systems (3.02.17.04),

RECOVERY OF PLUTONIUM--Ion Exchange (3.03.09).

GENERAL - FLOWSHEETS 6.17.17.01

3567 ✓ HW-61848
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
EUROCHEMIC QUESTIONS—PRELIMINARY REPORT. R. G. Geier. Aug. 24, 1959. Decl. Oct. 7, 1959. 30p. Contract AT(45-1)-1350. OTS.

Several questions asked by Eurochemic personnel on the operation of the Purex Plant are presented. (W.L.H.)

General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
EUROCHEMIC INFORMATION EXCHANGE—ANSWERS TO SPECIFIC QUESTIONS. O. F. Hill. May 5, 1959.

((SPECIAL) PROCESSES 6.17

PUREX 6.17.17

GENERAL - FLOWSHEETS 6.17.17.01

17p. Contract W-31-109-Eng-52. $3.30(ph), $2.40(mf) OTS. (HW-60281)

Answers to specific questions posed in relation to the Eurochemic processing plant are presented. The questions and information in the answers are largely in regard to Purex-type operation. Questions on extraction column details, TBP stability, use of elevated temperatures in the Purex Process, specifications for column plates, off standard streams and products, waste disposal, and others are included. (J.R.D.)

16878 HW-60116

General Electric Co. Hanford Atomic Products Operation, Richland, Wash.

DESCRIPTION OF PUREX PLANT PROCESS. E. R. Irish. Apr. 30, 1959. Decl. May 19, 1959. 16p. Contract [W-31-109-Eng-52]. $3.30(ph), $2.40(mf) OTS.

A brief summary, with reference literature for details, of pertinent and important process flowsheet conditions which are in use in the Purex Process is presented. (auth)

20003

DESIGN vs. PERFORMANCE OF PROCESS AND EQUIPMENT IN A LARGE-SCALE RADIOCHEMICAL SEPARATIONS PLANT. Presented at Nuclear Engineering and Science Conference, April 6-9, 1959, Public Auditorium, Cleveland, Ohio. Preprint V-71. A. W. Joyce, Jr., L. C. Peery, and E. B. Sheldon (Du Pont de Nemours (E. I.) & Co., Aiken, S. C.). New York, Engineers Joint Council, 1959. 37p. $0.50.

The primary plutonium separations system at the Savannah River Plant employs the Purex tributyl phosphate solvent extraction process in heavily-shielded remotely-maintained facilities. Process and equipment performance are referenced to the design bases with regard to product quality, yields, maintenance experience, and process hazards, within the limits of security regulations. (auth)

20992 NP-7827

European Company for the Chemical Processing of Irradiated Fuels, Mol, Belgium.

EUROCHEMIC PUREX FLOWSHEETS: NO. 1, DRAWING 89a AND NO. 2 DRAWINGS 89b AND 90b. 1959. $7.80(ph), $3.30(mf) OTS.

15238 TID-7569(p.44-53)

General Electric Co. Hanford Atomic Products Operation, Richland, Wash.

SEPARATIONS PLANT SILVER REACTOR INCIDENT. E. R. Irish. p.44-53 [of] PROCEEDINGS OF THE 1958 ATOMIC ENERGY COMMISSION AND CONTRACTOR SAFETY AND FIRE PROTECTION CONFERENCE, HELD AT ATOMIC ENERGY COMMISSION HEADQUARTERS BUILDING, GERMANTOWN, MARYLAND, JUNE 24-25, 1958. 10p. (HW-57048).

A description of the dissolver cell equipment in the Purex Plant is given. Some of the chemical reactions occurring in the silver reactor are given. The operating experience of the silver reactor that exploded is traced. A detailed description is presented of the incident. (W.L.H.)

1296 WASH-743

Division of Civilian Application, AEC.

SUMMARY REPORT: AEC REFERENCE FUEL-PROCESSING PLANT. Oct. 1957. 30p. $1.00(OTS).

A conceptual design is presented of a multipurpose chemical processing plant for the processing of spent reactor fuel elements. The concept chosen uses a number of parallel dissolution systems with a common aqueous extraction system based on the Purex Process. The dissolution systems were chosen to dissolve U-Zr, U-stainless steel, combination alloys such as U-Mo and U-Nb, and fuels involving combinations of U, Zr, and stainless steel in a single element. Schematic flow diagrams are included for the over-all plant, dissolution processes, a radioactive off-gas facility, the extraction system, and processing procedures for various fuel types. Data are tabulated from capitol cost estimates for the reference multipurpose plant, operating cost estimates, and the capital and operating costs for individual fuel types. A standard daily chemical processing charge of $15,300 was established. The charge is made for the time a fuel is using the plant for extraction plus time for shutdown, cleanup, and startup. (C.H.)

(SPECIAL) PROCESSES 6.17

PUREX 6.17.17

GENERAL - FLOWSHEETS 6.17.17.01

312 ✓
THE PUREX PROCESS-A SOLVENT EXTRACTION REPROCESSING METHOD FOR IRRADIATED URANIUM. E. R. Irish and W. H. Reas. May 1, 1957. p.83-106 of SYMPOSIUM ON THE REPROCESSING OF IRRADIATED FUEL HELD AT BRUSSELS, BELGIUM, MAY 20-5, 1957. 1165p. (TID-7534(Bks. 1-3); HW-49483A)

The Purex Process, which employs solvent extraction to separate and purify U and Pu from each other and from fission products contained in irradiated U fuel elements is described. A description of the over-all process, utilizing tri-butyl phosphate solvent (in a kerosene-type diluent) and HNO_3 salting agent, is provided along with chemical process flowsheets. The process chemistry of U, Pu, and fission products is discussed as affected by process variables. Methods of recovery of spent solvent and acid are briefly discussed. Alternate process arrangements are suggested. 12 references.

13020 ✓ ORNL-2020
Oak Ridge National Lab., Tenn.
EFFECT OF PLANT SIZE ON COSTS OF PUREX PROCESSING OF HETEROGENEOUS POWER REACTOR FUEL. J. W. Ullmann. Feb. 1, 1956. Decl. Apr. 26, 1957. 15p. Contract W-7405-eng-26. $0.30(OTS).

Cost estimates are given for 1- to 16-ton/day privately owned Purex processing plants. A 4-ton/day plant is estimated to be required to achieve 1 mill/kwh chemical reprocessing costs with fuel irradiated to 2000 Mwd/ton. Such a plant would handle the fuel discharged from sixteen 500-Mw reactors. If the same reactors discharged fuel irradiated to 10,000 Mwd/ton, a chemical reprocessing cost of 0.3 to 0.4 mill/kwh is anticipated. As plant sizes increase, the cost of shipping irradiated fuel becomes a larger fraction of the total reprocessing cost. (auth)

13021 TID-5318

RUTHENIUM IN THE PUREX PROCESS. R. E. Tomlinson and F. J. Leitz, Jr. Oct. 14, 1954. Decl. Mar. 11, 1957. 7p. (HW-33479(Rev.))

The difficulties encountered in the removal of Ru during the normal operation of the Purex production plant are outlined. Radiochemical properties and oxidation states of Ru are tabulated.

20994 ORNL-1558
Oak Ridge National Lab., Tenn.
A DESIGN AND COST ESTIMATE STUDY OF A PUREX PLANT. H. L. Hull and H. R. Zeitlin. Mar. 31, 1954. Decl. June 29, 1959. 80p. Contract W-7405-eng-26. $13.80(ph), $4.80(mf) OTS.

The preliminary design and cost study of a hypothetical Purex plant sized to process three metric tons of irradiated natural uranium per day and designed for direct maintenance are reported. (auth)

12375 KAPL-795(Del.)
Knolls Atomic Power Lab., Schenectady, N. Y.
AN INVESTIGATION OF THE $KMnO_4$-MnO_2 HEAD-END PROCEDURE FOR THE REMOVAL OF RUTHENIUM AND NIOBIUM-ZIRCONIUM. I. A PILOT-SCALE STUDY OF THE PROCEDURE WITH NATURAL URANIUM. J. K. Davidson, J. H. Holmes, and J. F. LaBonte. Aug. 22, 1952. Decl. with deletions Mar. 6, 1957. 30p. Contract W-31-109-Eng-52. $4.80(ph OTS); $2.70(mf OTS).

The results of a pilot-scale study with natural U to investigate the variables in the Purex head-end treatment are given. A simplification of the procedure is sought.

7480 ✓ CF-51-12-158
Oak Ridge National Lab., Tenn.
PUREX PROCESS FLOWSHEET NO. 3 IN TERMS OF PILOT PLANT FLOWS. D. O. Darby. Dec. 31, 1951. Decl. Feb. 15, 1957. 4p. $1.80(ph OTS); $1.80(mf OTS).

12401 ✓ ORNL-743(Del.)
Oak Ridge National Lab., Tenn.
APPLICATION OF THE PUREX PROCESS TO ORNL METAL WASTE RECOVERY. T. C. Runion and C. V. Ellison. Aug. 24, 1950. Decl. with deletions Mar. 1, 1957. 32p. Contract W-7405-eng-26. $6.30(ph OTS); $3.00(mf OTS).

(SPECIAL) PROCESSES 6.17

PUREX 6.17.17

CONSTRUCTION MATERIALS 6.17.17.02

16201 CF-59-1-15(Rev.1)
Oak Ridge National Lab., Tenn.
EUROCHEMIC ASSISTANCE: ORNL REVIEW OF EUROCHEMIC "STAINLESS STEEL MATERIALS." E. M. Shank. Jan. 14, 1959. 12p. $3.30(ph), $2.40 (mf) OTS.

Comments of qualified ORNL personnel on the proposed materials of construction for the Purex portion of the Eurochemic plant are reported. Persons commenting are E. C. Miller, G. M. Adamson, C. H. Wodtke, J. L. English, and P. Patriarca. Included is the condensation of information from a Eurochemic meeting to discuss these materials of construction. (auth)

HEAT TRANSFER AND FLUID FLOW

7162 HW-41860
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
STUDIES ON THE HYDRAULIC CHARACTERISTICS OF PUREX OFF-GAS JETS. L. R. Michels. Mar. 14, 1956. 27p. Contract [W-31-109-Eng-52]. $4.80(ph OTS); $2.70(mf OTS).

7965 HW-32466
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
RELATION OF SEPARATIONS PLANT EQUIPMENT EXPERIENCE TO THE PUREX DESIGN. Separations P. E. Report No. 48. F. A. Hollenbach. June 23, 1954. Changed from OFFICIAL USE ONLY May 22, 1957. 10p. Contract [W-31-109-Eng-52]. $3.30(ph OTS); $2.40(mf OTS).

The life experience of the various types of equipment currently in separations plant service (Redox, TBP, and $BiPO_4$) is recorded. Special features in the Purex plant equipment which resulted from consideration of the observed weaknesses are described. The sparing philosophy used for the various equipment groups in the Purex plant is also given. (T.R.H.)

CORROSION – PROCESS 6.17.17.03

CORROSION OF STAINLESS STEEL AND TITANIUM IN PUREX 2WW. M. C. Fraser. Mar. 24, 1958. 18p. (HW-43986)

Heat-transfer corrosion rate data are presented which compare the resistance of three types of stainless steel and one type of Ti to corrosion by boiling synthetic Purex 2WW. Specific corrosion parameters investigated were time and temperature. Laboratory and mock-up data had the same order of magnitude. Pilot plant data are presented which illustrate some aspects of the nature of corrosion on heat-transfer surfaces as affected by material composition and method of fabrication. Types 304-L, 347, and 312 stainless steel were tested in the annealed, sensitized, or as-welded condition, and type A-70 Ti was tested in the annealed condition. Their corrosion rates were linear with time after an induction period, and, with two exceptions, were linear with temperature. At one thousand hours of exposure and a metal temperature of 145°C, sensitized type 347 had a corrosion rate of about fifty-seven mils per year, sensitized 304-L—about thirty-nine mils per year, sensitized 312—about thirty-three mils per year, and annealed A-70 Ti—about three mils per year.

THE SUPPRESSION OF CHLORIDE VOLATILIZATION IN NITRIC ACID DISTILLATION. R. L. Moore. July 27, 1956. Decl. Mar. 14, 1957. 4p. (HW-44584)

Chloride-induced corrosion in Purex and similar processes may be reduced by the addition of $Hf(NO_3)_2$.

THE RESISTANCE OF VARIOUS BEARING MATERIALS TO CHEMICAL ATTACK BY NITRIC ACID AND URANYL NITRATE HEXAHYDRATE. Norman D. Groves. Mar. 11, 1954. Decl. Feb. 19, 1957. 20p. (HW-30500)

The chemical resistance of various bearing materials, used in Purex submerged pumps, to solutions of HNO_3 and $UO_2(NO_3)_2$ was studied. Results indicated that Corning glass, boron carbide, Ti or Chemelar M1-411 exhibited sufficient resistance to chemical attack by 60% HNO_3 and by 100% $UO_2(NO_3)_2$ solutions at boiling temperatures to warrant their use as bearings in Purex submerged rotating equipment.

(SPECIAL) PROCESSES 6.17

PUREX 6.17.17

Corrosion - Process 6.17.17.03

16201 CF-59-1-15(Rev.1)
Oak Ridge National Lab., Tenn.
EUROCHEMIC ASSISTANCE: ORNL REVIEW OF EUROCHEMIC "STAINLESS STEEL MATERIALS." E. M. Shank. Jan. 14, 1959. 12p. $3.30(ph), $2.40 (mf) OTS.

Comments of qualified ORNL personnel on the proposed materials of construction for the Purex portion of the Eurochemic plant are reported. Persons commenting are E. C. Miller, G. M. Adamson, C. H. Wodtke, J. L. English, and P. Patriarca. Included is the condensation of information from a Eurochemic meeting to discuss these materials of construction. (auth)

ION EXCHANGE 6.17.17.04

2056 ✓ HW-52796
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
REDUCTION AND STABILIZATION OF PLUTONIUM NITRATE IN CATION EXCHANGE FEED AND PRODUCT SOLUTIONS. A. Brunstad and R. C. Smith. Sept. 24, 1957. Decl. Nov. 10, 1958. 18p. Contract W-31-109-Eng-52. $3.30(ph OTS); $2.40(mf OTS).

In the concentration and purification of Pu by cation exchange, the feed solution must be in the plus three valence state, but the nitrate solution from a solvent extraction process contains a high % of Pu^{4+} and a small amount of Pu^{6+}. It was desired to find a reducing agent satisfactory for the reduction of Pu^{4+} and Pu^{6+} in Purex product streams and to make these streams suitable as cation exchange feed. The bisulfates met the requirements for speed and completeness of reduction in the feed material. (J.E.D.)

PROCESS CONTROL 6.17.17.05

16088 ✓ CF-58-10-126(Rev. I)
Oak Ridge National Lab., Tenn.
EUROCHEMIC ASSISTANCE: ANALYTICAL. L. T. Corbin. June 23, 1959. 5p. Contract [W-7405-eng-26]. $1.80(ph), $1.80(mf) OTS.

Information on the analytical requirements for a combination control and development laboratory which is to be associated with a Purex-type solvent extraction processing plant is presented. Additional comments by personnel of the ORNL Analytical Chemistry Division on questions directed to them for the Eurochemic Assistance Program are also given. (J.R.D.)

4431 HW-37272
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
IN-LINE ALPHA MONITOR PROTOTYPE PERFORMANCE. N. T. Hildreth. Oct. 11, 1955. Decl. Nov. 23, 1959. 31p. Contract [W-31-109-Eng-52]. OTS.

The performance of an in-line alpha monitor for continuously monitoring Pu in Purex Process waste streams is described. The instrument has a lower limit of measurement of 1×10^{-6} grams of Pu per liter and can measure concentrations as high as 7.9×10^{-2} g/liter. (C.J.G.)

15177 ✓ DP-336
Du Pont de Nemours (E. I.) & Co. Savannah River Lab., Aiken, S. C.
DETECTION OF NITRITE ION IN PLUTONIUM NITRATE SOLUTIONS. Myron O. Fulda. Dec. 1958. 7p. Contract AT(07-2)-1. $0.50(OTS).

Colorimetric and potentiometric methods were developed for the very rapid detection of nitrite ion in the 2AF Purex process stream. Both methods were designed to detect the presence of excess nitrite in the valence adjustment step. The presence of nitrite was detected at a concentration as low as 2×10^{-4} molar. (auth)

1202 ✓ HW-56674
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
DENSITY MONITOR FOR PUREX HA COLUMN. C. E. Huck. July 16, 1958. 17p. Contract W-31-109-Eng-52. $3.30(ph OTS); $2.40(mf OTS).

A densimeter system is described which was developed for use on HA column. Reproducibility of instrument readings is ± 2½% of recorder span for pulser frequencies of 44 to 104 cycles per minute. The effects of column pulsations and column static pressure at the feed point are small. Simulated column feed point static pressure of 10 psig caused improved precision due to added damping of float oscillations. (auth)

(SPECIAL) PROCESSES 6.17

PUREX 6.17.17

PROCESS CONTROL 6.17.17.05

SELECTED ANALYTICAL METHODS FOR PUREX PROCESS CONTROL. B. F. Rider—E. L. Bernier, ed. Apr. 30, 1953. Decl. Mar. 14, 1957. 142p. (KAPL-890(Rev.))

Step-by-step procedures used for control of the Purex process pilot plant at KAPL are given for use as a laboratory manual. Included are procedures for measuring acidity; for measuring density; for determining Pu; radiation measurements of α, β, and γ; radiochemical procedures for Am^{241} plus Cm^{242}, Ce^{144}, Cs^{137}, I^{131}, Np^{237}, Nb^{95}, Ru^{106}, Sr^{89}, Th^{234}, total rare earths, Zr^{95}, and total fission products; and U. Miscellaneous determinations include Al, C_4H_9OH, DBP, Fe^{3+} and Fe^{2+}, F^-, H_2O_2, $C_6H_4(OH)_2$, NH_2OH, MnO_2, Mn, NO_2^-, PO_4^-, Na, $NaNO_3$, sulfamate, SO_4^{2-}, TBP, and H_2O. Also included in the Appendix are safety rules and some laboratory techniques.

CONTINUOUS EVAPORATION OF THE PUREX PROCESS ICU STREAM IN THE ORNL PILOT PLANT. A. E. Wible. Feb. 27, 1952. Decl. Feb. 12, 1957. 12p. (CF-52-2-215)

Holdup in the continuous evaporator is given. Control is fully automatic. At 20% excess boil-up the evaporator recovers from sudden purges of feed in a reasonable time. Increasing the boildown required at the same U throughout has no effect on operation. A continuous chart record of product specific gravity at steady conditions showed fluctuations of ±0.01 specific gravity units around a given set point. The equipment is described in detail.

4361 TID-7581(p.52-60)

General Electric Co. Hanford Atomic Products Operation, Richland, Wash.

APPLICATION OF A RATIO METHOD OF PLUTONIUM RECEIPT MEASUREMENT IN A CONTINUOUS SEPARATIONS PLANT. W. H. Johnson. 9p.

The most accurate separations plant control system was based on acceptance of U weight and determining a Pu to U ratio, thereby arriving at a Pu value essentially independent of sampling and volume considerations. A discussion is presented of the events leading up to the adoption of a ratio method for routine receipt measurement, the details of its application in a Purex continuous process, method obstacles and their elimination, and the advantages to be realized with the ratio type measurement. (W.L.H.)

7393 PGR-64(W)

United Kingdom Atomic Energy Authority. Production Group. Chemical Services Dept., Windscale, Sellafield, England.

ANALYTICAL METHODS FOR THE INSPECTION OF ALUMINIUM NITRATE. 1959. 15p. BIS.

Water-insoluble matter and Al are determined gravimetrically. The total nitrate content is determined volumetrically, the chloride and sulfate turbidimetrically, and the boron and iron absorptiometrically. (auth)

PROCESS DATA 6.17.17.06

TEMPERATURE EFFECTS ON TBP SOLVENT EXTRACTION PROCESSES. D. G. Karraker. 11p. (A/CONF.15/P/519).

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

TBP extracts U (VI) and Pu (IV) well, and most of the fission products poorly. Ru, Zr, and Nb are the most difficult fission products to separate, and the radioactivity of the end products is determined by the extent to which these fission products are eliminated. The general behavior of these fission products indicates that slow changes among their species restrict their separation. An increase in temperature accelerates changes among these species, and results in an increased fission product separation for TBP processes. A combination of adsorption processes for removal of residual fission products with the improved, high temperature, Purex process achieves decontamination factors greater than 5×10^6 for the U product and greater than 5×10^7 for the Pu product. This performance equals the decontamination achieved in the normal Purex process (two or three solvent extraction cycles), with a considerable reduction in the required equipment. The decontamination of both cycles of the 25 process was improved by operation at 70°C. The Ru decontamination of the first cycle was a factor of 100 greater at 70°C than at 30°C, and the decontamination for Zr–Nb (combined) was improved by a factor of ten. The improvement in the second cycle decontamination varied with first cycle performance, and apparently was mainly limited by the activity present in the feed. Samples of the organic phase from the extraction-scrub contactor of the first cycle showed that the same amount of activity was extracted at both temperatures; but the extracted activity was removed much faster at higher temperatures. The radiolysis of TBP by the radioactivity of the feed solution was observed in experimental work on the 25 process. The results indicated that the radiolytic

(SPECIAL) PROCESSES 6.17

PUREX 6.17.17

PROCESS DATA 6.17.17.06

product, dibutylphosphate, impairs decontamination. These results were obtained with miniature solvent extraction equipment, operated continuously for extended periods, and processing feed solutions prepared from irradiated material. 5 references.

2818 DP-53
Du Pont de Nemours (E. I.) & Co. Savannah River Lab., Augusta, Ga.
EQUILIBRIUM DISTRIBUTION DATA FOR PUREX AND SIMILAR EXTRACTION PROCESSES. Thomas H. Siddall, III, Sidney G. Parker, and William E. Prout. July 1957. 27p. Contract AT(07-2)-1. $0.75(OTS).
Data are presented for the equilibrium distribution of U, Th, and HNO_3 between solutions of tri-n-butyl phosphate in kerosene and the various aqueous solutions encountered in the separation of Pu and U and in processes for the recovery of U^{235} from spent reactor fuel. (auth)

THERMOCHEMICAL MEASUREMENTS OF PUREX PROCESS SOLUTIONS. Glenn A. Burney. May 1956. 14p. (DP-158)
Data are presented for the specific heats of representative aqueous and organic solutions of the Purex separations process. An empirical equation, accurate within 4% of experimental values, permits the calculation of specific heats of aqueous solutions of $UO_2(NO_3)_2$ and HNO_3 throughout the ranges of process concentration Heats of reaction are reported for the extraction of water, HNO_3, and $UO_2(NO_3)_2$ into an organic phase composed of thirty vol.% tributyl phosphate in kerosene.

DETERMINATION OF THE SPECIFIC HEATS OF SOME PUREX SOLVENTS AND DILUENTS. Arthur Dreeben. Oct. 18, 1952. Decl. Oct. 4, 1955. 18p. (KAPL-834)
To aid in the calculation of probable temperature rises in the steam jetting of Purex organic streams, approximate specific heats have been determined for some pure solvents diluents, and U-solvent-diluent solutions.

7483 ✓ CF-52-4-13
Oak Ridge National Lab., Tenn.
PUREX 1A COLUMN STUDIES. J. R. Flanary. April 1, 1952. Decl. Feb. 20, 1957. 13p. Contract [W-7405-eng-26]. $3.30(ph OTS); $2.40(mf OTS).
The effects of aqueous-phase HNO_3 concentration and solvent saturation with U on the separation of fission products from Pu and U in the 1A column were investigated. Freezing point data of feed solutions and specific gravities of organic solvent phases containing U are reported. (C.W.H.)

7482 ✓ CF-52-2-215
Oak Ridge National Lab., Tenn.
CONTINUOUS EVAPORATION OF THE PUREX PROCESS ICU STREAM IN THE ORNL PILOT PLANT. A. E. Wible. Feb. 27, 1952. Decl. Feb. 13, 1957. 12p. $3.30(ph OTS); $2.40(mf OTS).
Holdup in the ICU continuous evaporator is equivalent to about $1\frac{1}{2}$-hr. production, and control is fully automatic. At 20% excess boil-up the evaporator recovers from sudden purges of feed in a reasonable time. Increasing the boildown required at the same U throughput has no effect on operation. A continuous chart record of product specific gravity at steady conditions showed fluctuations of ± 0.01 specific gravity units around a given set point. The equipment is described in detail. (J.E.D.)

5333 HW-58970
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
COMPOSITION OF SOLIDS FROM PUREX 1WW. H. H. Van Tuyl. Jan. 22, 1959. 9p. Contract [W-31-109-Eng-52]. $1.80(ph OTS); $1.80(mf OTS).
The solids in Purex 1WW were first observed during flowsheet tests for recovery of fission products from plant wastes. Since the nature of this solid was not apparent from the flowsheet composition of 1WW, some work was performed to characterize this material. Although this work was conducted over a period of about one year, it has been subordinate to the main one of testing flowsheets for fission product recovery. The solids have been observed in each of about six samples of plant 1WW that have been studied, and the centrifuged volume of solids has been about four percent in each case. (auth)

(SPECIAL) PROCESSES 6.17

PUREX 6.17.17

REAGENT ADJUSTMENT AND RECOVERY 6.17.17.07

14387 ✓ HW-50379
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
PUREX SOLVENT WASHING WITH BASIC POTASSIUM PERMANGANATE. G. L. Richardson. May 29, 1957. Decl. May 5, 1959. 6p. Contract [W-31-109-Eng-52]. $1.80(ph), $1.80(mf) OTS.

Purex Plant solvents were used in studies to investigate the fission product decontamination achieved by $KMnO_4$ scavenging of used solvent. Major variables studied were $KMnO_4$ concentration, volume ratio, successive scrubbing, temperature, and contact times. The distribution ratio of $KMnO_4$, the effective life of the scavenging solution, and the results of re-using the scavenged solvent in the Purex process were measured. (J.E.D.)

254 ✓ DP-333
Du Pont de Nemours (E. I.) & Co. Savannah River Lab., Aiken, S. C.
CONTINUOUS SOLVENT WASHING—HYDRAULIC OPERATING CHARACTERISTICS OF A LARGE-SCALE CONTINUOUS SOLVENT WASHER. Albert A. Kishbaugh. Apr. 1959. 17p. Contract AT(07-2)-1. OTS.

A large-scale continuous washer for Purex solvent was constructed from a design based upon tests on a laboratory-scale unit. The hydraulic operating characteristics and entrainment data were in reasonable agreement with the predicted performance. (auth)

360
RADIATION EFFECTS IN THE CHEMICAL PROCESSING OF FAST OXIDE BREEDER FUEL. Samuel S. Jones. May 31, 1956. Decl. Mar. 12, 1957. 19p. (KAPL-M-SSJ-3)

In processing spent fuel from the KAPL Plutonium Power Breeder Reactor in the Purex Process the effect of radiation damage becomes prominent. The effect of the radiation on the following process factors is discussed: temperature, gas evolution, viscosity, corrosion, solute oxidation states, solvent deterioration, and phase interface behavior.

13645 ✓ CF-56-1-18
Oak Ridge National Lab., Tenn.
ESTIMATION OF ACTIVITY IN HIGH LEVEL PUREX FEEDS. E. D. Arnold. Jan. 4, 1956. Decl. Mar. 13, 1957. 4p. Contract [W-7405-eng-26]. $1.80(ph OTS); $1.80(mf OTS).

Fission product activity levels and solvent damage rates for high level Purex solutions resulting from processing fast reactor fuels are estimated. (C.W.H.)

A TEST OF THE $Ca(OH)_2$ SOLVENT RECOVERY SYSTEM ON PUREX FIRST URANIUM CYCLE SOLVENT. P. A. Haas. June 11, 1953. Decl. Feb. 14, 1957. 9p. (CF-53-6-110)

A vertical solid-liquid mixer-settler developed for semi-continuous recovery of used TBP solvents by contacting the solvent with a $Ca(OH)_2$ slurry effectively removed large amounts of crud forming materials present in used Purex first U cycle solvent from Purex Pilot Plant run HCP-20. This was evidenced by the $Ca(OH)_2$ shake test which indicated by the clarity of the solvent phase and lack of crud formation at the interface that the solvent had been stripped of all crud formers. This system for Purex solvent recovery is thus recommended for consideration as an alternative to the carbonate wash system for crud former removal. Decontamination of the Purex solvent was low. Irreversible combination of radioactive ions occurred with solvent degradation products which continued to build up during the two-month period following the Pilot Plant run and within which this test was made. Further chemical evaluation is required before a satisfactory indication can be obtained of the decontaminability of the $Ca(OH)_2$ procedure, and before a recommendation can be made concerning use of this system for decontamination. Ru and Zr were the chief activities left in the treated solvent. No I was detected in the solvent at the time of the test.

11610 ✓ CF-53-3-102
Oak Ridge National Lab., Tenn.
USE OF "HOT" SOLVENT IN THE PUREX SECOND URANIUM CYCLE. R. W. Vest. Mar. 11, 1953. Decl. Feb. 16, 1957. 16p. Contract [W-7405-eng-26]. $3.30 (ph OTS); $2.40(mf OTS).

Laboratory investigation of the possibility of using first-cycle solvent in the second U cycle has yielded results which

(SPECIAL) PROCESSES 6.17

PUREX 6.17.17

REAGENT ADJUSTMENT AND RECOVERY 6.17.17.07

indicate that the present dual-solvent recovery system is necessary if product activity specifications are to be met, unless a new solvent recovery system which will more effectively remove the fission products from the organic waste streams is developed. (auth)

11568 ✓ HW-26517
Hanford Works, Richland, Wash.
NITRATION REACTIONS OF SHELL SPRAY BASE UNDER PUREX WASTE RECOVERY PROCESS CONDITIONS. R. M. Wagner. Dec. 5, 1952. Decl. Mar. 4, 1957. 13p. Contract W-31-109-Eng-52. $3.30(ph OTS); $2.40(mf OTS).

Experiments were performed to characterize the types of nitro compounds that might be formed by reaction of Shell Spray Base with nitric acid, to determine the extent of their formation, if any, and their path under conditions of nitric acid recovery in the Purex Process. It is concluded that the instability of Shell Spray Base to nitration, under conditions to prevail in the plant, presents neither a disposal problem nor a safety problem. (auth)

9661 ✓ ORNL-1138
Oak Ridge National Lab., Tenn.
THE HYDROLYSIS OF TRIBUTYL PHOSPHATE AND ITS EFFECT ON THE PUREX PROCESS. V. J. Reilly. Dec. 13, 1951. Decl. Mar. 2, 1957. 22p. Contract W-7405-eng-26. $0.30(OTS).

The rate of hydrolysis of TBP and the effect of the hydrolysis products in the Purex Process have been studied. Hydrolytic conditions may be encountered in the process which would lead to formation of dibutyl phosphoric acid, causing significant losses of Pu^{4+} in stripping. This situation may be easily alleviated by reducing and stripping the Pu^{3+}. (auth)

SOLVENT EXTRACTION 6.17.17.08

A STABLE DILUENT FOR PUREX PROCESS EXTRACTANTS. Henry Pollock. June 1958. 8p. (DP-294)

The chemical stability and physical properties of n-dodecane make it a superior diluent for extractants such as are used in the Purex Process. The chemical stability of commercial n-dodecane can be increased by purification with synthetic zeolites (molecular sieves). 2 references.

19814 HW-34501
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
CHEMICAL STABILITY OF PUREX AND URANIUM RECOVERY PROCESS SOLVENT. R. H. Moore. Mar. 2, 1955. Decl. July 15, 1959. 15p. Contract [W-31-109-Eng-52]. $3.30(ph), $2.40(mf) OTS.

The desirability of operating the Purex columns at higher temperatures, 50 to 70°C, made it necessary to obtain data on the stability of solvents at these higher temperatures. Since the present diluent will not be entirely safe at the upper temperature limit, a number of diluents with higher flash points were investigated. (J.E.D.)

THE APPLICATION OF MIXER-SETTLERS TO THE PUREX PROCESS. J. K. Davidson, A. C. Schafer, Jr., and W. O. Haas, Jr. May 1, 1957. p.130-151 of SYMPOSIUM ON THE REPROCESSING OF IRRADIATED FUELS HELD AT BRUSSELS, BELGIUM, MAY 20-5, 1957. (TID-7534(Bks. 1-3); KAPL-1809)

Considerable experience with mixer-settler equipment of several sizes has demonstrated the applicability of this type of contactor to the Purex process. It has been found that the design considerations and operating characteristics which apply to Purex apply equally well to other systems. Experience with Redox, for instance, is similar to that with Purex. For this reason, description of design considerations will be covered in general terms without specific reference to the solvent system. 26 references.

313 ✓
APPLICATION OF THE PULSE COLUMN TO THE PUREX PROCESS. R. G. Geier. May 1, 1957. p.107-29 of SYMPOSIUM ON THE REPROCESSING OF IRRADIATED FUELS HELD AT BRUSSELS, BELGIUM, MAY 20-5, 1957. 1165p. (TID-7534(Bks. 1-3); HW-49542A)

General aspects, including the effects of factors influencing performance, of pulsed solvent extraction columns are described. The original design concept, i.e., H.T.U. requirements, column heights, cartridge geometry, and expected operable volume velocities of the pulse columns for use in the Purex process is presented. Subsequent improvements in column internals resulting from development studies directed toward extending the range of column operability by increasing amplitude-frequency limitations and/or reversing the choice of continuous phase are also included.

(SPECIAL) PROCESSES 6.17

PUREX 6.17.17

SOLVENT EXTRACTION 6.17.17.08

5295 HW-40322
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
PUREX PLANT SMALL PULSE GENERATOR OPERATION. P. B. McCarthy. Dec. 7, 1955. 9p. Contract [W-31-109-Eng-52]. $1.80(ph OTS); $1.80(mf OTS).
The leakage rate past the piston of a small Purex pulse generator is not affected significantly by pulse frequency. With the piston weep hole plugged, about 0.05 gal./min. leaks by the piston. With a 3/64-in.-diameter weep hole, the leakage rate does not exceed 0.20 and 0.22 gal./min. for organic (30 vol. % TBP in hydrocarbon diluent) and water, respectively. Not more than one standard cfm of air is required to maintain a static balance (no liquid leakage past the piston) between the pulse leg and the pulse column. All air in the pulse leg is vented to the column in less than 3 min. (auth)

PUREX PULSE GENERATOR OPERATION. P. B. McCarthy. May 11, 1955. Changed from OFFICIAL USE ONLY May 16, 1957. 11p. (HW-36836)
A large (Size 2) Purex pulse generator was installed in the 321 Building Tank Farm to pulse solutions in the prototype Purex HA Column. Flow sheet considerations indicated a need for information on leakage rates with various size weep holes in the pulse generator piston. In addition to leakage tests, experiments were conducted to determine the air required under the piston to hold the column contents away from the pulse generator piston thereby preventing leakage during shut down and to determine the time required to bleed the air trapped in the pulse leg when the column is first filled.

PUREX 2A COLUMN INTERFACE CONTROL COMPONENT SPECIFICATIONS. R. E. Smith. Mar. 29, 1954. 6p. (HW-31429)

THE USE OF PHOSPHATE TO SUPPRESS EXTRACTION OF NIOBIUM AND ZIRCONIUM IN A MODIFIED PUREX PROCESS SYSTEM. Report of Invention. Eugene E. Voiland. June 16, 1953. Decl. Apr. 4, 1957. 2p. (HW-28405)

3487 CF-51-10-28
[Oak Ridge National Lab., Tenn.]
PUREX PROCESS. VAPOR PRESSURE OF AMSCO 123-15 AND OF 30% TBP IN AMSCO 123-15. L. E. Line, Jr. Oct. 3, 1951. Decl. Dec. 15, 1955. 5p. Contract [W-7405-eng-26]. $1.80(ph OTS); $1.80(mf OTS).
Since Amsco 123-15 diluent is a potential industrial hazard, the vapor pressures of 100% Amsco 123-15 and 30% tributyl phosphate – 70% Amsco 123-15 were determined over the range 20 to 185°C. Data are tabulated and graphed. (C.H.)

1539 HW-38263(Del.)
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
THE STABILITY OF PUREX SOLVENT TO RADIATION AND CHEMICAL ATTACK. J. L. Swanson. May 24, 1955. Decl. with deletions Aug. 25, 1959. 30p. Contract W-31-109-Eng-52. OTS.
The effects of variables on the rate of Purex solvent deterioration were investigated with the emphasis on those deterioration products which cannot be removed by carbonate washing. The deterioration rate was found to be directly proportional to the acid and nitrate ion concentrations, and proportional to the square root of the concentration of nitrite ion. Other observations on the effects of temperature, relative merits of brand name solvents, and the effects of radiation are included. A method of calculating the equilibrium level of the solvent deterioration products based on study conclusions and various estimates is outlined. (J.R.D.)

WASTE 6.17.17.09

12430 HW-58587
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
THE PILOT PLANT DENITRATION OF PUREX WASTES WITH FORMALDEHYDE. T. F. Evans. Feb. 23, 1959. 32p. Contract W-31-109-Eng-52. $1.00(OTS).
The reaction between formaldehyde and nitric acid, in which the acid is destroyed with the production of predominantly gaseous products, has been recognized as of great potential value in the processing of radioactive fuels, particularly during waste treatment. Laboratory studies of the reaction at Harwell and at Hanford have shown that a major fraction of the nitric acid can be

PUREX 6.17.17

WASTE 6.17.17.09

readily removed from an acidic solution containing nitrates by the addition of formaldehyde. The process possesses the advantages of low chemical cost; recoverability of nitric acid; and, in the case of waste treatment, the production of a solution relatively low in inert salt concentration suitable for fission product recovery or ultimate disposal. The primary purpose of the study was to confirm and extend existing information on the formaldehyde reaction to the destruction of nitric acid in Purex type waste (1WW) through operation of pilot plant scale apparatus. Operational behavior, formaldehyde utilization efficiency, and safety considerations were particular subjects of study. In addition, destruction of nitric acid in a Darex-type dissolver solution was investigated. (auth)

DP-291

Du Pont de Nemours (E. I.) & Co. Savannah River Lab., Augusta, Ga.

ADSORPTION OF RUTHENIUM BY ION EXCHANGE RESINS. Charles A. Prohaska. June 1958. 16p.

After a preliminary treatment with thiourea, at least 90% of the ruthenium remaining in the product uranium stream from the Purex Process can be removed by ion exchange.

1174

RECOVERY OF RADIOACTIVE CESIUM AT HANFORD. B. F. Judson, R. L. Moore, H. H. Van Tuyl, and R. W. Wirta (General Electric Co., Richland, Wash.). Chem. Eng. Progr. 55, Symposium Ser. No. 23, 1-4(1959)

The raffinate stream from the Purex plant is neutralized with caustic and treated with gaseous ammonia which precipitates the iron hydroxides. Slow precipitation of the iron carries essentially all the fission products except cesium. After centrifugation and disposal of the cake, the pH of the solution is adjusted and the cesium is precipitated as $Cs_2ZnFe(CN)_6$. After washing, the cesium compound is hydrolyzed by steam. Cesium is water-leached from the solids and evaporated in the presence of chloride ions to produce a stable cesium chloride powder. The conversion of an unused chemical reprocessing plant for the recovery of cesium is discussed. Economic analyses indicate that bulk quantities of cesium-137 could be made available at prices as low as 50 cents/curie. (C.J.G.)

12243 HW-51399

General Electric Co. Hanford Atomic Products Operation, Richland, Wash.

CALCULATION OF PUREX A-8 CRIB CAPACITY. H. L. Brandt. July 9, 1957. 10p. Contract [W-31-109-Eng-52]. $1.80(ph), $1.80(mf) OTS.

Strontium-90 was found to be the critical isotope for disposal purposes in the A-8 crib waste. A linear relationship was found between the probability distribution of C/C_0 for Sr^{90} and the logarithm of the volume of A-8 waste run through 80 cm laboratory soil columns. Owing to the inaccuracy of Sr^{90} analyses in concentrations less than 4.5×10^{-6} μc/cc, an extrapolation of the breakthrough curve was required to the limiting concentration of 8×10^{-8} μc/cc to determine column capacity. If it may be assumed that the observed linear relationship is independent of the length of the soil column, a method becomes available for calculating the usable capacity of the 200-foot column of soil underlying the A-8 crib. Calculations by this means show that 285 million gallons of A-8 waste may be discharged to this crib before the Sr^{90} would exceed 8×10^{-8} μc/cc in the ground water. (auth)

12242 HW-49728

General Electric Co. Hanford Atomic Products Operation, Richland, Wash.

THE EFFECT OF GROUND-WATER MOUNDS ON THE PUREX OPERATION. William H. Bierschenk. Apr. 18, 1957. 18p. Contract [W-31-109-Eng-52]. $3.30(ph), $2.40(mf) OTS.

A review of the changes in the size and shape of the ground-water mounds consequent upon disposal of cooling water from both the former B Plant operation and the present Purex operation is reported. The results of pertinent ground-water and waste monitoring studies and recommendations and a forecast of future hydrologic conditions attendant upon disposal of Purex cooling water are summarized. Channels of rapidly moving

(SPECIAL) PROCESSES 6.17

PUREX 6.17.17

WASTE 6.17.17.09

ground water were identified flowing eastward along the northern and southern flanks of Gable Mountain and flowing southeastward from Chemical Separations Areas toward 300 Area. Contaminated ground water from beneath 200-East disposal cribs routed to these channels would be rapidly transported to the Columbia River.

WASTE PROCESSING DEVELOPMENT. p. 32-7 of PROGRESS REPORT, MAY 16-NOVEMBER 15, 1955. Decl. Apr. 8, 1957. 74p. (BNL-380)

The following topics are presented. Tank Surge Test Experiment using a simulated Savannah River Purex-type waste; Resistivity of waste streams to current heat generations; metallic corrosion tests; melt corrosion studies; scaling characteristics of wastes; and Ru electroplating studies.

FIELD CORROSION TESTS IN PUREX ACID URANIUM AND WASTE CONCENTRATORS. N. D. Groves and K. M. Haws. June 28, 1956. 10p. (HW-42884)

Field corrosion test heat exchangers have been installed in the F-6, F-11, H-4, J-8, and K-4 concentrator overflow chambers in the Purex facility. The test units are sealed down, single tube calandria type heat exchangers operating in parallel and under the same conditions as the production units. These units will be removed for examination just before or just after failure. The tests are designed to provide corrosion data on 304-L and 347 stainless steel, 308-L and 347 welding alloys, plus 3 candidate construction materials: 312 stainless steel, Ta, and Ti in Purex acid, U, and waste concentrators.

PUREX WASTE STORAGE. PART 1. 241-A WASTE STORAGE FACILITIES. G. L. O'Neill and W. H. Swift. Mar. 7, 1956. 20p. (HW-41791(Pt.1))

Storage of the fission products separated from the product streams of the Purex process is being accomplished using a smaller volume of accompanying solution than any other process here-to-fore used at HAPO. The operating technique and control mechanisms which are needed to store large quantities of these highly radioactive wastes are not as yet fully understood, but considerable insight into the problem has been gained from the experience at Redox during the last 36 months. The basic intentions of the 241-A Storage Facility design is to control the boiling wastes by providing suitable tanks to contain the liquid and a vapor system provided with suitable seals to control the vapors. This document (Part I) will present a somewhat detailed description of the Purex Storage Facility

PRODUCTION OF ACIDITY IN STORED WASTE. L. L. Burger. Oct. 26, 1955. 3p. (HW-39658)

Nitrogen fixation in the air by radiation, highest in the case of Purex wastes, should contribute at most a few millimoles of acid per liter of solution per year. An air rate of 5×10^{-6} cfm per liter of waste would introduce acidity from the CO_2 present at a rate of 1.7 millimoles per liter per year, also an insignificant rate. More acidity may conceivably be produced by radiation decomposition of organic matter present.

1166 ✓ HW-54624

General Electric Co. Hanford Atomic Products Operation, Richland, Wash.

PROPOSAL FOR PLANT-SCALE SUBMERGED COMBUSTION TEST. G. Rey and R. J. Sloat. Sept. 16, 1958. 21p. Contract W-31-109-Eng-52. OTS.

With a 1×10^6 Btu/hr submerged combustion concentrator installation, reproducible de-entrainment factors of greater than 4×10^5 were routinely achieved. On the basis of the encouraging test results, the installation of a plant scale 5×10^6 Btu/hr prototype burner for coating waste concentration in a 750,000 gallon waste tank is proposed. As presently visualized approximately 2.3×10^6 gallons of Purex coating waste could be processed through the initial prototype installation. (W.D.M.)

A LABORATORY STUDY OF THE EXTENT OF PITTING AND GENERAL CORROSION OF SAE-1010 STEEL IN SIMULATED NEUTRALIZED PUREX PROCESS WASTE SOLUTION. N. Endow and Kenneth L. Sanborn. Aug. 6, 1954. Decl. Feb. 20, 1956. 18p. (HW-32734)

The corrosion rates of SAE-1010 carbon steel in neutralized concentrated Purex process waste media were investigated as a function of time in laboratory corrosion tests at 220°F. In this series of tests: (a) polished coupons were exposed for periods of one, two and three months in the vapors over the simulated waste solution; (b) coupons

PUREX 6.17.17

Waste 6.16.17.09

prepared with a fine sandblasted surface were exposed for periods of one, two, three, and ten months in the vapors over the solution; and (c) coupons bearing a normal sandblasted surface were exposed for one, two, and three month periods directly in the solution. The results of these tests indicate: SAE-1010 steel exposed to vapors over neutralized Purex waste solution is subject to rather severe initial pitting attack, but that the rate of attack decreases rapidly with time. The average pitting rates after one, three and ten months exposure were 0.002, 0.0007, and 0.0004 inches per month, respectively; there is no significant difference between a polished steel surface and a fine sandblasted surface in their resistance to pitting type attack in the vapor environment; the general corrosion rates for steel exposed to the vapor, calculated on the basis that attack is uniform over the surface, are less than 0.0001 inches penetration per month for both types of steel surfaces; and that the general corrosion rates for SAE-1010 mild steel exposed in the liquid/sludge are less than 0.0001 ipm. No pitting was evident on the sandblasted surfaces and no cracks or other effects were noted.

237 HW-37642
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
FIELD CORROSION TESTS IN REDOX AND PUREX UNDERGROUND WASTE STORAGE TANKS. Norman D. Groves, Morris C. Fraser, and William L. Walker. June 28, 1955. Changed from OFFICIAL USE ONLY Mar. 22, 1957. 10p. Contract W-31-109-Eng-52. $1.80(ph OTS); $1.80(mf OTS).

A corrosion-testing program has been initiated in Purex and Redox storage tanks to obtain corrosion data on carbon steel and three associated materials in neutralized process wastes. (C.W.H.)

10833 HW-31405
Hanford Atomic Products Operation, Richland, Wash.
FOUR AND ONE-HALF MONTH CORROSION TESTS OF SPRING STEEL WIRE IN SIMULATED NEUTRALIZED PUREX PROCESS WASTE SOLUTION. N. Endow. Apr. 12, 1954. Decl. Feb. 1, 1956. 10p. Contract [W-31-109-Eng-52]. $1.80(ph OTS); $1.80(mf OTS).

Corrosion rates of 1×10^{-5} to 4×10^{-5} inch/min. were obtained for steel wire exposed to Purex waste solutions at 230°F. There was no measurable decrease in the tensile properties and no cracks were noted in the tested specimens. (C.W.H.)

436
RUTHENIUM DISTILLATION IN PUREX ACID WASTE EVAPORATION. J. R. Flanary and V. J. Reilly. July 20, 1953. Decl. Feb. 14, 1957. 8p. (CF-53-7-130)

Ru has been found to distill to a significant extent from boiling 7 N HNO_3. The formation of distillable Ru at this acidity requires an appreciable length of time, but is noticeable in 5 to 10 hours. There is some evidence from this work that Ru distills more readily from dilute acid containing $Fe_2(SO_4)_3$, H_2SO_4 and $NaNO_3$, the non-volatile components of Purex acid wastes.

DISTILLATION OF PUREX WASTES. R. G. Mansfield. Apr. 24, 1953. Decl. with deletions Mar. 5, 1957. 15p. (ORNL-1472(Del.))

Experiments on distillation of Purex waste containing fission products showed that the activity in the distillate is due principally to volatilized Ru. This can be minimized by keeping the still-pot HNO_3 concentration below 9M.

13007 KLX-1610
Vitro Corp. of America, New York.
HOMOGENEOUS REACTOR PROCESSING PROGRESS REPORT FOR FEBRUARY 1953. Job 57. Mar. 10, 1953. Decl. May 13, 1957. 11p. Contract AT(11-1)-217. $1.80 (ph OTS); $1.80(mf OTS).

In attempts to develop a better method of adsorbing fission-product poisons than the untreated fluorite method, studies were continued on the batch-scale removal of β-radioactive material from Purex wastes by adsorption on $Pb_3(PO_4)_2$, CaF_2, $LiAlO_2$, and PbO. (B.J.H.)

MULTI-STAGE EVAPORATION OF PUREX ACID WASTES. V. J. Reilly. Jan. 4, 1952. Decl. Feb. 14, 1957. 12p. (CF-52-1-22)

This report discusses the over-all decontamination factors obtainable in two or more stages of evaporation where no entrainment separator is used.

(SPECIAL) PROCESSES 6.17

RALA 6.17.18

For isolation and recovery of Ba^{140} from irradiated MTR fuel assemblies by precipitation and ion exchange.

See also, this section,
ION EXCHANGE--Equipment (6.12.01),
Exchangers (6.12.02),
PRECIPITATION (6.14),
and,
under FISSION PRODUCT, POISON, AND RADIOISOTOPE REMOVAL,
BARIUM (2.04).

2030 LA-2034
Los Alamos Scientific Lab., N. Mex.
FABRICATION PROCEDURE FOR RaLa SOURCE FUEL RODS. J. E. Hockett, J. D. Duncan, D. T. Doll, J. M. Taub, T. L. Crisler, and D. S. Fisher. Feb. 15, 1956. Decl. Apr. 2, 1957. 65p. Contract W-7405-eng-36. $0.50(OTS).

A new method to procure RaLa sources was proposed which depended upon irradiation of small-diameter U^{235} wire in the MTR. A procedure for cladding and sealing U^{235} wire in stainless steel tubing, by drawing, plugging the tube ends, and welding, was developed, tested, and proved to be satisfactory. The stainless steel-clad U^{235} wire was then encased in 2S Al tubing. A complete fuel rod assembly was fabricated by drawing a larger 2S tube over the inner 2S-stainless steel-U^{235} assembly. The outer 2S tube was then machined to provide longitudinal flutes. All bonds at metal-to-metal interfaces were determined to be satisfactory by radiographic examination. (auth)

13206 ✓ IDO-14348
Phillips Petroleum Co. Atomic Energy Div., Idaho Falls, Idaho.
RADIATION DAMAGE PROBLEMS IN THE ICPP BARIUM-140 SEPARATION PROCESS. R. L. Andelin. Nov. 1, 1955. Decl. Feb. 23, 1957. 72p. Contract AT(10-1)-205. $7.80(ph OTS); $3.30(mf OTS).

The feasibility of separating Ba^{140} from spent MTR fuel assemblies (activity 10^5 curies) by the modified RaLa Process was demonstrated. Radiation damage to process components and solutions was negligible. (C.W.H.)

2268 IDO-18004(Del.)
Blaw-Knox Co. Chemical Plants Div., Idaho Falls, Idaho.
IDAHO CHEMICAL PROCESSING PLANT DESIGN AND OPERATIONS MANUAL FOR RALA PROCESSING — L CELL. Nov. 1, 1955. Decl. with deletions Sept. 1957. 181p. Contract AT(10-1)-728. (BKC-4289(Del.).)

The RaLa Process was developed for the isolation and recovery of Ba^{140} from irradiated MTR fuel assemblies. Equipment was designed to process one MTR fuel assembly every 30 days and to recover approximately 50,000 c of Ba^{140}/mo. Provisions were also made to transfer the solution containing the uranium from the fuel assembly to a cell for U recovery, and holdup capacity has been provided for solutions containing Sr^{90} so that this fission product can also be recovered if desired after a suitable decay period. Equipment and operation procedures are described. 29 figures. (C.H.)

7556 ✓ ORNL-1148
Oak Ridge National Lab., Tenn.
LABORATORY DEVELOPMENT OF THE MTR-RaLa PROCESS FOR THE PRODUCTION OF BARIUM^{140}. R. E. Blanco. Aug. 19, 1952. Decl. Mar. 2, 1957. 70p. Contract W-7405-eng-26. $10.80(ph OTS); $3.90(mf OTS).

13637 ✓ CF-52-1-189
[Oak Ridge National Lab., Tenn.]
A PRECIPITATION PROCESS FOR THE SEPARATION OF RADIOBARIUM FROM IRRADIATED MTR ASSEMBLIES. Quarterly Report [For] November 1951 Through January 1952. J. H. Pannell. Jan. 31, 1952. Decl. Apr. 2, 1957. 15p. Contract [W-7405-Eng-26]. $3.30(ph OTS); $2.40 (mf OTS).

Separation of barium by precipitation has been tested as an alternative to ion exchange in the RaLa Process. The removal of Sr, Ce, and Al have been studied under optimum conditions for recovery and decontamination of Ba. (auth)

(SPECIAL) PROCESSES 6.17

RALA 6.17.18

9659 ORNL-620
Oak Ridge National Lab., Tenn.
LABORATORY DEVELOPMENT OF AN ION EXCHANGE RALA PROCESS. R. E. Blanco. June 2, 1950. Decl. Feb. 28, 1957. 55p. Contract W-7405-eng-26. $9.30(ph OTS); $3.60(mf OTS).

This report presents the laboratory development of an ion exchange process for the purification of radioactive barium. (auth)

REDOX 6.17.19

Solvent extraction method for recovering U and Pu from HNO_3 solution of irradiated uranium. U and Pu (IV) are extracted by hexone with $Al(NO_3)_3$ as the salting agent and stripped separately after the Pu has been reduced to Pu(III).

(See Purex Process)

See also, this section,

SOLVENT EXTRACTION--Solvents--Ketones (6.16.05.04),

and,

under FISSIONABLE MATERIAL--RECOVERY,

RECOVERY OF IRRADIATED TH AND U--Solvent Extraction--Nitrate Systems (3.02.17.04),

RECOVERY OF PLUTONIUM--Reduction (3.03.13),

Solvent Extraction (3.03.14).

GENERAL – FLOWSHEETS 6.17.19.01

33
REDOX PROCESS–A SOLVENT EXTRACTION REPROCESSING METHOD FOR IRRADIATED URANIUM.
S. Lawroski and M. Levenson. May 1, 1957. p.45-68 of SYMPOSIUM ON THE REPROCESSING OF IRRADIATED FUELS HELD AT BRUSSELS, BELGIUM, MAY 20-5, 1957. 1165p. (TID-7534(Bks. 1-3))

The Redox Process is a continuous solvent extraction process which has been developed and used very successfully for several years to recover and purify U and Pu from irradiated natural U fuel. This process uses Hexone (methyl isobutyl ketone) for solvent extraction, $Al(NO_3)_3$ for common ion salting effect, and $Fe(SO_3NH_2)_2$ and $Na_2Cr_2O_7$ for the reduction and oxidation of Pu, respectively.

1761
COMBINATION OF UNIT PROCESSES FOR ISOLATING PLUTONIUM. Jan Rydberg and Lars Gunnar Sillén (Research Inst. of National Defence, Sundbyberg, Sweden and Royal Inst. of Tech., Stockholm). Acta Chem. Scand. 9, No. 8, 1241-51(1955).

The unit processes, reduction-oxidation and separation, which have been compiled from various laboratory and industrial methods used for the separation of Pu from irradiated U and the corresponding flow sheets are discussed. (C.W.H.)

9636 MCW-254
Mallinckrodt Chemical Works, St. Louis.
PRODUCTION OF URANIUM METAL FROM UNH FROM HANFORD SLUGS DECONTAMINATED BY THE REDOX PROCESS. W. H. Keller. Apr. 11, 1950. Decl. Feb. 27, 1957. 10p. $0.25(OTS).

Denitration to UO_3 of decontaminated $UO_2(NO_3)_2$ liquor, reduction of UO_3 to UO_2, fluorination of UO_2 to UF_4, reduction of UF_4 to metal, and casting of the metal in vacuum are described. (W.L.H.)

CORROSION – PROCESS 6.17.19.02

See also, this section,

REDOX--Waste (6.17.19.06).

(SPECIAL) PROCESSES 6.17

REDOX 6.17.19

Corrosion - Process 6.17.19.02

CORROSION OF AUSTENITIC STAINLESS STEEL IN MIXED ACIDS. W. W. Koenig. June 5, 1950. Decl. Feb. 19, 1957. 8p. (HW-17981)

An investigation was made of the corrosion rates of several austenitic stainless steels in simulated Redox Process waste storage solution containing HF, HNO_3, and H_2SO_4 acids. Carpenter 20 and T-309 SCb steels showed good resistance to solutions of two concentrations, while T-304 ELC and T-347 showed poor resistance to one concentration and good to fair resistance to the other.

CORROSION OF STELLITE IN REDOX STREAMS. W. W. Koenig. May 15, 1950. Decl. Apr. 5, 1957. 18p. (HW-17784)

Depositions of Stellite Nos. 1, 6, 6-2A, 12, and 21 on austenitic stainless steel T-347 and depositions of Stellite No. 6 on T-309SCb were found to be resistant to Redox streams at room and boiling temperatures. They were not satisfactorily resistant to boiling 60% HNO_3.

7593 HW-38610(Del.)
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
CORROSION STUDIES CONCERNING THE REDOX F-2 AND D-14 CONCENTRATORS—PHASE II OPERATION. N. D. Groves, M. C. Fraser, and W. L. Walker. Apr. 30, 1956. Decl. with deletions Feb. 3, 1959. 18p. Contract W-31-109-Eng-52.

A laboratory investigation of the potential corrosion problems incurred by the process changes was deemed necessary to provide a basis for estimating the expected service life of the Redox F-2 and D-14 concentrators. The three fold purpose of this laboratory study was to: establish the order of magnitude of the corrosion problem existing in the Redox F-2 and D-14 concentrators, provide preliminary indications of the most suitable material of construction for the replacement of these vessels, and indicate which area of investigation has the greatest potential for solving these corrosion problems. Samples of types 304L, 312, 347 stainless steels were exposed in the sensitized and as-welded conditions to synthetic anticipated Redox process solutions, both as heat transfer surfaces and as static total immersion specimens. Samples of Grade A-55 titanium were similarly exposed. The corrosion rates determined by these experiments indicate, a severe corrosion problem potentially exists in the Redox F-2 and D-14 concentrators, titanium exhibits corrosion rates two orders of magnitude less than the stainless steels tested, type 304L stainless steel welded with type 308L welding alloy exhibit the greatest corrosion resistance of the stainless steels tested, and solution composition has a greater effect on the corrosion rate than the differences between the stainless steels. (auth)

13748✓ HW-14955
Hanford Works, Richland, Wash.
CORROSION INVESTIGATIONS OF REDOX PILOT PLANT EQUIPMENT AT OAK RIDGE NATIONAL LABORATORIES. W. W. Koenig. Nov. 3, 1949. Decl. Feb. 20, 1957. 14p. $3.30(ph OTS); $2.40(mf OTS).

Corrosion encountered in the Oak Ridge pilot plant was small with the exception of the dissolver which showed considerable corrosion of the inside weld and of the welds securing the slug basket to the bottom of the vessel. The slug crash plate and service tubes also showed weld corrosion. (F.S.)

HAZARDS 6.17.19.03

HAZARDOUS MATERIALS—REDOX PLANT. J. W. Underwood. May 22, 1956. 14p. (HW-43319)

Each hazardous material used at the Redox plant is discussed as to toxicity, danger to skin, eyes, lungs, nasal passages, and flammability and explosion hazards. Materials discussed include hexone, hexone-HNO_3 mixtures, hexone-HNO_3-HNO_2 mixtures, HF, H_2, H_2O_2, C_3H_8, HNO_3, Nitrogen oxides, O_3, U, Pu, $Al(NO_3)_3$, $Cr(NO_3)_3$, $Fe(NH_4)_2(SO_4)_2$, $(COOH)_2$, $KMnO_4$, $AgNO_3$, $Na_2Cr_2O_3$, NaOH, $NaNO_3$, NH_2SO_3H, and H_2SO_4. 32 References.

Bureau of Mines.
AMMONIUM NITRATE: ITS PROPERTIES AND FIRE AND EXPLOSION HAZARDS (A REVIEW WITH BIBLIOGRAPHY). G. S. Scott and R. L. Grant. June 1948. 32p. (BM-IC-7463).

(SPECIAL) PROCESSES 6.17

REDOX 6.17.19

10471 HW-55974
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
TEMPERATURE OBSERVATIONS—REDOX PLANT SILVER REACTORS. G. L. Henson. May 7, 1958. 10p. Contract W-31-109-Eng-52. $1.80(ph OTS); $1.80(mf OTS).

The effects of temperature on the Redox Plant silver reactors were studied. The results suggested that better control of radioiodine could be obtained in the silver reactors by operating the reactors at influent gas temperatures not exceeding 375°F during dissolving of the Al jackets, and operating the reactors at influent gas temperatures above 375°F (but below 414°F) during the dissolving of the U metal. In-plant tests were made of silver reactor operation as a function of gas inlet temperature to determine effects on iodine removal efficiency and silver reactor life expectancy. (W.L.H.)

PROCESS DATA AND CONTROL 6.17.19.04

13859
A STUDY OF REDOX POTENTIALS OF PLUTONIUM IN NITRIC ACID. P. I. Artyukhin, A. D. Gel'man, and V. I. Medvedovskii (Inst. of Physical Chemistry, Academy of Sciences, USSR). Doklady Akad. Nauk S.S.S.R. 120, 98-100(1958) May 1. (In Russian)

Formal redox potentials for various plutonium pairs in nitric acid solutions of various concentrations and the disproportion of Pu(IV) in nitric acid solutions were determined. The redox potentials were measured in a wide range of concentrations of Pu(III) and Pu(IV); formal potentials for Pu(IV)–Pu(III), Pu(VI)–Pu(IV) and Pu(VI)–Pu(III) are tabulated. (R.V.J.)

2340 HW-17822
[Hanford Works, Richland, Wash.]
REFRACTIVE INDEX MEASUREMENTS. L. L. Burger. May 17, 1950. Changed from OFFICIAL USE ONLY July 6, 1956. 4p. Contract [W-31-109-Eng-52]. $1.80(ph OTS); $1.80(mf OTS).

Current physical studies of Redox solutions and current metal waste solutions have included a number of refractometric investigations. Aside from information pertinent to the structural nature of solutions these data have revealed that the refractions of a number of constituents in process streams are sufficiently different from those of the solvents that analytical application is possible. The purpose of this note is to summarize some of these data and to point out a few limitations of the method. Refractometric methods for analysis and control in organic chemistry are well known. Lack of extensive use in analysis of inorganic solutions has resulted probably from the lack of specificity for individual ions and to the fact that other methods were available. (auth)

13671 KAPL-328
Knolls Atomic Power Lab., Schenectady, N. Y.
THE DETERMINATION OF PLUTONIUM IN URANIUM RECOVERED BY REDOX. Walter S. Andrus and David J. Fisher. Apr. 11, 1950. Decl. Apr. 4, 1957. 9p. Contract W-31-109-eng-52. $1.80(ph OTS); $1.80(mf OTS).

A modified lanthanum fluoride procedure is described for the determination of total Pu in Redox stream 1EU. This technique is applicable to analyses of ± 5 parts Pu per 10^9 parts U. (C.W.H.)

9747 HW-10960
Hanford Works, Richland, Wash.
CLARIFICATION OF REDOX FEED (1AF) BY FILTRATION. A Semi-Works Study Progress Report. Redox Technical Data Study No. 8. V. R. Cooper and E. M. Coleman. Oct. 15, 1948. Decl. Feb. 2, 1956. 25p. Contract [W-31-109-eng-52]. $6.30(ph OTS); $3.00 (mf OTS).

1901 SOD-19
Standard Oil Development Co., Elizabeth, N. J.
REMOVAL OF NITRIC ACID FROM AQUEOUS REDOX SOLUTIONS BY DISTILLATION. R. C. Morbeck and C. H. Worsham. July 29, 1949. Decl. Mar. 4, 1957. 14p. Contract AT-30-3-GEN-3. $3.30(ph OTS); $2.40(mf OTS).

12998 HW-11885
Hanford Works, Richland, Wash.
NEUTRALIZATION OF HNO_3 IN REDOX ICU AND IAW STREAMS. M. H. Curtis. Dec. 10, 1948. Decl. Feb. 26, 1957. 6p. $1.80(ph OTS); $1.80(mf OTS).

Factors influencing choice of procedure used, hexone removal by evaporation in the presence of a retardant, destruction of retardant, HNO_3 elimination by boiling with Al, and concentration of the remaining solution by evaporation are reported. (J.E.D.)

(SPECIAL) PROCESSES 6.17

REDOX 6.17.19

PROCESS DATA AND CONTROL 6.17.19.04

2382 ✓ HW-10137
Hanford Works, Richland, Wash.
REDOX PROCESS CHEMISTRY. FREEZING POINT DATA FOR UNH–NH_4NO_3–HNO_3–H_2O SYSTEMS. M. K. Harmon and V. R. Cooper. July 7, 1948. Decl. Dec. 2, 1955. 12p. Contract [W-31-109-Eng-52]. $3.30(ph OTS); $2.40(mf OTS).

Data are presented from studies on the freezing point of UNH–NH_4NO_3–HNO_3–H_2O systems. The compositions of the test samples ranged from zero to approximately 55% UNH, for each of a series of four HNO_3 concentrations (0.0, 0.3, 1.0, 3.0M). The NH_4NO_3 concentration in all solutions was as close to 4.0M as possible. Wherever possible, cooling curves were obtained, and the setting point was compared with the melting temperature obtained upon reheating. Where no supercooling occurred, the freezing point was taken as the point of incipient crystallization, determined with the aid of a strong light source. In general, the behavior of the systems investigated was similar to that of those solutions previously studied, and as expected, the curves exhibited approximately the same minimum freezing temperatures (−20°C). Due, however, to the greatly increased amount of solute present, these minima occurred uniformly at much lower UNH concentrations (26 to 30%) than the corresponding solutions containing no NH_4NO_3. With due regard to this shift caused by the presence of NH_4NO_3, the positions of these minima (a characteristic of all solutions below 32% UNH) are governed, as before, by the HNO_3 concentration. Moreover, it can be seen that between 1 and 3M HNO_3, an acid concentration is reached, above which, the minimum temperature is attained when the solution contains no UNH. Any increase of either HNO_3 or UNH in such systems can result only in a decided elevation of the freezing point. (auth)

2346 ✓ CN-2088
[Chicago. Univ.] Metallurgical Lab.
CHEMICAL RESEARCH — BASIC CHEMISTRY OF PLUTONIUM REPORT FOR MONTH ENDING SEPTEMBER 1, 1944. Sept. 21, 1944. Decl. Dec. 13, 1955. 31p. $6.30 (ph OTS); $3.00(mf OTS).

The formation and behavior of Pu^{4+} complexes are studied as a function of pH. The absorption spectrum of $Pu(NO_3)_6^{2-}$ was studied in nitrate solutions. The hydrolytic behavior of Pu^{6+} is compared with that of U^{6+}. A number of experiments were performed on the rates of Pu redox reactions. (K.S.)

SOLVENT EXTRACTION 6.17.19.05

APPLICATION OF THE PACKED COLUMN TO THE REDOX PROCESS. E. R. Irish. May 1, 1957. p.69-82 of SYMPOSIUM ON THE REPROCESSING OF IRRADIATED FUELS HELD AT BRUSSELS, BELGIUM, MAY 20-5, 1957. 1165p. (TID-7534(Bks. 1-3); HW-29967A)

A brief description is presented of the Redox process for Pu and U separation and decontamination from fission products contained in irradiated U fuel elements. Performance characteristics determined for packed solvent extraction columns during development work are discussed. These characteristics are interrelated and include processing capabilities, mass transfer effectiveness, and operating peculiarities. Instrumentation for control and observation of performance is described briefly. Inherent difficulties which may be encountered in such equipment operation can be minimized by adequate operating techniques and equipment design, so that excellent performance may be obtained.

2821 HW-45972
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
A SUMMARY OF THE HISTORY OF HANFORD REDOX PLANT SOLVENT. G. R. Kiel. Oct. 4, 1956. Decl. Mar. 19, 1957. 9p. Contract W-31-109-Eng-52. $0.50(OTS).

THE REACTIONS OF HEXONE IN THE REDOX PROCESS. B. R. Jones and L. L. Burger. Sept. 1, 1950. Decl. Feb. 19, 1957. 22p. (HW-19096)

The initial reaction in a HNO_3–hexone system appears to be the relatively rapid combination of hexone with small amounts of HNO_2 to form an intermediate which then reacts in secondary reactions to produce more HNO_2, resulting in an autocatalytic decomposition of hexone. If the initial HNO_2 concentration is very low, the secondary reactions do not become significant at room temperature.

(SPECIAL) PROCESSES 6.17

REDOX 6.17.19

Solvent Extraction 6.17.19.05

The limiting HNO_3 concentration for a sustained or autocatalytic reaction depends on the temperature. Reagents which produce HNO_2 accelerate the hexone decomposition while any mechanism for removing HNO_2 inhibits the reaction.

BATCH (PSEUDO-) COUNTER-CURRENT EXTRACTION. W. O. Haas. Jan. 14, 1949. Decl. Apr. 4, 1957. 7p. (KAPL-M-WOH-1; MEMO-WOH-1)

Because there has been some question as to the most convenient way to perform laboratory simulated continuous multi-stage extractions, brief outlines and comparisons of several satisfactory methods are given with schematic and flow diagrams for columns 1A and 1C following the Redox Flowsheet.

PROCESS DESIGN SPECIFICATIONS FOR SOD TYPE MIXER-SETTLER CONTACTING UNITS FOR APPLICATION TO REDOX RECOVERY PROCESS. Dec. 31, 1948. Decl. Mar. 5, 1957. 33p. (SOD-17)

WASTE 6.17.19.06

THE RESULTS OF THE D-12 BOIL UP TEST AND RECOMMENDATIONS TO IMPROVE THE PERFORMANCE OF BAYONET TUBE BUNDLES. M. W. Cook. Feb. 7, 1956. 37p. (HW-41295)

Tests performed on the D-12 Redox waste evaporator for evaluation of the bayonet tube bundles as removable expendable heat transfer surfaces are reported. To overcome the difficulty encountered in the production of secondary streams in the condensate, a D-11 condenser was designed and tested.

FIELD CORROSION TEST-SAE 1020 CARBON STEEL IN REDOX PROCESS WASTE SOLUTION TANK NO. 104, 241-S. N. Endow and K. L. Sanborn. Aug. 11, 1954. Decl. Jan. 6, 1956. 24p. (HW-32755)

Data collected in corrosion tests on SAE 1020 steel specimens exposed in the radioactive waste solution from the Redox plant indicate that the corrosion rates do not differ significantly under conditions of self-concentration of wastes, from those measured in similar solutions at temperatures less than boiling. This observation may be considered valid for Redox waste up to 250°F and may apply at temperatures somewhat above 300°F. The maximum temperature recorded for the waste solution in the tank in which the specimens were exposed was 250°F, although temperatures in excess of 300°F may have been attained during the test. One stressed specimen was included in the test for the purpose of evaluating possible effects of stress corrosion cracking, but no evidence of stress corrosion cracking was observed after nine months exposure.

A STUDY OF CERTAIN PROPERTIES OF RUTHENIUM COMPOUNDS FOUND IN THE REDOX PROCESS. W. E. Gill and D. E. Wisehart. June 23, 1954. Decl. Mar. 13, 1957. 8p. (HW-32175)

In a study of the penetration by Ru compounds, silica gel was found to have an efficiency of 99.9 + % for collection of gaseous RuO_4 and droplets and particles from the Ru off-gas line. A temperature of 90 to 95°C did not in itself cause decomposition of RuO_4 when it was distilled from an aqueous solution.

2472 KAPL-391
Knolls Atomic Power Lab., Schenectady, N. Y.
CONCENTRATION OF RADIOACTIVE LIQUID WASTE BY EVAPORATION. G. E. McCullough. Sept. 22, 1950. Changed from OFFICIAL USE ONLY June 26, 1956. 43p. Contract W-31-109-Eng-52. $4.80(ph OTS); $2.70(mf OTS).

This report reviews the progress made since this facility has been in operation, explains how the service building is operated and why it is done this way, and provides a record for reference. In order to segregate and separate Redox wastes and to prepare them for storage and future treatment, five neutralizers have been provided. The neutralizer equipment, duplicated five times in various sizes, consists of a jacketed vessel equipped with an agitator or recirculating pump, a packed column, a condenser, and a receiving tank. The function of this equip-

9624 HW-32175
Hanford Atomic Products Operation, Richland, Wash.
A STUDY OF CERTAIN PROPERTIES OF RUTHENIUM

(SPECIAL) PROCESSES 6.17

REDOX 6.17.19

WASTE 6.17.19.06

COMPOUNDS FOUND IN THE REDOX PROCESS. W. E. Gill and D. E. Wisehart. June 23, 1954. Decl. Mar. 13, 1957. 8p. Contract [W-31-109-eng-52]. $1.80(ph OTS); $1.80(mf OTS).

In a study of the penetration by Ru compounds, silica gel was found to have an efficiency of 99.9+% for collection of gaseous RuO_4 and droplets and particles from the Ru off-gas line. A temperature of 90 to 95°C did not in itself cause decomposition of RuO_4 when it was distilled from an aqueous solution. (F.S.)

1955 HW-18595
Hanford Works, Richland, Wash.
CORROSION OF REDOX WASTE STORAGE TANK CONSTRUCTION MATERIALS. W. W. Koenig and K. L. Sanborn. Aug. 21, 1950. Decl. Apr. 11, 1957. 43p. Contract W-31-109-eng-52. $7.80(ph OTS); $3.30(mf OTS).

Austenitic stainless steel showed excellent corrosion resistance to the IAW Redox Stream (synthetic) at pH = 0, 2, and 10 and should equal or better its performance in the present separations process. The resistance of the mild steel was primarily a function of the pH of the waste streams. Best results were obtained at pH>9.5. None of the protective coatings gave completely satisfactory results under all of the test conditions which were employed. (auth)

SULFEX 6.17.20

For Yankee Atomic Power Reactor SS fuels, selective dissolution of stainless steel cladding by sulfuric acid, with subsequent dissolution of the exposed UO_2 core in nitric acid yielding a concentrated uranyl nitrate solution for Purex solvent extraction.

See also, this section,

PUREX (6.17.17),

and,

under FISSIONABLE MATERIAL--RECOVERY,

HEAD-END TREATMENTS--Stainless Steel Fuel Elements (3.01.09).

2464 ORNL-2785
Oak Ridge National Lab., Tenn.
SULFEX PROCESS: DEPASSIVATION OF STAINLESS STEEL. T. A. Gens. Nov. 20, 1959. 12p. Contract W-7405-eng-26. OTS.

The time required to break the passivation of films on 304 stainless steel produced by 1 hr treatment in refluxing 15.8 M HNO_3–0.025 M Cr(III) decreased as the sulfuric acid concentration increased and increased greatly when nitric acid was added to the sulfuric acid. The films resisted refluxing 6 M H_2SO_4 for 7 min, 6 M H_2SO_4–0.001 M HNO_3 for 2 hr, and 6 M H_2SO_4–0.01 M HNO_3 for several days. In refluxing 12 M H_2SO_4 the films dissolved in about 10 sec with up to 0.001 M HNO_3 in the solution, but 0.5 hr was required with 0.01 M HNO_3. The addition of 0.1 M HCOOH to refluxing sulfuric acid solution effectively destroyed as much as 0.01 M HNO_3. Films on stainless steel specimens in contact with platinum resisted refluxing 6 M H_2SO_4 for several hours. Films on bundles of 304 stainless steel tubes brazed together with Nicrobraze-50 dissolved instantly in refluxing 6 M H_2SO_4. (auth)

258 ORNL-2714
Oak Ridge National Lab., Tenn.
LABORATORY DEVELOPMENT OF THE SULFEX PROCESS FOR THE DISSOLUTION OF CONSOLIDATED EDISON POWER REACTOR FUEL. L. M. Ferris and A. H. Kibbey. Nov. 2, 1959. 25p. Contract W-7405-eng-26. OTS.

Flowsheets given for dissolution of Consolidated Edison power reactor fuel, 96% ThO_2–4% UO_2 sintered pellets clad in type 304L stainless steel, include removal of the stainless steel cladding with a 200% stoichiometric excess of boiling 4 or 6 M H_2SO_4. The ThO_2–UO_2 core is then dissolved in boiling 13 M HNO_3–0.04 M NaF–0.04 M $Al(NO_3)_3$. Essentially complete dissolution of the stainless steel requires about 6 hr in the preferred reagent, 6 M H_2SO_4; only about 80% is dissolved in 6 hr with 4 M H_2SO_4. Uranium and thorium losses to the decladding solution were, in general, less than 0.2 and 0.1%, respectively. After decladding, about 93% of the core is dissolved in 6 to 7 hr when a 200% stoichiometric excess of dissolvent is used; the resulting solution is 1 M in thorium. The

SULFEX 6.17.20

ThO_2-UO_2 heel can be dissolved immediately in about 3 hr in fresh dissolver solution, or can remain through subsequent decladding steps. Aluminum nitrate added to the core dissolver solution to inhibit corrosion does not affect the pellet dissolution rate at concentrations below 0.1 M. The solubilities of the neutron poisons boron and cadmium in process solutions were determined. Boric acid solubility was 0.2 to 0.4 M in most solutions; that of cadmium was 0.2 to 1.2 M. (auth)

9846 ORNL-2461
Oak Ridge National Lab., Tenn.
DEVELOPMENT OF THE SULFEX PROCESS FOR DECLADDING STAINLESS-STEEL-CLAD POWER REACTOR FUEL ELEMENTS WITH SULFURIC ACID. J. R. Flanary, W. E. Clark, J. H. Goode, and A. H. Kibbey. Mar. 30, 1959. 29p. Contract W-7405-eng-26. $1.00(OTS).

A flowsheet, based on laboratory studies, was designed for processing the fuel proposed for the Yankee Atomic Power Reactor by the Sulfex process. This process consists of selective dissolution of the stainless steel cladding by sulfuric acid, with subsequent dissolution of the exposed UO_2 core in nitric acid. With appropriate modifications, the process should be applicable to other stainless steel-clad reactor fuels. With unirradiated fuel specimens, dissolution of type 304L stainless steel cladding proceeded at rates of 2-5 mg/cm^2/min in boiling 4 M H_2SO_4; the rate appeared somewhat lower with irradiated specimens. From 0.01 to 0.02% of the uranium was lost to the decladding solution. The spent decladding solution may be neutralized with lime to form a plasterlike solid waste; open-pit disposal of this waste appears feasible. Subsequent dissolution of the UO_2 core proceeded readily in nitric acid, yielding a concentrated uranyl nitrate solution which may be readily processed in existing Purex solvent extraction facilities. (auth)

3678 CF-58-11-96
Oak Ridge National Lab., Tenn.
MONTHLY PROGRESS REPORT FOR CHEMICAL DEVELOPMENT, SECTION B [FOR] OCTOBER 1958. R. E. Blanco. Nov. 7, 1958. 27p. Contract [W-7405-eng-26]. $4.80(ph OTS); $2.70(mf OTS).

The one-hour dissolution rate of 304L stainless steel in 4 or 6 M H_2SO_4 containing 5 g of stainless steel per liter is less than 0.004 mg/min/cm^2. Head-end treatments of synthetic Consolidated Edison fuel do not remove B or Cd which may be added as homogeneous criticality controls. Washing used solvent with 0.1 M KOH followed by a lime slurry has satisfactorily purified solvent which was more highly degraded than is anticipated in the present power reactor fuel processing program. M-388 alloy (99% Ar–1% Ni) dissolves in boiling NaOH solution at essentially the same rate as pure aluminum. Approximately 20 hours was required for dissolution of EBWR plate in 6 M NH_4F. Further sulfex process corrosion studies were performed on Carpenter 20 Cb stainless steel and type 304 stainless steel. Gas impingement specimens of INOR-1 and -8 exposed for 425 hours in fused 50% NaF–50% ZrF_4 at 650°C under a simulated off-gas consisting of 2.4 wt. % H_2–97.6% HF showed rates of 0.003 and 0.09 mils per month, respectively. When irradiated with 1-Mev electrons, diethyl carbonate decomposes to the extent of 5.5 molecules per 100 ev of energy absorbed. Thorium solubility in Hg was found to increase from 0.0024 to 0.0264 wt. % as the temperature was increased from 40 to 356°C. A flowsheet for the lithium amalgam reduction of UF_6 to U has been prepared and demonstrated on a 10-g U scale. (W.L.H.)

10482
RECOVERY OF URANIUM FROM STAINLESS STEEL FUEL ELEMENTS. Leonard W. Niedrach, Arthur C. Glamm, Mary E. Brennan, and Burton E. Dearing (General Electric Co., Schenectady, N. Y.). Ind. Eng. Chem. 50, 763-6(1958) May.

An investigation was undertaken to provide data required for the "head-end step" of a process for recovering uranium from spent uranium dioxide stainless steel fuel. The step consisted of dissolution of the stainless steel cladding in sulfuric acid, dissolution of the uranium oxide fuel in nitric acid–sulfuric acid mixture, and a single solvent extraction cycle using 10% tributyl phosphate in a hydrocarbon diluent to separate the uranium from the stainless steel components. This treatment was designed to be compatible with further solvent extraction processing to be carried out in the chemical processing facilities at the reactor test station in Idaho. (auth)

(SPECIAL) PROCESSES 6.17

SULFEX 6.17.20

1583 IDO-14493
Phillips Petroleum Co. Atomic Energy Div., Idaho Falls, Idaho.
ANALYTICAL PROGRAM FOR PROCESSING STAINLESS STEEL URANIUM DIOXIDE REACTOR FUEL ELEMENTS.
G. A. Huff. Sept. 18, 1959. 22p. Contract AT(10-1)-205. OTS.

Analytical programs have been described for uranium recovery processes for aluminum-uranium and zirconium-uranium reactor fuel elements. It is the purpose of this paper to describe the analytical program for the uranium recovery process involving stainless steel-uranium reactor fuel elements. The recovery process consists of a two step dissolution and the regular liquid-liquid solvent extraction using TBP in kerosene. Sulfuric acid is used to dissolve the stainless steel. As a result of the sulfuric acid dissolution the uranium is converted to a slurry. The uranium slurry is then dissolved in nitric acid. After the nitric acid dissolution the feed material is compatible with the extraction columns used at the Idaho Chemical Processing Plant for other type fuel elements previously described. In order to analyze samples of dissolver solutions for constituents necessary for plant operation, three new analytical methods were developed. These methods are a rapid method for determining uranium, one for nitrate and one for sulfate. Methods are described for uranium, acidity, specific gravity, nitrate, and sulfate in the dissolver solution; also for small amounts of uranium in the extraction column raffinates. Samples of dissolver solution and first cycle aqueous raffinate contain large amounts of fission products. These samples require shielded facilities for analytical work. The remote analytical facility described by Dykes and his co-workers was used for this work. Precision and accuracy data are given for the analytical methods described. (auth)

25-TBP 6.17.21

Solvent extraction of U^{235} from irradiated enriched uranium--aluminum fuel elements from a nitric acid solution of the fuel by TBP.

See also, this section,
SOLVENTS--TPB - General (6.16.05.08),
and,
under FISSIONABLE MATERIAL--RECOVERY,
AL-U FUELS (3.01.01).

GENERAL - FLOWSHEETS 6.17.21.01

2270 ORNL-1993(Rev.)(Del.)
Oak Ridge National Lab., Tenn.
CHEMICAL DEVELOPMENT OF THE 25-TBP PROCESS.
J. R. Flanary, J. H. Goode, A. H. Kibbey, J. T. Roberts, and R. G. Wymer. Apr. 9, 1957. Decl. with deletions Sept. 18, 1957. 72p. Contract W-7405-eng-26. $12.30(ph OTS); $4.50(mf OTS).

Laboratory development studies leading to a tributyl phosphate extraction process for recovering U^{235} from irradiated enriched U-Al alloy reactor fuel elements are described. The fuel is dissolved in excess HNO_3, and the solution is digested to eliminate emulsion-forming substances and then extracted with tributyl phosphate, $Al(NO_3)_3$ being the salting agent. The U is stripped with dilute HNO_3 and, after evaporation, is re-extracted in a second cycle. Nitric acid is used as the salting agent in the second cycle. The solvent diluent is pretreated to remove impurities. Procedures for recovering the solvent for process reuse and for reducing the volume of the radioactive $Al(NO_3)_3$ containing waste are proposed. (auth)

7504 CF-55-5-137
Oak Ridge National Lab., Tenn.
25 PROCESS ASSISTANCE. For Period of May 2, 1955 to May 13, 1955. F. L. Culler. May 23 1955. Decl. Feb. 14. 1957. 8p. Contract [W-7405-eng-26]. $1.80(ph OTS); $1.80(mf OTS).

Studies were continued on the radiation damage in recycled TBP solvent, organic emulsion formation in Al solutions, chlorination of thermally denitrated thorium nitrate hydrate, and reduction of $ThCl_4$ with sodium amalgam. (C.W.H.)

12991 ✓ CF-55-5-34(Del.)
Oak Ridge National Lab., Tenn.
25 PROCESS ASSISTANCE FOR PERIOD OF APRIL 18,

(SPECIAL) PROCESSES 6.17

25-TBP 6.17.21

General - Flowsheets 6.17.11.01

1955 TO APRIL 29, 1955. F. L. Culler. May 5, 1955. Decl. with deletions Feb. 14, 1957. 8p. Contract [W-7405-eng-26]. $1.80(ph OTS); $1.80(mf OTS).

Results are summarized from studies related to the development of the 25 Process and the Metallex Process. Data are included on the thermal decomposition of thorium nitrate and the chlorination of thorium oxalate using Cl_2 and CCl_4. (C.H.)

4273 HW-19165
Hanford Works, Richland, Wash.
CORROSION OF CONSTRUCTION MATERIALS IN TBP (HW-3 FLOWSHEET) PROCESS STREAMS. W. W. Koenig and K. L. Sanborn. Nov. 1, 1950. Decl. Dec. 8, 1955. 15p. Contract W-31-109-eng-52. $3.30(ph OTS); $2.40 (mf OTS).

A corrosion test program to determine the effect TBP process streams, which contain chlorides, have upon the corrosion resistance of several construction materials, was completed. Data are presented on the performance of Carpenter 20, T-309SCb, T-347, T-316 and tantalum in the RAF and RAW process streams and on the performance of titanium and zirconium in the RAW stream. (auth)

12362 CF-50-2-55
[Oak Ridge National Lab., Tenn.]
PROCESS FOR THE RECOVERY OF U^{235} REACTOR FUEL FOR ARCO CHEMICAL AREAS. C. E. Larson. Feb. 7, 1950. Decl. Feb. 13, 1957. 9p. Contract [W-7405-eng-26]. $0.25(OTS).

A description of the proposed 25 Process for the recovery and decontamination of U^{235} from irradiated reactor fuel elements is presented. Chemical flowsheets are included. (C.W.H.)

8508 MCW-1441
Mallinckrodt Chemical Works. Uranium Div., Weldon Spring, Mo.
DEVELOPMENT OF THE TBP-HEXANE PROCESS FOR URANIUM PURIFICATION. W. C. Philoon, R. M. Edwards, and R. H. Farris. Feb. 1, 1960. 16p. Contract W-14-108-eng-8. OTS.

The development of the TBP–hexane process for the Weldon Spring Plant is described. (W.L.H.)

HEAD-END AND FEED 6.17.21.02

12363 CF-56-3-52(Del.)
Oak Ridge National Lab., Tenn.
25 PROCESS: REMOVAL OF URANIUM OXIDE PARTICLES FROM CAUSTIC DISSOLVER SOLUTION. Terminal Report. T. A. Arehart and C. W. Hancher. Mar. 12, 1956. Decl. with deletions Mar. 14, 1957. 7p. Contract [W-7405-Eng-26]. $1.80(ph OTS); $1.80(mf OTS).

In preliminary studies, small-scale batch centrifugation was a satisfactory method of separating the uranium solids formed in caustic dissolution of Al–U alloy from the suspending medium. Neither continuous centrifugation nor filtration was able to separate the U with losses as low as the desired 0.1%. (auth)

7503 CF-55-4-149
Oak Ridge National Lab., Tenn.
25 PROCESS ASSISTANCE. For Period of April 4, 1955 To April 15, 1955. F. L. Culler. Apr. 26. 1955. Decl. Feb. 14, 1957. 7p. Contract [W-7405-eng-26]. $1.80(ph OTS); $1.80(mf OTS).

Work on organic emulsion formation under 1A column feed-plate conditions was continued. Results of tracer level runs indicate that Zr and Nb were the principal contaminants in the first cycle U product. Thorium oxychloride preparation, thorium nitrate chlorination, thorium(IV) chloride reduction with Mg, and Th dissolution in Hg were investigated. (C.W.H.)

9619 CF-55-4-72(Del.)
Oak Ridge National Lab., Tenn.
25 PROCESS ASSISTANCE FOR PERIOD OF MARCH 21, 1955 TO APRIL 1, 1955. F. L. Culler. Apr. 11, 1955. Decl. with deletions Feb. 14, 1957. 7p. Contract [W-7405-Eng-26]. $1.80(ph OTS); $1.80(mf OTS).

Use of Thorex-type high temperature feed digestion at high HNO_3 concentrations will prevent subsequent organic emulsion formation in the 25 extraction column, even when the dissolver solution is known to contain emulsion-

(SPECIAL) PROCESSES 6.17

25-TBP 6.17.21

HEAD-END AND FEED 6.17.21.02

producing impurities. Gross D.F.'s for three spiked-feed small pulse column runs were at least 3×10^4 β and 5×10^3 γ across both columns. Trickle dissolver tests using 2S Al sheet in the 5-in. D pipe unit yielded a 5 ft packed height for a processing rate of 200 kg Al/day, equivalent to ~20 kg U/day when dissolving SR tubular elements. The packed height dropped to just under 3 ft when the dissolving rate was cut in half. Thorium metal quality studies indicate that metal samples prepared by arc-melting sintered compacts are within target specifications except for the iron content. The Abbie "Dispersall" mixer has been tested briefly for the dry-$ThCl_4$ contacting with NaHg and probably will prove satisfactory for making 0.5 lb batches of Th as quasi amalgam. (auth)

580 CF-54-6-244
Oak Ridge National Lab., Tenn.
THE EFFECTS OF CAUSTIC DISSOLUTION ON THE 25 EXTRACTION PROCESS. J. R. Flanary. June 29, 1954. Decl. Sept. 26, 1958. 11p. $3.30(ph OTS); $2.40(mf OTS).

The dissolution of U–Al slugs in caustic is examined in relation to the 25 Process. Various first-cycle flowsheets are discussed and compared to the current 25 process flowsheet in which nitric acid is used. The flowsheets have not been evaluated. (J.R.D.)

13000 IDO-14204(Del.)
American Cyanamid Co. [Atomic Energy Div.], Idaho Falls, Idaho.
LABORATORY STUDIES OF THE PREPARATION AND USE OF DIBASIC ALUMINUM NITRATE FOR THE "25" PROCESS FEED ADJUSTMENT STEP. R. B. Lemon. Dec. 31, 1952. Decl. with deletions Feb. 27, 1957. 11p. $3.30(ph OTS); $2.40(mf OTS).

Because of the possible advantages of the use of dibasic aluminum nitrate in the Idaho Chemical Processing Plant "25" Process feed adjustment step, studies of its preparation and use were undertaken. This report summarizes the work completed to date on this project. The preparation of dibasic aluminum nitrate using the method recommended by ORNL has been demonstrated on a laboratory scale. A solution approximately 7.5N acid deficient and 4.0M aluminum results from this procedure. When 2S aluminum is used for the preparation, the resulting solution must be filtered to remove the insolubles originally contained in the metal and also to remove metallic mercury which is present in the solution—a result of the reduction of the $Hg(NO_3)_2$ catalyst. It was found that medium or fine sintered glass filters precoated with filter aid have a capacity of approximately 500/ml/in.^{2} for this solution. Dissolver solutions adjusted to 0.2N acid deficiency with dibasic aluminum nitrate have essentially the same filtration characteristics as ammonium-hydroxide adjusted dissolver solution. As found by ORNL and also in this work, no local precipitation occurs when an aluminum nitrate-containing dissolver solution is adjusted to acid deficiency with dibasic aluminum nitrate solution. Possible advantages to the use of dibasic aluminum nitrate solution for the feed adjustment step are elimination of high ammonium nitrate concentrations in the first cycle feed and thus elimination of a possible explosion hazard from the first cycle raffinate evaporators and, as no precipitate is formed upon addition of dibasic aluminum nitrate solution to dissolver solution, the four-hour agitation time required after the adjustment step when ammonium hydroxide is used may be greatly reduced. The principal disadvantages to the use of dibasic aluminum nitrate for feed adjustment are an increase in first cycle aqueous waste volume and a decrease in first cycle capacities. Use of dibasic aluminum for adjusting a 1.0M HNO_3 dissolver solution decreases the capacity for NP and MTR processing 25% and increases the first cycle aqueous waste volume 41%. (auth)

9656 ORNL-302
Oak Ridge National Lab., Tenn.
MANGANESE DIOXIDE AS A FILTER AID IN THE "25" PROCESS. R. E. Blanco. July 27, 1949. Decl. Mar. 4, 1957. 17p. Contract W-7405-eng-26. $3.30(ph OTS); $2.40(mf OTS).

An evaluation of MnO_2 as a filter aid for clarifying 25 Process metal solutions (U–Al fuel rods in HNO_3) is presented. (auth)

(SPECIAL) PROCESSES 6.17

25-TBP 6.17.21

SOLVENT EXTRACTION 6.17.21.03

7531 IDO-14358
Phillips Petroleum Co. Atomic Energy Div., Idaho Falls, Idaho.
EFFECT OF FIRST CYCLE SCRUB RATIO REDUCTION ON DECONTAMINATION IN THE "25" PROCESS. F. P. Schilling. Dec. 1, 1955. Decl. Feb. 27, 1957. 15p. Contract AT(10-1)-205. $0.25(OTS).

The scrub flow ratio in the first cycle of the hexone solvent extraction process for the recovery and decontamination of U from Al–U alloy fuels has been reduced by a factor of 10. A minimum flow ratio of scrub : feed : extractant of 1 : 79 : 67 has been plant tested. Uranium losses were unaffected, and overall fission product decontamination for three cycles was changed only slightly. (auth)

11584 HW-27162
Hanford Works, Richland, Wash.
THE ANALYSIS OF TBP PROCESS STREAMS FOR CALCIUM WITH THE FLAME PHOTOMETER. D. W. Brite. Feb. 20, 1953. Decl. Feb. 6, 1957. 4p. Contract W-31-109-Eng-52. $1.80(ph OTS); $1.80(mf OTS).

PILOT PLANT DESIGN REPORT; TBP PROCESS. R. P. Milford. Jan. 27, 1950. Decl. Mar. 1, 1957. 39p. (ORNL-543)

A set of stainless steel columns and accompanying process equipment, including a product evaporator were installed to demonstrate the TBP process for recovering U metal waste. Tributyl phosphate, diluted with Varsol, is used as an extractant in one cycle of continuous liquid-liquid counter-current extraction scrubbing and stripping. Flowsheets, and assembly of columns are included.

WASTE 6.17.21.04

1868 CF-55-11-97
Oak Ridge National Lab., Tenn.
PROPOSAL FOR "25" OR THOREX WASTE DISPOSAL: SELECTIVE RECOVERY OF FISSION PRODUCTS IN RELATION TO THE LONG-RANGE ASPECTS OF CHEMICAL WASTE ECONOMICS. A. T. Gresky and R. P. Wischow. Nov. 17, 1955. Decl. Mar. 4, 1957. 13p. Contract [W-7405-eng-26]. $3.30(ph OTS); $2.40(mf OTS).

Laboratory data and a preliminary flowsheet for partial recovery of fission products from Thorex and/or "25" process waste streams are presented for evaluation.

7529 HW-27489
[Hanford Atomic Products Operation, Richland, Wash.]
FACTORS AFFECTING THE CONCENTRATION OF TBP PLANT WASTES. D. F. Shepard. Mar. 25, 1953. Decl. Feb. 19, 1957. 11p. Contract [W-31-109-Eng-52]. $3.30(ph OTS); $2.40(mf OTS).

Control of neutralized waste pH at 9.5 has relieved the process difficulty of line plugging in the TBP plant waste disposal. (F.S.)

7477 CF-51-9-115
Oak Ridge National Lab., Tenn.
PRESSURE DROP CALCULATIONS FOR THE OFF-GAS SYSTEM. H. O. Weeren. Sept. 24, 1951. Decl. Feb. 20, 1957. 4p. Contract [W-7405-eng-26]. $1.80(ph OTS); $1.80(mf OTS).

Complete figures are tabulated for the pressure drop through the off-gas system for the 25 Process. (B.J.H.)

2399 HW-18476
Hanford Works, Richland, Wash.
APPARENT VISCOSITY OF NEUTRALIZED AND CONCENTRATED RAW SLURRY–TBP HW-NQ. 4 FLOWSHEET. A. W. Allen. Aug. 15, 1950. Decl. Dec. 3, 1955. 13p. Contract W-31-109-Eng-52. $3.30(ph OTS); $2.40(mf OTS).

Viscosity data for flow in 1-in. iron pipe were obtained for a fission-product and plutonium-free slurry, consisting of simulated TBP Waste Metal Recovery Process

197
RECOVERY OF NITRIC ACID IN THE TRIBUTYL PHOSPHATE PROCESS FOR URANIUM RECOVERY. PART 2. A. CORROSION STUDIES. B. PATH OF CHLORIDE ION. C. PATH OF TRIBUTYL PHOSPHATE. R. M. Wagner and C. Groot. May 29, 1950. Decl. Feb. 19, 1957. 12p. (HW-17937)

The corrosion rates for type 316 stainless steel are given for various parts of the distillation system. The path of TBP in distillation of column waste (RAW) and the path of chloride during RAW concentration and HNO_3 distillation are outlined.

(SPECIAL) PROCESSES 6.17

THOREX 6.17.22

Recovery of thorium and uranium from irradiated metal, Th and U extracted by TBP from HNO_3 solution. U^{233} isolated by ion exchange.

Formerly "Interim-23 Process."

See also, this section,

SOLVENTS--TBP - Nitrate Systems (6.16.05.10),

ION EXCHANGE--Exchangers (6.12.02).

GENERAL - FLOWSHEETS 6.17.22.01

20005
AN EVALUATION OF THE DESIGN AND PERFORMANCE OF THE THOREX PILOT PLANT. Presented at Nuclear Engineering and Science Conference, April 6-9, 1959, Public Auditorium, Cleveland, Ohio. Preprint V-73. W. R. Winsbro and G. S. Sadowski (Oak Ridge National Lab., Tenn.). New York, Engineers Joint Council, 1959. 33p. $0.50.

This paper discusses plant and equipment utilized for pilot plant development of the Thorex process. Included are plant layout, selection of materials of construction, welding criteria, and testing and inspection procedures. Selection criteria and performance of radive process equipment and instrumentation are evaluated. (auth)

ORNL-2591
Oak Ridge National Lab., Tenn.
LABORATORY DEVELOPMENT OF THE THOREX PROCESS. Progress Report [for] December 1, 1955 to January 1, 1958. R. H. Rainey, A. B. Meservey, and R. G. Mansfield. Feb. 27, 1959. 55p. Contract W-7405-eng-26. $1.50(OTS).

The Thorex process has been modified to increase decontamination so that thorium irradiated to more than 2000 g U^{233} per ton thorium and decayed less than 180 days may be recovered. Modifications include the conversion to a codecontamination flowsheet, sweep of I^{131} from the dissolver with iodine carrier, improved ruthenium decontamination by addition of bisulfite to the feed, addition of a U^{233} third-cycle solvent extraction, and improved solvent recovery by use of 0.2 M NaOH treatment. Activity remaining in products from pilot plant experiments processing thorium irradiated to 4000 g U^{233} per ton of thorium, decayed 27 days, by this modified process, exceeded tentative specifications by about a factor of two. (auth)

10975 ✓ CF-58-6-4
Oak Ridge National Lab., Tenn.
THOREX, ANALYSIS OF MODIFICATION COSTS. R. V. Foltz and W. R. Winsbro. June 1, 1958. 30p. Contract [W-7405-eng-26]. $6.30(ph), $3.00(mf) OTS.

Actual design and construction costs and schedules for a group of four short-cool processing modifications to the Thorex Pilot Plant are analyzed and summarized. (auth)

11377 ✓ ✓
THOREX SHORT COOLED PROCESSING. Presented at Nuclear Engineering and Science Conference, held at Chicago, March 17 to 21, 1958. Preprint 122, Session 37. A. B. Meservey and R. H. Rainey (Oak Ridge National Lab., Tenn.). New York, American Institute of Chemical Engineers, 1958. 7p.

The conventional Thorex flowsheet for the aqueous processing of long-decayed neutron-irradiated thorium slugs gives poor decontamination from iodine and ruthenium when short-decayed slugs are processed. Good decontamination is obtained after a head-end treatment with potassium iodide and the addition of sodium bisulfite to the feed. (auth)

9456 BNL-483(p.149-53)
Du Pont de Nemours (E. I.) & Co. Savannah River Lab., Augusta, Ga.
THOREX PROCESS DEVELOPMENT AT SAVANNAH RIVER. T. H. Siddall. p.149-53 [of] THORIUM-U^{233} SYMPOSIUM, SPONSORED BY THE UNITED STATES ATOMIC ENERGY COMMISSION, AT BROOKHAVEN NATIONAL LABORATORY, JANUARY 9-10, 1958. 5p.

Selected topics are discussed in SRL and KAPL development work in the processing of irradiated Th. (W.L.H.)

(SPECIAL) PROCESSES 6.17

THOREX 6.17.22

GENERAL – FLOWSHEETS 6.17.22.01

9457 BNL-483(p.154-6)
Oak Ridge National Lab., Tenn.
PILOT PLANT PROCESSING OF IRRADIATED THORIUM. E. M. Shank. p.154-6 [of] THORIUM–U^{233} SYMPOSIUM, SPONSORED BY THE UNITED STATES ATOMIC ENERGY COMMISSION AT BROOKHAVEN NATIONAL LABORATORY, JANUARY 9-10, 1958. 3p.

A summary is presented of the status of the Thorex program, a review of the process flowsheet as it now stands, a discussion of 30 months of pilot plant operation during the processing of long-decayed and short-decayed thorium, and a summary of the work still required to conclude the development of a short-decay Thorex flowsheet. (auth)

THE THOREX PROCESS. F. R. Bruce. May 1, 1957. p. 180-22 of SYMPOSIUM ON THE REPROCESSING OF IRRADIATED FUEL HELD AT BRUSSELS, BELGIUM, MAY 20-5, 1957. 1165p. (TID-7534(Bks. 1-3))

The Thorex process was developed to recover U^{233} and Th from irradiated Th. It may be used for reprocessing irradiated ThO_2 and ThO_2-UO_2 mixtures as well as the metals. Both Th and U products are sufficiently separated from fission products and Pa to permit their direct handling during fuel element fabrication. The principles of the Thorex process and a flowsheet were described in a paper by A. T. Gresky which appears in "The Proceedings of the International Conference on the Peaceful Uses of Atomic Energy." In this paper the Thorex Pilot Plant operating experience over the last two and a half years will be reviewed, the current flowsheet presented and evaluated, and recent developments in Thorex process chemistry described. 9 references.

THOREX PROCESS SUMMARY, FEBRUARY 1956. A. T. Gresky. Apr. 25, 1956. Decl. Feb. 14, 1957. 201p. (CF-56-2-157)

The Thorex process has the purpose of separating and decontaminating Th, U^{233}, and Pa^{233} from neutron-irradiated Th. Designed principally for the processing of Al-clad Th slugs, the process may be modified for use with other reactor materials, such as ThO_2 or $ThOCO_3$. It is a solvent-extraction process which employs TBP as the extractant, HNO_3 as the Th-dissolution agent, and either $Al(NO_3)_3$ or HNO_3 as the salting agent. Final isolation of the U^{233} product is accomplished by ion exchange.

Oak Ridge National Lab., Tenn.

OPERATING EXPERIENCE WITH TWO DIRECT-MAINTENANCE RADIOCHEMICAL PILOT PLANTS. F. R. Bruce, E. M. Shank, R. E. Brooksbank, J. R. Parrott, and G. S. Sadowski. 51p. (A/CONF.15/P/536)

Performance. Cost. Control.

8730 ✓
OPERATION OF THE THOREX PILOT PLANT WITH HIGHLY IRRADIATED THORIUM. E. M. Shank (Oak Ridge National Lab., Tenn.). pp. 279-301 in "Progress in Nuclear Energy. Series III. Process Chemistry. Volume 2."

The Thorex solvent-extraction flowsheet was converted from a one-cycle to a multi-cycle system to increase the product decontamination when processing highly irradiated thorium. The new "co-decontamination" flowsheet provides two solvent-extraction cycles for thorium and three such cycles plus an ion-exchange step for uranium. The Thorex Pilot Plant is mostly installed in concrete-shielded cells. It is directly maintained and remotely operated. Process equipment is unit-shielded, provisions for equipment decontamination are built in, radiochemical sampling equipment is remotely operated, and concatenated pulsed columns are used for the extraction. The plant has been in operation for 3 years, including 17 months with the co-

HEAD-END AND FEED 6.17.22.02

DISSOLUTION OF ALUMINUM-CANNED THORIUM. John G. Beach, William C. Schickner, and Charles L. Faust. July 26, 1955. Decl. Feb. 13, 1957. 16p. (BMI-1022)

From the standpoint of dissolution characteristics, single-dip-canned Th slugs present a system of four components based on dissolution characteristics: Al, AlSi, and $ThSi_2$ coatings, and the Th core. For reprocessing by the Thorex process, irradiated slugs are dissolved in a

(SPECIAL) PROCESSES 6.17

THOREX 6.17.22

mixture of HNO_3 and HF. Dissolution of the Th core, however, is prevented by the $ThSi_2$ layer, which is not attached by the acid mixture. The acid mixture corrodes type 309 stainless steel equipment, and introduces Cr into the reprocessing solutions. Chemical dissolution of the slug components was slow. Electrolytic dissolution with coated Th as the anode resulted in dissolution rates for the various components several times those rates obtained with simple chemical dissolution. The $ThSi_2$ layer was easily removed. HNO_3 with $Hg(NO_3)_2$ but without HF dissolved the Al can but not the AlSi. $ThSi_2$ and Th were disintegrated by anode electrolyses in HNO_3 alone. In HNO_3–HF, Th dissolved but some of the Th precipitated as ThO. Precipitation was greater with electrolytic dissolution. The results observed in these studies suggest a dissolution cycle that permits separation of the canned-slug components. Stoichiometric use of the acid might be more closely approached and problems of materials of construction could be lessened.

CENTRIFUGATION TESTS [ON] KAPL THOREX HEAD-END. Donald J. Smith. Mar. 30, 1955. Decl. Apr. 5, 1957. 15p. (KAPL-M-DJS-2)

A series of experiments testing the centrifuge properties of MnO_2 cake formed during the KAPL Thorex head-end is described. The effect of varying the acid concentration from 0.25M to 1.00M at 0.8M $Th(NO_3)_4$ and the effect of varying and Th concentration from 0.5M to 1.20M at 0.60M HNO_3 was measured. Centrifugation is satisfactory at greater than 0.5M HNO_3 and less than 1.00M $Th(NO_3)_4$.

439

PREPARATION OF ACID DEFICIENT ALUMINUM NITRATE BY ELECTRODIALYSIS. E. J. Parsi. Apr. 14, 1954. Decl. Feb. 14, 1957. 14p. (CF-54-4-131)

A continuous method for the production of acid deficient $Al(NO_3)_3$ was developed in a semi-pilot scale electrodialysis unit. Using this method a clear solution of 90% monobasic 0.6 M $Al(NO_3)_3$ has been produced at a rate of 0.37 liters/hour. A by-product 0.29 N HNO_3 containing less than 40 ppm Al was also produced at a rate of 0.57 liters/hour. From the data obtained in this run, a preliminary cost estimate for the continuous production of acid deficient $Al(NO_3)_3$ 0.6 M in Al and 0.44 acid deficient was made. It was based on the requirements of the Thorex pilot plant of approximately 24 liters/hour. The cost is largely dependent on the rate of corrosion of the anode and on the labor required. However, on scaling up labor costs will be reduced, but the cost of the anode replacement will not be changed. The cost of a unit to satisfy the needs of the Thorex pilot plant is of the order of $500.00 exclusive of anode costs. Adding up the cost of auxiliary equipment and installation, the total cost of the unit is approximately $3700.00. It will cost $3500.00 to use 0.001 in. Pt foils as anodes. At this low scale of production and assuming a Pt corrosion rate of 0.05 mg/a-hour, the cost per pound of Al is approximately $0.64 of which $0.37 is labor and $0.10 is Pt loss. To this must be added $1.49/lb of Al in the $Al(NO_3)_3$ feed. Scaling up by a factor of 20 over the Thorex pilot plant unit, the installed cost of the unit will be of the order of $43,500, and the investment in Pt about $70,000. Using the same assumption as in the smaller scale unit, the cost per pound of Al produced is approximately $0.35, of which $0.11 is labor and $0.10 is Pt loss. Again the cost of Al in the feed must be added to the above figure. All the equipment was amortized at 16% per annum, except for the ion exchange membranes and gaskets, for which a conservative 200% was used. This method is very attractive, but its economic feasibility hinges heavily on the rate of corrosion of Pt as an anode in HNO_3 or in the development of a suitable substitute anode.

13640 CF-53-3-208

Oak Ridge National Lab., Tenn.

THOREX PROCESS: CONTINUOUS DISSOLVER DEVELOPMENT PROGRAM QUARTERLY REPORT FOR PERIOD NOVEMBER 1, 1952–FEBRUARY 1, 1953. D. L. Foster. Mar. 23, 1953. Decl. Feb. 14, 1957. 11p. $1.80(ph OTS); $1.80(mf OTS).

SOLVENT EXTRACTION 6.17.22.

AECU-4056

Georgia Inst. of Tech., Atlanta, Engineering Experiment Station.

RADIATION CHEMISTRY OF ORGANIC SUBSTANCES. Final Report for January 1, 1957 to January 31, 1959. James A. Knight, Jr. Jan. 31, 1959. 38p. For Oak Ridge National Lab. Project No. A-323. Contract W-7405-eng-26, Subcontract 1082. $6.30(ph), $3.00(mf) OTS.

Radiation damage

Solvent

Thorex

The chemical processing of reactor fuels involves the use of organic substances as extracting agents and as diluents. When most organic substances are exposed to reactor fuels, radiation damage occurs. The objectives

THOREX 6.17.22

SOLVENT EXTRACTION 6.17.22.03

of this project were to provide more information regarding radiation damage to hydrocarbon solvents which are similar to those used as diluents in the Thorex Process and to determine if the radiation-damage diluent has a pronounced deleterious effect upon the extraction process. (W.L.H.)

16740 ✓ AAEC/E-26
Australia. Atomic Energy Commission Research Establishment, Lucas Heights, New South Wales.
DILUENTS FOR THE SOLVENT EXTRACTION OF THORIUM USING TRIBUTYL PHOSPHATE GENERAL CONSIDERATION AND THE THIRD PHASE PROBLEM.
M. S. Farrell and J. D. Goldrick. Sept. 1958. 19p.
An examination of three possible diluents available from Australian sources of supply, has shown that "odorless mineral spirits" from Vacuum Oil Co. Pty. Ltd., is the purest in alkane content and is the most suitable of the three for use with TBP for the chemical processing of irradiated fuel, for example by the Thorex process. A small quantity of sec-octyl alcohol will suppress third phase formation but further work is required to ensure that its use is otherwise compatible with the process. (auth)

7761 ORNL-2473
Oak Ridge National Lab., Tenn.
THOREX PROCESS: THIRD URANIUM CYCLE STUDIES.
J. R. Oliver. Apr. 14, 1958. 16p. Contract W-7405-eng-26. $0.75(OTS).
Laboratory studies indicated that a Thorex third cycle uranyl nitrate feed containing 50 g of uranium per liter should be 0.70 M in $Al(NO_3)_3$ and 0.50 M in HNO_3; the scrub, 0.50 M in $Al(NO_3)_3$; the extractant, 10 vol. % tributyl phosphate (TBP) in Amsco; and the feed/scrub/extractant flow ratios, 1/0.3/1.5. With these conditions the uranium loss was 0.005 g/liter, or 0.01%, in laboratory tests. Decontamination factors were about 600 for ruthenium and 400 for zirconium–niobium. (auth)

13817 CF-58-3-8
Oak Ridge National Lab., Tenn.
HYDROCLONE PERFORMANCE IN THE SOLVENT RECOVERY SYSTEM OF THE THOREX PILOT PLANT.
J. Cerny, III. Mar. 31, 1958. 23p. Contract [W-7405-eng-26]. $4.80(ph OTS); $2.70(mf OTS).
Tests on 1-gpm and 0.5-gpm hydroclones showed them to be unnecessary. Solvent-aqueous phase separation, although effective when optimized, was infrequently required. The 1-gpm hydroclone produced better water separation than the 0.5-gpm hydroclone for similar control parameters. Decontamination factors for widely varying flow conditions ranged from 1 to 3. Introduction of water into the hydroclone feed stream had no effect on decontamination or particle removal. (auth)

THOREX PILOT PLANT RUN AWD-1 SUMMARY. W. T. McDuffee and O. O. Yarbro. Jan. 6, 1958. 5p. (CF-58-1-56)
The processing of short-decayed Th metal results in the collection of first cycle extraction column raffinate which contains 20 to 30% of the original mass^{233} as Pa^{233}. Ultimate recovery of the U^{233} requires storage of the raffinate for decay of Pa^{233}. During the Thorex Pilot Plant short-decay scouting run HD-19, an estimated 27 g of Pa^{233} was collected and stored as extraction column raffinate. The data resulting from the processing (run AWD-1) of the first cycle raffinate from run HD-19 are summarized. A period of 346 days elapsed between the completion of run HD-19 and run AWD-1. A one-cycle solvent-extraction flowsheet was used to separate U^{233} from fission products and other contaminants contained in the raffinate. The U^{233} was extracted into 6% tributyl phosphate in Amsco 125-82 and subsequently stripped into dilute HNO_3. Feed for run AWD-1 was prepared by concentrating and adjusting the 1A-column raffinate from run HD-19 to feed specifications.

CHEMICAL PROCESSING OF ThO_2. E. M. Shank. p. 155-63 of HRP CIVILIAN POWER REACTOR CONFERENCE HELD AT OAK RIDGE NATIONAL LABORATORY, MAY 1-2, 1957. R. B. Briggs, comp. July 1957. 306p. (TID-7540)
The Thorex Pilot Plant has been in operation 28 months and has processed irradiated Th using the originally developed 1-7 cycle flowsheet and a recently developed 2-cycle flowsheet. In addition, a short scouting run with short decayed Th has been made using the 1-cycle flowsheet. While ThO_4 has not been processed, the discussion is directly applicable to ThO_4. The capacity of the pilot plant is sufficient to process the blanket from 3-450 Mw homogeneous reactors.

(SPECIAL) PROCESSES 6.17

THOREX 6.17.22

SOLVENT EXTRACTION 6.17.22.03

12966 CF-56-10-126(Del.)
Oak Ridge National Lab., Tenn.
STATUS REPORT FOR CHEMICAL DEVELOPMENT SECTIONS A AND B [FOR] WEEK ENDING OCTOBER 12, 1956. R. E. Blanco and D. E. Ferguson. Oct. 19, 1956. Decl. with deletions Mar. 13, 1957. 14p. $3.30(ph OTS); $2.40(mf OTS).

Progress is reported on studies related to the processing of the fuel and blanket for the Homogeneous Reactor Test. Data are included from studies on the dissolution rate of Zr in H_2SO_4; radiation effects on samples of dry Amsco 125-82 and Thorex Process solvent; an analysis of UF_4 produced by the Excer Process from Vitro solvent extraction concentrate; and the precipitation of UO_4 from UO_2SO_4 solutions. (C.H.)

12996 ✓ CF-56-9-119
Oak Ridge National Lab., Tenn.
THOREX: SECOND THORIUM CYCLE MANPOWER AND COST SUMMARY. W. R. Winsbro and B. F. Bottenfield. Sept. 24, 1956. Decl. Mar. 13, 1957. 27p. Contract [W-7405-eng-26]. $4.80(ph OTS); $2.70(mf OTS).

A second Th decontamination cycle, consisting of two solvent extraction pulse columns, a natural-convection evaporator stripper, and auxiliary mixing, storage, and surge vessels, was installed. Specifications, scheduling, and design and construction costs are reported. (L.T.W.)

THOREX PILOT PLANT: EVALUATION OF EQUIPMENT PERFORMANCE FOR THE PERIOD OCTOBER 1954 THROUGH MARCH 1955. A. J. Pecquet, W. R. Winsbro, and G. S. Sadowski. Oct. 14, 1955. Decl. Feb. 16, 1957. 58p. (CF-55-10-124)

Descriptions and performances of the mechanical equipment (pulse columns, pulse generators, pumps, etc.) used in the Thorex Process Pilot Plant are presented. Information is also presented on the case of direct maintenance of unit-shielded equipment.

7505 ✓ CF-55-7-49
Oak Ridge National Lab., Tenn.
THOREX SECOND THORIUM CYCLE. R. P. Wischow. July 5, 1955. Decl. Feb. 14, 1957. 11p. Contract [W-7405-eng-26]. $0.25(OTS).

Operating data and flowsheets are given for the Thorex second thorium cycle. This cycle is being added to the original Thorex extraction system to ensure adequate Th decontamination. (C.W.H.)

7502 ✓ CF-55-3-120
Oak Ridge National Lab., Tenn.
RECOVERY OF U-235 FROM HRE FUEL BY THE INTERIM-23 FACILITY. K. H. McCorkle. Mar. 17, 1955. Decl. Feb. 14, 1957. 20p. Contract [W-7405-eng-26]. $3.30(ph OTS); $2.40(mf OTS).

The recovery and decontamination of U^{235} from spent HRE fuel solution by solvent extraction with TBP was investigated. Recoveries of 91.1% U^{235} were obtained. The gross β and gross γ-decontamination factors were 2.8×10^5 and 4.9×10^4 respectively. (C.W.H.)

THOREX SOLVENT RECOVERY: DEVELOPMENT OF CALCIUM HYDROXIDE SLURRY PROCESS. P. A. Haas and A. C. Jealous. Feb. 16, 1955. Decl. Mar. 2, 1957. 30p. (ORNL-1784)

$Ca(OH)_2$ slurries, approximately 360 g of $Ca(OH)_2$ per liter of H_2O, effectively removed impurities from Thorex process solvent (42% TBP in Amsco 123-15) which had been used in nonradioactive pulse column studies, so that it could be reused in extraction columns. Contact between solvent and slurry was made in mixer settlers. The volume of waste from this recovery process was 1% or less of the volume of solvent treated. An equipment diagram and a flowsheet are given.

7499 ✓ CF-54-11-117
Oak Ridge National Lab., Tenn.
PRELIMINARY STUDY OF "COTTON" AND THORIUM "BACKGROUNDS" IN THE THOREX PROCESS. S. A. Reynolds. Nov. 22, 1954. Changed from SECRET Dec. 16, 1955. Decl. Feb. 14, 1957. 14p. Contract [W-7405-eng-26]. $3.30(ph OTS); $2.40(mf OTS).

The natural beta and gamma backgrounds associated with Th and U^{233} in the Thorex Process have been approximated. Corrections were made for the self-absorption of U^{233} betas in Th and in $SnCl_2$. (C.W.H.)

(SPECIAL) PROCESSES 6.17

THOREX 6.17.22

SOLVENT EXTRACTION 6.17.22.03

13019 ORNL-1610(Rev.)
Oak Ridge National Lab., Tenn.
LABORATORY DEVELOPMENT OF THE THOREX PROCESS. Progress Report [for] February 1, 1953 to July 31, 1953. A. T. Gresky, M. R. Bennett, S. S. Brandt, W. T. McDuffee, B. G. Ryle, J. E. Savolainen, and R. P. Wischow. Apr. 22, 1954. Decl. Mar. 25, 1957. 97p. Contract W-7405-eng-26. $0.60(OTS).

Laboratory-scale studies on the Thorex process have led to the recommendation that excess acid be removed from the organic stream in a slightly longer B-column with very dilute nitric acid rather than in a separate column with "acid-deficient" aluminum nitrate as formerly. A head-end evaporation-reflux treatment of the feed is recommended to prevent loss of thorium, to coagulate colloidal siliceous material and thus minimize emulsions in later steps, and to decrease the ruthenium in the product stream. Pa^{233} adsorption by silica gel was inhibited by niobium, ferric, or chromic ions. (auth)

7490 CF-53-6-138
Oak Ridge National Lab., Tenn.
RECOMMENDED PULSE COLUMN DIMENSIONS AND ACCESSORIES FOR THE THOREX PILOT PLANT. A. C. Jealous. June 17, 1953. Decl. Feb. 20, 1957. 6p. Contract W-7405-eng-26. $1.80(ph OTS); $1.80(mf OTS).

7488 ✓ CF-53-6-105
Oak Ridge National Lab., Tenn.
SOME INSTRUMENTATION DEVELOPMENT FOR THOREX PULSE COLUMNS. Tom M. Gayle. June 12, 1953. Decl. Feb. 14, 1957. 13p. Contract [W-7405-eng-26]. $3.30(ph OTS); $2.40(mf OTS).

Special instruments were developed for and applied to the two-inch vertical pulse column and the two-inch concatenated pulse column of the Thorex process. The instruments were intended for a hot pilot plant operating under similar conditions. Instruments described include a pneumatic detector for interface level; a pneumatic detector for pulse amplitude (illustrated); an oscilloscope for pulse monitoring (illustrated); an instrument for interface level and amplitude detection by capacity measurement (illustrated); a flowmeter for radioactive liquids; and an automatic control for column feeds and withdrawals (illustrated). (C.H.)

12990 CF-52-10-26
Oak Ridge National Lab., Tenn.
$Ca(OH)_2$ CONTACTORS FOR THOREX SOLVENT RECOVERY SYSTEM. W. R. Winsbro. Oct. 3, 1952. Decl. Apr. 2, 1957. 8p. Contract [W-7405-eng-26]. $1.80(ph OTS); $1.80(mf OTS).

9653 ✓ ORNL-20
Oak Ridge National Lab., Tenn.
COMPARISON OF BATCH AND CONTINUOUS 23 PROCESSES. G. H. Hanson. March 17, 1948. Decl. Mar. 7, 1957. 39p. $15.30(ph OTS); $5.40(mf OTS).

The separation of U and Th by solvent extraction has been studied at X-10 and other sites for several years. In connection with the recovery and decontamination of U^{233} produced by the irradiation of Th in the proposed Research Pile and other piles, both batch and continuous solvent extraction processes have been proposed. For a plant capacity of fifteen kg of Th per day, a continuous column process is more favorable than one using intermittent batch extractions. This recommendation is contingent upon the economic solution of the crude problem and the demonstration of trouble-free operation with packed columns for long periods of time. The estimated investment and operating costs for each process are essentially the same. (D.E.B.)

ION EXCHANGE – U^{233} 6.17.22.04

12368 ✓ CF-56-11-27
Oak Ridge National Lab., Tenn.
THOREX PILOT PLANT: BENCH TESTS ON RESIN SORPTION COLUMNS FOR THE NEW URANIUM ISOLATION SYSTEM. J. H. Walker. Nov. 8, 1956. Decl. Mar. 12, 1957. 15p. Contract [W-7405-eng-26]. $3.30(ph OTS); $2.40(mf OTS).

Bench tests were conducted on new ion exchange equipment installed in the Thorex pilot plant for the isolation and removal of U and Th from the process. Variables studied include the following: pressure drop through the column and bed expansion vs. volumetric flow rate of water, maximum bed contraction when treated with 6M HNO_3, and loss of resin from the column through the retaining-plug

(SPECIAL) PROCESSES 6.17

THOREX 6.17.22

ION EXCHANGE – U^{233} 6.17.22.04

sc .en and Teflon gasket. A Th column was loaded to maximum capacity with Th to determine the resin-bed contraction at this condition. (L.T.W.)

582 CF-55-12-88
Oak Ridge National Lab., Tenn.
INVESTIGATION OF COUNTERCURRENT ION EXCHANGE FOR ISOLATION OF URANIUM-233. R. E. Leuze and V. C. A. Vaughen. Dec. 15, 1955. Decl. Sept. 26, 1958. 17p. Contract [W-7405-eng-26]. $3.30(ph OTS); $2.40(mf OTS).

A simplified procedure of operating the ion exchange columns for isolation of uranium-233 in the Thorex Pilot Plant was developed and tested on a laboratory scale. This procedure is based upon volume measurement instead of flowing stream analyses during elution. The essential features are upflow sorption and downflow elution at 70°C with a calculated volume of elutriant containing 3.0 M $NH_4C_2H_3O_2$, 1.5 M $HC_2H_3O_2$, and 0.75 M $(NH_4)_2HC_6H_5O_7$ followed by 5 column volumes of demineralized water. Countercurrent flow makes it possible to essentially eliminate acetate recycle through solvent extraction by leaving a small uranium heel on the resin instead of complete elution with large volumes of acetate tailings containing small amounts of uranium. The

11628 ✓ KAPL-M-DJS-1
Knolls Atomic Power Lab., Schenectady, N. Y.
A CONTINUOUS ION EXCHANGE SYSTEM FOR URANIUM CONCENTRATION AND DECONTAMINATION IN THE THOREX PROCESS. Donald J. Smith. Apr. 1, 1955. Decl. Mar. 15, 1957. 17p. Contract W-31-109-Eng-52. $3.30(ph OTS); $2.40(mf OTS).

The design for a continuous ion exchange system for use in the concentration and decontamination of the U stream following the solvent extraction cycles in the Thorex Process is given. The moving bed technique is compared to a fixed bed system under similar conditions. (W.L.H.)

PROCESS DATA AND CONTROL 6.17.22.05

10976 ✓ CF-58-7-53
Oak Ridge National Lab., Tenn.
THOREX PILOT PLANT: CRITICALITY REVIEW OF THE THOREX PILOT PLANT USING THE INT-23 PROCESS. W. T. McDuffee and O. O. Yarbro. July 8, 1958. 5p. Contract [W-7405-eng-26]. $1.80(ph), $1.80(mf) OTS.

The results of a criticality review of the ORNL Thorex pilot plant are presented for the condition where a low TBP (INT-23) flowsheet is used in recovering U^{233} from irradiated thorium. Criticality control will be maintained by limiting the U^{233} inventory in the solvent extraction system to less than 550g by means of stream-volume and U^{233} material balances determined at 8 hr intervals. Equipment modification and improved operating procedures for safe plant operation are outlined. (auth)

12703 ✓ ORNL-2332
Oak Ridge National Lab., Tenn.
A CRITICALITY STUDY OF THE THOREX PILOT PLANT. O. O. Yarbro. Sept. 17, 1957. 29p. Contract W-7405-eng-26. $0.25(OTS).

A recent criticality study has indicated that the Thorex Pilot Plant is critically safe for the processing of thorium irradiated to less than 10,000 grams of U^{233} per ton. As the size of equipment exceeds the geometrically safe size and the total uranium inventory exceeds the minimum critical mass, minor equipment modifications and changes in operational procedures were necessary to provide safety under adverse operating conditions. (auth)

348 ✓
SELECTED METHODS OF CHEMICAL ANALYSIS FOR THOREX PROCESS CONTROL. W. W. Sabol and B. F. Rider. Sept. 1, 1956. 77p. (KAPL-1477)

Step-by-step procedures for control of the Thorex process miniature pilot plant at KAPL are given for use as a laboratory manual. Included are procedures for measuring acidity, density, Th content, radiation (α, β, and γ), U^{233} and U^{232}. General radiochemical procedures are given for determination of Ce^{144}, Cs^{137}, Pa^{233}, Ru^{106}, Sn^{89}, Th^{234}; total rare earths (TRE) and Zr^{95} + Nb^{95}. Miscellaneous

(SPECIAL) PROCESSES 6.17

THOREX 6.17.22

Process Data and Control 6.17.22.05

determinations cover n-butanol, DBP, F^-, fluorimetric U, MnO_2, Mn, PO_4^-, TBP, and unstable constituents in TBP diluent.

5423 / CF-55-6-43
Oak Ridge National Lab., Tenn.
CALCULATIONS OF GAMMA COUNTING RATE OF THORIUM. S. A. Reynolds. June 8, 1955. Decl. Dec. 20, 1955. 12p. Contract [W-7405-eng-26]. $3.30(ph OTS) $2.40(mf OTS).

The natural gamma activity associated with thorium in the Thorex Process has been estimated on the basis of several calculations and scintillation counter measurements. The relative activity of Th as a function of time after purification is tabulated. (C.W.H.)

DECONTAMINATION 6.17.22.06

DECONTAMINATION OF THE ORNL THOREX PILOT PLANT. K. H. McCorkle and W. R. Winsbro. July 25, 1956. 31p. (ORNL-2058)

Decontamination of the Thorex Pilot Plant is described. Twenty-four major pieces of highly contaminated stainless-steel processing equipment were decontaminated in 19 days to safe working levels by alternate treatments with 20% NaOH—2% $Na_2C_4H_4O_6$, and 20% HNO_3. $H_2C_2O_4$ or acid was substituted for $H_2C_4H_4O_6$ in about half the treatments. The average cost per treatment was $222; the total cost was $36,000.

11617 ✓ CF-56-4-184
Oak Ridge National Lab., Tenn.
THOREX PILOT PLANT: DECONTAMINATION RUN IX-16. J. H. Walker and G. S. Sadowski. Apr. 18, 1956. Decl. Mar. 14, 1957. 14p. Contract [W-7405-eng-26]. $3.30(ph OTS); $2.40(mf OTS).

The feed adjustment tank was decontaminated to a final radiation level of 950 mr/hr at contact with 30% HNO_3 and 20% NaOH–2% sodium tartrate solution. The top of the partitioning column was easily decontaminated to 13 mr/hr, but decontamination of the BT evaporator was limited by contaminants on the walls of the cubicle. The radiation level at the evaporator was only decreased from ~ 600 to 400 mr/hr. Two acid treatments alternated with two caustic–tartrate treatments were sufficiently effective to reduce the radiation level at the bottoms of the stripping column from 2,300 to 230 mr/hr. An elution of the silica gel column with 0.5 M HNO_3 followed by 4% oxalic acid brought the radiation level of the column from 23,000 to 4000 mr/hr. Exposure rates and levels for maintenance personnel are given. (L.T.W.)

ThO_2 PRODUCTION 6.17.22.07

12318 CF-56-1-50
Oak Ridge National Lab., Tenn.
PILOT PLANT PREPARATION OF THORIUM OXIDE. W. H. Carr. Jan. 4, 1956. Decl. Mar. 13, 1957. 27p. Contract [W-7405-eng-26]. $4.80(ph OTS); $2.70(mf OTS).

ThO_2 is prepared in the Thorex pilot plant by converting thorium nitrate to thorium oxalate, then calcining the oxalate to the oxide. Reagent-grade oxalic acid is added to a thorium nitrate solution at 40°C, precipitating thorium oxalate. After agitation for one hour, the slurry is filtered and the oxalate washed. The oxalate is calcined at 370, 520, and 800°C. The resulting ThO_2 is blended and packed for shipment. (auth)

CORROSION 6.17.22.08

5261 ✓ ORNL-2844
Oak Ridge National Lab., Tenn.
THOREX PILOT PLANT CORROSION STUDIES: II. CORROSION OF TYPES 304L, 309SCb, AND 347 STAINLESS STEEL DURING DEVELOPMENT AND PRODUCTION-DEVELOPMENT PERIODS. J. L. English. Jan. 20, 1960. 50p. Contract W-7405-eng-26. OTS.

Corrosion data for types 304L, 309SCb, and 347 stainless steel were obtained in a number of process vessels in the ORNL Thorex pilot plant during the development and the production-development periods of operation occurring between December 1954 and September 1956. Stressed corrosion-test specimens were exposed in the batch dissolver tank, the feed adjustment tank, the BT vapor separator, the A-column feed tank, and the BTC catch tank. Generally, types 304L and 309SCb stainless steel exhibited comparable corrosion resistance in all environments examined. Most of the studies were conducted with these two alloys. Severe corrosion damage was encountered in the vapor phase of both the batch dissolver tank, operated at a maximum temperature of 115°C, and the feed adjustment tank, operated at a maximum temperature of 155°C. Corro-

(SPECIAL) PROCESSES 6.17

THOREX 6.17.22

Corrosion 6.17.22.08

sion rates for types 304L and 309SCb stainless steel varied from approximately 30 to 55 mpy in the batch dissolver tank during the development and the production-development periods. Vapor-phase corrosion rates in the feed adjustment tank during the last half of the development period ranged from 85 to 100 mpy. Severe corrosion attack was experienced also in the BT vapor separator, which operated at a maximum temperature of 115°C. Rates were 30 to 55 mpy for type 304L stainless steel, 25 mpy for type 309SCb stainless steel, and a maximum rate of 150 mpy for a composite specimen of types 309SCb and 347 stainless steel. Solution-phase corrosion rates for types 304L and 309SCb stainless steel in the batch dissolver tank ranged from 13 to 28 mpy, with the lower rates observed on the type 309SCb steel. The latter corroded at an average rate of 41 mpy in the feed adjustment tank, however. No data were available for type 304L stainless steel in the solution phase of this vessel. The corrosion of types 304L and 347 stainless steel was negligible in the A-column feed tank and the BTC catch tank; observed rates were less than 0.1 mpy. (auth)

CORROSION STUDIES FOR THE THOREX PROCESS. J. L. English. Mar. 27, 1953. Decl. Feb. 14, 1957. 21p. (CF-53-3-246)

Stainless steel specimens were abraded and exposed to Thorex Process solutions and vapors at atmospheric and boiling temperatures. Weight losses and penetration measurements are recorded.

13744 ✓ CF-53-1-256
Oak Ridge National Lab., Tenn.
RESULTS OF CORROSION TESTS OF TYPE 347 STAINLESS STEEL WELDMENTS IN SOLUTIONS SUPPLIED BY THE CHEMICAL TECHNOLOGY DIVISION. C. C. Vreeland. Jan. 24, 1953. Decl. Apr. 2, 1957. 8p. Contract [W-7405-eng-26]. $1.80(ph OTS); $1.80(mf OTS).

Stainless steel weldments for the Thorex Pilot Plant were corrosion tested in Thorex AW stream and 3-20 decontamination solutions. Uniform weight losses were observed for 3 specimens which were examined metallographically and with a low-power microscope. No excessive attack was noted on the weld metal or base metal. (T.R.H.)

UAP PROCESS 6.17.23

Developed for recovery of U from aged Hanford metal waste. Waste is dissolved at 85° C. in presence of a small amount of H_3PO_4 by the careful addition of 60% HNO_3.

~90% of the U is precipitated as UAP at a pH of 0.1 to 0.2 by the addition of NH_4NO_3. Slow cooling permits the crystals to perfect themselves and expel fission products.

See also, under FISSIONABLE MATERIAL--RECOVERY,

RECOVERY OF IRRADIATED TH AND U--Precipitation --Phosphate (3.02.15.07).

5267 ✓

THE URANYL-AMMONIUM PHOSPHATE PROCESS FOR RECOVERY OF URANIUM FROM SLAG SCRAP. E. R. Johnson, E. O. Rutenkroger, A. B. Kreuzmann, and B. C. Doumas (National Lead Co. of Ohio, Cincinnati). Chem. Eng. Progr. 53, 56F-9F(1957) Feb.

The process developed involves the following steps: roasting and grinding of the scrap material, leaching the roasted and ground material with HCl to solubilize the U, precipitating the U from the filtered leach liquors as $UO_2NH_4PO_4$, and converting the $UO_2NH_4PO_4$ to $Na_2U_2O_7$ by slurring of the former with a NaOH solution. A detailed description of the process and equipment for plant-scale processing is presented. (W.L.H.)

URANIUM RECOVERY FROM METAL WASTES. p.130-134 of CHEMICAL SEPARATIONS PROCESSES. PART I. CHAPTERS 1-11. Sigfred Peterson, comp. and ed. Dec. 1953. Decl. with deletions Mar. 20, 1957. 137p. (CF-53-12-150(pt.1)(Del.2))

The uranyl ammonium phosphate (UAP) and the tributyl phosphate (TBP) processes for the recovery of U^{235} from metal waste solutions are described in detail. The process for the recovery of Pu from old $BiPO_4$ waste by the modified Purex process is also given.

(SPECIAL) PROCESSES 6.17

UAP PROCESS 6.17.23

8805 K-621
Carbide and Carbon Chemicals Corp. K-25 Plant, Oak Ridge, Tenn.
LABORATORY DEVELOPMENT OF THE UAP PROCESS. J. C. Barton and R. H. Rainey. Aug. 4, 1950. Decl. Dec. 27, 1955. 79p. Contract [W-7405-eng-26]. $0.45(OTS).

The laboratory research and development of a process for the recovery of U from aged Hanford metal waste by precipitation as uranyl ammonium phosphate (UAP) is described. The product UAP, representing 99% of the U in the waste, can be handled in conventional equipment and has an inhalation hazard only 31% greater than normal U. It can easily be converted to UF_6 or to U for re-use in project installations. In the standard UAP Process, aged Hanford metal waste is dissolved at 85°C in the presence of a small amount of H_3PO_4, by the careful addition of 60% HNO_3. Approximately 95% of the U is precipitated as UAP at a pH of 0.1 to 0.2 by the addition of NH_4NO_3. Slow cooling permits the crystals to perfect themselves and to

9724 ✓ K-405
Carbide and Carbon Chemicals Corp. K-25 [Plant], Oak Ridge, Tenn.
CORROSION OF STAINLESS STEELS IN URANYL AMMONIUM PHOSPHATE FILTRATES FROM HANFORD WASTE. H. A. Bernhardt, W. Davis, Jr., and J. R. Flanary. June 1, 1949. Decl. Feb. 23, 1957. 13p. Contract [W-7405-eng-26]. (KLO-103). $3.30(ph OTS); $2.40(mf OTS).

Storage of acid filtrates from the UAP process for uranium recovery from alkaline Hanford waste has imposed the problem of determining the type of metal that can be used for storage tanks. Two stainless steels, 302 and Nb stabilized, have been tested in synthetic filtrates and found to corrode at the rates 0.007 and 0.045 mils/year, respectively. These values are much smaller than an estimated maximum allowable corrosion rate of 10 mils/year. It is concluded that either of these stainless steels or 304 or 316 stainless steels could be used in the construction of tanks for storing acid filtrates from the UAP process. (auth)

ZIRCEX 6.17.24

See also, this section,
CHLORINATION (6.03),
and,
under FISSIONABLE MATERIAL--RECOVERY,
HEAD-END TREATMENTS--Zirconium-Uranium Fuel Elements (3.01.12),
and,
under HAZARDS AND PROTECTION,
PYROPHORICITY (4.01.10),
and,
under PLANTS AND EQUIPMENT,
HIGH TEMPERATURE SYSTEMS--Materials (5.10.02), Vessels (5.10.04).

21805 ORNL-2631(Del.)
Oak Ridge National Lab., Tenn.
STATUS OF DEVELOPMENT OF THE ZIRCEX PROCESS. J. J. Perona, J. B. Adams, J. E. Savolainen, and T. A. Gens. Mar. 31, 1959. Decl. with deletions July 24, 1959. 61p. Contract W-7405-eng-26. $10.80 (ph), $3.90(mf) OTS.

The Zircex Process is a chemical method for separating zirconium and uranium by reacting spent fuels with anhydrous hydrogen chloride at >500°C, producing volatile zirconium tetrachloride and a nonvolatile uranium-containing residue. The hydrochlorination step was found feasible from the chemical and engineering viewpoints. Reaction times for fully enriched fuel assemblies would probably be less than 10 hr and for decladding UO_2-containing fuels less than 3 hr. For the thick plate Experimental Boiling Water Reactor fuel assemblies, reaction times of 30 hr or more would probably be required. Glass wool filters packed to a density of about 0.35 g/cc kept uranium losses to the $ZrCl_4$ sublimate below 0.1% for all fuels. Unirradiated fuel specimens were used, except for a single tracer-level run. A satisfactory method for the condensation of highly radioactive $ZrCl_4$ has not yet been developed. (auth)

(SPECIAL) PROCESSES 6.17

ZIRCEX 6.17.24

AQUEOUS REPROCESSING OF NUCLEAR FUELS. Charles L. Peterson, et al. (Battelle Memorial Inst., Columbus, Ohio) and Walter E. Clark (Oak Ridge National Lab., Tenn.). Ind. Eng. Chem. 51, 32-7(1959) Jan.

A feasible corrosion-resistant container material for the single-unit Zircex hydrochlorinator-dissolver is not apparent at present. Of the materials studied, the best choice is Haynes 25. Its use would require that penetration rates of approximately 3 mils per month be tolerated. The process should be run in two steps. Several materials, such as Inconel, Haynes 25, Type S-816, Illium R, and nickel, appear promising for construction of the hydrochlorinator. The cold residue would then be dissolved by transferring it to a container made from titanium or tantalum. Preliminary experiments in the alternate liquid-phase Zircex hydrochlorination system employing fused aluminum chloride–ammonium chloride indicate that nickel and the Hastelloys may be satisfactory for construction of the hydrochlorinator. Molybdenum appears even better

ZIRCEX FLUORIDE VOLATILITY COMBINATION. P. R. Ammann, D. A. Madden, and D. L. Swift. Mar. 20, 1958. Decl. with deletions June 19, 1958. 29p. (AECD-4267;KT-339(Del.))

In using the Zircex Process head-end to recover Zr from fuel elements before recovery of U by fluoride volatility, loss of U as UCl_4 is a problem. The suitability of using a Ni wire filter for trapping entrained UCl_4, and recovery of the trapped U by direct fluorination of filter and residue is investigated. It is recommended from these studies that water vapor and O_2 should be kept out of the reactor, and that the filter and filter material should be further investigated.

8434

A PROCESS FOR THE PREPARATION OF METALLIC URANIUM FROM URANIUM TETRACHLORIDE BY FUSED SALT ELECTROLYSIS. (to Ministry of International Trade and Industry). British Patent 828,258. Feb. 17, 1960.

A process for fused salt electrolysis of UCl_4 to produce U is described which avoids disadvantages of similar processes by maintaining UCl_4 concentration above 10%. In one example the bath contained 1300 g UCl_4, 830 g KCl, 130 g NaCl, and 640 g LiCl. A graphite anode and Mo-wire cathode and a pyrex vessel were used. The electrolytic current was 20 amps, the voltage 6 to 9 for 322.5 amp-hr at 450 ± 20°C. In the example 575 g of very pure U was obtained at a current efficiency of 81%. (T.R.H.)

11355 ✓ AECU-3750

Massachusetts Inst. of Tech., Oak Ridge, Tenn. Enginnering Practice School.

ZIRCEX URANIUM SUBLIMATE LOSS. T. R. Jaworowski and C. J. King, III. Jan. 24, 1958. 23p. For [Oak Ridge Gaseous Diffusion Plant. Contract W-7405-eng-26, Subcontract No. 70]. (KT-310). $4.80(ph OTS); $2.70(mf OTS).

One of the limitations of the Zircex Process is the large percentage loss of U which is carried off with the volatile $ZrCl_4$. Since the ratio of U to Zr in the fuel element is quite small (< 1%), a large percentage of U loss can be caused by small amounts of U in the $ZrCl_4$ sublimate. The U loss must be kept below 1% if the Zircex Process is to be adapted to industrial scale processing. (W.L.H.)

EVALUATION OF CONTAINER MATERIALS FOR ZIRCEX AND DAREX NUCLEAR-FUEL-RECOVERY PROCESSES. Paul D. Miller, Charles L. Peterson, Earl L. White, and Frederick W. Fink. Dec. 11, 1957. 44p. (BMI-1242)

The selection of a suitable material for the processing equipment is most important in fuel-recovery processes. The problem arises because of the aggressive nature of the reagents that must be used for disintegrating or dissolving some spent fuel elements. In this investigation, corrosion tests were made under conditions simulating those that may occur in several proposed processes in order that candidate materials of construction could be evaluated. No material has been found which would allow both the hydrochlorination and dissolution steps of the vapor-phase Zircex process to be carried out in a single vessel. For a two-vessel process, several materials, such as Inconel, Haynes 25, Type S-816 alloy, Illium R, and the Hastelloys B and C, appear promising for construction of a hydrochlorinator; the dry residue would then be transferred to another vessel, perhaps of Ti or Ta, for dissolution. In the alternate liquid-phase Zircex process,

(SPECIAL) PROCESSES 6.17

ZIRCEX 6.17.24

preliminary tests have given promising results with Ni and Ni–Cr alloys. However, the only metals studied, which withstand the corrosive boiling $AlCl_3 \cdot NH_4Cl$ plus gaseous HCl environment with but negligible weight losses have been Pt, Au, W, and Mo. A variety of laboratory studies have shown that Ti will apparently withstand any condition likely to occur in the critical areas of the Darex process, including the dissolver and chloride-stripping column. It will undergo a severe attack in HCl vapor, but this is easily prevented by the presence of a small amount of HNO_3 vapor or oxides of N. Ta is another promising material disclosed by these tests, but its use in the dissolver seems negated because embrittlement was detected under certain conditions. No signs of cracking or other attack have been observed on Type 347 stainless steel specimens exposed at room temperature with various levels of stress for over 6 months to Purex-type acid extraction systems containing chlorides far in excess of the concentrations expected during operation of either the Zircex or Darex head-end processes.

11356 AECU-3751
Massachusetts Inst. of Tech., Oak Ridge, Tenn.
Engineering Practice School.
ZIRCEX KINETICS AND URANIUM LOSS STUDY. T. J. Carter, Jr., and R. A. Stone. Nov. 13, 1957. 23p. For [Oak Ridge Gaseous Diffusion Plant. Contract W-7405-eng-26, Subcontract No. 70]. (KT-319). $4.80 (ph OTS); $2.70(mf OTS).

A study was made of the effects of temperature, impurities in the hydrogen chloride, flow rates, and niobium content on both the reaction rates and insoluble uranium losses in the Zircex Process is presented. (W.L.H.)

ZIRCONIUM FOR NUCLEAR REACTORS. I. E. Newnham (C.S.I.R.O., Melbourne). Research (London) 10, 424-8 (1957) Nov.

A method for preparing reactor grade Zr is outlined. The process consists of conversion of $ZrCl_4$ to $ZrCl_3$, condensation of unreduced $HfCl_4$ on the lid of the vessel, and transfer of sublimed $ZrCl_4$ to another vessel for Mg reduction. (T.R.H.)

12367 ✓ CF-56-10-41
Oak Ridge National Lab., Tenn.
ZIRCEX: HEP DESIGN AND RECOMMENDATIONS. C. E. Guthrie. Oct. 8, 1956. Decl. Mar. 13, 1957. 14p. Contract [W-7405-eng-26.] $3.30(ph OTS); $2.40(mf OTS).

The high-temperature gaseous hydrochlorination (Zircex) system for processing Zr-containing fuel elements consists of a batch hydrochlorinator followed by a batch aqueous feed adjustment tank. The preliminary design is presented along with an outline of additional development work required and alternate processes which must be considered. (auth)

11618 ✓✓CF-56-8-101 (Del.)
Oak Ridge National Lab., Tenn.
POWER REACTOR FUEL PROCESSING STATUS REPORT FOR AUGUST 1956. R. E. Blanco, W. K. Eister, and D. E. Ferguson. Aug. 15, 1956. Decl. with deletions Mar. 20, 1957. 20p. $3.30(ph OTS); $2.40(mf OTS).

Hermex Process. Fernald scrap uranium was significantly decontaminated by Hermex processing. Zircex Process. Removal of zircaloy-2 jackets from UO_2 cores by hydrochlorination did not form UO_2Cl_2 or affect the solubility of the cores seriously. A hydrochlorinator for

13815 AECD-3744
Massachusetts Inst. of Tech., Oak Ridge, Tenn.
Engineering Practice School.
RECOVERY OF ZIRCONIUM TETRACHLORIDE POWDER. L. A. Lee and M. A. Welt. Aug. 6, 1956. Decl. Aug. 11, 1958. 20p. For Union Carbide Nuclear Co. [K-25 Plant, Contract W-7405-eng-26, Subcontract 70]. (KT-226). $3.30(ph OTS); $2.40(mf OTS).

The Zircex Process for the recovery of zirconium from zirconium clad fuel elements is presented. Various types of apparatus were investigated for desublimation and collection of the solid zirconium tetrachloride. Of primary importance is the determination of particle size and distribution of the $ZrCl_4$ condensed for, in general, the larger and more uniform the particle size, the easier the collection. (W.L.H.)

13585 ✓ CF-56-5-193 (Del.)
Oak Ridge National Lab., Tenn.
HEAT REMOVAL FROM ZIRCONIUM HYDROCHLORINATION. R. J. McNamee. May 22, 1956. Decl. with deletions Mar. 13, 1957. 5p. Contract [W-7405-eng-26]. $1.80(ph OTS); $1.80(mf OTS).

(SPECIAL) PROCESSES 6.17

ZIRFLEX 6.17.25

See also, under FISSIONABLE MATERIAL--RECOVERY

HEAD-END TREATMENTS--Zirconium-Uranium Fuel Elements (3.01.12),

and,

under HAZARDS AND PROTECTION,

PYROPHORICITY (4.01.10).

4389 ✓ ORNL-2713
Oak Ridge National Lab., Tenn.
MODIFIED ZIRFLEX PROCESS FOR DISSOLUTION OF ZIRCONIUM- AND NIOBIUM-BEARING NUCLEAR FUELS IN AQUEOUS FLUORIDE SOLUTIONS: LABORATORY DEVELOPMENT. T. A. Gens and F. G. Baird. Dec. 22, 1959. 29p. Contract W-7405-eng-26. OTS.

Modified Zirflex process flowsheets were developed for recovering uranium from the newer power reactor fuel alloys after discharge from the reactor. The STR (1% U-97% Zr-2% Sn) and EBWR Core-1 (93.5% U-5% Zr-1.5% Nl clad in Zircaloy-2) fuels are used as examples of low- anc high-uranium fuels, respectively. A dissolvent of 6 M NH_4F yields a solution of zirconium and a precipitate of ammonium uranous fluoride. In one process, ammonium hydroxide is added to produce insoluble hydrous oxides of uranium, zirconium, and niobium. The NH_4F-NH_4OH supernatant is removed by filtration, partially evaporated and recycled as dissolvent. The uranium and zirconium oxides are dissolved in nitric acid to yield a solvent extraction feed solution of low fluoride content. In an alternative process nitric acid and aluminum nitrate are added to the ammonium fluoride fuel solution to oxidize U(IV) to soluble U(VI) and prepare a stable solution suitable for solvent extraction. Chromic acid is also added in the case of the STR fuel. In a variation of this flowsheet for the EBWR fuel, only enough 6 M NH_4F is added to dissolve the cladding. Nitric acid and aluminum nitrate are then added to dissolve the core. Insoluble niobic oxide, which carries about 0.03% of the uranium from the EBWR fuel, is removed by filtration from the solvent extraction feed solutions in the EBWR flowsheets. (auth)

7594 ✓ HW-56752(Del.)
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
PRELIMINARY ZIRFLEX FLOWSHEET FOR THE DISSOLUTION OF ZIRCALOY CLAD FUELS. C. R. Cooley and A. M. Platt. July 10, 1958. Decl. with deletions Feb. 3, 1959. 7p. Contract W-31-109-Eng-52. $1.80 (ph), $1.80(mf) OTS.

A preliminary flowsheet for processing Zircaloy-clad uranium dioxide fuels is presented. The process (Zirflex) employs a mixture of boiling ammonium fluoride and ammonium nitrate for the selective dissolution of the fuel cladding. Most of the uranium dissolved during coating removal is precipitated as UF_4 by cooling. Centrifugation recovers the precipitate which is later dissolved in nitric acid and aluminum nitrate. After decladding the remaining cores of metallic uranium or uranium dioxide are dissolved in a conventional manner with nitric acid. Likewise, the customary stainless steel equipment may be used.

14715 ✓ A/CONF.15/P/2429
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
THE ZIRFLEX PROCESS. J. L. Swanson. 20p. $0.50 (OTS).

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

The Zirflex Process is designed to accomplish selective dissolution of zirconium or Zircaloy fuel cladding. The process involves the dissolution of the cladding material in solutions of ammonium fluoride alone or in mixture with ammonium nitrate. Mixtures of ammonium fluoride and ammonium nitrate are preferred since they

14388 HW-57582(Del.)
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
TECHNOLOGY OF NON-PRODUCTION REACTOR FUELS REPROCESSING—BUDGET ACTIVITY. Quarterly Report. V. R. Cooper. Sept. 25, 1958. Decl. with deletions Apr. 2, 1959. 11p. Contract W-31-109-Eng-52. $3.30(ph), $2.40(mf) OTS.

Mechanical processing activities for the period are briefly reviewed. Dejacketing and dissolution studies for the Zirflex Process and Niflex Process are outlined. Redox and TBP solvent extraction studies and packed TBP solvent extraction columns are discussed. Zirflex corrosion studies are reported. Analytical developments are briefly outlined. (For preceding period see HW-56477.) (J.E.D.)

SINTERING 6.18

16994 ✓ NYO-8087
Carnegie Inst. of Tech., Pittsburgh. Metals Research Lab.
FUNDAMENTAL STUDY OF THE EARLY STAGES OF SINTERING. Final Report. June 30, 1959. 131p. Contract AT(30-1)-1826. $21.30(ph), $6.90(mf) OTS.

The process of sintering was examined in the early stages with emphasis on changes occurring at the very outset. Uncompacted, coarse powder was used. Methods used in the investigation include microscopic examination of fractured samples, quantitative metallography, and closed surface topology. A discussion of the bases of the topological principles is given including a relationship for evaluation of a powder mass genus, a description of sintering stages, graphical representations of topological surface, modes of shape change during sintering, and mechanisms of metal transport. Specifications of the powders used are listed along with data on density and sintering densification rate in relation to genus. Also, data from experiments in which relatively large masses of powder were sintered, sectioned, and surveyed internally with respect to density are presented. Sources of error are examined, and methods of removal of side influences which have no bearing on sintering are discussed. The true rate of densification, evidenced by the moving together of particle centers, is of greatest technical interest, and problems associated with its determination are examined. Aspects considered include studies of impure densification, early growth, and the effects of gas expulsion in initial sintering stages due to surface and internal oxides. The microstructures of powders during sintering are examined, and photomicrographs are included. Data indicate that a kind of plastic deformation promotes grain growth. (J.R.D.)

FINE PARTICLE MEASUREMENT, SIZE, SURFACE, AND PORE VOLUME. Clyde Orr, Jr. and J. M. Dallavalle. New York, The MacMillan Company, 1959. 365. $10.50.

A detailed discussion of the important techniques currently used in research laboratories for the measurement of size, surface, and pore volume is presented. The theory and application of the measurements are treated, and a comprehensive description is provided of more than seventy techniques including automatic scanning, the fixed-time method, the Hauser-Lynn method, gravimetric analysis, and liquid-phase sorption. A bibliography of approximately 400 references complements the text. (W.D.M.)

NOTE ON CORRELATION OF SURFACE CHARACTERISTICS OF URANIUM DIOXIDE POWDERS WITH THEIR SINTERING BEHAVIOR. D. R. Stenquist, B. Mastel, and R. J. Anicetti (General Electric Co., Richland, Wash.). J. Am. Ceram. Soc. 41, 273-4(1958) July.

The surface characteristics of Mallinckrodt PWR-grade and ceramic-grade UO_2 powders and Hanford-depleted UO_2 powder were investigated. The UO_2 powders were compacted at 15 tons/in^2. into pellets 0.600 in. in diameter and about 1 in. in height and then sintered for 8 hr at 1750°C. The percent change in density was used as a measure of sinterability. (W.D.M.)

2196 AEC-tr-3395
POROUS SINTER METALS, THEIR PRODUCTION AND UTILIZATION. R. Palme. Translated for Oak Ridge Gaseous Diffusion Plant from Metall 12, 28-32(1958).

12377 NYO-8085
Carnegie Inst. of Tech., Pittsburgh. Metals Research Lab.
FUNDAMENTAL STUDY OF THE EARLY STAGES OF SINTERING. Annual Report. June 15, 1957. 29p. Contract AT(30-1)-1826. $4.80(ph OTS); $2.70(mf OTS).

During early stage sintering the number of interparticle contact points (the connectivity of the metal surface) remains constant. Meanwhile the metal surface area diminishes, the rate of mass transfer apparently being proportional to the number of particle contacts and to the surface free energy, that is to the surface area in any one example. At the outlet of sintering there is a sharp decrease in density followed by a subsequent and continuing increase in density; this reversal is thought to be associated with a reversal in the proportions of positive and negative curvature of the metal surface. A revision of the particle counting method is presented. (auth)

10620 ✓ NYO-8526
Penn-Texas Corp., New York and Stevens Inst. of Tech., Hoboken, N. J. Powder Metallurgy Lab.
COMPACTING AND SINTERING OF METAL POWDERS WITHOUT THE APPLICATION OF PRESSURE.
Henry H. Hausner, Donald P. Ferriss, and Frank W. Heck. May 25, 1957. 20p. Contract AT(30-1)-1991. $3.30(ph OTS); $2.30(mf OTS).

The properties of a mass of loose powders are compared with those of powders compacted under pressure, and it has been shown that uncompacted powders have many properties which facilitate sintering. The rate of sintering, expressed in terms of densification, has been determined for uncompacted 316 stainless steel powders for the sintering temperature 1300°C. Various mixtures of fine and coarse powder particles were prepared in order to determine the mixtures which result in maxi-

763 SEP-250
Sylvania Electric Products Inc. Research Labs., Bayside, N. Y.
FUNDAMENTALS OF SINTERING—III. Third Annual Progress Report for June 30, 1957 to June 30, 1958.
A. L. Pranatis, L. S. Castleman, and L. Seigle.

STEAM SINTERING OF URANIUM DIOXIDE. C. Arne Arenberg and Paul Jahn (Illinois Inst. of Tech., Chicago). J. Am. Ceram. Soc. 41, 179-83(1958) May.

The following variables were considered in an investigation of the sintering of uranium dioxide: (1) sintering aids, i.e., small additions of substances that might aid the densification process, (2) soaking temperature, (3) soaking time, (4) furnace atmosphere, and (5) particle size of the uranium dioxide. The incipient sintering temperature, the effect of binders, and the effect of fabricating pressure also were determined. The data obtained in the investigation led to the following optimum conditions for obtaining dense UO_2 by cold pressing and sintering: (1) preparing the UO_2 as a submicron powder but not fine enough to be pyrophoric, (2) cold pressing at 20 to 30 tons per sq. in. without the use

11424

SINTERING UNDER PRESSURE OF ALUMINA AND BERYLLIA. R. Caillat and R. Pointud (Centre d'Etudes nucleaires, Saclay, France). Rev. met. 54, No. 4, 277-82(1957). (In French)

The influence of temperature and pressure sintering, together with the time of application of this pressure, on the density of sintered discs made from pure alumina and beryllia powder is examined. Densities close to the maximum theoretical density are quickly, reached from 1,600 °C onwards, under pressures of about 200 kg/cm^2. The crystalline texture of the products obtained is studied simultaneously. The experimental results are discussed according to several advanced theories. (auth-tr)

11840 NYO-7440
Carnegie Inst. of Tech., Pittsburgh. Metals Research Lab.
FUNDAMENTAL STUDY OF THE EARLY STAGES OF SINTERING. Progress Report. F. N. Rhines, C. W. Spencer, and R. J. Knight. June 14, 1956. 18p. Contract AT(30-1)-1826. $3.30(ph OTS); $2.40(mf OTS).

The geometric changes occurring at the very beginning of sintering were studied. Very coarse atomized oxygen-free spherical porous Cu particles were sintered without compaction. After sintering, the fracture surface produced by breakage was observed, and measurements were made on polished sections through the sinterings. The number of contact points between powder particles, the growth of the weld areas, changes in specific volume, and particle surface area and curvature were observed. (F.S.)

747 ✓ AECU-3848
Utah. Univ., Salt Lake City. Inst. for the Study of Rate Processes.
EFFECT OF OXIDE ADDITIONS ON THE SINTERING OF MAGNESIA. Technical Report No. II. James W. Nelson and Ivan B. Cutler. June 1, 1956. 48p. Contract AT(11-1)-82. (M-5953). $7.80(ph OTS); $3.30 (mf OTS).

The effect of transition, alkali, alkaline earth and heavy metal oxide additions on the sintering of magnesia was determined by measurements of bulk densities and shrinkages. Zirconium dioxide and the transition metal oxides were found to increase the bulk densities, especially in the region of low sintering temperatures. The alkali, alkaline earth and the volatile heavy metal oxides hindered low temperature sintering. (auth)

9277
ON THE RATE OF SINTERING. Gerhard Bockstiegel (Sintermetallwerk Krebsöge, GmbH, Krebsöge, Germany). J. Metals 8, 580-5(1956) May.

Kuczynski's formula has been derived for the case of non-spherical particles. Two formulas of Kuczynski's type have been derived, one describing the increase in tensile strength, the other describing the progress of shrinkage of a powder compact. It has been shown that the exponents of all three formulas each contain two magnitudes of different physical characters, viz, the geometrical factor α and the kinetic factor β. The interrelationships between the three exponents are stated. (auth)

4614 ISC-288(Suppl.)
Ames Lab., Ames, Iowa.
SINTERING MECHANISM AS APPLIED TO REFRACTORY OXIDES: A BIBLIOGRAPHY. SUPPLEMENT I, 1952–1955. D. R. Wilder. Jan. 15, 1956. 20p. Contract W-7405-eng-82. $0.20(OTS).

Reference and abstracts are given for much of the technical literature published during the years 1952 to 1955 pertaining to the sintering mechanism of refractory oxides. Some prior work which is concerned specifically with later publications has been included. Many of the abstracts have been taken directly from abstract journals or from author abstracts. The listing is alphabetically by authors. (auth)

2743
INTERACTION BETWEEN METALS AND ATMOSPHERES DURING SINTERING. John T. Norton (Massachusetts Inst. of Tech., Cambridge). J. Metals 8, 49-53(1956) Jan.

A study was made of the various types of interaction between metals to be sintered and the atmosphere which surrounds them, depending upon whether the phenomena are essentially chemical or physical in nature. (auth)

1829✓
THE MECHANISM OF SINTERING OF NONVOLATILE METALS AND OXIDES. Leslie L. Seigle and A. L. Pranatis (Sylvania Electric Products Inc., Bayside, N. Y.). Metal Progr. 68, No. 6, 86-90(1955) Dec.

Various sintering mechanisms are discussed, and illustrations are given. It is concluded that volume diffusion is the rate-controlling process in all but the initial stages of sintering of normally sized particles of nonvolatile metals and oxides. The influence of grain boundaries on sintering rates is also discussed. (B.J.H.)

1783
FUNDAMENTAL STUDY AND EQUIPMENT FOR SINTERING AND TESTING OF CERMET BODIES. VII. FABRICATION, TESTING, AND PROPERTIES OF 34 Al_2O_3–66 Cr–Mo CERMETS. Thomas S. Shevlin and Charles A. Hauck (Ohio State Univ., Columbus, Ohio). J. Am. Ceram. Soc. 38, 450-4(1955) Dec.

The properties of a cermet solid body containing 34% Al_2O_3 and 66% 80Cr–20Mo alloy (50Al_2O_3–50 alloy by volume) are given as firing shrinkage, density, modulus of rupture, tensile strength, stress-rupture life, modulus of elasticity, oxidation resistance, thermal-shock resistance, and thermal expansion. It is shown that substitutions of the 80Cr–20Mo alloy for Cr in a similar cermet developed earlier substantially improved thermal-shock resistance. (auth)

13739 WAPD-126
Westinghouse Electric Corp. Atomic Power Div., Pittsburgh.
THE FABRICATION OF DENSE URANIUM DIOXIDE CYLINDRICAL COMPACTS BY COLD PRESSING AND SINTERING. J. Glatter, H. R. Hoge, and B. E. Schaner. Oct. 14, 1955. Decl. Mar. 12, 1957. 23p. Contract AT-11-1-GEN-14. $0.35(OTS).

Pressing and sintering characteristics of Mallinckrodt hydrogen-reduced UO_2 powder are presented. Punch and die materials and factors influencing punch and die design are described. Density and shrinkage data for sintered UO_2 are given. A graphite resistor sintering furnace is described. (auth)

12434 SEP-110
Sylvania Electric Products, Inc., Bayside, N. Y.
EQUIPMENT AND PROCEDURES FOR HOT-PRESSING URANIUM AND URANIUM HYDRIDE POWDERS. H. M. McCullough and J. L. Zambrow. Apr. 1953. Decl. Mar. 6, 1957. 27p. Contract AT-30-1-Gen-366. $0.35(OTS).

Equipment and procedures are described for hot-pressing uranium and uranium hydride powders. The methods of loading dry boxes, construction of vacuum pots and vacuum systems, and die and punch designs are given in detail. The hot-pressing operations are described, showing die preparation, method of die fill, pressing, heating, ejection, and inspection procedures. (auth)

5294 SEP-33
Sylvania Electric Products Inc., Bayside, N. Y.
THE PROCESS OF PULSE SINTERING. Bernard Kopelman. Dec. 22, 1949. Decl. Dec. 6, 1955. 12p. Contract AT-30-1-Gen-366. $0.15(OTS).

The process of pulse sintering is described whereby metal powders or their mixtures are simultaneously pressed and sintered in a matter of seconds to yield materials of essentially theoretical density. This is accomplished by passing momentary surges of electrical current through the pressed compact while the pressure is being applied. The resulting material shows a grain size essentially that of the particle size of the starting powder, with complete random orientation of the grains. Several examples of the usefulness of this method to problems of interest in the atomic energy program are discussed and photomicrographs depict the results that may be obtained. (auth)

THERMAL DIFFUSION 6.19

THERMAL DIFFUSION FACTORS FROM COLUMN OPERATION. T. I. Moran and W. W. Watson (Yale Univ., New Haven). Phys. Rev. 111, 380-3(1958) July 15.

Extending the measurements of Corbett and Watson, the performance of a carefully-constructed, all-metal, hot-wire thermal diffusion column was determined for isotope separations in neon, argon, krypton, and xenon. The same quantitative agreement with theory for normal argon gas at the low wall-temperature ratio of 2 is again found. This is probably fortuitous, for in general there is a discrepancy between the calculated and observed separation factors, with a trend in the data indicating that the assumptions of the theory that (1) the molecules are Maxwellian and (2) the thermal diffusion factors are constant, independent of temperature, are at fault. (auth)

8551
HIGHER APPROXIMATIONS FOR THE TRANSPORT PROPERTIES OF BINARY GAS MIXTURES. III. ISOTOPIC THERMAL DIFFUSION. S. C. Saxena and E. A. Mason (Univ. of Maryland, College Park). J. Chem. Phys. 28, 623-5(1958) Apr.

Tables of the reduced isotopic thermal diffusion factor as a function of a reduced temperature are given for the exp-six potential with potential parameter α = 12, 13, 14, 15, and for the Lennard-Jones (n-6) potential with potential parameter n = 12, ∞. These tables are based on recently derived higher approximations and are intended to supersede previous tabulations, which are of lower accuracy. (auth)

262 UCRL-5116(Pts.I and II)
California. Univ., Livermore. Radiation Lab.
GASEOUS THERMAL DIFFUSION. PART I. BIBLIOGRAPHY OF OPEN LITERATURE, 1940-57. Cecilia McFadden, comp. PART II. A BIBLIOGRAPHY OF THE REPORT LITERATURE, 1941-1957. Frederick E. Frost, comp. Jan. 1958. (Pt. I, 29p. and Pt. II, 7p.) Contract W-7405-eng-48. Pt. I, $4.80(ph OTS); $2.70 (mf OTS); Pt. II, $1.80(ph OTS); $1.80(mf OTS).

These two parts were issued separately, but are cataloged as a unit.

This bibliography includes references on separation of gases by thermal diffusion, thermal diffusion column technique, theory of separating gases by thermal diffusion, and design of thermal diffusion equipment. (W.L.H.)

8970
THE THERMO-OSMOSIS OF THE RARE GASES THROUGH A RUBBER MEMBRANE. Richard J. Bearman (Stanford Univ., Calif.). J. Phys. Chem. 61, 708-13(1957) June.

A theory of thermo-osmosis based on the thermodynamics of irreversible processes is developed. Results on the thermo-osmosis through a rubber membrane of the rare gases (with the exception of radon) and of carbon dioxide are presented and then interpreted. A discussion of various sources of experimental error also is given. (auth)

5373
MASS TRANSFER. C. R. Wilke and John M. Prausnitz (Univ. of California, Berkeley). Ind. Eng. Chem. 49, 577-82(1957) March.

A review is presented on mass transfer. Molecular diffusion, turbulent diffusion, diffusion and convection, interfacial resistance, transfer between two liquid phases, solids, and diffusive separations are the sub-topics considered. 73 references. (T.R.H.)

THERMAL DIFFUSION 6.19

PARAMETERS OF THERMAL DIFFUSION COLUMNS. G. R. Grove, K. W. Foster, and R. E. Vallee (Mound Lab., Miamisburg, Ohio). Chapter 35 of Proc. Symposium on Isotope Separation, Amsterdam, 462-70(1957).

Estimations of hot-wire thermal diffusion separations of non-Maxwellian gases have associated uncertainties which are due in part to the use of cylindricity corrections calculated for a Maxwellian gas ($N = 1$). Separation with no draw-off of mixtures of hydrogen and helium ($N \approx 2/3$) were studied. From data obtained for pressures of maximum separation, equilibrium separation values, and rates of approach to equilibrium, thermal column shape factors were determined in the geometric range $15 < r_1/r_2 < 100$ and the temperature range $2 < T_2/T_1 < 4$. The effective values of the transport, convection, and diffusion shape factors were found to be sufficiently different from those of a Maxwellian gas. Thus, the effect of the non-Maxwellian characteristic of gas mixtures should be taken into account in the optimum design of a critical separation facility. (auth)

5812 ✓

PERFORMANCE OF A HOT-WIRE THERMAL DIFFUSION COLUMN. J. W. Corbett and W. W. Watson (Yale Univ., New Haven, Conn.). Phys. Rev. 101, 519-22(1956) Jan. 15.

The performance of a carefully constructed, metal, hot-wire, thermal diffusion column is examined in the regions of laminar and turbulent gas flow in the column for several hot-wire temperatures using the normal argon isotopic mixture as a column gas. A new experimental method is employed which simplifies and increases the accuracy of the experiment. At the lowest temperature examined, quantitative agreement with theory is observed without the use of a parasitic remixing factor. The effect of spacers on the center wire on column performance was also examined. No significant effect was observed, this being attributed to the careful construction of the column. (auth)

MASS TRANSFER. C. R. Wilke (Univ. of California, Berkeley). *Ind. Eng. Chem.* 47, 658-64(1955).

This review includes references on molecular diffusion in gases and liquids, turbulent diffusion, mass transfer resistance in one phase and two phases, interfacial resistance, mass transfer to and within solids, and diffusive separation processes. 64 references.

7701 ✓ A-531

Columbia Univ., New York. Div. of War Research. THEORETICAL CONSIDERATIONS ON LIQUID THERMAL DIFFUSION EXPERIMENTS. Karl Cohen. Feb. 5, 1943. Decl. Feb. 12, 1957. 22p. Contract OEMsr-412. (4-R-21). $4.80(ph OTS).

ULTRASONICS 6.20

See also, this section,

SOLVENT EXTRACTION--Ultrasonics, Use of (6.16.07).

16311 NYO-7934

Aeroprojects, Inc., West Chester, Penna. APPLICATIONS OF ULTRASONIC ENERGY. Progress Report No. 13 Covering Period from December 1, 1958 to January 31, 1959. Feb. 1959. Changed from OFFICIAL USE ONLY May 1, 1959. 31p. Contract AT(30-1)-1836. $4.80(ph), $2.70(mf) OTS.

Investigations of additive aerosols as supplements in the ultrasonic coalescence of KCl were continued, and of the several additives utilized, the antistatic agent G-3300 appeared to be the most effective. Preliminary experiments were also carried out with submicron NaCl aerosol as a simulant for radioactive particulates in calciner off-gases. Greater analytical sensitivity obtainable with NaCl permitted the observation that particulate concentration could be reduced to less than 8 μg/cu ft through ultrasonic coalescence in a single stage. Controlled experiments indicate ultrasonic dissolution rates of thorium in boiling mercury (350°C) to be 14.8 $mg/cm^2/hr$, with 95% confidence limits of 0.8 $mg/cm^2/hr$. This represents a 1000-fold increase over the non-ultrasonic control, and a 2-fold increase over the ultrasonic rate at 150°C reported earlier. Ultrasonic leaching for 21 hours of a simulated cesium-bearing alumina waste was better than 99% effective with a continuous flow treatment of 1 ml/min of 0.01 N HNO_3 using a ratio of waste to liquid of 1:100. Promising results were also obtained at higher power levels (1000 watts) and shorter time periods in batch-type leaching treatment. (For preceding period see NYO-7933.) (auth)

ULTRASONICS 6.20

8403 NYO-2569
Aeroprojects, Inc., West Chester, Penna.
APPLICATIONS OF ULTRASONIC ENERGY. Progress Report No. 15 Covering Period April 1, 1959 to May 31, 1959. June 1959. 28p. Contract AT(30-1)-1836. OTS.

Further testing of ultrasonic multistage equipment for the agglomeration of dilute radioactive particulates has established a configuration generally suitable for field test work. Experimentation over a limited range of flow rates indicates removal of sodium chloride test aerosols to a level approaching the limit of flame photometric analysis. The ultrasonically activated tantalum crucible obtained for pyrometallurgical extraction has proven to be satisfactory when evaluated by a room-temperature simulant system—ultrasonic extraction of copper from a copper–mercury alloy with dilute nitric acid. Experiments in the ultrasonic disintegration of fuel element cladding in mercury have shown that a direct-coupling technique is superior in effectiveness to close proximity treatment. The elevation of the treatment temperature to the boiling point of mercury has increased the erosion rate of Zircaloy-2 (2.5 mg/cm^2/hr) by approximately 10-fold over that at temperatures up to 200°C. Work with two other types of ultrasonically activated ion-exchange columns indicates increased ultrasonic activity in the resin bed. This permits operation nearer equilibrium conditions and at higher flow rates than with non-ultrasonically activated columns. Both the breakthrough capacity and the apparent total capacity of the resin have been increased. Preliminary work has been initiated on the application of ultrasonic vibration to ceramic processing. (For preceding period see NYO-2568.) (auth)

4710 NAA-SR-2798
Atomics International Div., North American Aviation, Inc., Canoga Park, Calif.
THE FINISHING AND INSPECTION OF URANIUM FUEL SLUGS IN THE PROCESSING REFABRICATION EXPERIMENT. J. Guon, J. R. Foltz, and W. Cockrell. Dec. 15, 1958. 65p. Contract AT(11-1)-GEN-8. $1.75 (OTS).

Methods of remote finishing and inspection of cast, radioactive, Sodium Reactor Experiment-type fuel elements were investigated. Cold short fracture, abrasive, electrical discharge, and ultrasonic methods of machining were studied. Mechanical and optical methods of dimensional inspection were investigated. Subsurface porosity effects were analyzed. Radiographic and ultrasonic techniques were used. (auth)

17465
ULTRASONIC STUDIES IN ELECTROLYTE SOLUTIONS. SALTS OF HEAVY METALS. Bh. Krishnamurty and M. Satyanarayana Murty (Andhra Univ., Waltair). J. Sci. Ind. Research (India) 17B, 216-18(1958) June.

Ultrasonic velocities in aqueous solutions of salts of some heavy metals, viz. lead, thallium, thorium, and uranium, were determined at varying concentrations. Based on these determinations, the adiabatic compressibilities of the solutions were calculated. The ultrasonic velocities were found to decrease with increasing concentration of the salts in the solutions, contrary to the general variation of ultrasonic velocities in the case of other salts which show an increase in velocity. Ultrasonic velocities determined in a solution of a mixture of lead nitrate and acetate are lower than those obtained in solutions of individual salts. This confirms the view that the decrease in ultrasonic velocity in solutions of these salts is due to the presence of heavy metal ions. (auth)

12450 NYO-7930
Aeroprojects, Inc., West Chester, Penna.
APPLICATIONS OF ULTRASONIC ENERGY. Progress Report No. 9 Covering Period from April 1, 1958 to May 31, 1958. June 1958. 63p. Contract AT(30-1)-1836. $10.80(ph OTS); $3.90(mf OTS).

Studies in aqueous precipitation of thorium oxalate under ultrasonic influence were continued. The smallest and most uniform oxalate particles, many of which were in the submicron region, were produced by fast addition of solid oxalic acid to thorium nitrate solution at a temperature of about 20°C followed immediately by 5 min. of ultrasonic treatment, or by fast addition of oxalic acid solution to thorium nitrate solution at 20°C followed by 30 min. of ultrasonic agitation. The shorter treatment time favors the former method. Ultrasonic leaching of a simulated cesium-bearing alumina waste with 0.01 N nitric acid solution was most effectively accomplished in a "fixed-bed" treatment vessel. Extrapolation of experimental data suggested that close to 100% of the radioactive material could be removed from 5 grams of waste in 24 hours by uninterrupted batch-type ultrasonic treatment, using 300 milliliters of nitric acid solution, or in 10 to 12 hours by continuous-flow treatment using 9 to 10 liters of leaching liquid. With the view of applying vibratory energy to accelerate magnesium extraction of plutonium from uranium, preliminary experiments indicated tantalum to be a

potentially satisfactory container material for the molten metal system; the tantalum resisted attack, under ultrasonic influence, by molten uranium–chromium alloy at temperature up to 950°C. Design and construction of a treatment array incorporating a tantalum vessel were initiated. Agglomeration of a dilute aerosol of solid submicron KCl particles (simulating a highly radioactive aerosol waste stream) in an ultrasonic standing-wave field, using an additive liquid aerosol to promote coalescence, indicated that close to 100% of the aerosol could be trapped by this method, although optimum conditions have not yet been established. Ultrasonic agglomeration in liquid systems using an annular standing-wave treatment vessel appeared promising. (auth)

9860 NYO-7924
Aeroprojects, Inc., West Chester, Penna.
ULTRASONICALLY PRODUCED DISPERSIONS OF THORIUM BISMUTHIDE IN MOLTEN BISMUTH. M. L. Bromberg, J. G. Thomas, and W. B. Tarpley. Mar. 1958. 29p. Contract AT(30-1)-1836. $4.80(ph OTS); $2.70(mf OTS).

Ultrasonic energy was applied to the production of finely dispersed thorium bismuthide of potential use in the blanket preparation for the LMFR. A controlled-atmosphere ultrasonic-treatment furnace was assembled for operation at temperatures up to 1200°C, taking advantage of previously developed principles of ultrasonic furnace construction. Using 5% thorium–95% bismuth systems, it was found that when ultrasonic vibration was applied during cooling from complete solution temperature (1050°C) to 450°C, vibratory energy suppressed the normal precipitation of one-centimeter-long thorium bismuthide platelets and produced more nearly equiaxed particles of a size generally less than 50 μ in diameter. When applied to melts containing large platelets formed by cooling under the above conditions without applied ultrasonics, vibratory energy applied isothermally at 450 to 500°C fractured the platelets into generally equiaxed particles smaller than 100 μ. Increased treatment time under these conditions appeared to increase the effectiveness of particle breakup. (auth)

6522 NYO-7927
Aeroprojects, Inc., West Chester, Penna.
APPLICATIONS OF ULTRASONIC ENERGY. Progress Report No. 7 Covering Period from December 1, 1957 to January 31, 1958. Feb. 1958. 50p. Contract AT(30-1)-1836. $7.80(ph OTS); $3.30(mf OTS).

Ultrasonic precipitations of thorium oxalate, carried out by the addition of solid oxalic acid to thorium nitrate solution, yielded smaller and more regularly shaped particles than did non-ultrasonic precipitations involving slow addition of oxalic acid solutions. The importance of digesting the precipitate and of thorough washing to remove excess reactants was emphasized. Preliminary experiments were undertaken in the reaction of solid thorium nitrate and oxalic acid with concomitant water addition and ultrasonic application. Studies were undertaken in the ultrasonic disintegration of composite fuel elements in mercury. Rapid erosion of Hastelloy C, stainless steel, mild steel, molybdenum, and graphite couplers indicate a problem in selection of an ultrasonic coupler material, although it may be possible to use a sacrificial coupler. Relatively short exposure resulted in extensive pitting and erosion of stainless steel sheet. The high erosion rates of the above materials indicate that such claddings may be effectively disrupted in mercury with applied ultrasonics. Ultrasonic leaching of cesium-bearing alumina waste with 0.01N nitric acid indicated a positive ultrasonic effect on rate of leaching in a continuous-flow or fixed-bed array. Undesirable breakup of the calcined waste pellets obtained under certain conditions suggests that non-cavitation leaching should be investigated. Investigation was initiated on ultrasonic application to the magnesium extraction of plutonium from uranium–chromium alloys. Preliminary studies in a room-temperature simulant system indicated high efficiency in emulsifying mercury in water and a substantially increased rate of extraction of copper from mercury with aqueous reagents. The possible use of a tantalum ultrasonic coupler will be investigated prior to tests with the actual system of interest. A theoretical evaluation and experimental program has been initiated on ultrasonic agglomeration of submicron radioactive particles in very dilute gas streams. A new approach, adding an aerosol of particles in the 2 to 10 μ size range of a suitable wetting agent and superimposing ultrasonic vibration, appears promising both theoretically and practically. The production of additive aerosol particles from a viscous non-evaporating surfactant liquid in this size range with the Aeroprojects atomization techniques has been demonstrated. (For preceding period see NYO-7926.) (auth)

7197 NYO-7916
Aeroprojects, Inc., West Chester, Penna.
APPLICATIONS OF ULTRASONIC ENERGY. Progress Report No. 2 Covering Period from December 16, 1956 to February 15, 1957. Apr. 1957. 24p. Contract AT(30-1)-1836. $4.80(ph OTS); $2.70(mf OTS).

Significant increase in dissolution rates of Type 304 stainless steel and of Zircaloy-2 was observed as a direct result of ultrasonic treatment. Investigation of the effect of temperature on the ultrasonic dissolution rate of stainless steel indicated a minor increase in rate with increased temperature. An ultrasonic controlled-atmosphere furnace has been designed to permit treatment of melts of thorium bismuthide varying in depth from $^1/_{32}$ inch to 2 inches. Fabrication of the furnace is about one-third complete. Ultrasonic transducer-coupler-punch systems for treatment of powdered metal compacts have been designed and partially fabricated for installation in a 100-ton hydraulic press, and much of the auxiliary equipment has been assembled. Metal powders to be used in the experimental investigation have been procured. (auth)

18905 NYO-7932
Aeroprojects, Inc., West Chester, Penna.
APPLICATIONS OF ULTRASONIC ENERGY. Progress Report No. 11 Covering Period from August 1, 1958 to September 30, 1958. Oct. 1958. 33p. Contract AT-(30-1)-1836. $4.80(ph), $2.70(mf) OTS.

Continuous aqueous precipitation of thorium oxalate was accomplished using impinging jet streams of the reactants in an ultrasonically activated zone. Evaluation of the influence on resultant particle size was obtained through photomicrographs and centrifugal sedimentation size analysis. Log probability plots showed a significant decrease in particle size and a narrowing of the size distribution for the ultrasonically prepared material as compared to the control. Leaching of a cesium-bearing alumina waste with 0.01 N nitric acid in the presence of vibratory energy was less effective at a frequency of 800 kc than at 20 kc. The effect of treatment time, power level, and ratio of leach liquid to waste was investigated with a 20-kc "fixed-bed" treatment vessel. Leaching efficiency was increased at the higher power levels and the same efficiency maintained when the ratio of waste to acid was increased. Intermittent treatment may be effective, with promise for further equipment economies. Several liquid additive aerosols were evaluated in the ultrasonic agglomeration of KCl aerosol, a simulant for radioactive particulates in calciner off-gas. Silicone oils of several viscosities and two liquid surfactants produced essentially the same degree of agglomeration as the glycerine-water mixture previously used. Development of a larger unit for field test was initiated. Studies of ultrasonic coalescence in stabilized kerosene-water emulsions led to the conclusion that a longer exposure time is indicated. Therefore fabrication was undertaken on a new annular treatment vessel which will permit a two-fold increase in treatment time at the same linear velocity previously found to be near optimum. (For preceding period see NYO-7931.) (auth)

18904 NYO-2568
Aeroprojects, Inc., West Chester, Penna.
APPLICATIONS OF ULTRASONIC ENERGY. Progress Report No. 14 Covering Period from February 1, 1959 to March 31, 1959. Apr. 1959. 45p. Contract AT(30-1)-1836. $7.80(ph), $3.30(mf) OTS.

Several surfactants were evaluated in the ultrasonic agglomeration of KCl aerosol, a simulant for radioactive dispersions. These experimental studies indicate that the extent of coalescence is probably influenced by chemical properties of the aerosols. Multistage equipment for gaseous coalescence was assembled, and preliminary tests indicated it to be more effective than single-stage agglomeration. The tantalum crucible assembly for the ultrasonic extraction of plutonium from uranium with molten magnesium has been received. In preliminary testing of this vessel, at room temperature, microphone measurements revealed a relatively uniform ultrasonic intensity field across the crucible and slightly decreasing intensity from the bottom to the top of the vessel. Additional measurements of the ultrasonic dissolution rate of thorium in mercury showed the rate of attack to be significantly higher at the boiling point of mercury than at lower temperatures. Under the conditions of the experiment, the rate of attack was independent of the exposure time. The rate of ultrasonic erosion of AISI 304 stainless steel in boiling mercury was found to be somewhat lower than the rate at 150°C, confirming the previously observed trend toward reduced erosion rate with increasing temperature. On the basis of current data, it appears that ultrasonic leaching of a simulated alumina waste containing cesium can be effectively accomplished at a power level of 100 watts in 24 hours. Continuous flow treatment was ef-

fective, using 0.01 N HNO_3 and a waste-to-leaching liquid ratio of 1:100. Batch treatment was less effective, even at higher power levels. Preliminary work has indicated an increase in the total capacity of an ion exchange resin in a test column operation when ultrasonic energy is applied. In addition, the breakthrough capacity was increased, and the column performed more nearly at equilibrium conditions than in a control run without ultrasonics. (For preceding period see NYO-7934.) (auth)

ZONE MELTING 6.21

See also, under FISSIONABLE MATERIAL--RECOVERY;

RECOVERY OF IRRADIATED TH AND U

--Fused Salts and Liquid Metals (3.02.11),

--Zone Melting (3.02.19).

12731 ✓
METHODS OF AND APPARATUS FOR TREATING SOLID–LIQUID SYSTEMS. (to Western Electric Co., Inc.). British Patent 810,448. Mar. 18, 1959.

Apparatus for electromagnetically suspending and zone refining a metal rod is described. An electric current is passed through a rod of metal to be purified enclosed in a quartz tube. A carriage is provided for passing the rod between the pole faces of a strong electromagnet. The interaction of the magnetic field and electric field opposed at 90° causes a portion of the rod to be melted and suspended. Thus as this molten zone is caused to move along the rod impurities are exchanged across the liquid-solid interface. The regulation of currents and control of the apparatus are discussed along with some of the difficulties involved. A means for cooling the pole-faces is given. (T.R.H.)

11469 ✓
ATTEMPT TO PURIFY METALS BY ZONE MELTING WITH AN ARC. G. Cabane (Dept. de Métallurgie et de Chimie Appliquée, Paris). J. Nuclear Energy 6, 269-74(1958) May. (In French)

The most reactive metals can be melted without contamination by arc-melting on a water-cooled copper hearth. For the purification of these metals, the zone melting process was applied with an apparatus in which a zone of a metallic rod resting in the groove of a copper hearth is melted by a tungsten arc. Efficiency of arc zone melting was checked for uranium, zirconium, and thorium purification. Advantages and limitation of the process are outlined. (auth)

7864
THE ULTIMATE DISTRIBUTION OF IMPURITY IN THE ZONE-MELTING PROCESS. L. W. Davies C.S.I.R.O., Sydney). Phil. Mag. (8) 3, 159-62(1958) Feb.

The ultimate distribution of a given impurity in a metal ingot after the passage of an infinite number of molten zones was determined analytically. (A.C.)

7865
ELECTROMAGNETIC STIRRING IN ZONE REFINING. I. Braun, F. C. Frank, S. Marshall, and G. Meyrick (Univ. of Bristol, Eng.). Phil. Mag. (8) 3, 208-9(1958) Feb.

The efficiency of the zone refining process can obviously be increased by stirring the molten zone to disperse the impurity-rich layer at the solid-liquid surface. Induction heating is sometimes preferred to radiant heat because it produces more convection; but no marked improvement has been reported. Pfann and Dorsi(1957) have described a method of stirring the melt by passing an electric current through the ingot and impressing a magnetic field across the molten zone. Preliminary results obtained by using a rotating magnetic field as the stirring agent during the purification of aluminum are described. (A.C.)

ZONE MELTING. William G. Pfann. New York, John Wiley & Sons, Inc., 1958. 246p.

The normal freezing and the distribution coefficient in zone melting are presented. The theory of zone refining is discussed. Various techniques of zone refining are presented. The applications of zone refining to semiconductors, metals, and chemicals are discussed. The principles of continuous zone refining are presented. Zone leveling and single-crystal growth are discussed along with methods of perturbing the concentration and temperature-gradient zone melting. (W.L.H.)

ZONE MELTING 6.21

DEVELOPMENTS IN MELT REFINING OF REACTOR FUELS. L. Burris, N. R. Chellew, S. Lawroski, G. A. Bennett, A. A. Chilenskas, M. Ader, H. M. Feder, J. B. Knighton, I. O. Winsch, J. Wolkoff, and W. A. Rodger. 12p. A/CONF.15/P/538

Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.

After investigation of various oxides, zirconia were selected as the crucible material. Data are presented on Ce removal rates from up to 5 kg of material, determined as a function of temperature. Studies were also made on the possibility of effecting separation of Zr as a carbide or nitride. Separation of purified metal is effected by top pouring. On a 2 to 5 kg scale purified ingot yields were slightly greater than 95% using charges of small reactor fuel pins. Methods are discussed for handling fission product gases and volatilized metals including Na which may accompany the fuel pin charges and the volatile fission product metals such as Cs. A brief description is given of the auxiliary process under development for processing crucible residues and material deliberately removed to provide the necessary periodic removal of noble elements. A description of the equipment and tests of various materials of fabrication are discussed. The results of preliminary experiments to ascertain the behavior of plutonium are also described.

20446

SOLID STATE PHYSICS. VOLUME 4. ADVANCES IN RESEARCH AND APPLICATIONS. Frederick Seitz and David Turnbull, eds. New York, Academic Press, Inc., 1957. 549p.

Research activity is presented of solid state physics on ferroelectrics and antiferroelectrics, mobility of electrons in solids, orthogonalized plane-wave method, bibliography of atomic wave functions, and techniques of zone melting and crystal growing. (W.D.M.)

5664 ✓ OSR-TN-56-70

Franklin Inst. Labs. for Research and Development, Philadelphia.

ZONE PURIFICATION OF REACTIVE METALS. Interim Report. R. L. Smith and J. L. Rutherford. Feb. 1, 1956. 37p. (I-A1878-1; AD-81522).

Apparatus and techniques used to zone-melt reactive, high-melting-point metals by the floating zone method are described. The use of radioactive tracers to determine the redistribution of solute during zone melting is discussed and results are shown for P^{32} in iron, Au^{198} in titanium and zirconium, and Zn^{65} in titanium. Some tensile results on Fe at liquid N_2 and liquid He temperatures are given which indicate that the removal of trace impurities may increase markedly the low temperature ductility of Fe. (auth)

PELLETIZING 6.22

3287 ✓ UCRL-4629

California. Univ., Livermore. Radiation Lab.

RELATIONSHIPS BETWEEN APPLIED PRESSURE, DENSITY, AND LENGTH IN PRESSED COMPACTS. Barney Rubin. Dec. 15, 1955. 12p. Contract W-7405-eng-48. $0.15(OTS).

Equations have been derived for expressing the variations with length of true and average densities of a die-pressed compact. For any given set of pressing conditions and materials whose parameters can be determined, these expressions permit calculation of the variation of density within such compacts as functions of pressure, compact area, compact perimeter, and compact length. (auth)