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CHEMISTRY—SEPARATION PROCESSES
FOR PLUTONIUM AND URANIUM

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BIBLIOGRAPHY ON NUCLEAR REACTOR FUEL REPROCESSING AND WASTE DISPOSAL

Volume 3

Fissionable Material—Recovery

T. F. Connolly

Oak Ridge National Laboratory
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Union Carbide Corporation

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T. F. Connolly

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July 30, 1962

United States Atomic Energy Commission
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PROCESSING OF NUCLEAR REACTOR FUELS – A BIBLIOGRAPHY

Introduction

This volume includes Section 3.0 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).

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FISSIONABLE MATERIAL-RECOVERY 3.00

HEAD-END TREATMENTS 3.01

Al-FUELS 3.01.01

6362
REMOVAL OF ALUMINUM COATINGS. J. H. Peterson (to U. S. Atomic Energy Commission). U. S. Patent 2,901,343. Aug. 25, 1959.

A process is presented for dissolving aluminum jackets from uranium fuel elements without attack of the uranium in a boiling nitric acid–mercuric nitrate solution containing up to 50% by weight of nitric acid and mercuric nitrate in a concentration of between 0.05 and 1% by weight.

2484
PROCESS FOR REMOVING ALUMINUM COATINGS. J. Flox (to U. S. Atomic Energy Commission). U. S. Patent 2,893,863. July 7, 1959.

A process is presented for removing aluminum jackets or cans from uranium slugs. This is accomplished by immersing the aluminum coated uranium slugs in an aqueous solution of 9 to 20% sodium hydroxide and 35 to 12% sodium nitrate to selectively dissolve the aluminum coating, the amount of solution being such as to obtain a molar ratio of sodium hydroxide to aluminum of at least 1.65.

CONTINUOUS DISSOLUTION OF URANIUM–ALUMINUM FUELS IN A TRICKLE-TYPE COLUMN DISSOLVER. J. C. Bresee (Oak Ridge National Lab., Tenn.); D. L. Foster (Convair, Fort Worth, Tex.); and E. O. Nurmi (Engelhard Industries, Inc., Newark, N. J.). Chem. Eng. Progr. 55, Symposium Ser. No. 22, 25-32(1959).

The performance of a full-scale continuous dissolver with suitable dimensions for power reactor enriched fuel dissolution was investigated. Results on a full-scale continuous trickle-type column dissolver for enriched uranium–aluminum fuels are presented. Both cast and extruded unirradiated alloys were dissolved, and correlations relating dissolving rate and product concentration to feed acid rate and concentration for both types of alloy are given. The application of the correlations to column design and operation is discussed. (auth)

15176 ✓ DP-313
Du Pont de Nemours (E. I.) & Co. Savannah River Lab., Aiken, S. C.
DISSOLVER FOR NATURAL URANIUM FUEL ELEMENTS. Vincent P. Caracciolo. Dec. 1958. 23p. Contract AT(07-2)-1. $0.75(OTS).

Sufficient data were obtained with a semiworks-scale dissolver to permit design of plant-scale equipment for the dejacketing and subsequent dissolving of aluminum-clad uranium fuel elements of various shapes and sizes. The aluminum dejacketing reaction occurred as fast as caustic was added and was independent of the shape of the elements. The rate of uranium dissolving increased with increasing acidity, was proportional to the exposed surface area, and was independent of the shape of the elements. A mechanism for uranium dissolution is discussed that explains the variation of acid consumption and the rate of gas evolution. (auth)

6335 TID-7583(p.187-94)
Du Pont de Nemours (E. I.) & Co., Wilmington, Del.
CHEMICAL PROCESSING OF CHALK RIVER FUELS. H. L. Hull. 8p.

One of the existing Savannah River Plant dissolvers will be modified to accommodate eleven-foot long fuel elements from Chalk River, Canada. The aluminum cladding will be removed with sodium hydroxide, and the uranium cores will be dissolved in nitric acid. The Purex process, operated in mixer settlers, will be used to recover and decontaminate the uranium and plutonium in the core solution. (auth)

5257 IDO-14468
Phillips Petroleum Co. Atomic Energy Div., Idaho Falls, Idaho.
A GELATIN-FILTRATION HEADEND FOR FUEL REPROCESSING SOLUTIONS FROM SILICON-CONTAINING ALUMINUM ALLOYS. Bill J. Newby and Bernice E. Paige Feb. 6, 1959. 54p. Contract AT(10-1)-205. OTS.

A laboratory study of a gelatin headend process for feed from silicon-containing aluminum fuels and plant salvage solutions is described. The optimum conditions for the gelatin treatment of fuel solutions were to boil a 0.1 to 0.5N nitric acid solution with 100 milligrams of gelatin per liter for 30 minutes. This treatment improved filtration rates and decreased the surface activity of the filtrate for TBP extraction. A number of possible flowsheets for fuel solutions are presented using gelatin treatment and filtration. An adequate treatment was not found for salvage solutions of unknown composition because a gelatin dosage which was satisfactory for all solutions could not be selected. The optimum treatment for a salvage solution which was grossly contaminated with zirconium, soluble and colloidal silica, and dibutyl phosphate was to boil a 1N acid deficient solution with 600 milligrams of gelatin per liter, filter, and use a Hexone extraction system. A silicic colloid in fuel processing solutions was characterized as a surface active material by this study. (auth)

HEAD-END TREATMENTS 3.01

Al-FUELS 3.01.01

2819
URANIUM–ALUMINUM ALLOY DISSOLUTION. R. G. Wymer and R. E. Blanco (Oak Ridge National Lab., Tenn.). Ind. Eng. Chem. 49, No. 1, 59-61(1957) Jan.

Dissolution of heterogeneous nuclear reactor fuel elements is the initial step in aqueous processes for the recovery of fissionable and fertile materials. This step converts the solid fuel to a liquid process feed material. Common fuel materials such as uranium–aluminum alloy and aluminum jackets for uranium or thorium core materials are readily soluble in nitric acid catalyzed by mercuric nitrate or in sodium hydroxide solutions. Jacket removal by caustic dissolution has the advantage of selective dissolution in that the core materials remain untouched. Sodium nitrate can be added to the caustic to suppress hydrogen evolution and reduce the explosion hazard. This paper reports a study of the nitric acid and caustic systems as applied to uranium–aluminum alloy dissolution.

5334 IDO-14451
Phillips Petroleum Co. Atomic Energy Div., Idaho Falls, Idaho.
CONTINUOUS DISSOLVER THEORY. II. APPLICATION OF TUBE-FLOW DISSOLVER THEORY TO DATA FROM A 2-INCH, CONTINUOUS FLOODED DISSOLVER. E. E. Erickson. Jan. 14, 1959. 51p. Contract AT(10-1)-205. $1.50(OTS).

General relationships for the performance of a continuous flooded dissolver, derived earlier, were applied to available pilot plant data on the mercury-catalyzed dissolution of 2S aluminum in nitric acid for four shapes of metal elements. The relationships were based on nonmixing flow of the liquid phase, uniform metal packing characteristics, and a first-order reaction rate law. The metal elements were round rods ¼ inch in diameter and 3 inches long, 5 × 0.625 × 0.0625-inch flat plates, tubes 0.625 inch in diameter and 3 inches long with a 0.028-inch wall thickness, and the same tubes flattened so as to have about a 0.03-inch gap between sides. The dissolver was a 2-inch Pyrex column, and the effective height ranged from 8 to 10 feet. Some data were available also for the dissolution of the round rods and tubes in which the height of the bed of elements varied from about 1 to 10 feet. The average nitric acid concentration in the dissolvent feed was 5.6 molar. The data for round rods were used to determine effective reaction velocity constants for catalyst concentrations ranging from 1.5×10^{-6} to 3.75×10^{-4} molar Hg^{2+}. The general equation was used to predict the effect of the shape of metal elements, the flow rate of the dissolvent, and the height of bed of elements on the dissolution rate. The calculated rate followed the trends in the data and showed the model as expressed by the dissolver equation to give a picture generally consistent with experimental results from the pilot plant. (auth)

12974 DP-297
Du Pont de Nemours (E. I.) & Co. Savannah River Lab., Augusta, Ga.
DISSOCIATION OF NITRIC ACID IN ALUMINUM NITRATE SOLUTIONS. Robert C. Axtmann and Bruce B. Murray. June 1958. 11p. Contract AT(07-2)-1. $0.50(OTS).

The proton magnetic resonance spectra of the HNO_3–H_2O–$Al(NO_3)_3$ system indicate that the dissociation of nitric acid is repressed significantly by aluminum nitrate. Values for α, the dissociation constant of nitric acid, obtained from these spectra, are given as a function of aluminum ion concentration and of nitric acid concentration. (auth)

7122 IDO-14425
Phillips Petroleum Co. Atomic Energy Div., Idaho Falls, Idaho.
EFFECT OF GEOMETRICAL SHAPE ON THE CONTINUOUS DISSOLUTION OF ALUMINUM IN MERCURY–CATALYZED NITRIC ACID. A. F. Boeglin and J. A. Buckham. Dec. 31, 1957. 26p. Contract AT(10-1)-205. $4.80(ph OTS); $2.70(mf OTS).

The results of a study of the effect of geometrical shape of aluminum reactor fuel elements on the dissolution rate in mercury-catalyzed nitric acid are presented. Four shapes, round rods, tubes, flat plates, and flattened tubes, were tested at five catalyst concentrations (from 1.5×10^{-6} to 7.5×10^{-4} molar mercury), and at each of three feed rates (280, 140, and 70 gram-moles of nitric acid per hour). Statistical analysis of the data showed that the main effects, feed rates, catalyst concentration and shape, and all first order interaction effects had a significant effect on dissolution rate. A mathematical relationship between dissolution rate and feed rate, catalyst concentration and the catalyst concentration by feed rate interaction was graphically developed for each shape. At the higher catalyst concentrations, all shapes dissolve at practically the same rate. However, flat plates and flattened tubes are much less dependent on catalyst concentration and at lower catalyst concentrations dissolve over twice as fast as round rods and tubes. Correlation of the dissolution rates of round rods with the rates obtained in previous work on cylindrical uranium–aluminum alloy fuel elements showed very good agreement. A comparison of the dissolution rate obtained in a stainless steel vessel and that obtained in a glass vessel was made; no appreciable difference was noted. (auth)

HEAD-END TREATMENTS 3.01

Al-FUELS 3.01.01

10470 IDO-14407
Phillips Petroleum Co. Atomic Energy Div., Idaho Falls, Idaho.
STATUS REPORT ON STUDIES OF URANIUM–ALUMINUM ALLOY DISSOLUTION TO A DIBAN DISSOLVER PRODUCT. R. L. Andelin, F. H. Tingey, and C. M. Slansky. June 17, 1957. 45p. Contract AT(10-1)-205. $0.55(OTS).

A series of statistically designed, batch dissolution experiments was conducted with Al–U fuel elements to determine their optimum dissolution rate in HNO_3, with Hg and fluoride catalysts, to an acid-deficient dissolver product. The optimum, average rate to a 4.0M Al, 3.5M acid-deficient solution is 69.8 mg/(cm^2)(hr). The optimum average rate to a 3.0M Al, 2.1M acid-deficient solution is 196.3 mg/(cm^2)(hr). These rates are considered feasible for application to plant processes. (auth)

4139 Y-1176
Union Carbide Nuclear Co. Y-12 Plant, Oak Ridge, Tenn.
AN IMPROVED METHOD OF MEASURING URANIUM CONCENTRATION IN URANIUM–ALUMINUM ALLOY BY GAMMA COUNTING. P. F. Haughton, C. R. Fultz, and L. E. Burkhart. Mar. 29, 1957. 17p. Contract W-7405-eng-26. $0.75(OTS).

A scintillation counter has previously been used to measure U concentration in U–Al alloy reactor slugs. The present method is an improvement in that the entire slug is now being counted, using a larger crystal and multiplier phototube. The U distribution in the individual slugs is discussed. This, plus improvements in equipment used, gives a somewhat lower limit of error. Data on 105 cast and 75 machined slugs are given and analyzed. Various applications are discussed. (auth)

6601
CONTINUOUS DISSOLUTION OF URANIUM–ALUMINUM REACTOR FUELS. A. F. Boeglin, J. A. Buckham, L. Chajson, R. B. Lemon, D. M. Paige, and C. E. Stoops (Phillips Petroleum Co., Idaho Falls, Idaho). A.I.Ch.E. Journal 2, 190-4(1956) June.

Extensive pilot plant studies of the continuous, mercury-catalyzed nitric acid dissolution of uranium–aluminum alloy materials similar to possible reactor fuel elements were carried out. Marked differences were observed in the dissolution rates of cast and wrought alloys. Optimum feed-acid concentrations varied with the type of alloy. At constant acid feed conditions dissolving rates varied approximately with the cube root of catalyst concentration up to a limiting concentration. The metal dissolving rate was proportional to the 0.8 power of the nitric acid feed rate. A general empirical correlation was developed. (auth)

196
SEPARATION OF HIGH SPECIFIC ACTIVITY Na^{22} FROM IRRADIATED ALUMINUM. N. Hollbach and L. Yaffe (McGill Univ., Montreal, Quebec, Canada). Can. J. Chem. 34, 1508-9(1956) Oct.

A method has been developed for the separation of high specific activity Na^{22} from irradiated Al by a cation exchange resin. The process recovered 99.8% of the original Na^{22} activity and was radiochemically pure. (W.L.H.).

2744
EXAMINING IRRADIATED FUELS UNDER WATER. Henry J. Bellarts, John L. Spencer, and Roy S. Peterson (Hanford Atomic Products Operation, Richland, Wash.). Nucleonics 14, No. 1, 30-3(1956) Jan.

An underwater facility is described which consists of a primary inspection station, equipment for cleaning slugs, and a dejacketer, where the Al can is removed from the U. The facility is designed to examine 128 slugs per 8-hr shift. The equipment and procedures are described and illustrated, and the advantages of underwater facilities over hot cells are summarized. (M.P.G.)

1328 ORNL-1966
Oak Ridge National Lab., Tenn.
THE REMOVAL OF FISSION PRODUCTS FROM AN ACID ALUMINUM NITRATE SOLUTION BY CO-PRECIPITATION METHODS. Herman L. Krieger, Bernd Kahn, and Conrad P. Straub. Oct. 20, 1955. 19p. Contract W-7405-eng-26.

The removal of the major long-lived fission products by co-precipitation from an acid aluminum nitrate solution has been studied. Of the methods investigated the most efficient and economical treatment was found to be the co-precipitation of Ru and Cs with copper ferrocyanide; of Sr with barium sulfate; of Zr and Nb with zirconyl phosphate; and of the rare earths and Y with cerium oxalate. Approximately 95% of the added tracers and carriers were removed from solution in a precipitate of about 50 g/l at a cost of 1.5 cents per liter. (auth)

DEVELOPMENT OF A METHOD FOR PREDICTING CONTINUOUS TRICKLE-TYPE COLUMN DISSOLVER BEHAVIOR FROM BATCH DATA. R. G. Wymer. May 14, 1954. Decl. Apr. 2, 1957. 10p. (CF-54-5-74)

A method for determining dissolver solution concentration as a function of length of column traversed for trickle-type continuous dissolvers has been developed. An illustrative example is worked out for the Hg-catalyzed HNO_3 dissolution of U–Al cast alloy slugs. The method employs a graphical integration of simple functions of dissolution rates and acid concentrations. These rates and concentrations are easily obtained from batch experiments made over the important ranges of concentration.

HEAD-END TREATMENTS 3.01

Al-FUELS 3.01.01

METALLURGICAL FACTORS AFFECTING THE DISSOLVING RATE OF 7.5% U–92.5% Al ALLOY. D. E. Hamby. Dec. 6, 1950. Decl. Feb. 20, 1957. 11p. (CF-50-12-23)

It was shown that there is a marked difference in the dissolving rate of cast slugs and extruded slugs of U–Al alloys. A metallographic study of the slugs reveals a possible explanation of the observed results. The compound UAl_4 is known to be dissolved by acid more rapidly than is pure Al. The UAl_4 compound particles are distributed differently depending upon the mode of solidification and fabrication. The rate of solution may depend to a great extent upon the relative surface areas of the Al and UAl_4. Photomicrographs show the distribution of the eutectic in the cast structure and in the extruded material. In the extruded material the compound particles are more or less uniformly distributed in a matrix of Al, whereas in the cast slug, the compound is present in the form of a eutectic network around the grain boundaries of pure Al. The acid can attack the alloy as a whole more readily if the easily dissolved UAl_4 is uniformly dispersed as in the case of the extruded material, than when segregated at the grain boundaries as in the cast slugs.

Bi-U FUELS 3.01.02

See also, this section,

FUSED SALTS AND LIQUID METALS-- Fused Salts (3.02.11.01)

Fused salts containing lithium undesirable. Li (a good neutron absorber) exchanges with the Bi in the fuel.

14420

METHOD OF SEPARATING FISSION PRODUCTS FROM FUSED BISMUTH-CONTAINING URANIUM. R. H. Wiswall (to U. S. Atomic Energy Commission). U. S. Patent 2,840,464. June 24, 1958.

A process is described for removing metal selectively from liquid metal compositions. The method effects separation of fission product metals selectively from dilute solution in fused bismuth, which contains uranium in solution without removal of more than 1% of the uranium. The process comprises contacting the fused bismuth with a fused salt composition consisting of sodium, potassium and lithium chlorides, adding to fused bismuth and molten salt a quantity of bismuth chloride which is stoichiometrically required to convert the fission product metals to be removed to their chlorides which are more stable in the fused salt than in the molten metal and are, therefore, preferentially taken up in the fused salt phase.

REMOVAL OF FISSION PRODUCTS FROM URANIUM–BISMUTH FUELS. O. E. Dwyer, A. M. Eshaya, and F. B. Hill. 24p. (A/CONF.15/P/1781)

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

Processing of fuel, consisting of U, Mg, and Zr in Bi, for the LMFR is described, and recent experimental results are discussed. The volatile fission products to be removed are Xe and Kr, which are removed by the same methods. Non-volatile fission products include high cross-section rare earths; these can be removed from the fuel by fused chloride melts. Two methods of removal of Zr to minimize corrosion are described. A method is given for removal of fission products whose chlorides are less stable than $BiCl_3$ and those whose chlorides are more stable than $BiCl_3$ but less stable than UCl_3.

6599

PROCESS FOR FISSION-PRODUCT REMOVAL FROM URANIUM–BISMUTH REACTOR FUELS BY USE OF FUSED-SALT EXTRACTION. O. E. Dwyer (Brookhaven National Lab., Upton, N. Y.). A.I.Ch.E. Journal 2, 163-8(1956) June.

The Liquid Metal Fuel Reactor under development at the Brookhaven National Laboratory uses a fuel which is a solution of U^{233}, Mg, and Zr in liquid bismuth. For a power breeder thermal reactor, high neutron economy is essential, and this calls for low concentrations of those fission products in the fuel which are high neutron capturers. Roughly 45% by weight of the fission products can be continuously removed from the fuel by salt extraction with alkali and alkaline-earth fused-salt mixtures. These fission products contain the highly "poisonous" rare earths. A discussion of process design considerations and proposed flow sheets is presented. (auth)

HEAD-END TREATMENTS 3.01

DECAY STORAGE 3.01.03

See also, under HAZARDS AND PROTECTION,

SHIPPING AND HANDLING OF RADIOACTIVE MATERIALS (4.02).

FAST REACTOR FUEL PROCESSING—THE ECONOMIC IMPORTANCE OF COOLING TIME. F. Roberts and K. M. Towers. Feb. 1957. 9p. (RCTC/P-92)

The economic implications of shortening fuel cooling time have been studied for a 1000 Mw fast reactor power station using a Pu alloy fuel in the core. The likely saving on annual interest charges by reducing cooling time from 120 down to 30 days would be equivalent to a reduction in processing plant costs of 50%, if a Pu cost of 85 per gram is assumed. It is considered that a pyrometallurgical process should be developed, as this represents the most favorable approach to shortening cooling times.

13399 ✓ HW-50156
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
SOLUBLE POISONS FOR SLIGHTLY ENRICHED URANIUM SYSTEMS. N. Ketzlach. May 9, 1957. Decl. May 5, 1959. 12p. Contract [W-31-109-Eng-52]. $3.30(ph), $2.40(mf) OTS.

A study of B and Th poisoning of slightly enriched U^{235} heterogeneous and homogeneous systems has been made. This study indicates large processing plant capacity increases are possible by the incorporation of soluble neutron poisons. A tabulation of other readily available neutron poisons together with their poisoning effects has been made. The importance of being able to remove the neutron poisons when desired as well as having them present under all conditions where nuclear safety is dependent upon them has also been presented. (auth)

Decay-Heat Cooling Requirements of Spent Fuel from EBR-2, C.H. Scheibelhut. Nucleonics 14, 12, Dec 1956, 57-60, 62.

After removing reactor fuel subassembly from sodium, fuel still generates about 2 kw of decay heat; hence cooling is needed to prevent damage that would complicate recovery of fuel from subassembly; tests are described which show that best way to provide this cooling is by forced convection of argon, which is atmosphere in disassembly cell.

23032 ✓ CF-59-8-103
Oak Ridge National Lab., Tenn.
A PRELIMINARY REPORT ON IRRADIATED ORR FUEL ELEMENT TEMPERATURES IN AIR. J. F. Wett, Jr. Aug. 26, 1959. 11p. $3.30(ph), $2.40(mf) OTS.

Irradiated ORR fuel elements were raised into the hot cell at the ORR and their plate temperatures recorded. It was concluded that an ORR element can be safely cooled by stagnant air after a decay of approximately one day. (J.E.D.)

12577 NAA-SR-Memo-113
North American Aviation, Inc., [Downey, Calif.].
THE EFFECT OF DECAY TIME ON HEAT GENERATION IN AN URANIUM-IMPREGNATED GRAPHITE FUEL ELEMENT AFTER REMOVAL FROM A REACTOR. S. Nakazato. [195?]. Decl. Feb. 27, 1957. 11p. $3.30 (ph OTS); $2.40(mf OTS).

The equation which relates the excess temperature of a fuel rod with the radioactivity decay time has been derived. The approximation equation for the maximum excess temperature as a function of cooling time has been obtained. The heat liberation from decay of fission product has been estimated from the Borst-Wheeler curve. The results show that a high rate of removal from the rod should be maintained by a rapid circulation of fluid and by an increase of surface area. The results also show that the transfer period may be the factor which determines the cooling period. Radiation damage on the solvent may be the greatest determining factor in the selecting of the appropriate cooling interval. (auth)

DECAY AND STORAGE OF IRRADIATED FUEL. J. W. Ullmann and E. D. Arnold. Apr. 9, 1956. 19p. (CF-56-4-51)

Reasons for storage to allow decay of irradiated fuel elements and possible methods of storage are given. The effects on storage and inventory costs of fuel element composition, plant size, element geometry, reactor type, methods of irradiation and recycle, and type of metallurgical handling are discussed. Estimates and comparisons are included for the decay for several typical fuels. The special problems associated with Th fuel elements are considered.

HEAD-END TREATMENTS 3.01

DECAY STORAGE 3.01.03

Choosing Storage Time to Minimize Processing Costs, J.W. Ullmann, E. D. Arnold. Nucleonics 15, 6 June 1957, p 80-3.

Problems attending reprocessing of irradiated fuel; to determine optimum fuel cycle, cost of storing spent fuel must be balanced against processing, refabrication and shielding costs; analysis of these factors for various fuels and reactor types; two hypothetical cases considered.

9617 CF-52-3-215
Oak Ridge National Lab., Tenn.
HOMOGENEOUS REACTOR CHEMICAL PROCESSING. Quarterly Report for the Period Ending March 31, 1952. D. E. Ferguson. Mar. 25, 1952. Decl. Feb. 20, 1957. 25p. Contract [W-7405-eng-26]. $4.80(ph OTS); $2.70 (mf OTS).

The development of homogeneous reactor chemical processing has been based on a Pu producer using a UO_2SO_4 heavy water solution as fuel. The general approach has been to study application of tributyl phosphate solvent extraction to this separation problem, and to use this process as a yardstick by which to evaluate attractive alternate methods of separation. The work has now progressed to the point at which a reliable chemical flowsheet has been obtained; however, this flowsheet does not necessarily represent the optimum method of chemical processing. For instance, the inherent advantage of starting with U in solution is lost because of the necessity of heavy water recovery, and the long cooling period for Np decay necessitates large storage facilities and inventories of valuable materials. (auth)

HRT-CP: DESIGN AND OPERATION OF THE RECOMBINERS FOR THE HRT CHEMICAL PROCESSING PLANT. William L. Carter. Mar. 8, 1956. 20p. (CF-56-3-139)

Radiolytic decomposition of heavy water in the decay-storage vessels of the HRT chemical processing plant requires that a recombiner be installed for D_2 and O_2 recovery. Decomposition gases will be diluted with steam, passed through an entrainment separator, superheated and passed over a platinized Al_2O_3 catalyst. D_2 and O_2 will be present in stoichiometric amounts at concentrations well into the non-explosive region. After recombination the D_2O vapor will be totally condensed and refluxed to the decay-storage tank. The rate of water decomposition in the chemical plant is quite small when compared to the same reaction in the HRT core system. In fact sufficient steam will be produced by absorption of radiation in the fuel solution to dilute the combustibles below their explosive concentration. However, auxiliary heat has been provided for additional boil-up. To avoid poisoning the catalyst surface with condensed moisture, the recombiner is cradled in a mantle heated by 250 psig steam. Pertinent data on the recombiner design are tabulated.

HRT SOLIDS PRODUCTION RATE CONSIDERATIONS IN CHEMICAL FACILITY DESIGN. H. E. Williamson. July 12, 1955. Decl. June 21, 1957. 9p. (CF-55-7-42)

The effects of solids production rate on the design of the chemical facility for the HRT are discussed. It is necessary to know the degree to which solids can be concentrated in the evaporator without limiting operation or rendering a slurry too difficult to handle. Additional decay storage capacity in the chemical process cell seems desirable.

IMPLICATIONS OF THE U-232 DECAY CHAIN ACTIVITY FOR THE PROCESSING OF IRRADIATED THORIUM AND RECOVERED U-233. R. C. Feber. Feb. 4, 1954. Decl. Apr. 3, 1957. 32p. (KAPL-1271)

The problems introduced by the associated beta and gamma radiation from the U^{232} decay chain in U^{233} processing are of sufficient magnitude to introduce new considerations with respect to handling the U^{233} product, processing and recycling the irradiated Th, and setting decontamination specifications. The U^{233} from an MTR slug had a U^{232} content which would result in a γ-ray dosage of about 600 r/hr at the surface of a 1 kg sphere of U^{233} after secular equilibrium was reached in ten years. More than half as much radiation would be present at the end of two years. Remote handling of such U^{233} would be required at even shorter periods after isolation. If this high U^{232} content cannot be tolerated, much lower g/ton levels must be used, since the fraction of the U^{232} in U^{233} is roughly proportional to the g/ton level. The bulk of the β- and γ- emitting daughters of U^{232} which are associated with the separated Th grows in during the cooling period prior to processing. A 4-in.-diameter billet of thorium 10 in. in length recovered from the MTR irradiated slug which has been cooled 200 days would have a γ-ray dosage of 1.1 r/hour at the surface of the cylinder. This radiation would decay with the 1.9 year half life of Th^{228}. However, if the Th were infinitely recycled with 270 days irradiation at the MTR flux, 200 days cooling and 100 days allowed for slug refabrication, the dose rate at the surface of the billet would reach a limiting value of 2.1 r/hour. This would require remote handling facilities for slug refabrication procedures. The β and γ activities of the U^{232} decay chain must be taken into account in setting decontamination specifications for Th.

HEAD-END TREATMENTS 3.01

DECAY STORAGE 3.01.03

11611 CF-53-7-45(Rev.)
Oak Ridge National Lab., Tenn.
CALCULATIONS OF DECONTAMINATION OF MTR MATERIALS BY THE 25 PROCESS. F. R. Bruce. July 8, 1953. Decl. Mar. 7, 1957. 8p. Contract [W-7405-eng-26]. $1.80(ph OTS); $1.80(mf OTS).

The major activities associated with ICPP product from MTR assemblies are calculated as a function of cooling time. (T.R.H.)

DISSOLUTION (GENERAL) 3.01.04

2453 BMI-1375
Battelle Memorial Inst., Columbus, Ohio.
CONSTRUCTION MATERIALS FOR VARIOUS HEAD-END PROCESSES FOR THE AQUEOUS REPROCESSING OF SPENT FUEL ELEMENTS. Charles L. Peterson, Paul D. Miller, James D. Jackson, and Frederick W. Fink. Aug. 28, 1959. 121p. For Oak Ridge National Lab. Contract [W-7405-eng-26, Subcontract No. 988]. OTS.

Materials of construction were evaluated for use in critical areas of head-end processes for the aqueous reprocessing of spent nuclear fuel elements. The Sulfex-Thorex, Darex-Thorex, Darex, Zirflex, and Zircex processes were considered. The effect of varying heat treatments on the resistance of the materials was also evaluated. Dissolution of unirradiated fuel pins was carried out in vessels of promising materials. The corrosion rate of Ni-o-nel was about 5 mils per month during actual fuel-pin dissolution by the Sulfex-Thorex process. Stabilization and heat treatment are necessary to prevent intergranular attack at welds. Carpenter 20 Cb is subject to stress-corrosion cracking by the Sulfex decladding solution and Illium R behaves similarly to Ni-o-nel in Thorex solutions. Titanium shows promise as a construction material for a Darex-Thorex dissolver. However, several questions remain concerning a vapor-phase attack observed around certain weldments. Carpenter 20 Cb, Ni-o-nel, and Types 309 and 309S Cb stainless steel appeared worthy of further study for the Zirflex dissolver. Preliminary evaluations show that at least Ni-o-nel and Carpenter 20 Cb should be studied further as possible construction materials for a single vessel for Zirflex and Sulfex-Thorex processes. Illium R, Hastelloy C, and nickel were not attacked by hydrochlorination conditions of the Zircex process. Minor attack was found on Inconel and Type S-816 alloy. The corrosion of titanium was negligible in the Darex dissolver and feed-adjustment system. The use of Type 304 ELC stainless steel lines for transferring Darex dissolver solution is not recommended. No corrosion of Type 347 stainless steel was observed after exposure for 2 years to chloride-contaminated Purex extraction systems at room temperature. (auth)

9840 CF-59-3-7
Oak Ridge National Lab., Tenn.
POISON ROD REQUIREMENTS FOR A SOLID-FUEL LEACHER TANK. B. E. Prince. Mar. 2, 1959. 10p. Contract [W-7405-eng-26]. $1.80(ph), $1.80(mf) OTS.

Estimates were made of the amount of neutron poisoning, in the form of boron rods, required to maintain subcriticality in a tank used for dissolution of solid fuel elements. The tank under consideration was a cylinder 19.5 inches in diameter and 6 ft in height. Maximum concentrations of fuel and fertile material resulting from dissolution of various type fuel elements are tabulated. Below the maximum concentrations listed, it was found that the limiting poisoning requirements are associated with dissolution of the NS Savannah elements. About five 1-inch diameter rods appear necessary: a centrally located rod and four rods in a square array each about 5 inches from the central rod. (auth)

5475 HW-58049
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
NUCLEAR SAFETY IN PROCESSING LESS THAN 5.0% U-235 ENRICHED REACTOR FUELS. N[orman] Ketzlach. Dec. 11, 1958. 23p. Contract W-31-109-Eng-52. $4.80(ph OTS); $2.70(mf OTS).

Processing of reactor fuels in which the initial enrichment is less than 5% U^{235} is considered. The critical parameters for heterogeneous systems of fuel in water or in uranium solutions as well as homogeneous solutions of fuel and water are examined. In addition, nuclear safety in the use of boron poisoning to increase safe batch sizes and in the use of safe vessel geometries is discussed. A cartridge-type dissolver system for fuel elements is described, and it is pointed out that experiments to determine the neutron reflecting properties of enriched uranium solutions surrounding vessels are justified. (J.R.D.)

HEAD-END TREATMENTS 3.01

DISSOLUTION (GENERAL) 3.01.04

IMPROVEMENTS IN OR RELATING TO VESSELS FOR PROCESSING NUCLEAR FUEL. Ronald Tunstall Ackroyd, Dennis Kenyon, and Derek Edmund Abel (to United Kingdom Atomic Energy Authority). British Patent 811,790. Apr. 15, 1959.

A dissolver for highly enriched fuel is described. The vessel consists of a central column with a steam jacket surrounded by a neutron absorbing material. A tank encloses this arrangement so that the central column is connected with the surrounding tank. Thus fuel elements in the central column are covered with HNO_3 and heated. The liquid flows by convection up the column and down around the outside of the neutron absorbing material. (T.R.H.)

11689 IDO-14452
Phillips Petroleum Co. Atomic Energy Div., Idaho Falls, Idaho.
CONTINUOUS DISSOLVER THEORY. III. DEVELOPMENT OF GENERAL TANK-FLOW RELATIONSHIPS WITH APPLICATION TO PILOT PLANT DATA FOR A FLOODED COLUMN DISSOLVER. E. E. Erickson. Apr. 1, 1959. 34p. Contract AT(10-1)-205. $1.00(OTS).

General relationships are developed for the performance of continuous flooded dissolvers based on complete mixing, such as tank flow, uniform packing characteristics of metal, and a first-order rate law. The form of the final equation is the same as that derived for the non-mixing or tube-flow case discussed in earlier reports of this series, with differences appearing only in the term that expresses the concentrations of metal and dissolvent in the liquid phase. The relationships are applied to available pilot plant data on the mercury-catalyzed dissolution of four shapes of 2S aluminum elements.

CONTINUOUS DISSOLVER EXTRACTOR FOR PROCESSING METAL. R. B. Lemon and J. A. Buckham (to U. S. Atomic Energy Commission). U. S. Patent 2,872,296. Feb. 3, 1959.

An apparatus is presented for the continuous dissolution of metal slugs in an aqueous acid and sequential continuous extraction of selected metal values from the acid solution by counter-current contact with an organic solvent. The apparatus comprises a cylindrical tank divided into upper and lower sections. Dissolution of the metal slug takes place in the lower section and the solution so produced is continuously fed to the topmost plate of the upper extraction section. An immiscible organic extractant is continuously passed by a pulsing pump into the lowermost unit of the extraction section. Suitable piping and valving permits of removing the aqueous raffinate solution from the lowermost portion of the extraction section, and simultaneous removal of organic solvent extractant containing the desired product from the uppermost portion of the extraction section.

15176 DP-313
Du Pont de Nemours (E. I.) & Co. Savannah River Lab., Aiken, S. C.
DISSOLVER FOR NATURAL URANIUM FUEL ELEMENTS. Vincent P. Caracciolo. Dec. 1958. 23p. Contract AT(07-2)-1. $0.75(OTS).

Sufficient data were obtained with a semiworks-scale dissolver to permit design of plant-scale equipment for the dejacketing and subsequent dissolving of aluminum-clad uranium fuel elements of various shapes and sizes. The aluminum dejacketing reaction occurred as fast as caustic was added and was independent of the shape of the elements. The rate of uranium dissolving increased with increasing acidity, was proportional to the exposed surface area, and was independent of the shape of the elements. A mechanism for uranium dissolution is discussed that explains the variation of acid consumption and the rate of gas evolution. (auth)

European Company for the Chemical Processing of Irradiated Fuels, Mol, Belgium.
HEAD END PROCESS. PART 1. REVIEW OF THE LITERATURE. PART 2. PROPOSAL FOR LABORATORY SCALE DISSOLUTION UNITS. Technical Report No. 8. E. Detilleux. May 16, 1958. 19p. $3.30(ph), $2.40(mf) OTS. (NP-7665)

A literature survey on head-end operations for liquid-liquid extraction of irradiated fuels was conducted. A study of the third and fourth operations for natural or slightly enriched fuel elements containing Al, Zr, Mg, and stainless steel is emphasized. Purex-type flowsheets for fuel dejacketing and dissolution are given along with an examination of clarification and adjustment of solutions. Also the design of a fumeless dissolution installation for laboratory use is described and diagrammed. (J.R.D.)

DISSOLUTION EQUIPMENT. J. A. Buckham and C. E. Stevenson. May 1, 1957. p.831-47 of SYMPOSIUM ON THE REPROCESSING OF IRRADIATED FUELS HELD AT BRUSSELS, BELGIUM, MAY 20-5, 1957. 1165p. (TID-7534(Bks. 1-3))

Batch and continuous dissolution equipment used in the United States is briefly described. Certain design criteria for dissolvers are given. A brief comparison is made between continuous and batch dissolvers.

HEAD-END TREATMENTS 3.01

DISSOLUTION (GENERAL) 3.01.04

DISSOLUTION AND FEED ADJUSTMENT. R. E. Blanco. May 1, 1957. p. 22-44 of SYMPOSIUM ON THE REPROCESSING OF IRRADIATED FUELS HELD AT BRUSSELS, BELGIUM, MAY 20-5, 1957. 1165p. (TID-7534(Bks. 1-3))

A brief summary of the chemistry of the dissolution and feed adjustment steps along with chemical flowsheets for these operations is presented. The intent of this presentation is to touch briefly on each topic. Detailed discussions may be found in the references cited. 23 references.

19035 ✓ NP-7754
European Company for the Chemical Processing of Irradiated Fuels, Mol, Belgium.
PRETREATMENT OF FUEL ELEMENTS. Technical Report No. 48; Chapter 130. E. Detilleux. [1958]. 10p. $1.80(ph), $1.80(mf) OTS.

A review is presented of pretreatment methods with special attention to those useful at Eurochemic. Mechanical and chemical decanning methods presently in use or proposed for Al-, Mg-, stainless steel-, and Zr- or Zr alloy-jacketed fuels are discussed. Darex, Sulfex, and Zirflex processes are included along with special methods for U–Mo and U–Zr fuels. (T.R.H.)

DISSOLUTION OF URANIUM METAL AND ITS ALLOYS. Robert P. Larsen (Argonne National Lab., Lemont, Ill.). Anal. Chem. 31, 545-9(1959) Apr.

The most useful methods for the dissolution of uranium metal and its alloys are reviewed, with particular emphasis on the preparation of solutions for analysis. The behavior of the metal and its alloys in the common acids, ethyl acetate solutions of bromine and hydrogen chloride, and sodium hydroxide–peroxide mixtures is described. Recommendations for dissolving each of a wide variety of uranium alloys are summarized in tabular form. (auth)

8788
HOW WILL WE PROCESS ADVANCED REACTOR FUELS? F. S. Martin (United Kingdom Atomic Energy Authority, Springfield Works). Atomic World 10, 18-21, 40(1959) Jan.

Information is presented on head-end steps for processing advanced reactor fuel elements which are not directly soluble in nitric acid. Discussions are included on the application of the following head-end steps: Darex, anodic dissolution, carburization, molten metal, hydrofluoric acid, and Zircex. (J.H.M.)

AUTO-RINSE CYCLE FOR FUEL ELEMENT RECOVERY. Completion Report. E. N. Wells. June 10, 1957. 12p. (HW-50675)

A rinse system has been designed to provide for rinsing the fuel elements, after they have been removed from the treater tank, in an enclosed, vented tank, and recovering the U from the rinse water. After each load of fuel elements has been placed in the rinse tank, the rinse cycle is automatically timed and controlled.

14712 A/CONF.15/P/1930
Oak Ridge National Lab., Tenn.
DISSOLUTION AND FEED PREPARATION FOR AQUEOUS RADIOCHEMICAL SEPARATION PROCESSES. F. L. Culler and R. E. Blanco. 85p. $0.50(OTS).

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

Most irradiated power reactor fuels can be radiochemically processed by the well established solvent extraction methods provided that the corrosion resistant cladding materials can be removed or dissolved and that the fuel element cores can be dissolved in or converted to an aqueous solution highly salted with nitrate anions. Thus, the aim of much of the current aqueous processing development is to devise compatible mechanical and chemical dissolution procedures for all power reactor fuels. Mechanical processing is being studied as an alternate to non-nitric-acid head-end systems to facilitate associated waste storage and corrosion problems. Mechanical operations include disassembly of complex fuel bundles and dejacketing of fuel rods and pins to expose nitric acid soluble cores. Mechanical techniques under study include chopping, roll decladding, sawing, and breaking. Chemical removal of zirconium cladding can be done with aqueous ammonium fluoride, hydrofluoric acid, sulfuric acid, or by anhydrous hydrochlorination or chlorination in gas-solid or anhydrous organic media. Stainless steel may be declad by dissolution in sulfuric acid. (auth)

DEVELOPMENT OF AQUEOUS PROCESSES FOR HEAD END TREATMENT OF FUELS FROM THE POWER REACTOR DEMONSTRATION PROGRAM. C. W. Pierce and B. Manowitz. July 1957. 19p. (BNL-3334)

The wide diversity of fuels resulting from the Power Reactor Demonstration Program presents many problems in reactor fuel reprocessing. In order to minimize the initial investment necessary to change over and convert existing facilities to handle these new fuels, aqueous processes will be desirable. This report summarizes an approach to cope with these problems emphasizing aqueous head end processes.

HEAD-END TREATMENTS 3.01

DISSOLUTION (GENERAL) 3.01.04

5270

PREPARATION OF POWER REACTOR FUELS FOR AQUEOUS PROCESSING. J. E. Savolainen and R. E. Blanco (Oak Ridge National Lab., Tenn.). Chem. Eng. Progr. 53, 78F-81F (1957) Feb.

New methods, Darex and Zircex Processes, for converting fuels fabricated of HNO_3-insoluble metals to HNO_3 solutions suitable for reprocessing by solvent extraction are discussed. (W.L.H.)

8728

PREPARATION OF POWER REACTOR FUELS FOR PROCESSING BY SOLVENT EXTRACTION. R. E. Blanco (Oak Ridge National Lab., Tenn.). pp. 223-46 in "Progress in Nuclear Energy. Series III. Process Chemistry. Volume 2."

Power reactor reprocessing problems are discussed from the viewpoint of converting heterogeneous fuels to a nitric acid solution suitable for purification by solvent extraction. Current and proposed fuels are classified into types based on reprocessing principles. Flowsheets are presented for the solution of aluminum-clad uranium or thorium fuels in nitric acid along with procedures under development for processing zirconium and stainless steel clad fuels. Methods under development include mechanical processing, solution in sulfuric, hydrofluoric or dilute hydrochloric–nitric acids, hydrochlorination, electrolytic solution, oxidation and carburization. (auth)

Continuous Dissolution of Uranium-aluminum Reactor Fuels, A.F.BOEGLIN, J.A.BUCKHAM, et al. Am Inst Chem Engrs-J 2, 2, June 1956, p 190-4.

Marked differences were observed in dissolution rates of cast and wrought alloys; optimum feed-acid concentrations varied with type of alloy; at constant acid feed conditions, dissolving rates varied approximately with cube root of catalyst concentration up to limiting concentration; metal dissolving rate was proportional to 0.8 power of nitric acid feed rate; general empirical correlation developed.

2501

METHOD OF DISSOLVING METALLIC URANIUM. W. W. Schulz (to U. S. Atomic Energy Commission). U. S. Patent 2,897,047. July 28, 1959.

A process is presented for more rapidly dissolving metallic uranium which comprises contacting the uranium with a mixture of nitric and phosphoric acids. The preferred concentration is a mixture which is about 10 M in nitric acid and between 0.1 to 0.15 M in phosphoric acid.

3576

CONTINUOUS DISSOLUTION OF URANIUM–ALUMINUM FUELS IN A TRICKLE-TYPE COLUMN DISSOLVER. J. C. Bresee, D. L. Foster, and E. O. Nurmi (Oak Ridge National Lab., Tenn.). Chem. Eng. Progr. 55, Symposium Ser. No. 22, 25-32(1959).

A continuous column dissolver may be operated with critically safe dimensions; hence this type of dissolver may have an unlimited charge of metal per unit, with no theoretical upper limit to the capacity. With the added advantage that a continuous dissolver may better serve a continuous solvent-extraction process than a batch dissolver, the continuous column dissolver is potentially the most satisfactory type for short fuel elements or sections of elements. A study was conducted to investigate the performance of a full-scale continuous dissolver with suitable dimensions for enriched-power-reactor-fuel dissolution and to determine the capacity of this scale of equipment at a specified product concentration. Results on a full-scale continuous trickle-type column dissolver for enriched uranium-aluminum-alloy fuels are presented. Both cast and extruded unirradiated alloys were dissolved, and correlations are given relating dissolving rate and product concentration to feed acid rate and concentration for both types of alloy. The application of the correlations to column design and operation is discussed. (auth)

11624 IDO-14226

American Cyanamid Co. Atomic Energy Div., Idaho Falls, Idaho.

SETTLING RATES OF PARTICLES IN DISSOLVER SOLUTION 3002 UM-1Fu. G. K. Cederberg. Feb. 24, 1953. Decl. Mar. 13, 1957. 9p. Contract AT-(10-1)-177. $1.80(ph OTS); $1.80(mf OTS).

The settling rates at room temperature of the smallest and largest particles in the difficult filtering MTR dissolver solution 3002UM–1Fu were found to be 0.66 and 80.0 cm/hr, respectively. The particle sizes of the residue, which were estimated from photomicrographs of the particles, compared favorably with those calculated by Stoke's law from these experimental settling rates. (auth)

HEAD-END TREATMENTS 3.01

DISSOLUTION (GENERAL) 3.01.04

7407 ✓✓ANL-5466
Argonne National Lab., Lemont, Ill.
CHEMICAL ENGINEERING DIVISION SUMMARY REPORT [FOR] APRIL, MAY, AND JUNE 1955. July 27, 1955.
Decl. Feb. 7, 1957. 92p. Contract W-31-109-eng-38. $0.60(OTS).

Progress is reported on the following investigations: direct-cycle boiling-reactor entrainment studies; dissolution of U, Zr, and Al in BrF_3 solutions; volatilization of U from UF_4 with O_2; corrosion of Ni in fused-salt solutions; kinetics of the U–BrF_3 reactions; development of a vacuum casting furnace for slagging; extraction of Pu and U with liquid Mg; corrosive effects of molten U and Mg on container materials; oxidative slagging process for purification of spent fuel elements; denitration of uranyl nitrates; effect of reduction step on hydrofluorination of UO_2; calcination of fuel-processing waste solutions; spectrophotometric determination of Pu; and volatilization of UF_6 from KF–BrF_3 systems. (C.W.H.)

2740 RDB(W)TN-14
United Kingdom Atomic Energy Authority. Industrial Group. Windscale Works, Sellafield, Cumb., England.
SURGING IN THE CONTINUOUS METAL DISSOLVING PROCESS. T. I. M. Crofts. 1958. Date of MS. Jan. 15, 1952. 5p.

In order to ascertain the probable causes of surging in the continuous dissolvers, a review has been made of the conditions leading to surging during the development work. It is concluded that surging is due to vigorous evolution of gas causing an increase in the liquor hold up in the dissolver, followed by a reduction in gas evolution accompanied by a surge of liquor from the overflow pipe. These conditions are met when the acid strength and temperature of the dissolver liquor (and hence the dissolving rate) are high. (auth)

18911 ✓ RISLEY-5057/2
Gt. Brit. Div. of Atomic Energy (Production), Risley, Lancs, England.
THE DISSOLUTION OF CAST URANIUM RODS IN 3N NITRIC ACID CONTAINING 0.3GM. URANIUM PER MILLILITRE. (Second Interim Report). J. H. Tonkin. Feb. 16, 1949. 22p.

On the basis of information obtained from laboratory experiments, work has been carried out on a 20 liter scale to establish the rate of dissolution of uranium on a continuous basis such as to yield a liquor containing 0.3 g of uranium per ml in 3 N nitric acid and to provide information for a larger scale trial on a one-third production scale plant. (auth)

13363 ✓ HW-57271
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
THE DISSOLUTION OF I. AND E. FUEL ELEMENTS. N. Ketzlach. Aug. 25, 1958. 10p. Contract W-31-109-Eng-52. $1.80(ph), $1.80(mf) OTS.

A study involving calculation of the reactivity parameters for water lattices of 1.007% enriched I and E fuel elements whose OD varied from 1.37 to 1.17 in. and whose ID varied from 0.48 to 0.68 in. was conducted. The investigation was aimed at determining whether the reactivity of 1.37 OD by 0.48 in. ID increases or decreases during dissolution. It was concluded that nuclear safety based on maximum OD fuel processed for both I and E as well as solid fuel rods can be attained at the 1.0% U^{235} enrichment level. Data are included. (J.R.D.)

5254 HW-61482
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
RATES OF REACTION OF IRRADIATED UO_2 AND URANIUM METAL WITH SULFURIC ACID. J. L. Swanson. Aug. 13, 1959. 8p. Contract AT(45-1)-1350. OTS.

The rates at which samples of irradiated UO_2 and U dissolve in H_2SO_4 were determined. Parallel experiments were performed with similar unirradiated samples. Data and observations on the dissolutions under various conditions are included. (J.R.D.)

A KINETIC STUDY OF THE DISSOLUTION OF UO_2 IN SULFURIC ACID. Theron L. Mackay and Milton E. Wadsworth (Univ. of Utah, Salt Lake City). Trans. Met. Soc. AIME 212, 597-603(1958) Oct.

Sintered UO_2 samples were leached in sulfuric acid solutions of various concentrations. A pressurized system was used so that it was possible to investigate the kinetics of the reaction to 270°C with oxygen overpressures as high as 900 psi. The rate was observed to be a function of the concentration of hydrogen ions and directly proportional to the partial pressure of oxygen. Evidences are presented which indicate that a UO_2 surface site reacts with a molecule of water to form a hydroxyl complex which in turn can dissociate with the characteristics of a weak acid. A rate determining step has been proposed which involves the reaction between an oxygen molecule and the hydroxyl complex on the surface of UO_2. (auth)

HEAD-END TREATMENTS 3.01

DISSOLUTION (GENERAL) 3.01.04

9718 ANL-RCV-SL-1090
Argonne National Lab., Lemont, Ill.
DISSOLUTION OF THORIUM OXIDE–URANIUM OXIDE FUEL ELEMENTS. R. P. Larsen. Aug. 9, 1956. Decl. Mar. 9, 1957. 2p. Contract [W-31-109-eng-38]. $1.80 (ph OTS); $1.80(mf OTS).

2828 RCTC/P-87
Gt. Brit. Atomic Energy Research Establishment, Harwell, Berks, England.
SIMPLE MELTING AND CASTING FOR THE PROCESSING OF FAST REACTOR FUEL ELEMENTS. B. A. J. Lister and R. A. Davies. Feb. 14, 1957. 10p.
A critical examination is made of methods of processing fast reactor fuel elements by simple melting and casting. An outline scheme is considered for a possible process. A suggestion is made for continuous processing in the presence of molten calcium. (auth)

20166 NAA-SR-3910
Atomics International Div., North American Aviation, Inc., Canoga Park, Calif.
CHEMICAL PULVERIZATION OF SINTERED URANIUM DIOXIDE BODIES. PART I. (Preliminary Small-Scale Studies). S. Stransberg and T. E. Luebben. Aug. 1, 1959. 61p. Contract AT-11-1-GEN-8. $1.75(OTS).
A preliminary study was conducted to develop a non-mechanical method for the pulverization of sintered uranium dioxide fuels suitable for use as an economical procedure for the low decontamination reprocessing of such nuclear fuels. Pulverization of ceramic shapes, to a −400 mesh powder, was accomplished by oxidation to U_3O_8 in air, or pure O_2 (at a reduced pressure). The U_3O_8 was reduced back to UO_2 with hydrogen, or by thermal decomposition at 1200°C in vacuum. The resultant UO_2 powder should be suitable for re-enrichment by dry blending with $U^{235}O_2$, and can be refabricated to high density bodies. The optimum treatment for pulverization of sintered, UO_2 pellets consisted of oxidation, in air at 1 atmosphere pressure, at a temperature of ~375°C. Satisfactory reduction of the finely divided U_3O_8 powder to UO_2 was accomplished in flowing hydrogen, at 1 atmosphere pressure, at about 650°C. (auth)

2457 DP-421
Du Pont de Nemours (E. I.) & Co. Savannah River Lab., Aiken, S. C.
REPROCESSING POWER REACTOR FUELS. Quarterly Progress Report No. 7 [for]April 1 to July 1, 1959. Leon H. Meyer and Edward S. Occhipinti, comps. Sept. 1959. 10p. Contract AT(07-2)-1. OTS.
Stainless steel cladding on power fuel elements can be dissolved electrolytically with equipment and reagents that may be compatible with the present SRP separations plant. Plutonium and uranium were extracted effectively from simulated dissolver solutions used for processing of Detroit-Edison core elements. A molar ratio of aluminum to total fluoride of 2.7:1 was found necessary to extract Pu(IV) with an extractant of tributyl phosphate in n-dodecane. For preceding period see DP-393. (auth)

20990 DP-396
Du Pont de Nemours (E.I.) & Co. Savannah River Lab., Aiken, S. C.
ELECTROLYTIC DISSOLUTION OF STAINLESS STEEL. Hugh E. Henry. July 1959. 11p. Contract AT(07-2)-1. $0.50(OTS).
Stainless steel rods and a stainless steel clad uranium rod were dissolved satisfactorily by anodic oxidation in nitric acid. Dissolving rates of stainless steel varied linearly with current density for both direct current and alternating current. Acidity, salt concentration, and temperature had very little or no effect on dissolving rates. The major gaseous product from electrolytic dissolution of stainless steel in nitric acid was nitric oxide with small amounts of carbon monoxide, nitrogen, and nitrous oxide. Samples of the off-gas contained no hydrogen. (auth)

4382 DP-439
Du Pont de Nemours (E.I.) & Co. Savannah River Lab., Aiken, S. C.
REPROCESSING OF POWER REACTOR FUELS. Quarterly Progress Report No. 8 [for] July 1 to October 1, 1959. E. S. Occhipinti, comp. Nov. 1959. 10p. Contract AT(07-2)-1. OTS.
Niobium can be used as an inert metal connector for electrolytic dissolution of stainless steel; zirconium metal and zirconium–10 wt.% uranium alloy can be electrolytically disintegrated in 8 to 13M HNO_3. Criticality calculations show that it is safe to dissolve a group of four subassemblies of PRDC elements in the large plant dissolvers, provided that the subassemblies are held in a single row. (auth)

HEAD-END TREATMENTS 3.01

DISSOLUTION (GENERAL) 3.01.04

7464
STRIPPING PROCESS FOR PLUTONIUM. M. Kolodney (to U. S. Atomic Energy Commission). U. S. Patent 2,907,700. Oct. 6, 1959.

A method for removing silver, nickel, cadmium, zinc, and indium coatings from plutonium objects while simultaneously rendering the plutonium object passive is described. The coated plutonium object is immersed as the anode in an electrolyte in which the plutonium is passive and the coating metal is not passive, using as a cathode a metal which does not dissolve rapidly in the electrolyte, and passing an electrical current through the electrolyte until the coating metal is removed from the plutonium body.

MECHANICAL PREPARATION 3.01.05

See also, under HAZARDS AND PROTECTION,

DUSTS AND POWDERS (4.01.03),

PYROPHORICITY (4.01.10).

6339 ✓ TID-7583(p.306-99)
Oak Ridge National Lab., Tenn.
MECHANICAL PROCESSING OF SPENT POWER REACTOR FUEL AT OAK RIDGE NATIONAL LABORATORY. C. D. Watson, J. B. Adams, G. K. Ellis, G. A. West, F. L. Hannon, W. F. Schaffer, and B. B. Klima. 94p.

The mechanical processing of spent power reactor fuels as an aid to or as a substitute for chemical head ends is being actively investigated by Oak Ridge National Laboratory in two development programs: 1. Underwater mechanical equipment program for the predissolution treatment of fuel to eliminate massive inert hardware and disassembly of fuel (if required) to facilitate processing of fuel on a production level in the Power Reactor Fuel Processing Pilot Plant now being erected. 2. Dry cave mechanical equipment program to determine the feasibility of dejacketing stainless steel-clad NaK- or Na-bonded fuels of uranium and uranium-molybdenum and of shearing (chopping) and leaching metallic-clad tubular fuel arrays of the oxides of uranium and/or thorium. An underwater facility to receive, store, remove inert hardware, and disassemble and charge fuel to charger carriers has been designed and most of the features accepted for final design. Full-sized equipment, consisting of carriers, a traversing table, disassembly saw, hydraulic dejacketer, auxiliary dejacketer, slug washer, and slug canner for dejacketing and handling the stainless steel-clad NaK-bonded fuel of the SRE Core 1 has been built but is not yet evaluated. Disassembly, chopping, and leaching tubular assemblies has been shown to be feasible in laboratory investigations. A prototype shear is being designed and full-scale batch and continuous leachers have been fabricated for testing. (auth)

20985 ✓ CF-59-8-88(Suppl. 1)
Oak Ridge National Lab., Tenn.
POWER REACTOR FUEL REPROCESSING: MECHANICAL PHASE. B. B. Klima. Sept. 2, 1959. 36p. Contract [W-7405-eng-26]. $9.30(ph), $3.60(mf) OTS.

An occurrence at the SRE which caused approximately 7 fuel elements to part when being pulled from the reactor complicated reprocessing, however the bulk of the elements may still be reprocessed as planned. New or reworked parts for the mechanical decladder were supplied by the fabricators for rebuilding in ORNL shops. Administrative actions dictated by budgetary considerations include reduction of UO_2 pellet order to the quantity needed this fiscal year, reduction of Mark III prototype fuel element to 12, stoppage of all work by REE Division personnel, and stoppage of all work not related to SRE fuel element reprocessing. (auth)

10487
MECHANICAL DISASSEMBLY OF SPENT POWER-REACTOR FUELS. Presented at Nuclear Engineering and Science Conference, held at Chicago, March 17 to 21, 1958. Preprint 39, Session 34. W. W. Barton and J. J. Brooks (American Machine and Foundry Co., Greenwich, Conn.). New York, American Institute of Chemical Engineers, 1958. 30p.

Mechanical disassembly appears to be a promising alternative to chemical head-end methods for the preparation of spent power-reactor fuels for reprocessing. Most of the inert structural material is removed and the fuel itself is exposed for rapid dissolution in nitric acid. Such difficult-to-dissolve materials as stainless steel and Zircaloy-2 are eliminated from the subsequent chemical separation process, and the total quantity of material to be handled in the separation process is reduced to a minimum. In addition, the inert material of the fuel element assemblies is disposed of as solids instead of as processed liquid wastes. Conceptual designs of proposed mechanical disassembly machines and facilities are presented. Metallic-pin, metallic-rod, metallic-plate and ceramic fuel element assemblies are handled. Throughput is one ton per day of natural or slightly enriched uranium and 25 kilograms per day of highly enriched uranium. (auth)

HEAD-END TREATMENTS 3.01

MECHANICAL PREPARATION 3.01.05

Underwater Mechanical Treatment for Plate-Type Fuel Elements, J.A. MARSH, D. HUMPHREYS. Nuclear Power 2, 16, Aug 1957, p 331-3.

At Dounreay, surplus sheathing material on fuel elements from Materials Testing Reactor is milled and sheared off before chemical processing; pond receives fixed charge of irradiated fuel element boxes from reactor in biologically shielded flask; as much aluminum as possible is then removed mechanically underwater and fixed quantities of active material fed to dissolver, ensuring safe processing.

10608 ✓ KAPL-1774
Knolls Atomic Power Lab., Schenectady, N. Y.
DEVELOPMENT OF A NEW PROCESS FOR CUTTING STAINLESS STEEL. R. A. Gahr. Apr. 22, 1957. Changed from OFFICIAL USE ONLY June 4, 1958. 27p. Contract W-31-109-Eng-52. $4.80(ph OTS); $2.70(mf OTS).

6537 ✓ A/CONF.15/P/2182
SOME PECULIARITIES IN TREATING IRRADIATED FUEL ELEMENTS AT THE FIRST ATOMIC POWER STATION IN THE U.S.S.R. V. B. Shevchenko, N. S. Povitskii (Povitsky), and A. S. Solovkin (U.S.S.R.). 10p.
Some features of processing irradiated fuel elements at the first atomic power station in the U.S.S.R. are presented. It is impossible to remove mechanically a stainless steel can from the surface of the fuel element. Therefore, to remove a can partially the atomic fuel element is cut in the transverse direction into pieces ≥ 10 mm long by a special device. The dissolution of the alloy in nitric acid takes place in an inert gas atmosphere. Additional chelating agents are used to prevent the formation of sediments when atomic fuel is being dissolved. Uranium and Pu(IV) are extracted from nitric acid solutions by 20% tri-n-butyl phosphate solution. To increase the uranium distribution ratio, extraction may be carried out at low temperatures. If fuel elements are to be processed without prolonged cooling, then deposition procedures are used for the initial (preliminary) separation of uranium and plutonium from macro-contaminants and fission products. (auth)

3813 HW-49555
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
HEAD-END FACILITY FOR POWER REACTOR FUEL ELEMENTS. J. G. Bradley and H. E. Hanthorn. Apr. 5, 1957. Decl. Nov. 21, 1958. 10p. Contract [W-31-109-Eng-52]. $3.30(ph OTS); $2.40(mf OTS).
A head-end facility for mechanically dismembering and chopping up PRPR fuel elements is described. Additional facilities are described for opening the ends only of valuable Zr-clad fuel elements so that the claddings may be recovered for reuse. Dissolver facilities are provided in each case. An integrated facility capable of recovering U and Pu from PRPR fuels and power reactor fuels, accepting irradiated fuel elements, and reprocessing the recovered U and Pu into recycle fuel is also described briefly. (W.L.H.)

14343 ✓ TID-7556(p.27-44)
Westinghouse Electric Corp. Bettis Plant, Pittsburgh.
THE DESIGN OF THE IDAHO EXPENDED CORE FACILITY. C. E. Langlois. p.27-44 [of] SIXTH HOT LABORATORIES AND EQUIPMENT CONFERENCE, MARCH 19-21, 1958, INTERNATIONAL AMPHITHEATRE, CHICAGO, ILLINOIS. 18p.
It has been recognized for some time that there is much to be learned from a core after it has been taken out of operation. A plant is under construction at NRF, Idaho which will be equipped to cut up, and analyze cores as well as to prepare the fuel-bearing material for recovering of fuel by a chemical processing plant. A brief description of the special design features of the facility and the reasons for them are given. (auth)

13019 NAA-SR-2687
Atomics International Div., North American Aviation, Inc., Canoga Park, Calif.
MODIFIED HOT-CAVE FACILITY FOR REPROCESSING EXPERIMENTS. S. Strausberg, W. J. Gardner, J. Guon, T. E. Luebben, and T. H. Mills. June 30, 1958. 81p. Contract AT-11-1-GEN-8. $2.25(OTS).
The system consists of two hot-caves, a transfer cave, and associated utilities. Cave 1 (west) is used primarily for kilogram-scale processing experiments involving chemical reactions of irradiated reactor fuels at temperatures up to 1800°C. Cave 2 (east) is used primarily for mechanical operations such as decanning, component disassembly, and inspection. The transfer unit is for sample handling, waste removal, intercave transfer, and the storage of kilocurie sources. (auth)

HEAD-END TREATMENTS 3.01

MECHANICAL PREPARATION 3.01.05

15751 ✓

CUTTING AND WEDGING JACKET REMOVER. Melvin Freedman and S. Raynor (to U. S. Atomic Energy Commission). U. S. Patent 2,880,636. Apr. 7, 1959.

A tool is presented for stripping cladded jackets from fissionable fuel elements. The tool is a tube which fits closely around the jacket and which has two cutting edges at opposite sides of one end. These cutting edges are adjusted to penetrate only the jacket so that by moving the edges downward the jacket is cut into two pieces.

5256 HW-62490

General Electric Co. Hanford Atomic Products Operation, Richland, Wash.

HIGH-SPEED SAW TESTS. POWER REACTOR FUELS REPROCESSING DEVELOPMENT. V. P. Kelly. Nov. 18, 1959. 15p. Contract AT-(45-1)-1350. OTS.

The basic scheme for processing power reactor fuels at Hanford includes the removal of inert end fittings from the active sections of the fuels. High-speed saw blades, including abrasive, diamond, and friction blades, were tested under conditions similar to those encountered in the power fuels reprocessing program. In these tests, the greater durability of the steel friction blade established its superiority over the other two types. The greater durability was demonstrated in both cutting ability and tolerance for abuse. In addition to its primary use for the removal of end fittings and hardware from the power reactor fuels, some studies were made to determine whether high speed saws could be used for cutting of the main fuel cores into small segments for charging into the dissolver. The results of these experiments are also presented. (W.L.H.)

6387 ✓

HYDRAULIC POWER HACKSAW FOR RADIOACTIVE MATERIAL. Machinery 95, 235-7(1959) Aug. 12.

The design and operation of a hydraulically-operated power hacksaw for radioactive materials are given. The machine is designed for remote control by a manually-operated mechanical manipulator and a hydraulic system with a pressure supply of 1500 psi. (C.J.G.)

A BIBLIOGRAPHY ON CUTTING OF METALS (1864-1943). Orlan W. Baston. New York, American Society of Mechanical Engineers. 1945. 547p.

This extensive bibliography is arranged chronologically and most of the references are annotated. Subject and author indexes are included.

5555 ✓ AD-158469

Battelle Memorial Inst., Columbus, Ohio.

HIGH-TEMPERATURE-ALLOY CUTTING PROGRAM. Report No. 3. J. E. Clifford, D. E. Semones, and J. McCallum. Mar. 31, 1958. 36p. Contract AF33 (600)-35440.

A method is described for cutting titanium and other high-temperature alloys by electrolytic action alone. Experimental tests on 1 to 1½-inch-diameter stock using three rotating metal disks mounted on a common spindle have demonstrated that multiple-disk electrolytic cutting is technically feasible. Successful development of: production machine utilizing 60 rotating cathode disks (2 per cut) indicates the following time per cut for an 8-inch-diameter billet, based on experimental cutting rates: unalloyed titanium, 17 minutes; M-252 (or Waspalloy), 14 minutes; A-286, 8 minutes; low-carbon steel, 6 minutes; AISI 4340 steel (550 Bhn), 12 minutes; and AISI 4340 (180 Bhn), 12 minutes. The test on 4340 steel, which indicated that cutting rate is independent of hardness and crystal structure, met one of the project objectives of a cutoff method suitable for metal strength up to 250,000 psi. Squareness of cut (0.006-inch deviation per inch of diameter) was indicated to be satisfactory for forging operations. The main economic advantage appears to be in saving valuable metal by permitting narrow cut widths of 1/16 inch or less. Good surface finish, lack of heat-damaged-or work-hardened surface, absence of burrs, etc., eliminate the need for subsequent machining operations. (auth)

8103

5 ELECTROLYTIC CUTTING OF METALS. G. L. Kehl and I. Moch, Jr. (to U. S. Atomic Energy Commission). U. S. Patent No. 2,739,935, Mar. 27, 1956.

A method and apparatus are described for the electrolytic cutting of those metals and alloys which are subject to electrolytic attack. The apparatus consists of a tank, containing the metal to be cut mounted so that it may be rotated, the electrolyte, the cutting arm made up of a central cutting electrode, and two exterior robber electrodes separated by insulators, a means to adjust this cutting arm in relation to the metal, circuitry to impress a potential between the cutting electrode and the robber electrodes, and a nozzle directing a flow of electrolyte against the cutting arm. This method and apparatus will produce a clean straight edged cut, free of mechanically imposed stresses, burrs or jagged edges, and no metal is lost by dust, shavings or metal being burned away. (auth)

HEAD-END TREATMENTS 3.01

MECHANICAL PREPARATION 3.01.05

A DEVICE TO REMOVE ALUMINUM JACKETS MECHANICALLY FROM BONDED URANIUM SLUGS. H. W. Alter, C. J. Beck, and B. V. Coplan. June 1, 1954. Decl. with deletions Feb. 25, 1957. 9p. (KAPL-1139(Del.))

A description is given of a simple device to remove Al jackets from bonded U slugs. The successful application of the device to Hanford slugs is illustrated.

15428 ✓

IMPROVEMENTS IN OR RELATING TO SHIELDING DEVICES FOR MACHINING TOXIC AND EXPENSIVE MATERIALS AND THOSE HAVING A CRITICAL MASS. Ronald Tunstall Ackroyd and Dennis Kenyon (to United Kingdom Atomic Energy Authority). British Patent 813,928. May 27, 1959.

A device which permits machining of toxic, expensive, or fissionable materials safely and without loss is described. It consists of a tube which encloses the piece to be machined and is equipped with rubber sleeves to seal to the tailstock of the lathe. An opening with a flapseal is provided for inserting tools; other openings are provided for inert gas sweeping or cutting oil application. (T.R.H.)

Battelle Memorial Inst. Defense Metals Information Center, Columbus, Ohio.

BAND SAWING OF TITANIUM AND TITANIUM ALLOYS. Carl T. Olofson. July 1, 1959. 15p.

Band-sawing is being accomplished under conditions adapted to the general machining behavior of titanium. The difficulties encountered in other metal-cutting operations are also encountered in sawing. They result from galling, smearing, abrasiveness, and the generation of high temperatures in the cutting zone. Many difficulties can be minimized by selecting the proper saw pitch and feeding pressure for the work thickness involved. Also, a proper relationship of band velocity and feed must exist for economic tool life. (auth)

OFF-GAS 3.01.06

See also, under HAZARDS AND PROTECTION,
AIR CLEANING (4.01.01),
and,
under WASTE TREATMENT AND DISPOSAL,
GASEOUS FISSION-PRODUCT DISPOSAL (8.03.02).

GENERAL 3.01.06.01

See also, under FISSION PRODUCT, POISON, AND RADIOISOTOPE REMOVAL,
Krypton (2.28),
Iodine (2.23),
Xenon (2.51).

HNO_3 3.01.06.02

2461 NAA-SR-2309

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

GAS-HANDLING SYSTEM FOR THE PROCESSING REFABRICATION EXPERIMENT. H. Bernard. Aug. 15, 1958. 79p. Contract AT-11-1-GEN-8. OTS.

In the process proposed by the Processing Refabrication Experiment (PRE) as outlined various gases, fumes, and particulates are evolved during the melting operation and are carried off in a helium gas stream. The contaminants are removed from the carrier gas by either electrostatic or impingement filters and liquid metal or solid adsorbers. This project was undertaken to review the literature of adsorption of inert off-gases on silica gel, and to design, construct, and operate adsorption test equipment to ascertain the design parameters for a PRE pilot plant. (auth)

20004

DESIGN AND OPERATING CONSIDERATIONS FOR OFF-GAS SYSTEMS IN NUCLEAR PROCESSING PLANTS. Presented at Nuclear Engineering and Science Conference, April 6-9, 1959, Public Auditorium, Cleveland, Ohio. Preprint V-72. Lloyd R. Michels (General Electric Co., Richland, Wash.). New York, Engineers Joint Council, 1959, 29p. $0.50.

Highlights of integrated facilities for the control of air-borne radio-iodine and fission product particulate material at the Hanford Purex Plant are presented. Design and operating information is provided on improved silver reactors, fibrous glass filters, auxiliary iodine and nitric acid absorbers and improved nitric acid rectification equipment. (auth)

HEAD-END TREATMENTS 3.01

OFF-GAS 3.01.06

HNO_3 3.01.06.02

THE ECONOMIC ASPECTS OF HNO_3 RECOVERY FROM DISSOLVER OFF-GASES. J. L. Schwennesen. May 1, 1953. Decl. Sept. 27, 1957. 8p. (LWS-24822; CRD-T4C-151)

The dissolving of irradiated U is accomplished with the evolution of large quantities of nitrogen oxides. This report is intended to indicate the economic feasibility for recovering the nitrogen oxides or HNO_3 as a function of acid value and the tonnage rate of U processed in a separations plant.

15693 CF-59-3-92(Rev.)(Del.)
General Electric Co. Hanford Atomic Products Operation, Richland, Wash. and Oak Ridge National Lab., Tenn.
ANSWERS TO QUESTIONS ON OFF-GAS TREATMENT. Eurochemic Assistance Program: Comments by HAPO, dated March 12, 1959, on Questions by E. L. Nicholson. L. P. Bupp. Mar. 26, 1959. 7p. $1.80(ph), $1.80(mf) OTS.

Information relative to the performance and operation of silver reactors is presented. The information is given as answers to questions submitted in the USAEC-Eurochemic Company coöperative program. The answers to 8 questions are presented along with the basis for comments. (J.R.D.)

DESIGN AND ECONOMIC STUDY OF HOT OFF-GAS STORAGE SYSTEM. C. S. Johnson and T. J. Carter. Dec. 20, 1957. Decl. with deletions June 27, 1958. 24p. (AECD-4269; KT-327(Del.))

A study was made of the various gas disposal methods that might be used to handle the radioactive fission product gases, Kr, Xe, and I_2. The practicality of atmospheric disposal is dependent on the ability to control radioactive gas release so that only concentrations below the tolerance level are encountered by humans. When meteorological conditions are favorable, the high activity dissolver off-gas can be discharged from tall stacks. However, when meteorological conditions are unfavorable, discharge to the atmosphere is not possible and temporary storage facilities must be provided. These storage facilities, in addition to preventing discharge of radioactive gases to the atmosphere, act as a "holdup" tank so that the shorter-lived activities can decay away before discharge to the atmosphere. However, since a considerable portion to the activity is the contribution of 10.27 year half-life Kr^{85}, reduction of activity levels by this means is relatively insignificant. Storage of the radioactive gases for safe release to the atmosphere can be accomplished by means of ambient temperature charcoal adsorbers, low temperature charcoal absorbers, high pressure tanks, or low pressure gas holders. With the present information available, it was not felt an intelligent design using charcoal adsorbers could be made. Also, because of the relatively large gas volume and the risk (from accidental release of the gas) involved in high pressure storage, this technique was not considered practical. Accordingly, it was felt that a slightly subatmospheric storage system offered the most promise at the present time for providing safe and economic temporary storage of the radioactive gases. Therefore, the technical feasibility of such a system has been studied and a preliminary design and cost analysis are submitted.

RADIOLOGICAL DEVELOPMENT ACTIVITIES. Health and Safety Unit Semiannual Progress Report for January-June 1952. July 1952. Decl. Mar. 29, 1957. 42p. (KAPL-814)

Data are reported from a study of the total activity, the size distribution of the particulate material, and isotopic composition of the radioactive components discharged from the stack during the dissolving, head-end, and extraction operations of the separations process. The atmospheric dilution of the stack effluent was also estimated. Information obtained on the radioisotopic composition of evaporator condensate provided the basis for revision of the maximum permissible limits for discharge to the Mohawk River. A series of tests established a maximum detection efficiency of 19% for a scintillation probe in detecting γ radiation in a liquid medium. A smoke generator suitable for the evaluation of the efficiency of laboratory fume hoods is described, and a schematic sketch is included.

318
TREATMENT OF GASEOUS EFFLUENTS. Walter C. Schmidt. Apr. 10, 1957. p.362-76 of SYMPOSIUM ON THE REPROCESSING OF IRRADIATED FUELS HELD AT BRUSSELS, BELGIUM, MAY 20-5, 1957. May 1, 1957. 1165p. (TID-7534(Bks. 1-3); HW-49549A)

Equipment for the removal of noxious radioactive waste gases from chemical processing facilities is described. The discussion is separated into 2 parts to describe the equipment necessary for the removal of particulate radioactivity present as an aerosol and other radioactivity which is present as a true gas. As the discussion develops on the decontamination of waste gases, actual data and empirical equations for design calculations are presented. Also presented is a short section on the difficulties experienced in operating the equipment as described.

HEAD-END TREATMENTS 3.01

OFF-GAS 3.01.06

HNO_3 3.01.06.02

OFF-GAS TREATMENT IN BERKELEY ENCLOSURES. M. D. Thaxter, H. P. Cantelow, and C. Burk. Jan. 7, 1957. 16p. (UCRL-3635)

Developments at UCRL in off-gas treatment are reviewed. A multiple-purpose gas scrubber and a total capture system for slug-dissolver off gas are described.

BIBLIOGRAPHY AND ABSTRACTION OF DEENTRAINMENT LITERATURE. R. H. Bond and J. H. Murray. Nov. 13, 1956. Decl. with deletions June 3, 1958. 49p. (HW-46701(Del.))

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.

8505 ORNL-2116
Oak Ridge National Lab., Tenn.
MEASUREMENT AND ANALYSIS OF THE HOLDUP OF GAS MIXTURES BY CHARCOAL ADSORPTION TRAPS. W. E. Browning and C. C. Bolta. Aug. 10, 1956. 23p. Contract W-7405-eng-26. $0.25(OTS).

The holdup behavior of gaseous fission products in charcoal traps was investigated as a function of trap geometry, type and amount of charcoal, trap temperature, flow rate, and type of inert gas used as purging agent. An analytical expression is presented for the calculation of holdup curves for gases when trap geometry and the adsorption isotherm for the gases on charcoal are known. (C.W.H.)

12623 CF-56-6-127
Oak Ridge National Lab., Tenn.
FUEL MELTING EXPERIMENT—IRRADIATION REQUEST ORNL-MTR-28, PHASE A. F. H. Neill. June 20, 1956. Decl. Feb. 22, 1957. 9p. Contract [W-7405-eng-26]. $1.80(ph OTS); $1.80(mf OTS).

The irradiation program to obtain information about gaseous fission product release from the melting of an APPR fuel element is presented. (T.R.H.)

4114 CF-56-1-106
Oak Ridge National Lab., Tenn.
SHIELDING FOR THE HRT CHARCOAL PIT AND OFF-GAS LINE. James O. Kolb. Jan. 20, 1956. 13p. Contract [W-7405-eng-26]. $3.30(ph OTS); $2.40(mf OTS).

Calculations for corrections for shielding of the HRT charcoal pit, off-gas line from the east valve pit to the charcoal pit and dose rates for various dirt and concrete thicknesses are given. (F.S.)

RADIOACTIVITY OF DISSOLVER GAS. W. A. Brooksbank and E. J. Fuller. Aug. 26, 1953. Decl. Nov. 22, 1955. 32p. (ORNL-1064)

An apparatus for counting β activity in off-gas is described. It consists of 3 parts; the dilution section, the absorption section, and the sample counting tubes. The dilution apparatus provides a rapid and simple way of reducing activity concentration. The absorption section is used to absorb the I_2 activity from the diluted sample. The sample counting tubes hold the gas under known conditions while it is being counted. Geometry for the sample counting tubes was determined to be 3.5% for Kr^{85}. I_2 dissolver gas was analyzed with the ion chamber, and the proportions of Xe^{133} and X^{135} contained in it were measured. The proportions of Kr^{85} and Xe^{133} in a mixture of the 2 were checked by use of the apparatus with a fair degree of accuracy. A detailed procedure for noble gas analysis is given in the Appendix.

21438
SURVEY OF RECENT WORK ON THE VISCOSITY, THERMAL CONDUCTIVITY AND DIFFUSION OF GASES AND GAS MIXTURES. P. E. Liley (Purdue Univ., Lafayette, Ind.). p.40-69 of "Thermodynamic and Transport Properties of Gases, Liquids and Solids."

Recent theoretical and experimental studies of transport properties of gases and gas mixtures are reviewed. Emphasis is given to data reported for high temperatures while no data for temperatures below the ice point are reviewed. Summary tables are given to enable easy access to references for the pressure and/or temperature dependence for a particular property and gas. Suggestions are appended for work urgently required and a bibliographic listing of 550 references given. (auth)

HEAD-END TREATMENTS 3.01

OFF-GAS 3.01.06

HNO_3 3.01.06.02

Containment of Radioactive Fission Gases by Dynami Adsorption, R. E. Adams, W. E. Browning, Jr. and R. D. Ackley, Ind. and Eng. Chem., 51, 12, p.146 Dec. 1959.

1165 HW-20332
Hanford Works, Richland, Wash.
DECONTAMINATION OF DISSOLVER VENT GASES AT HANFORD. A. G. Blasewitz, R. V. Carlisle, B. F. Judson, M. F. Katzer, E. F. Kurtz, W. C. Schmidt, and B. Weidenbaum. Feb. 16, 1951. Decl. July 6, 1959. 17p. Contract W-31-109-Eng-52. OTS.

The dissolver vent gases constitute the most highly contaminated gas stream in the present Hanford Separations Plants. From the health hazard viewpoint there are two principal contaminants, radio-iodine and an aerosol composed of other fission products. An extensive study has been made at Hanford of methods and equipment to remove effectively these materials. This investigation has led to the development and adoption of the silver reactor and Fiberglas filter for iodine and particulate matter removal, respectively. Plant scale equipment has been designed and installed in the dissolver cells. The first silver reactor–Fiberglas filter assembly was placed in operation on October 26, 1950. The efficiencies of the two components have been evaluated. Caustic scrubbing monitors were used to determine the radio-iodine content of the dissolver vent gases upstream and downstream from the silver reactor, and CWS Type 6 monitoring filters were used to determine the particulate decontamination of the Fiberglas filter. The monitoring data have established the I^{131} removal efficiency of the silver reactor to be 99.9% and the filtration efficiency of the Fiberglas filter bed to be greater than 99.9% and probably near 99.99%. A conservative estimate of the minimum useful lives of the two components (under Hanford conditions) is at least one year. (auth)

IMPROVEMENTS IN OR RELATING TO THE SEPARATION OF GASEOUS MIXTURES. Schofield Labrow, James Findlay Dunn, Robert Lipscomb, and Andrew Craig (to United Kingdom Atomic Energy Authority). British Patent 811,930. Apr. 15, 1959.

A device for separating gases by differential freezing is described. Gas is introduced at the bottom of a cooled container, and a part of it freezes out on the walls to be scraped off by an oscillating scraper. The unfrozen portion passes out the top. An arrangement at the bottom allows reheating and removal of the scraped-off portion without losing pressurization of the container. This arrangement consists of a series of openings in a plate coupled to the oscillating scraper. (T.R.H.)

2643 AERE-C/M-260
Gt. Brit. Atomic Energy Research Establishment, Harwell, Berks, England.
AN APPARATUS FOR THE DISPOSAL OF FLUORINE ON A LABORATORY SCALE. G. Long. Oct. 1955. 5p.

The apparatus in which F_2 is burnt in a coal-gas flame and the combustion products adsorbed in caustic soda solution is described. Flow rates of up to 15 l/hr have been handled. (auth)

RECOVERY OF BY-PRODUCT FLUORINE. Grady Tarbutton, Thad D. Farr, Thomas M. Jones, and Harry T. Lewis, Jr. (Tennessee Valley Authority, Wilson Dam, Ala.). Ind. Eng. Chem. 50, 1525-8(1958) Oct.

Practical methods for recovering fluorine compounds from effluent gas in phosphate processes and conversion of these compounds to useful products were investigated. Recovery as cryolite was encouraging in a plant-scale test producing specification grade material. Recovery as aluminum fluoride was evaluated, and recovery as hydrogen fluoride with a solid bed absorption method is described. (J.R.D.)

HEAD-END TREATMENTS 3.01

OFF-GAS 3.01.06

KRYPTON 3.01.06.03

16550 CF-59-6-47
Oak Ridge National Lab., Tenn.
REMOVAL OF FISSION PRODUCT GASES FROM REACTOR OFF-GAS STREAMS BY ADSORPTION (PRESENTED AT AMERICAN NUCLEAR SOCIETY MEETING, DETROIT, MICHIGAN, DECEMBER 10, 1958).
W. E. Browning, R. E. Adams, and R. D. Ackley.
June 11, 1959. 36p. Contract [W-7405-eng-26]. $6.30 (ph), $3.00(mf) OTS.

A disposal process is described in which the noble gas fission products, krypton and xenon, are delayed relative to the sweep gas by physical adsorption as they pass through an adsorbent such as activated charcoal. A theoretical expression describing this process was developed, using a theoretical plate analysis, and was verified experimentally. The retention time for a gas present in trace concentration is proportional to the amount of charcoal in the adsorber bed and to the adsorption coefficient which is evaluated experimentally for a particular combination of materials and conditions. The retention time is inversely proportional to the volume flow rate of the sweep gas. The retention times of experimental adsorbers have been measured by a radioactive tracer technique using krypton-85 and xenon-133 to typify fission gases. Retention times were measured for various adsorbents including different grades of activated charcoal, silica gel, activated alumina, and molecular sieve materials. Activated charcoal was the most effective adsorbent. Retention times were measured using helium, hydrogen, argon, nitrogen, oxygen, air, CO_2, Freon-12, and krypton as sweep gases, individually. Water vapor reduces the retention time for krypton on charcoal as does CO_2. Krypton interferes negligibly up to 1000 microns partial pressure. Provision must be made for the dissipation of heat from radioactive decay. Retention time decreases logarithmically as temperature increases. Ignition temperatures in oxygen were determined and methods for prevention and control of charcoal fires were investigated. Experimental results described were used to design off-gas adsorber systems for in-pile experiments and to analyze the performance of an adsorber for a homogeneous circulating fuel reactor. These adsorber systems performed satisfactorily for times in excess of one year.

7062 A/CONF.15/P/309
THE RECOVERY OF RADIO KRYPTON FROM DISSOLVER WASTE GASES BY FRACTIONAL EXTRACTION IN SOLVENTS. R. W. McIlroy, E. Glueckauf, H. J. de Nordwall, and F. C. W. Pummery (Atomic Energy Research Establishment, Harwell, Berks, Eng.). 23p.

The use of solvents for scrubbing out soluble gases from mixtures is well known, but the separation of mixtures of relatively insoluble gases has not been studied very closely. A special case occurs, however, when small quantities of valuable gases occur in a mixture of less soluble gases. The mixture nitrogen, oxygen, argon, krypton, xenon, and nitrous oxide which constitutes dissolver waste gas is a pertinent example. The last three have an abundance of 1% or less, and the required products are krypton (stable and radioactive) and xenon (stable). The solubilities of nitrogen, oxygen, and argon in carbon tetrachloride are <0.35 v/v and those of krypton, xenon, and nitrous oxide are >1 v/v. A plant designed to separate the first three from the last three components is described. In principle the system is similar to any two phase extraction system, but differs from the normal gas/liquid column in having a low gas flow and continuous liquid phase, and from a normal solvent extraction column in having bottom reflux. With low gas flows, back diffusion in a continuous gas phase would be very disadvantageous, consequently a continuous liquid phase was used. In this case steady operation with low gas flows requires considerable care and control. The feed concentration of the required product gases is low, and the effective separation factors are comparatively low. This requires an efficient reflux system at the foot, and the reflux ratio must be high. Equations were derived for the behavior of each component on the assumption that each gas has the same plate height. The calculation is complicated by the fact that the flow rate (of gas in both phases) is not constant along the length of the column but increases towards the foot due to the accumulation there of the more soluble components. This also leads to maxima in the concentration gradients of all gases in the mixture except those of greatest and least solubility, an effect which can only occur to any large extent in mixtures of more than two components having widely different solubilities.

HEAD-END TREATMENTS 3.01

OFF-GAS 3.01.06

KRYPTON 3.01.06.03

2830 CF-52-11-82(Del.)
Oak Ridge National Lab., Tenn.
PILOT PLANT RADIOACTIVE GAS SEPARATION PROCESS EQUIPMENT AND OPERATING INSTRUCTIONS. W. B. Watkins, W. L. Poe, and D. R. Lindow. Nov. 12, 1952. Decl. with deletions Nov. 21, 1958. 45p. Contract [W-7405-eng-26]. $7.80(ph OTS); $3.30 (mf OTS).

The purpose of the Radioactive Gas Separation Process (RAGS) is to remove radioactive elements, particularly krypton-85, from the gas evolved during the dissolution of irradiated fuel units. The process was developed by the ORNL Chemical Development and Unit Operations Sections and Linde Air Products Co., and was then installed in the ORNL pilot plant to confirm the feasibility of the laboratory flowsheet and to secure sufficient data to support an economic study of a plant scale installation. The process was operated in three distinct phases; first, the dissolver off-gas was continuously processed through the entrainment separator, condenser, iodine tower, filter, acid tower, and into the gas holder. Second, the gas was continuously processed through the caustic scrub tower, alumina dryer, cold trap, and silica gel adsorption unit. Finally, the krypton and xenon were desorbed from the silica gel bed and stored. The off-gases from 24 dissolvings were processed through the equipment. The adsorption of radioactive krypton on silica gel at −183°C was demonstrated in six successive runs. (auth)

IODINE 3.01.06.04

2240 HW-42408(Del.)
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
CHEMISTRY OF THE "SILVER REACTOR." PART A. INFLUENCE OF NO_2 CONCENTRATION AND TEMPERATURE DURING SLUG DISSOLVING. G. B. Barton. PART B. REACTIONS WITH AMMONIA GAS. E. D. McClanahan, Jr. Mar. 5, 1956. Decl. with deletions June 11, 1959. 15p. OTS.

A study was conducted to improve the efficiency of iodine removal from chemical processing waste gases. The investigation revealed that no single chemical equation expresses the reaction of silver nitrate with iodine in the presence of various compositions of air−nitrogen−oxygen systems under varied conditions. A study was also made on the effects of coating removal gases such as ammonia on the silver reactor. Observations on conditions under which absorbed iodine may be released due to the presence of ammonia are included as well as a discussion of chemical aspects of ammonia reactions. It was concluded that the silver nitrate reactor will not reduce the iodine concentration in plant gas streams to the level required by future needs. It is noted that the investigations were halted before completion. (J.R.D.)

13419 AECU-4120
Idaho Operations Office. Health and Safety Div., AEC.
IODINE MONITORING AT THE NATIONAL REACTOR TESTING STATION. Claude W. Sill and J. Kenneth Flygare, Jr. Mar. 23, 1959. 19p. $3.30(ph), $2.40(mf) OTS.

Operations at the National Reactor Testing Station generally involve new types of fuel elements or reactors or the chemical processing of spent fuel elements. Because the isotopes of iodine are produced in high yield during fission and their short half-lives permit equilibrium concentrations to be built up rapidly, considerable inventories of these isotopes will be present after a reactor has operated for a relatively short length of time. The gaseous nature of the element allows its escape after rupture of the element cladding and particularly during chemical dissolution of short-cooled fuel. Iodine is one of the chief problems at this installation from the standpoint of radiological protection. Some of the problems encountered and the way in which they were solved are described. Of particular interest is the development of a continuous monitor for gaseous iodine that permits immediate detection and records the buildup of activity as a function of time. (auth)

HOMOGENEOUS REACTOR FUEL REPROCESSING. Quarterly Progress Report [for] April 1−June 30, 1956. Vitro Job 1087. Harold F. Reichard, John D. McAdoo, Stanley J. Klach, Gloria K. Koenig, and Paul E. Smith. July 31, 1956. Decl. Mar. 29, 1957. 14p. (KLX-10034)

The behavior of volatile fission products in aqueous homogeneous reactor core solution was studied. Particular attention is given to the chemistry of I_2, with the specific objective of removing I_2 rapidly from the fuel as a means of controlling Xe^{135} poisoning. Feasibility of high-pressure O_2 stripping of I_2 with an ejector is being studied as a means of implementing this process.

HEAD-END TREATMENTS 3.01

OFF-GAS 3.01.06

XENON 3.01.06.05

5953 ✓
THE DIFFUSION OF FISSION-Xe FROM U METAL. K. E. Zimen and L. Dahl. Z. Naturforsch. 12a, 167-9(1957) Feb. (In German)

OTHER FUELS AND MATERIALS 3.01.07

See also, under SURVEYS (GENERAL),

FUELS AND FUEL ELEMENTS (7.04).

ORNL-2460
Oak Ridge National Lab., Tenn.
HYDROFLUORIC ACID DECLADDING OF ZIRCONIUM-CLAD POWER REACTOR FUEL ELEMENTS. W.E. Clark and A.H. Kibbey. Nov. 7, 1958. 17p.

A detailed flowsheet is presented for the removal of Zircaloy-2 cladding from a UO_2 core (as in the Commonwealth Edison fuel- by dissolution in hydrofluoric acid to give a F/Zr ratio of 4.5/1 in the solution. The core is dissolved in 8 M HNO_3 for subsequent removal of fission products by solvent extraction. In tests on unirradiated prototypes, uranium loss to the fluoride solution was as little as 0.16%. Dissolution data on uranium metal and U-10% Mo alloy indicate that these cores can be declad similarly. U-Zr alloys cannot be so stripped while U-Nb is somewhat doubtful. It appears that pure zirconium cladding can be removed about as easily as Zircaloy-2. One container constructed of Carpenter 20 (niobium stabilized) stainless steel can probably be used for both dissolution steps. (auth)

9461 BNL-483(p.168-71)
Oak Ridge National Lab., Tenn.
PROCESSING OF THE AQUEOUS HOMOGENEOUS REACTOR AND THE MOLTEN SALT REACTOR. D. E. Ferguson. p.168-71 [of] THORIUM–U^{233} SYMPOSIUM, SPONSORED BY THE UNITED STATES ATOMIC ENERGY COMMISSION AT BROOKHAVEN NATIONAL LABORATORY, JANUARY 9-10, 1958. 4p.

A brief description and flowsheet are given for reprocessing homogeneous reactor fuel and blanket slurries. A brief description is also given of the fluoride volatility process which will be used to reprocess fuel and blanket systems of the molten salt reactor. (W.L.H.)

5253 HW-61106
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
PRELIMINARY CHEMICAL FLOWSHEETS FOR THE DECLADDING AND DISSOLUTION OF NON-PRODUCTION FUELS. M. K. Harmon. July 24, 1959. 9p. OTS.

Preliminary chemical flowsheets are presented for cladding and core dissolution of Zircaloy-clad UO_2 and stainless steel-clad U–Mo non-production fuels. (W.L.H.)

9460 BNL-483(p.165-7)
Brookhaven National Lab., Upton, N. Y.
CORE AND BLANKET PROCESSING IN THE LMFR. O. E. Dwyer. p.165-7 [of] THORIUM–U^{233} SYMPOSIUM, SPONSORED BY THE UNITED STATES ATOMIC ENERGY COMMISSION AT BROOKHAVEN NATIONAL LABORATORY, JANUARY 9-10, 1958. 3p.

A proposed process for reprocessing irradiated Liquid Metal Fuel Reactor core and blanket systems is briefly discussed. (W.L.H.)

European Company for the Chemical Processing of Irradiated Fuels, Mol, Belgium.
PRACTICAL CONSIDERATIONS ON THE DECANNING AND THE DISSOLUTION OF ALUMINUM- OR MAGNESIUM-CLAD ELEMENTS. Technical Report No. 26. E. Detilleux. Dec. 4, 1958. 21p. $4.80(ph), $2.70(mf) OTS.

Data in various reports indicate that it is possible to process Al- and Mg-clad fuel elements in the same vessel. The conditions of such an operation are considered, and proposed chemical flowsheets are presented. Treatment of Al-clad elements are discussed, and operational procedures are presented in tabular form. Small changes in the Al-clad element procedure for use with Mg-clad elements are pointed out. The apparatus flowsheet resulting from this study is presented in TR-25. (J.R.D.)

HEAD-END TREATMENTS 3.01

OTHER FUELS AND MATERIALS 3.01.07

1229
ACID LEACHING AND ANODE DISSOLUTION OF GRAPHITE-MODERATED FUELS. K. R. Hyde and J. L. Woodhead (Atomic Energy Research Establishment, Harwell, Eng.) and D. J. O'Connor (Australian Atomic Energy Commission Research Establishment, Lucas Heights, New South Wales). p.420-8 of "Australian Atomic Energy Symposium, 1958."

Results of nitric acid leaching and anodic disintegration processing studies of two types of homogeneous graphite moderated fuels are reported. A 2N nitric acid leaching at 90 to 100°C appears suitable as the primary processing method, whereby uranium and thorium are obtained in solution suitable for solvent extraction. Anodic disintegration in 2N nitric acid is a satisfactory wet method of breaking down the graphite fuel assemblies, but the disintegration process does not give the required uranium and thorium recoveries. It was not possible to assign any electrochemical action to the anodic solution of uranium and thorium carbides. (auth)

7692 HW-32309
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
THE REMOVAL OF NICKEL PLATES FROM URANIUM. R. L. Dillon. June 25, 1954. Decl. Jan. 28, 1960. 5p. Contract [W-31-109-Eng-52]. OTS.

Nickel is removed from U by immersing for 15 min in one part 70% HNO_3, and one part water at 80°C with cold water rinsing. U dissolves in 50% by volume commercial HNO_3 at the rate of 0.1 mil/min at 80°C. A suitable construction material for the tank is corrosion-tested stainless steel. (F.S.)

9637 NAA-SR-213
North American Aviation, Inc., Downey, Calif.
THE USE OF BROMINE TRIFLUORIDE IN THE RECOVERY OF URANIUM FROM URANIUM IMPREGNATED GRAPHITE FUEL MATERIALS. F. D. Rosen. Dec. 29, 1952. Decl. Feb. 27, 1957. 34p. $0.30(OTS).

Removal of 99.8% of the U as UF_6 from graphite impregnated with uranium oxide or carbide has been accomplished by treating the material with at least 0.5 mole of BrF_3 per mole of graphite. A preliminary investigation of the reaction of BrF_3 with graphite indicated that the following reactions may occur, depending on the experimental conditions: $BrF_3 + C \rightleftharpoons C_nBrF_3$ ($n \geq 1$), $BrF_3 + C \longrightarrow (CF)_x + Br_2$, $BrF_3 + C \xrightarrow{UC_2} CF_4 + Br_2$. Graphite impregnated with UC_2 has been found to be considerably more reactive toward BrF_3 than the oxide-impregnated materials, but preliminary treatment with liquid Br_2 ensured a smooth reaction on subsequent addition of BrF_3. Safety requirements for handling the BrF_3-graphite system are reviewed, with special reference to the dry graphite residue left after BrF_3 treatment. (auth)

11699 HW-28282
Hanford Atomic Products Operation, Richland, Wash.
REMOVAL OF THE COMPOUND LAYER FROM DE-CANNED HANFORD URANIUM SLUGS BY THE USE OF SODIUM HYDROXIDE. H. L. Brandt and H. P. Kraemer. May 15, 1953. Decl. Feb. 19, 1957. 6p. Contract [W-31-109-Eng-52]. $1.80(ph OTS); $1.80(mf OTS).

In a laboratory-scale investigation of sodium hydroxide removal of the compound layer from de-canned, triple-dipped, and lead-dipped U slugs, a 50% caustic solution readily removed the compound layer. (F.S.)

7510 CF-56-6-27
Oak Ridge National Lab., Tenn.
HRP-BP: DISSOLVING SOLUTION FOR SOLIDS REMOVED FROM UO_2SO_4 BLANKET OF HRT. William L. Carter. June 4, 1956. Decl. Feb. 14, 1957. 5p. Contract [W-7405-eng-26]. $1.80(ph OTS); $1.80(mf OTS).

When the HRT is converted from a heavy water blanket to a UO_2SO_4 blanket, a processing plant will begin operation to remove the Pu, fission, and corrosion products from the blanket. A short discussion of the expected corrosion rates of the stainless steel parts in the blanket system and the composition of the blanket before processing is presented. (W.L.H.)

7475
PLUTONIUM CLEANING PROCESS. M. Kolodney (to U. S. Atomic Energy Commission). U. S. Patent 2,915,387. Dec. 1, 1959.

A method is described for rapidly removing iron, nickel, and zinc coatings from plutonium objects while simultaneously rendering the plutonium object passive. The method consists of immersing the coated plutonium object in an aqueous acid solution containing a substantial concentration of nitrate ions, such as fuming nitric acid.

4470 AEC-tr-2820
THE REACTION OF METALLIC TITANIUM, ZIRCONIUM, HAFNIUM AND THORIUM WITH ACIDS, IN PARTICULAR HYDROFLUORIC ACID. M. E. Straumanis and J. I. Ballass. Translated by D. J. Beernsten from Z. anorg. u. allgem. Chem. 278, 33-41(1955). 6p.

An abstract of the paper appears in Nuclear Science Abstracts as NSA 9-2783. (W.L.H.)

HEAD-END TREATMENTS 3.01

PLUTONIUM – ALUMINUM FUEL ELEMENTS 3.01.08

See also, under PROCESS CHEMISTRY AND ENGINEERING,

SPECIAL PROCESSES--Bismuth Phosphate (6.17.02),
Butex (6.17.03),
Metal Recovery (6.17.15),
Purex (6.17.17),
Redox (6.17.19).

7737 HW-57342
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
PLUTONIUM FUELS DEVELOPMENT—PLUTONIUM METALLURGY OPERATION QUARTERLY REPORT FOR APRIL, MAY, JUNE 1958. O. J. Wick. Sept. 10, 1958. 51p. Contract W-31-109-Eng-52. $4.80(ph), $2.70(mf) OTS.

Radiometallurgical examination of the last two capsules irradiated in the MTR to 55 to 60% burnup of the Pu atom is complete. These capsules contained Al–1.65 wt. % Pu and Al–12 wt. % Si–1.65 wt. % Pu alloy cores. Assembly is in progress on two four-rod Al–8 wt. % Pu and Al–12 wt. % Si–8 wt. % Pu alloy clusters for irradiation in the 250°C water of the KER loop facility. A pyrometallurgical process for recycling irradiated Pu–Al fuels has been tested on unirradiated materials. Injection casting of Al–Pu alloy into Zircaloy-clad tubes is being investigated as a fuel fabrication technique. Mixed crystal oxides of U and Pu in a ratio of one to five have been processed to form pellets having a green density of 63% of theoretical with an L/D value of 1.3. Assembly was completed on the first model of a new 19-rod cluster design designated the Mark-IB. Improved techniques were devised for the welded closures on Zircaloy tubing and for the attachment of Zircaloy spacer wires to the Zircaloy end caps by Heliarc spot welding techniques. (W.L.H.)

22157
TREATMENT OF PLUTONIUM–ALUMINUM FUEL RODS. (to Atomic Energy of Canada, Ltd.). British Patent 820,660. Sept. 23, 1959.

A process is presented for Pu recovery from spent Pu–Al fuels. The fuel material is heated with Bi above the melting point, fission product concentration in the Bi phase, and separation of Bi phase. (T.R.H.)

PROBLEMS ASSOCIATED WITH THE CHEMICAL PROCESSING OF ALUMINUM–SILICON–PLUTONIUM FUEL MATERIALS. E. E. Voiland. Apr. 1, 1958. 13p. (HW-54819)

The Al–Pu–Si system having been proposed as fuel material for the Plutonium Recycle Test Reactor, its behavior in chemical processes was investigated. The Si reduced the dissolution rate and would not dissolve in HNO_3. An HF–HNO_3 system would dissolve it but equipment corrosion, volatile fluorides and other problems result. Dissolution in boiling NaOH results in a colloidal solution which resists coagulation. These and other findings lead to a recommendation not to use Si.

14694 A/CONF.15/P/546
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
THE PREPARATION AND REPROCESSING OF PLUTONIUM–ALUMINUM ALLOY. W. L. Lyon. 7p. $0.50(OTS).

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

An alloy of plutonium and aluminum offers an attractive reactor fuel material since the technology of the matrix is sound, the alloy is readily produced and fabricated, it has good nuclear and physical properties, and it is acceptable with water coolant as regards heat transfer and corrosion rate. The alloy may be produced by direct alloying of the metals, or by reduction of plutonium fluoride or oxide in aluminum. The latter method, wherein PuO_2 and cryolite mixtures are added to molten aluminum, employing the technique of Saller and Runnalls, is expected to find extensive application in the production of fuel loadings for power reactors. The chemical reprocessing of Pu-Al fuel may be accomplished by adapting aqueous methods for which sound technical bases exist. On the other hand, this material may lend itself to processing by one of several interesting pyrochemical schemes. Some of these are described together with brief comments regarding the economics of plutonium fuel processing. (auth)

11622 ✓ HW-21442
Hanford Works, Richland, Wash.
COATING REMOVAL WASTE LOSS REDUCTION. Final Report. Production Test 221-B-8. B. E. Kirkendall. June 25, 1951. Decl. Mar. 4, 1957. 13p. Contract W-31-109-Eng-52. $3.30(ph OTS); $2.40(mf OTS).

It was demonstrated that the Pu and U losses associated with the Al jacket dissolution could be reduced by substituting a water wash for the 5% HNO_3 wash following the coating removal and that this change would not adversely affect the product yield or decontamination in subsequent process steps. (D.E.B.)

HEAD-END TREATMENTS 3.01

PLUTONIUM – ALUMINUM FUEL ELEMENTS 3.01.08

9234 CRC-639
Atomic Energy of Canada Ltd. Chalk River Project, Chalk River, Ont.
THE EXTRACTION OF PLUTONIUM FROM PLUTONIUM–ALUMINUM ALLOYS BY MOLTEN BISMUTH. D. E. McKenzie, J. W. Fletcher, and T. Bruce. Apr. 1956. 13p. (AECL-314)

The distributions of Pu, Am and certain fission products between neutron-irradiated Al–Pu alloys and molten Bi have been examined in small scale experiments. An equal volume of Bi extracts 78% of the Pu, 93% of the Am, the majority of the fission product Ce, but essentially no Ru or Zr from the Al phase. The fraction of Pu in the Bi phase decreases slightly with increasing temperature in the range 800 to 1050°C but is independent of the Pu concentration. A possible process, utilizing these results, is discussed. (auth)

9233 CRC-636
Atomic Energy of Canada Ltd. Chalk River Project, Chalk River, Ont.
SOME EXPERIMENTS RELATED TO THE PROCESSING OF PLUTONIUM–ALUMINUM ALLOY BY HIGH TEMPERATURE METHODS. D. E. McKenzie, J. W. Fletcher, and T. Bruce. Apr. 1956. 13p. (AECL-310).

A number of high temperature methods have been investigated for the decontamination of Pu–Al alloys from fission products. The methods include oxidative slagging, treatment of the alloy with molten Pb, Tl, or cryolite, and solution of the alloy in PbF_2–NaF mixtures. Of these techniques, the simplest and most effective is the treatment of the molten alloy with molten cryolite. The fission products Sr and Ce can be removed but Zr and Ru remain in the Al phase with essentially all of the Pu. (auth)

STAINLESS STEEL FUEL ELEMENTS 3.01.09

See also, under PROCESS CHEMISTRY AND ENGINEERING,

FUSED SALTS AND LIQUID METALS--Liquid Metals (6.10.02),

SPECIAL PROCESSES--Darex (6.17.04),

Fused Salt-Fluoride Volatility (6.17.11),

Sulfex (6.17.20).

HEAD-END PROCESSES FOR DISSOLVING STAINLESS STEEL–UO_2 DISPERSION TYPE FUEL ELEMENTS. I. CARBIDE AND NITRIDE TREATMENTS. II. ANODIC DISSOLUTION. F. S. Martin and M. J. Waterman. Jan. 1958. 17p. (AERE-C/R-2454)

The treatment of 18-8-1 type stainless steel plate with NH_4 or CH_4 at about 1000°C renders the metal more susceptible to dissolution in HNO_3. The rate of penetration (of carbide or nitride) is very slow, and the method does not seem to be very suitable, except for the treatment of very thin plates (~0.025″ thickness). Stainless steel and stainless steel–UO_2 cermets dissolve readily in HNO_3 if an anodic potential is applied. High current efficiencies are obtainable with d-c and moderate efficiencies with a-c. A wide range of initial HNO_3 concentrations may be used, and dissolution may be carried to the point where large quantities of $Fe(OH)_3$ are precipitated. U recoveries are > 99.5%. The bulk of the fission product activity is found in solution, but fission product Nb is associated with traces of insoluble residue. The method would be suitable for the dissolution of large pieces of stainless steel (e.g., whole fuel elements) where individual electrical contacts may be made external to the solution; it would not be suitable where large numbers of small pieces have to be held (e.g., in a Pt basket), due to the almost negligible current efficiencies that are obtained in such cases.

ELECTROLYTIC DISSOLUTION OF STAINLESS STEEL. E.C. Pitzer (to U.S. Atomic Energy Commission). U.S. Patent 2,865,832. Dec. 23, 1958.

A method is described for electrolytic dissolution of stainless steel in a nitric acid solution. The stainless steel object is kept in contact with either a tantalum, titanium, or zirconium anode in the electrolytic cell. Some constituents, including niobium, niobium carbide, and silicon, are not dissolved and must be removed from the anode surface by agitation in order to maintain a prescribed current.

12658
HAZARD CONTROL IN PROCESSING STAINLESS STEEL AND COPPER CONTAMINATED WITH URANIUM. D. M. Davis, J. C. Hart, and A. D. Warden (Oak Ridge National Lab., Tenn.). Am. Ind. Hyg. Assoc. Quart. 18, 235-41(1957) Sept.

HEAD-END TREATMENTS 3.01

STAINLESS STEEL FUEL ELEMENTS 3.01.09

4753 CF-54-4-39(Del.)
Oak Ridge National Lab., Tenn.
RECOVERY OF URANIUM FROM THE SPENT STAINLESS STEEL FUEL ELEMENTS OF THE PACKAGE REACTOR. J. E. Savolainen. Apr. 6, 1954. Decl. with deletions May 7, 1957. 26p. Contract [W-7405-eng-26]. $4.80(ph OTS); $2.70(mf OTS).

A solvent extraction process for recovering uranium from spent stainless-steel clad Package Reactor fuel elements is proposed, and the results of limited laboratory work establishing its feasibility reported. The process differs from those previously developed for stainless-steel clad fuel elements principally in the method used to bring the fuel into solution prior to solvent extraction recovery and purification. This process is based on the original concept of destroying the passivity of the stainless steel by the introduction of carbon into the metal to form chromium carbide. (auth)

REVISED EBR-II FLOWSHEET FOR FUEL CYCLE WITH FUEL PURIFICATION BY OXIDATIVE SLAGGING. L. Burris, Jr. and I. G. Dillon. Feb. 23, 1956. Decl. Mar. 11, 1957. 15p. (ANL-LB-SL-1016)

7445 HW-62199
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
DISSOLUTION OF STAINLESS STEEL CLAD POWER REACTOR FUELS WITH NITRIC ACID AND HYDROFLUORIC ACID. C. R. Cooley. Oct. 12, 1959. 18p. Contract AT(45-1)-1350. OTS.

The application of the Niflex Process for the dissolution of stainless steel clad fuel elements was studied on a pilot plant scale. A summary of pilot plant data and supporting laboratory information is presented. It was concluded that the process is reasonably successful in laboratory and pilot unit tests; however, more suitable construction materials are needed, and the reaction mechanisms must be studied further for optimization purposes. (J.R.D.)

THORIUM – URANIUM FUEL ELEMENTS 3.01.10

See also, under PROCESS CHEMISTRY AND ENGINEERING,

SPECIAL PROCESSES--Hermex (6.17.13),
Metallex (6.17.14),
Thorex (6.17.22).

2059 ORNL-2519
Oak Ridge National Lab., Tenn.
DISSOLUTION OF SINTERED THORIUM–URANIUM OXIDE FUEL IN NITRIC ACID–FLUORIDE SOLUTIONS. W. D. Bond. Nov. 14, 1958. 19p. Contract W-7405-eng-26. $0.75(OTS).

Boiling 13 M HNO_3–0.04 M NaF appears to be a suitable dissolvent for the 96% ThO_2–4% UO_2 fuel element core material similar to that to be used by Consolidated Edison. Dissolution of unirradiated ¼-in.-diam pellets was 98% complete in 5.3 hr, producing a final product solution of 8.6 M HNO_3–1 M $Th(NO_3)_4$–0.041 M $UO_2(NO_3)_2$ at an over-all average dissolution rate of 3.4 mg/cm^2 · min. Extrapolation of the rate data indicates that complete dissolution could be achieved in 6 to 7 hr. A stepwise procedure with fresh dissolvent would produce faster dissolution rates. The total dissolution time was 1 to 2 hr for pellets that had been crushed to a powder. Sulfuric, nitric, and hydrofluoric acids and aqua regia were ineffective dissolution agents. (auth)

NITRIC ACID DISSOLUTION OF THORIUM. KINETICS OF FLUORIDE-CATALYZED REACTION. Robert Lee Moore, C. A. Goodall, J. L. Hepworth, and R. A. Watts, Jr. (Hanford Atomic Products Operation, Richland, Wash.). Ind. Eng. Chem. 49, 885-7(1957) May.

The penetration rate of Th metal by boiling HNO_3 was measured as a function of the concentrations of acid, fluoride catalyst, and dissolved Th. The fluoride concentration was the most important variable, the dissolving rate in 13M HNO_3 increasing from 1 mg per hour per sq cm in the absence of fluoride to over 10,000 mg per hour per sq cm at 0.1M fluoride. The rate also increased with increasing nitric acid and reached a maximum at about 13M. Dissolved Th inhibited the reaction, presumably by complexing the free fluoride. Aluminum had a similar effect. The dissolution rate of thoria was found to be much less than that of Th. "Integral" dissolvings were also made, and an empirical mathematical equation was derived to correlate these results and facilitate application. (auth)

SEPARATION OF URANIUM FROM FOREIGN SUBSTANCES. B. J. Fontana (to U. S. Atomic Energy Commission). U. S. Patent 2,780,517. February 5, 1957.

A method for separating U from neutron irradiated Th is presented. The irradiated mass is dissolved in HNO_3 and a concentrated aqueous solution is formed wherein the $Th(NO_3)_4$ is present in a ratio by weight of $Th(NO_3)_4$ to H_2O of approximately 1:1. The U, however, is present in concentration of less then 1% that of the Th. This aqueous solution is then extracted with ether which preferentially takes up 70 to 80% of the U present and only 0.5% of the Th.

HEAD-END TREATMENTS 3.01

THORIUM – URANIUM FUEL ELEMENTS 3.01.10

DISSOLUTION OF THORIUM OXIDE–URANIUM OXIDE FUEL ELEMENTS. R. P. Larsen. Aug. 9, 1956. Decl. Mar. 9, 1957. 2p. (ANL-RCV-SL-1090)

INDUCTION MELTING OF THORIUM. P. D. Corzine (Nuclear Metals Inc., Cambridge, Mass.). p.63-5 in "Progress in Nuclear Energy. Series V. Metallurgy and Fuels. Volume 2."

Methods and equipment for the induction melting of thorium and thorium alloys are described and compared. Beryllia remains the most useful material, not only for the containers themselves, but for slip linings and washes in crucibles and molds of graphite. The choice of a furnace is shown to be dependent upon prod uct specifications; the advantages of several types are given. Problems of segregation, sub-surface gas, and cracking in alloy melts are treated briefly, and a summary analysis of sixteen 75-lb billets of Th–5.4 wt. % U alloy is included. (auth)

7449 ORNL-2821
Oak Ridge National Lab., Tenn.
PROCESS FOR DISSOLUTION OF BORAX IV REACTOR FUEL: LABORATORY DEVELOPMENT. L. M. Ferris. Feb. 1, 1960. 21p. Contract W-7405-eng-26. OTS.

Flowsheets are presented for the dissolution of Borax IV reactor fuel (6.35% $UO_{2.3}$–ThO_2 pellets encased in 1% nickel–aluminum alloy and bonded with lead). In the preferred method the aluminum is dissolved first in boiling 2 M NaOH–1.78 M $NaNO_3$, with a uranium loss of ~0.07%. The lead and nickel are then dissolved in boiling 1.5 M HNO_3, with uranium losses of <0.2%. The oxide core is dissolved in two successive digestions with boiling 13 M HNO_3–0.04 M NaF–0.1 M $Al(NO_3)_3$ to produce a solution 0.6 M in thorium and 0.04 M in uranium. Dissolution of only the aluminum in sodium hydroxide or sodium hydroxide–sodium nitrate solution prior to core dissolution is unattractive since the rate of core dissolution is lowered greatly if lead is present and a product solution containing greater than 0.2 M thorium is unattainable owing to the low solubility of lead nitrate in nitric acid–thorium nitrate solutions. Simultaneous dissolution of aluminum and lead in mercury-catalyzed nitric acid appears costly since a mercury concentration of at least 0.5 M is required to ensure an adequate dissolution rate. The solubility of lead nitrate in nitric acid and nitric acid–thorium nitrate solutions was determined. (auth)

7448 NAA-SR-4660
Atomics International Div., North American Aviation, Inc., Canoga Park, Calif.
ELECTROREFINING OF IRRADIATED THORIUM–URANIUM ALLOY. W. N. Hansen, W. L. Long, and E. W. Murbach. Jan. 15, 1960. 18p. Contract AT-11-1-GEN-8. OTS.

An investigation of the electrorefining of irradiated thorium–uranium alloy was carried out. The alloy is dissolved anodically into a fused salt bath of NaCl–KCl eutectic and deposited in a molten zinc cathode. Experiments with irradiated material resulted in the separation of 80 to 90% of the total fission product activity. The product is an alloy of thorium, uranium, and zinc. The zinc can be removed by vacuum distillation. (auth)

4381 DP-399
Du Pont de Nemours (E. I.) & Co. Savannah River Lab., Aiken, S. C.
DISSOLUTION OF THORIUM IN MIXTURES OF HNO_3 AND HF. David G. Karraker. Sept. 1959. 16p. Contract AT(07-2)-1. OTS.

Laboratory measurements of dissolution rates of thorium in mixtures of nitric and hydrofluoric acids show that thorium can be dissolved with practicable time cycles in large-scale processing. The corrosion of stainless steel during the dissolution of thorium is acceptable, except that when the thorium is coated with Al–Si alloy, frequently used as a bonding material for aluminum cans, severe local corrosion of the stainless steel occurs at areas of contact with residual Al–Si. This contact corrosion does not occur when the cans are hot-press-bonded to the thorium without Al–Si. (auth)

ULTRASONICS 3.01.11

8804 HW-39080
Hanford Atomic Products Operation, Richland, Wash.
EFFECT OF ULTRASONICS ON DISSOLUTION OF NICKEL IN NITRIC ACID. R. W. Wirta and R. C. Smith. Sept. 20, 1955. 6p. Contract W-31-109-eng-52. $1.80 (ph OTS); $1.80(mf OTS).

Ultrasonic energy was found to increase the rate of dissolution of Ni in HNO_3 markedly. (auth)

HEAD-END TREATMENTS 3.01

ULTRASONICS 3.01.11

13916 NYO-7931
Aeroprojects, Inc., West Chester, Penna.
APPLICATIONS OF ULTRASONIC ENERGY. Progress Report No. 10. Covering Period from June 1, 1958 to July 31, 1958. Aug. 1958. 26p. Contract AT(30-1)-1836. $4.80(ph OTS); $2.70(mf OTS).

Further investigation of mercury disintegration of fuel-element cladding materials under ultrasonic influence revealed a measurable rate of ultrasonic attack for Zircaloy-2, corresponding with previously obtained results with Type 304 stainless steel. Ultrasonic attack rates on thorium in mercury at 72° to 88°C ranged to 6.92 mg/cm^2/hr under the experimental conditions used. Ultrasonic application produced a positive and significant effect on the flow of thixotropic thorium amalgam through an orifice, the rate of flow being markedly increased with modest increase in ultrasonic power. From experimentation to date, it appears that ultrasonic agglomeration supplemented by the use of additive liquid aerosol particles can achieve sufficient decontamination in calciner off-gas to materially reduce the health hazard. Using a solid KCl simulant aerosol, a reference was established for evaluating improvements in operating techniques, and test data indicate that collection efficiency improves with large volumes of additive aerosol, low flow rates, and a relatively low ultrasonic frequency. Studies of ultrasonic coalescence in stabilized kerosene/water emulsions, using apparatus permitting the development of annular standing waves, indicated effective emulsion breaking on a continuous basis at flow rates in the range of 144 to 360 cc/min. The annular geometry apparently leads to superior results through minimization of circulation effects. (auth)

5889 NYO-7789
Aeroprojects, Inc., West Chester, Penna.
APPLICATIONS OF ULTRASONIC ENERGY. Progress Report No. 1 Covering Period from October 16, 1956 to December 15, 1956. Feb. 12, 1957. 22p. Contract AT(30-1)-1836. $3.30(ph OTS); $2.40(mf OTS).

Investigations were initiated in applying elastic vibratory energy to three problems of the Atomic Energy Commission: fuel element dissolution, compound crystallization, and powder metallurgy. The dissolution of disks of Mo, Zircaloy-2, and Type 304 stainless steel sheet in hot acid solutions was markedly accelerated by close-proximity ultrasonic treatment. Preparatory to studies of the ultrasonic effect on Th bismuthide crystal size and shape, , acoustic intensity in molten Bi was found to be high in the cavitation zone adjacent to the ultrasonic coupler face, but to drop off rapidly with distance. Significant factors in ultrasonic application to several powder metallurgy problems were examined, and designs for suitable experimental equipment were projected. (auth)

8943 NYO-7917
Aeroprojects, Inc., West Chester, Penna.
APPLICATIONS OF ULTRASONIC ENERGY. Progress Report No. 3 Covering Period from February 16, 1957 to April 15, 1957. May 1957. 32p. Contract AT(30-1)-1836. $6.30(ph OTS); $3.00(mf OTS).

Continued experimentation in the dissolution of stainless steel and Zircaloy-2, using close-proximity ultrasonic treatment, has resulted in the accumulation of substantial amounts of data on the effect of various reagent solutions. The use of fluoboric acid appears promising for the dissolution of Zircaloy-2; a solution of 5M nitric acid plus 1M hydrochloric acid effects satisfactory dissolution of stainless steel with applied ultrasonics. Studies on the effect of pressure and applied r-f power on the rate of dissolution of Zircaloy-2 in an 80% sulfuric acid–20% nitric acid solution indicated retardation of the corrosion rate as the pressure was increased from atmospheric up to 270 psig. Increased power at atmospheric pressure produced significantly increased corrosion rates. Subatmospheric pressure showed a negligible effect on rate of corrosion. Exploratory work indicated promise for the technique involving direct coupling of ultrasonic energy to the material to be dissolved. Fabrication of the resistance-heated, controlled-atmosphere ultrasonic furnace for the treatment of thorium bismuthide melts has been completed. Ultrasonic transducer-coupling-punch systems for introducing both axial and shear-type vibration into powdered metals during compaction have been fabricated and assembled in a 100-ton hydraulic press. (For preceding period see NYO-7916.) (auth)

ZIRCONIUM – URANIUM FUEL ELEMENTS 3.01.12

606
DESHEATHING OF URANIUM FUEL RODS. (to Atomic Energy of Canada, Ltd.). British Patent 820,670. Sept. 23, 1959.

The decladding of Zr-clad U rods by exposing the U and treating with steam at 340 to 380°C is described. (T.R.H.)

HEAD-END TREATMENTS 3.01

ZIRCONIUM - URANIUM FUEL ELEMENTS 3.01.12

See also, under HAZARDS AND PROTECTION,
PYROPHORICITY (4.01.10),
and,
under PROCESS CHEMISTRY AND ENGINEERING,
FUSED SALTS AND LIQUID METALS--Liquid Metals (6.10.02),
SPECIAL PROCESSES--DeBoer (6.17.05),
Fused Salt-Fluoride Volatility (6.17.11),
HCL-Alcohol (6.17.12),
Purex (6.17.17),
Zircex (6.17.24),
Zirflex (6.17.25).

19986 NAA-SR-3591
Atomics International Div., North American Aviation, Inc., Canoga Park, Calif.
REMOVAL OF IRRADIATED UO_2 FUEL FROM THE CLADDING BY CONTROLLED OXIDATION. L. A. Hanson. Aug. 1, 1959. 26p. Contract AT-11-1-GEN-8. $0.75(OTS).

A method using remote operations for removing irradiated UO_2 fuel from the zircaloy cladding has been investigated. The oxidation reaction of UO_2 to U_3O_8 accomplishes pellet and particle breakup by an increase in lattice dimension, and the oxidized sample falls from the cladding. Removal rate vs. temperature curves have been plotted for both air flow and oxygen flow. Optimum removal rate was obtained at 620 ± 10°C with oxygen and at 560 ± 10°C with air as the flow gas. The removal rate with oxygen is more than twice the rate obtained with air flow. This process indicates promise as a scaleup operation because of the simplicity of essential apparatus and of the techniques involved. (auth)

19983 DP-393
Du Pont de Nemours (E. I.) & Co. Savannah River Lab., Aiken, S. C.
REPROCESSING OF POWER REACTOR FUELS. Quarterly Progress Report No. 6 [for] January 1 to April 1, 1959. Edward S. Occhipinti, comp. June 1959. 11p. Contract AT(07-2)-1. $0.50(OTS).

Welds of 309SCb corrode at a rate much faster than that of the wrought metal in boiling HF-HNO_3 systems, although the weld and wrought metal corrode at about qual rates in HF alone. When a 309SCb dissolver is used for processing Detroit-Edison core elements, a satisfactory alternative to dissolving in HNO_3 containing a low concentration of HF is to remove the cladding of zirconium with dilute HF, and then to dissolve the core alloy (10% Mo-90% U) by addition of HNO_3 containing a small amount of $Al(NO_3)_3$ to complex free fluoride. (For preceding period see DP-338.) (auth)

4492
CONCENTRATED NITRIC AND DILUTE HYDROFLUORIC ACID MIXTURES IN DISSOLUTION OF ZIRCONIUM METAL. E. M. Vander Wall and E. M. Whitener (Phillips Petroleum Co., Idaho Falls, Idaho). Ind. Eng. Chem. 51, 51-4(1959) Jan.

In mixtures of nitric and hydrofluoric acids the rate of dissolution of zirconium metal is first-order with respect to the hydrofluoric acid. Varying the nitric acid concentration from 0 to 13M causes a small change in the initial rate of dissolution, which indicates that nitric acid is not necessary for dissolution. However, the presence of 13M nitric acid produces stable solutions which have a final total fluoride-zirconium molar ratio of 2. Rate constants are given for the initial rate of dissolution of zirconium at 40, 50, 60, and 115°C, and for the disappearance of hydrofluoric acid during dissolution at 40, 50, and 60°C in the region where the total fluoride-zirconium molar ratio is 6 or greater. Evi-

19984 IDO-14478
Phillips Petroleum Co. Atomic Energy Div., Idaho Falls, Idaho.
SALT-PHASE CHLORINATION OF REACTOR FUELS. I. DISSOLUTION OF ZIRCONIUM ALLOYS IN LEAD CHLORIDE. H. T. Hahn and E. M. Vander Wall. May 22, 1959. 23p. Contract AT(10-1)-205. $0.75 (OTS).

HEAD-END TREATMENTS 3.01

ZIRCONIUM – URANIUM FUEL ELEMENTS 3.01.12

Dissolution of zirconium-based fuel elements by a salt-phase chlorination process is proposed. Scoping experiments show that lead chloride rapidly dissolves zirconium, Zircaloy-2, and uranium-zirconium alloys; that zirconium tetrachloride readily volatizes from the melt; and that uranium loss by volatility can be reduced to a negligible amount. Salient advantages offered by the ARCO (Alloy Reguline Chloride Oxidation) process include early separation of matrix material simultaneously with dissolution, reduction in size of final uranium decontamination equipment, and compact waste storage. (auth)

DP-338
Du Pont de Nemours (E. I.) & Co. Savannah River Lab., Augusta, Ga.
REPROCESSING OF POWER REACTOR FUELS. Fourth Quarterly Progress Report [for] July 1 to October 1, 1958. Edward S. Occhipinti, comp. Feb. 1959. 12p. Contract AT(07-2)-1. $0.50(OTS).

Increased concentrations of nitric acid increased the dissolution rate of zirconium in hydrofluoric–nitric acid mixtures having concentrations of hydrofluoric acid less than approximately 1.0 molar. Simulated waste solutions, obtained from the processing of zirconium-clad fuel elements of uranium–molybdenum alloy, could be evaporated to one-half their original volume without the formation of undesirable amounts of precipitate. The dissolution rate of stainless steel by electrolysis in nitric acid was primarily a function of the current density; variations in the acidity, salt concentration, and temperature were ineffective over wide ranges. (For preceding period see DP-319.) (auth)

67 ORNL-2558
Oak Ridge National Lab., Tenn.
DECLADDING OF PWR BLANKET FUEL ELEMENTS WITH AQUEOUS AMMONIUM FLUORIDE SOLUTIONS. L. M. Ferris. Oct. 9, 1958, 20p. Contract W-7405-eng-26. $0.75(OTS).

A flowsheet is presented for decladding PWR blanket fuel elements with boiling 6 M NH_4F–1.0 M NH_4NO_3. After decladding, the pelleted UO_2 core is dissolved in 10 M HNO_3 to produce a solution suitable for recovery of uranium by solvent extraction. A 30-mil Zircaloy-2 jacket can be completely dissolved in about 2 hr with a uranium loss of less than 0.02%. The total uranium loss after decladding and core dissolution was less than 0.022%. All experiments were performed with unirradiated fuel specimens. Ammonium nitrate was added to the decladding solution to dissolve the tin in Zircaloy-2 and thus prevent excessively high uranium losses (~0.8%) to a nitric acid–insoluble residue comprised mainly of SnO_2. Another solvent for tin, cupric chloride, caused higher uranium losses (0.13%) to the dejacketing solution. (auth)

REPROCESSING OF POWER REACTOR FUELS. First Quarterly Progress Report to January 1, 1958. Woodfin E. Shuler and Thomas H. Siddall, III. Apr. 1958. 14p. (DP-283)

This is the first quarterly progress report on investigations of chemical processing methods for ZrU fuel systems. Progress on removing Zr jackets with HF and NH_4F; recovery of fissile material from decladding solutions; dissolving U–Mo alloy in HNO_3 and mixtures of HNO_3 and HF; stability of dissolver solutions; codissolving Zr and U–Mo alloy with mixtures of HNO_3 and HF; and methods for separating Zr and U before solvent extraction are summarized.

20988 DP-335
Du Pont de Nemours (E. I.) & Co. Savannah River Lab., Aiken, S. C.
PROCESSING OF FUEL ASSEMBLIES THAT CONTAIN ZIRCONIUM. Woodfin E. Shuler. Jan. 1959. 33p. Contract AT(07-2)-1. $1.00(OTS).

Fuel elements that are clad but not alloyed with zirconium can be declad with ammonium fluoride solution and the uranium core can then be dissolved in nitric acid. If zirconium is also present as an alloying element in the core, a satisfactory approach is the combined dissolution of the fuel in a fluoride-nitric acid solution. Aluminum ion must then be added to the solution to complex the fluoride and permit recovery of uranium by Purex-type solvent extraction. Alternatively, the zirconium and the fluoride can be separated from the dissolver solution by precipitation as barium fluozirconate. (auth)

1120 ORNL-2460
Oak Ridge National Lab., Tenn.
HYDROFLUORIC ACID DECLADDING OF ZIRCONIUM-CLAD POWER REACTOR FUEL ELEMENTS. W. E. Clark and A. H. Kibbey. Nov. 7, 1958. 17p. Contract W-7405-Eng-26. $0.75(OTS).

A detailed flowsheet is presented for the removal of Zircaloy-2 cladding from a UO_2 core (as in the Commonwealth Edison fuel) by dissolution in hydrofluoric acid to give a F/Zr ratio of 4.5/1 in the solution. The core is dissolved in 8 M HNO_3 for subsequent removal of fission products by solvent extraction. In tests on unirradiated prototypes, uranium loss to the fluoride solu-

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ZIRCONIUM – URANIUM FUEL ELEMENTS 3.01.12

tion was as little as 0.16%. Dissolution data on uranium metal and U-10% Mo alloy indicate that these cores can be declad similarly. U–Zr alloys cannot be so stripped while U–Nb is somewhat doubtful. It appears that pure zirconium cladding can be removed about as easily as Zircaloy-2. One container constructed of Carpenter 20 (niobium stabilized) stainless steel can probably be used for both dissolution steps. (auth)

563

DECLADDING OF PWR BLANKET FUEL ELEMENTS WITH AQUEOUS AMMONIUM FLUORIDE SOLUTIONS. L. M. Ferris. Oct. 9, 1958. 20p. (ORNL-2558)

A flowsheet is presented for decladding PWR blanket fuel elements with boiling 6 M NH_4F—1.0 M NH_4NO_3. After decladding, the pelleted UO_2 core is dissolved in 10 M HNO_3 to produce a solution suitable for recovery of U by solvent extraction. A 30 mil Zircaloy-2 jacket can be completely dissolved in about 2 hr with a U loss of less than 0.02%. The total U loss after decladding and core dissolution was less than 0.022%. All experiments were performed with unirradiated fuel specimens. NH_4NO_3 was added to the decladding solution to dissolve the Sn in Zircaloy-2 and thus prevent excessively high U losses (~0.8%) to a HNO_3—insoluble residue comprised mainly of SnO_2. Another dissolvent for Sn, $CuCl_2$, caused higher U losses (0.13%) to the dejacketing solution. 11 references.

2833 DP-318

Du Pont de Nemours (E. I.) & Co. Savannah River Lab., Augusta, Ga.

REPROCESSING OF POWER REACTOR FUELS. Second Quarterly Progress Report [for] January 1 to April 1, 1958. E. S. Occhipinti, comp. Sept. 1958. 8p. Contract AT(07-2)-1. $0.50(OTS).

A zirconium-clad alloy containing 90 wt. % uranium and 10 wt. % molybdenum can be dissolved in nitric acid containing low concentrations of fluoride ion. Uranium was extracted satisfactorily from a typical dissolver solution by a solvent of 10% tributyl phosphate in "Ultrasene," but further work will be required to extract plutonium satisfactorily. (auth)

PRECIPITATION OF ZIRCONIUM, NIOBIUM, AND RUTHENIUM FROM AQUEOUS SOLUTIONS. A. S. Wilson (to U. S. Atomic Energy Commission). U. S. Patent 2,847,278. Aug. 12, 1958.

An improvement on the "head end process" for decontaminating dissolver solutions of their Zr, Ni, and Ru values. The process consists in adding a water soluble symmetrical dialkyl ketone, e.g., acetone, before the formation of the manganese dioxide precipitate. The effect is that upon digestion, the ruthenium oxide does not volatilize, but is carried on the manganese dioxide precipitate.

2371 ORNL-2824

Oak Ridge National Lab., Tenn.

ELECTROLYTIC DISINTEGRATION OF ZIRCALOY-2 IN NITRIC ACID SOLUTIONS. Walter E. Clark and Sigfred Peterson. Nov. 27, 1959. 18p. Contract W-7405-eng-26. OTS.

Zircaloy-2 is anodically converted to scaly ZrO_2 at 60°C in 8 M HNO_3. About 0.5 mole of acid is consumed per faraday, and after saturation of the electrolyte with nitrogen oxides about 0.3 mole of gas is evolved per faraday. The nitric acid is reduced to hydrogen, NO, and NO_2, with hydrogen predominating if the cathode is Zircaloy and NO if the cathode is platinum. Corrosion specimens of HRT metals were exposed to the electrolysis conditions. From determinations of the decomposition potential of nitric acid it appears that a metal container for the electrolytic process can be protected from stray-current corrosion by holding it at a potential ~0.5 volt positive to a platinum cathode operating at a current density of 5 to 10 ma/cm^2. Practical laboratory experiments tended to confirm this conclusion. (auth)

12774

REPROCESSING URANIUM–ZIRCONIUM ALLOY REACTOR FUEL ELEMENTS. Presented at Nuclear Engineering and Science Conference, held at Chicago, March 17 to 21, 1958. Preprint 139, Session 37. C. B. Leek, R. B. Lemon, and F. K. Wrigley (Phillips Petroleum Co., Idaho Falls, Idaho). New York, American Institute of Chemical Engineers, 1958. 22p.

The process employed for the recovery of uranium from uranium–zirconium alloy reactor fuel elements at the Atomic Energy Commission's Idaho Chemical Processing Plant is described and the results of the initial processing and decontamination operations summarized. (auth)

HEAD-END TREATMENTS 3.01

ZIRCONIUM – URANIUM FUEL ELEMENTS 3.01.12

ZIRCONIUM ALLOY FUEL REPROCESSING. D. G. Reid, C. E. Stevenson, R. B. Lemon, and F. K. Wrigley. 20p. (A/CONF.15/P/526)

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

The fuel elements are dissolved in HF and the resulting solution adjusted with $Al(NO_3)_3$ to a composition suitable for solvent extraction in stainless steel equipment. Three solvent extraction cycles are used to separate the U from the Zr, fission products, and transuranic elements. U recovery efficiency is better than 99.5%, and decontamination factors greater than 10^6 are realized over the three extraction cycles. It is indicated that processing costs for 1% U–Zr fuel could be reduced to about $1.75/g of U for a unit capable of processing 7.5 kg U per day.

1293 IDO-14413

Phillips Petroleum Co. Atomic Energy Div., Idaho Falls, Idaho.

ANALYTICAL PROGRAM FOR PROCESSING ZIRCONIUM–URANIUM REACTOR FUEL ELEMENTS. James E. Rein and Ralph C. Shank. Aug. 15, 1957. 28p. Contract AT(10-1)-205. $1.00(OTS).

Fuel elements fabricated with zirconium as the alloying material are rapidly gaining in popularity. Recently, a high zirconium content, enriched uranium fuel was processed in the Idaho Chemical Processing Plant for recovery of the unfissioned uranium. The process was based on dissolution in hydrofluoric acid, oxidation of uranium(IV) to uranium(VI) with chromium(VI), complexation of the free fluoride with aluminum(III), and separation of the uranium by pulsed column extraction with tributylphosphate in kerosene. It is the purpose of this paper to describe the analytical program for this process. Because of the high burnup of uranium, the samples contained high concentrations of fission products which are chemically complex and interfere with conventional methods of analysis. The high activity level of the samples required use of a special remote handling facility. Methods are described for uranium, acidity, zirconium, aluminum, fluoride, and specific gravity in the dissolver and column feed solutions; for small amounts of uranium in the extraction column raffinates; and for tributylphosphate in the extractant. Typical reliability data are given for each of the described analytical methods. (auth)

STUDIES ON NON-AQUEOUS METHODS OF SEPARATION. S. Ruby. June 20, 1956. Decl. Mar. 1, 1957. 14p. (SEP-214)

The most promising approaches to non-aqueous fuel processing found were hydriding as a means of physical reconstitution of a damaged fuel (no fission-product separation effected) and hydrochlorination as a pre-solvent extraction treatment for U–Zr fuels.

ALTERNATE PROCESSING METHODS FOR ZIRCONIUM AND STAINLESS STEEL CONTAINING FUELS. R. E. Blanco. May 1, 1957. p. 251-261 of SYMPOSIUM ON THE REPROCESSING OF IRRADIATED FUELS HELD AT BRUSSELS, BELGIUM, MAY 20-5, 1957. 1165p. (TID-7534(Bks. 1-3))

The objective of the development program for alternate processing methods is to develop universal methods which could convert all fuels of a given type, as for example Zr or stainless steel, to a nitrate form suitable for purification by solvent extraction. A number of methods are being surveyed for this purpose. These programs are in the laboratory or small engineering scale at present and have not been tested on highly irradiated fuel samples. Consequently the fate of Pu is unknown. These processes must be tested on highly irradiated fuels to determine their over-all relative efficiencies. 13 references.

PRESENT DISSOLUTION METHODS FOR ZIRCONIUM AND STAINLESS STEEL. C. M. Slansky. May 1, 1957. p.244-50 of SYMPOSIUM ON THE REPROCESSING OF IRRADIATED FUELS HELD AT BRUSSELS, BELGIUM, MAY 20-5, 1957. 1165p. (TID-7534(Bks. 1-3))

U recovery processes are described for nuclear fuels alloyed and clad with Zr and with type 304 and 347 stainless steels. The Zr process utilizes HF dissolution, HNO_3 oxidation and $Al(NO_3)_3$ complexing and salting prior to TBP extraction. The 18-8 Ni–Cr stainless steels are dissolved in boiling 6M H_2SO_4, oxidized with HNO_3, adjusted for salting strength and extracted with TBP.

5922 IDO-14372(Del.)

Phillips Petroleum Co. Atomic Energy Div., Idaho Falls, Idaho.

EVALUATION OF ICPP FOR DETROIT-EDISON COMPANY FBR II FUEL PROCESSING. H. Schneider. Oct. 18, 1954. Decl. with deletions Feb. 25, 1957. 22p. Contract AT(10-1)-205. (CPP-54-97(Del.)). $4.80(ph OTS); $2.70(mf OTS)

The processing of a 2% Zr–U alloy type fuel is feasible. Recovery of core and blanket can be accomplished at rates compatible with reactor discharge rates with only minor process and equipment modifications and additions. Cost data are summarized. (F.S.)

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9638 NAA-SR-214
North American Aviation, Inc., Downey, Calif.
THE SEPARATION OF URANIUM FROM ZR–U FUEL BY FLUORINATION METHODS. W. Gardner. Dec. 29, 1952. Decl. Mar. 2, 1957. 18p. Contract AT-11-1-GEN-8. $0.25(OTS).

Two fluorination processes are reported for the separation of U from U–Zr fuel material (96 wt%). The first is a two-step fluorination in which the fuel alloy is dissolved in very concentrated aqueous HF, the product fluorides are dried, and then the UF_4 is converted to UF_6 with liquid BrF_3. The second method is a direct reaction of solid U–Zr alloy at 800°C with elemental F to produce ZrF_4 and volatile UF_6. A preliminary investigation of corrosion problems is reported for both processes. (auth)

12663 BNL-3335
Brookhaven National Lab., Upton, N. Y.
DISSOLUTION OF ZIRCONIUM-TYPE FUEL ELEMENTS IN SULFURIC ACID CATALYSED WITH HF. J. J. Reilly, W. H. Regan, R. H. Wiswall, and L. P. Hatch. [1957]. 19p. Contract [AT-30-2-gen-16].

Some experiments relating the rate of dissolution of Zircaloy-2 in H_2SO_4 to temperature, HF concentration, and H_2SO_4 concentration are described. (T.R.H.)

RECOVERY OF IRRADIATED Th AND U

ADSORPTION 3.02.01

16025
EXTRACTION OF METALS FROM AQUEOUS SOLUTION. N. F. Kember and U. K. Atomic Energy Authority. British Patent 803,264. Nuclear Eng. 4, 284(1959) June.

Thorium, uranium, zirconium, and hafnium are extracted by an adsorption method based on the observation that these metals are strongly adsorbed from aqueous mineral acid solution by phosphorylated cellulose. The aqueous solution containing the metals should have a concentration at least 10-N with respect to mineral acid.

55 CF-58-9-69
Oak Ridge National Lab., Tenn.
STATUS REPORT ON HF AND UF_6 ADSORPTION EXPERIMENT. Mack Tyner. Sept. 2, 1958. 16p. Contract [W-7405-eng-26]. $3.30(ph OTS); $2.40(mf OTS).

A study of the adsorption of HF and UF_6 on NaF pellets in the presence and absence of nitrogen diluent was made. The adsorption reaction was studied in an adiabatic bed by means of thermocouples placed at known heights. Equations were written for the numerical calculation of compositions and adsorption rates as functions of height and time in the bed. Data from nine experiments on HF is available for numerical analysis. (auth)

12542 CEA-tr-X-125
SÉPARATION DE L'URANIUM PAR EXTRACTION AU MOYEN D'UNE COLONNE ET ANALYSE QUANTITATIVE PAR PHOTOMÉTRIE. (Separation of Uranium by Extraction in a Column and Quantitative Analysis by Photometry.) T. Hara. Translated into French by E. Hara from Nippon Kagaku Zasshi 78, 337-40(1957). 10p.

A method for the extraction of small quantities of uranium is described. It consists in the absorption of the solution containing the uranium by silica. The silica is placed in a separatory funnel containing glass fiber and synthetic silica. Ether is used as uranium eluant. The addition of sodium acetate to the uranium solution improves the reproducibility of the method. The presence of thorium, zirconium, and phosphoric ion interfers with the uranium extraction, and methods of preventing the interference are given. (J.S.R.)

SEPARATION OF U AND Pu FROM Zr, Nb, Ru, AND Cs ON A COLUMN OF MANGANESE DIOXIDE. D. Cvjeticanin and N. Cvjeticanin. Apr. 11, 1958. 10p. (JENER-54)

The adsorption of U, Pu, Zr, Nb, Ru, Sr–Y, and Cs was examined to provide a good separation of U and Pu from the fission products mentioned. It was established that it is quantitatively possible to wash UO_2^{2+} and PuO_2^{2+} from a column of MnO_2 with 0.1 N HNO_3. Under the same conditions, 99.2% of Zr^{95}, Nb^{95}, Ru^{106}, and Cs^{137} was adsorbed.

RECOVERY OF IRRADIATED TH AND U 3.02

ADSORPTION 3.02.01

9754
THE DEVELOPMENT AND PROPERTIES OF AN ADSORBENT FOR URANIUM. Leslie A. McClaine, Paul Noble, Jr., and Edward P. Bullwinkel (Arthur D. Little, Inc., San Francisco). J. Phys. Chem. 62, 299-30(1958) Mar.

An adsorbent for uranium from sulfuric acid solutions by formation of a uranyl chelate compound on the surface of a modified porous char was developed. The capacity for uranium is limited by the size of the chelate molecule formed, and the pore size distribution of the adsorbent. (J.R.D.)

6361
REMOVAL OF URANIUM FROM ORGANIC LIQUIDS. S. P. Vavalides (to U. S. Atomic Energy Commission). U. S. Patent 2,901,314. Aug. 25, 1959.

A process is described for recovering small quantities of uranium from organic liquids such as hydrocarbon oils, halogen-substituted hydrocarbons, and alcohols. The organic liquid is contacted with a comminuted alkaline earth hydroxide, calcium hydroxide particularly, and the resulting uranium-bearing solid is separated from the liquid by filtration. Uranium may then be recovered from the solid by means of dissolution in nitric acid and conventional extraction with an organic solvent such as tributyl phosphate.

6435 A/CONF.15/P/326
FIXATION DU BROME ET DE L'HEXAFLUORURE D'URANIUM SUR LES CARBONES. (Fixation of Bromine and of Uranium Hexafluoride on Carbon). J. Maire (Societe "Le Carbone-Lorraine," France). 14p.

The fixation of bromine on pitch coke, paraffin coke, petroleum, coke, and various blacks was studied. The sorption curves were determined as a function of the bromine partial pressure. The results obtained with petroleum coke and pitch coke are analogous to those obtained with graphite. The paraffin cokes and blacks give isotherms sharply different from those of other carbons. In the study of fixation of uranium hexafluoride on carbon, it was found that the hexafluoride is adsorbed by graphites and graphitized carbons and gives a lamellar compound. The stability of this compound was determined. Uranium hexafluoride can be removed from a nitrogen stream by passage over graphite, and the hexafluoride is purified of traces of HF. (J.S.R.)

2503
ADSORPTION METHOD FOR SEPARATING THORIUM VALUES FROM URANIUM VALUES. G. E. Boyd, E. R. Russell, and J. Schubert (to U. S. Atomic Energy Commission). U. S. Patent 2,898,185. Aug. 4, 1959.

An improved ion exchange method is described for recovery of uranium and thorium values as separate functions from an aqueous acidic solution containing less than 10^{-3} M thorium ions and between 0.1 and 1 M uranyl ions. The solution is passed through a bed of cation exchange resin in the acid form to adsorb all the thorium ions and a portion of the uranyl ions. The uranium is eluted by means of aqueous 0.1 to 0.4 M sulfuric acid. The thorium may then be stripped from the resin by elution with aqueous 0.5 M oxalic acid.

10787 BMI-JDS-196
Battelle Memorial Inst., Columbus, Ohio.
ADSORPTION OF URANIUM FROM PHOSPHATE SOLUTIONS BY ACTIVATED CARBON. Iver Igelsrud and E. F. Stephan. June 30, 1949. Decl. Mar. 12, 1956. 45p. Contract W-38-094-eng-27. $0.30(OTS).

The work done on U adsorption from phosphate solutions from the time the initial experiments were made in May, 1948, to June 1949, is described. During this time, a total of fifty-six carbons, all but four of which were activated, have been tried under a variety of conditions on phosphate solutions ranging in U content from 15 to over 300 mg./l. and over a range of acidities up to over 40% of free H_3PO_4. The initial results, obtained on superphosphate solutions with a pH of 2.7, indicated nearly quantitative, over 90%, adsorption on a minus 200-mesh C. Efforts to confirm this with duplicates of this C and with many other carbons have been fruitless. There are some indications that, by increasing the ratio of C to U, the total adsorption might be increased from superphosphate solutions. This has not

2322 BMI-JDS-187
Battelle Memorial Inst., Columbus, Ohio.
QUARTERLY REPORT. SECTION I. URANIUM FROM PHOSPHATE ROCK. Iver Igelsrud and James F. Shea. SECTION II. FUNDAMENTAL INVESTIGATION OF THE SORPTION OF URANIUM FROM SOLUTIONS. J. C. Goodrich and R. Belcher. Mar. 15, 1949. Decl. Mar. 7, 1956. 60p. Contract W-38-094-eng-27. $9.30(ph OTS); $3.60(mf OTS).

RECOVERY OF IRRADIATED TH AND U 3.02

ADSORPTION 3.02.01

6363 A/CONF.15/P/1388
L'EMPLOI DES RADIOELEMENTS DANS L'ETUDE DES COEFFICIENTS DE DIFFUSION DES ELECTROLYTES DANS LES GELS. (The Use of Radioelements in a Study of the Diffusion Coefficients of Electrolytes in the Gels.) G. Cetini and F. Ricca (Univ. of Turin). 12p.

A report is presented on the results obtained in the experimental determination of the diffusion coefficients of phosphates in a gelatin gel. The measurements were carried out in a chemically homogeneous medium to rule out the perturbation caused by the presence of two distinct phases, which, as a rule, is a feature of the work on diffusion in gels. The experimental method adopted affords a determination of the values of the diffusion coefficients at various points in the gel and a means of following the development of the phenomenon as a function of time. (auth)

CALCINATION 3.02.02

See also, under PROCESS CHEMISTRY AND ENGINEERING,

SPECIAL PROCESSES:

Thorex--ThO_2 Production (6.17.22.07)

315
CONTINUOUS CALCINATION EQUIPMENT FOR CONVERTING UNH TO UO_3. R. G. Geier. May 1, 1957. p.286-95 of SYMPOSIUM ON THE REPROCESSING OF IRRADIATED FUELS HELD AT BRUSSELS, BELGIUM, MAY 20-5, 1957. 1165p. (TID-7534(Bks. 1-3); HW-49652A)

The continuous conversion of UNH to UO_3 in an agitated trough calciner has been demonstrated. This paper contains a description of a pilot plant unit. The range of operating conditions tested and their influence on UO_3 quality and pilot plant equipment operability are discussed.

9675 HW-26531
Hanford Works, Richland, Wash.
PRODUCTION OF UO_3 BY CALCINATION OF URANYL PEROXIDE. R. L. Moore and R. A. Watts, Jr. Dec. 10, 1952. Decl. Feb. 28, 1957. 3p. Contract [W-31-109-Eng-52]. $1.80(ph OTS); $1.80(mf OTS).

1494
ROASTING TO REMOVE FLUORIDES FROM AMMONIUM DIURANATE. E. R. Johnson, E. O. Rutenkroger, H. M. Beers, and A. B. Kreuzmann (National Lead Co. of Ohio, Fernald). Chem. Eng. Progr. 52, 474-6(1956) Nov.

Fluoride contamination in $(NH_4)_2U_2O_7$ formed in a U recovery process can be volatilized by calcination above 1,250°F. As a result of tests described, it is concluded that this calcination can be carried out more effectively in a fluidized bed reactor than in either a muffle furnace or a raked-tray-type roaster. (auth)

3052
PROCESSES OF PRODUCING URANIUM TRIOXIDE. C. E. Larson (to U. S. Atomic Energy Commission). U. S. Patent 2,723,181. November 8, 1955.

Uranium(VI) oxide, that may be readily converted to UCl_4 by reacting with CCl_4, can be prepared by controlled calcination of UO_4 at a temperature of 200 to 325°C, the UO_4 having been thoroughly washed with an aqueous solution of NH_3 after precipitation by H_2O_2 from an aqueous solution of a uranyl salt. (auth)

CHELATION 3.02.03

7497 GAT-186
Goodyear Atomic Corp., Portsmouth, Ohio.
PURIFICATION AND DETERMINATION OF URANIUM IN IMPURE SOLUTIONS USING VERSENE. O. A. Vita, C. F. Trivisonno, and F. S. Voss. July 6, 1956. 12p. Contract AT-(33-2)-1. $0.15(OTS).

The experimental results and the results obtained from production samples indicate that the Versene method can be used to quantitatively separate and determine U from solutions containing interfering cations. Versene is used to chelate U and all interfering impurities such as Fe, Ni, Al, Zn, Cu, SiO_2, Ag, Co, sulfate, chloride, phosphate, and fluoride. The U is selectively precipitated as uranyl ammonium phosphate using ammonium dibasic phosphate. The precipitate is digested and centrifuged. The supernatant liquid is decanted and the precipitate is dissolved in concentrated HNO_3. Finally, the U content is determined colorimetrically by the basic peroxide method. (auth)

RECOVERY OF IRRADIATED TH AND U 3.02

CHELATION 3.02.03

5263

SOME QUANTITATIVE REMARKS ON EXTRACTION EQUILIBRIA. III. M. Oosting (Analytical Research Inst., T.N.O., Rijswijk, Netherlands). Anal. Chim. Acta 21, 505-10(1959) Dec. (In English)

The solubility products of Al, Cu, Fe Ni, Th, and U chelates are determined. The conditions for separation of Cu/UO_2, Fe/UO_2, and Fe/Al by extraction are calculated and shown to be correct. The separation of Al/UO_2 and UO_2/Th by extraction with oxine-chloroform was proved impossible under any conditions. (auth)

16205

HYDROLYSIS AND OLATION OF Th(IV) CHELATES OF POLYAMINOPOLYCARBOXYLIC ACIDS. Raymond F. Bogucki and Arthur E. Martell (Clark Univ., Worcester, Mass.). J. Am. Chem. Soc. 80, 4170-4(1958) Aug. 20.

The ethylenediaminetetraacetate and the 1,2-diaminocyclohexanetetraacetate chelates of Th(IV) were found to form binuclear diolate chelates in aqueous solution. Through measurement of the influence of concentration on the potentiometric titration curves, the acid dissociation constants of the normal chelate compounds and the dimerization constants of the hydrolysis products were determined. The Th(IV) chelate of heptadentate or octadentate diethylenetriaminepentaacetic acid forms a completely coördinated, monohydroxo chelate which shows no tendency to form polynuclear substances. (auth)

10102

THE INFLUENCE OF THE STRUCTURE OF CHELATING AGENTS ON THE COMPLEXITY AND DISTRIBUTION CONSTANTS OF THORIUM COMPLEXES. David Dyrssen (Research Inst. of National Defence, Sundbyberg, Sweden). Rec. trav. chim. 75, 753-8(1956) June.

A table of coordination compounds, the value of the acid dissociation complexes pKa and mean complexity constants are presented. It has been observed that 5-membered chelate rings are more stable than 6 or 4-membered ones. The stability of the chelate complexes of H^+ and Th^{4+} are discussed. (W.L.H.)

STUDIES ON THORIUM-AMMONIUM AURINTRICARBOXYLATE CHELATE: PART I. PHYSICO-CHEMICAL INVESTIGATION OF THE COMPOSITION, STRUCTURE AND STABILITY. Anil K. Mukherji and Arun K. Dey (Univ. of Allahabad, India). J. Sci. Ind. Research (India) B17, 312-17(1958) Aug.

Ammonium aurintricarboxylate forms a colored chelate complex with tetravalent thorium.

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)

1903 TID-10002

California. Univ., Berkeley. [Radiation Lab.] THE CHELATE PROCESS I. J. R. Thomas and H. W. Crandall. Dec. 12, 1946. Decl. 14, 1957. 58p. Contract W-7405-eng-48b. $0.40(OTS).

The chelate process for the extraction and decontamination of plutonium has been investigated. The process is designed as an alternate process for the Hanford type operation. The basic chemical factors have been studied and two flowsheets have been developed and tested for recovery of plutonium. An over-all yield of plutonium in excess of 99% is reported for one flowsheet using benzene as the organic solvent and 98.5% when toluene was substituted for benzene. The general factors involved in establishing all details of the flowsheets are thoroughly discussed and decontamination studies are indicated to complete the study in the present equipment. (auth)

CHLORINATION 3.02.04

See also, under PROCESS CHEMISTRY AND ENGINEERING,

SPECIAL PROCESSES--Darex (6.17.04),

Zircex (6.17.24).

RECOVERY OF IRRADIATED TH AND U 3.02

CHLORINATION 3.02.04

12510 ✓
PROCESS FOR PRODUCING URANIUM TETRACHLORIDE. (to United Kingdom Atomic Energy Authority.) British Patent 812,792. Apr. 29, 1959.

An apparatus for producing UCl_4 by reacting CCl_4 with UO_2 is described. Powdered UO_2 is introduced at the high end of an inclined, rotating reaction tube, and CCl_4 vapor enters at the low end. A heating muff maintains reaction vessel temperature at 450 to 475°C. The UCl_4 emerges at the lower end. (T.R.H.)

PROCESS FOR PRODUCING URANIUM TETRACHLORIDE. (To United Kingdom Atomic Energy Authority.) British Patent 812,121. Apr. 22, 1959.

A method and laboratory apparatus for making UCl_4 from UO_2 and CCl_4 are described. Hot CCl_4 vapor (500°C) is passed over UO_2 powder at 450°C to produce UCl_4, CO, CO_2, and $COCl_2$. Unreacted CCl_4 and $COCl_2$ are recycled until completion of the reaction. The apparatus consists of a tubular furnace with tubing for heating the CCl_4 and also for the reaction. (T.R.H.)

PROCESS FOR RECOVERING URANIUM. G. E. MacWood, Coleman D. Wilder, and D. Altman (to U. S. Atomic Energy Commission). U. S. Patent 2,879,130. Mar. 24, 1959.

A process is described for recovering uranium from deposits on stainless steel liner surfaces of calutrons. The deposit is removed from the stainless steel surface by washing with aqueous nitric acid. The solution obtained containing uranium, chromium, nickel, copper, and iron is treated with excess of ammonium hydroxide to precipitate the uranium, iron, and chromium and convert the nickel and copper to soluble ammonia complexions. The precipitated material is removed, dried, and treated with carbon tetrachloride at an elevated temperature of about 500 to 600°C to form a vapor mixture of UCl_4, UCl_5, $FeCl_3$, and $CrCl_4$. The UCl_4 is separated from this vapor mixture by selective fractional condensation at a temperature of about 300 to 400°C.

6359
APPARATUS FOR CHARGING A RECEPTACLE WITH A DENSE SUBLIMATE FORM OF URANIUM CHLORIDE. P. H. Davidson (to U. S. Atomic Energy Commission). U. S. Patent 2,900,237. Aug. 18, 1959.

An apparatus for filling a tubular storage receptacle with a dense massive form of uranium chloride is described. The apparatus includes an evacuated housing divided into a vaporizing chamber and a portion adapted to receive the receptacle. A nozzle conducts vaporized uranium chloride from the chamber to the interior of the receptacle. The nozzle is withdrawable to progressively deposit the uranium chloride under controlled conditions to produce a dense sublimate which fills the receptacle.

4380 CF-59-4-9
Oak Ridge National Lab., Tenn.
RECOVERY OF URANIUM FROM RESIDUES PRODUCED IN HYDROCHLORINATION OF NUCLEAR FUEL ALLOYS. T. A. Gens. Apr. 10, 1959. 9p. Contract [W-7405-eng-26]. OTS.

Chlorination at 550 to 600°C of Zircex residues with carbon tetrachloride resulted in greater than 99.99% recovery of the uranium. Both the hydrochlorination residue and the solids which remain after leaching the hydrochlorination residue with nitric acid were chlorinated with similar results. By this technique, loss of 1 to 6% of the uranium to the nitric acid insoluble residue, which occurs in simple hydrochlorination and dissolution of STR fuel, is avoided. Other advantages include reduced corrosion and the elimination of possible explosions between U-Zr alloys and nitric acid. A modified flowsheet is proposed. (auth)

PROCESSES OF CHLORINATION OF URANIUM OXIDES. S. Rosenfeld (to U. S. Atomic Energy Commission). U. S. Patent 2,852,337. Sept. 16, 1958.

An improvement is described in the process for making UCl_4 from uranium oxide and carbon tetrachloride. In that process, oxides of uranium are contacted with carbon tetrachloride vapor at an elevated temperature. It has been found that the reaction product and yield are improved if the uranium oxide charge is disposed in flat trays in the reaction zone, to a depth of not more than ½ centimeter.

5043 ✓
METHOD OF PRODUCING URANIUM TETRACHLORIDE. H. R. McCombie and E. L. Wagner (to U. S. Atomic Energy Commission). U. S. Patent 2,735,746, February 21, 1956.

Uranium tetrachloride has been prepared by introducing finely divided UO_2 into an elongated reaction zone having a temperature gradient between the input and discharge points from 440 to 475°C continually advancing the UO_2 through the reaction zone, introducing a counter current of gaseous CCl_4 within the advancing mass of UO_2 so as to produce finely divided crystalline UCl_4 and means of continuously withdrawing the UCl_4 from the chamber. Carbon tetrachloride may be recovered from the gaseous reaction products and recycled. (auth)

RECOVERY OF IRRADIATED TH AND U 3.02

CHLORINATION 3.02.04

12402 SEP-214
Sylvania Electric Products Inc. Atomic Energy Div., Bayside, N. Y.
STUDIES ON NON-AQUEOUS METHODS OF SEPARATION. S. Ruby. June 20, 1956. Decl. Mar. 1, 1957. 14p. Contract AT-30-1-GEN-366. $0.30(OTS).

The most promising approaches to non-aqueous fuel processing found were hydriding as a means of physical reconstitution of a damaged fuel (no fission-product separation effected) and hydrochlorination as a pre-solvent extraction treatment for U–Zr fuels. (auth)

5047
CYCLIC PROCESSES OF PRODUCING UCl_6. J. M. Carter (to U. S. Atomic Energy Commission). U. S. Patent 2,737,439, March 6, 1956.

Uranium hexachloride has been produced from UCl_6 by converting the UCl_6 into UCl_4 and UCl_4, removing the UCl_6, converting the remaining UCl_4 into a uranium oxychloride by reaction with a dry air stream, converting the uranium oxychloride into UCl_5, and then repeating the cycle employing the UCl_5 thus obtained. (auth)

5033
MANUFACTURE OF URANIUM TETRACHLORIDE. S. Rosenfeld (to U. S. Atomic Energy Commission). U. S. Patent 2,733,124, January 31, 1956.

Uranium oxides such as UO_2 and UO_3 can be converted to UCl_4, by contacting a mixture of CCl_4 and $CHCl_3$ substantially in the ratio of 3:1 with UO_2, UO_3, or a mixture of the same while maintaining a temperature in the range of 400 to 500°C. (auth)

5041
PROCESS FOR PREPARATION OF CHLORIDES OF URANIUM. Ernest C. Evers, et al (to U. S. Atomic Energy Commission). U. S. Patent 2,734,795, February 14, 1956.

Uranium chlorides can be prepared by reacting an U oxide such as UO_3, with liquid CCl_4 in the presence of initially added extraneous UCl_5 at an elevated temperature in the range from 80 to 200°C. The reaction mixture is agitated during the reaction period to prevent caking. Uranium pentachloride is the principal product, which may readily be converted to CCl_4 by bleeding off the evolved by-product gases during the reaction period. (auth)

3055
PREPARATION OF URANIUM HEXACHLORIDE. Ross E. Van Dyke and Ernest Charles Evers (to U. S. Atomic Energy Commission). U. S. Patent 2,725,279. November 29, 1955.

Uranium hexachloride has been produced by treating UCl_5, UCl_4, UO_3, or mixtures of two or more of these reactants with a mixture of liquid CCl_4 and Cl_2. (auth)

3054
MANUFACTURE OF URANIUM TETRACHLORIDE. M. J. Polissar (to U. S. Atomic Energy Commission). U. S. Patent 2,725,278. November 29, 1955.

A process is outlined for manufacturing UCl_4 of high purity and of crystal size and structure in a vacuum apparatus comprising producing CCl_4 vapor from a liquid source in a vaporizing chamber above a reaction chamber, introducing CCl_4 vapor by gravity into the reaction chamber at 400 to 500° C. to react with UO_2 or UO_3 to product UCl_4, and returning unused CCl_4 for recycling. (auth)

3537 AECD-3937
Tennessee Eastman Corp., Oak Ridge, Tenn.
DIRECT CONVERSION OF TF_6 [UF_6] TO TCl_4 [UCl_4]. Vincent P. Calkins. Sept. 25, 1945. Decl. Dec. 21, 1955. 6p. Contract W-7401-eng-23. (C-0.350.4). $1.80(ph OTS); $1.80(mf OTS).

Results indicate that the conversion of UF_6 to UCl_4 proceeds through a two-step process involving UF_4. Uranium (IV) chloride can be prepared by the direct reaction of UF_4 with $AlCl_3$ or BCl_3. (C.W.H.)

6412 A/CONF.15/P/2195
ISSLEDOVANIE REAKTSII KHLORIROVANIYA DVUOKISI URANA I DVUOKISI PLUTONIYA CHETYREKHKHLORISTYM UGLERODOM. (Investigations of Uranium Dioxide and Plutonium Dioxide Chlorination with Carbon Tetrachloride.) I. V. Budaev and A. N. Vol'skii (U.S.S.R.). 48p.

The results of investigations of uranium dioxide and plutonium dioxide chlorination with carbon tetrachloride are described. A number of experiments were made using carbon tetrachloride to study the mechanism and rate of uranium dioxide and plutonium dioxide chlorination at various temperatures. The thermodynamic analysis of the most important reactions taking place during uranium dioxide chlorination with carbon tetrachloride is discussed. Standard free energy and equilibrium constant equations were deduced as a function of temperature. The solids and gases obtained in the chlorination process were subjected to thorough qualitative and quantitative analyses. The investigations show that the composition of the resulting gases is considerably changed, depending on the temperature and duration of chlorination. The compositions of the gases and solid compounds point to the mechanism of this process and to its dependence on temperature. The differences in the kinetics of chlorination in carbon tetrachloride flow and in a closed space were established. An account of the study of the plutonium dioxide chlorination with carbon tetrachloride is also given.

RECOVERY OF IRRADIATED TH AND U 3.02

COMPLEX FORMATION 3.02.05

See also, this section,

ION EXCHANGE (3.02.13),
PRECIPITATION (3.02.15),
SOLVENT EXTRACTION (3.02.17).

THORIUM 3.02.05.01

1483 ✓
THORIUM FLUORIDE COMPLEX FORMATION REACTIONS IN SOLUTION. I. V. Tananaev and Lu Chao-ta. Zhur. Neorg. Khim. 4, 2122-6(1959) Sept.

The composition and stability of thorium fluoride ions in solutions were determined by solubility and optical methods. The solubility of CaF_2 in $Th(NO_3)_4$ solutions and in $Th(NO_3)_4$ + NaF mixtures was investigated. It was found that ThF^{3+} and ThF^{2+} ions are formed in the solution. $K_{ThF^{3+}} = 1.2 \times 10^{-6}$ (solubility method) and 1.3×10^{-6} (optical method); $K_{ThF_2^{2+}} = 1.8 \times 10^{-3}$ (solubility method). (R.V.J.)

THE THORIUM-APANS COMPLEX. A Spectrophotometric Study. A. R. Palmer (Australian Atomic Energy Commission Research Labs., Sydney). Anal. Chim. Acta 19, 458-61(1958) Nov.

The compound formed from thorium and Apans has been studied by spectrophotometric methods and shown to contain thorium and reagent in the ratio 1:2. The apparent stability constant of this complex has been measured as $1.4 \pm 0.1 \cdot 10^{10}$. (auth)

COMPLEX COMPOUNDS OF THORIUM OXALATE WITH AMMONIUM OXALATE. K. N. N. Kurup, K. V. Nair, and S. S. Moosath (Univ. of Kerala, Trivandrum, India). Proc. Indian Acad. Sci. A47, 373-8(1958) June.

The system thorium oxalate-ammonium oxalate-water was investigated by physico-chemical methods. A new procedure is adopted in preparing the solution for these studies by the mono-variation method. Data are presented to prove the existence of two complexes in solution from measurements of electrical resistance, pH, and relative viscosity. (auth)

5580
STUDIES ON THE EXTRACTION OF METAL COMPLEXES. XXI. THE COMPLEX FORMATION OF THORIUM WITH TROPOLONE. David Dyrssen (Research Inst. of National Defence, Sundbyberg, Sweden). Acta Chem. Scand. 9, No. 10, 1567-74(1955).

Uranium(VI) and Th^{4+} can be separated from trivalent ions (e.g. La^{3+} and Y^{3+}) by extraction with chloroform solutions of tropolone at pH = 2. Data on the formation constants of thorium tropolonate are included. (C.W.H.)

URANIUM 3.02.05.02

1484 ✓
INVESTIGATION OF URANYL COMPLEX FORMATION IN OXALATE SOLUTION BY THE SOLUBILITY METHOD. A. I. Moskvin and F. A. Zakhorova. Zhur. Neorg. Khim. 4, 2151-60(1959) Sept. (In Russian)

Processes of uranyl complex formation with oxalate ions take place with hydrogen ion concentrations of < 2 mole/l. The solubility of $UO_2(C_2O_4) \cdot 3\ H_2O$ is equal to 2.2×10^{-9} in chloric acid solutions and 3.0×10^{-8} in nitric acid solutions Complexes of $UO_2(C_2O_4)$ and $UO_2(C_2O_4)_2^{2-}$ with concentration instability constants equal to $K_n^2 = (1.7 \pm 0.3) \times 10^{-7}$ and $K_n^3 = (1.0 \pm 0.3) \times 10^{-12}$ and acetolysis constants equal to 2.5×10^{-2} and 2.1×10^{-2}, respectively, were found. (R.V.J.)

22171
RECOVERY OF URANIUM BY CYCLOALKYLDITHIOCARBAMATE COMPLEXING. O. K. Neville (to U. S. Atomic Energy Commission). U. S. Patent 2,892,855. June 30, 1959.

The separation of uranium-233 from an aqueous nitric acid solution of neutron irradiated thorium by selectively complexing the uranium is described. The separation is carried out by contacting the thorium solution with a non-aromatic organic dithiocarbamate selected from the group which consists of alkali and alkaline earth cycloalkyldithiocarbamates and recovering the resulting uranyl cycloalkyldithiocarbamate complex by organic solvent extraction such as with methyl ethyl ketone. The complexed uranium may be stripped from the separated organic phase by scrubbing with one normal nitric acid solution.

RECOVERY OF IRRADIATED TH AND U 3.02

COMPLEX FORMATION 3.02.05

URANIUM 3.02.05.02

2506

RECOVERY OF URANIUM BY AROMATIC DITHIOCARBAMATE COMPLEXING. O. K. Neville (to U. S. Atomic Energy Commission). U. S. Patent 2,899,451. Aug. 11, 1959.

A selective complexing organic solvent extraction process is presented for the separation of uranium values from an aqueous nitric acid solution of neutron irradiated thorium. The process comprises contacting the solution with an organic aromatic dithiocarbamate and recovering the resulting uranodithiocarbamate complex with an organic solvent such as ethyl acetate.

22165

ENHANCING PRECIPITATIONS BY APPLYING SOLUBLE COMPLEX FLUORINE-CONTAINING REAGENTS. G. W. Stahl (to U. S. Atomic Energy Commission). U. S. Patent 2,886,408. May 12, 1959.

The use of fluo-reagents in processes for the recovery of transuranic elements such as plutonium for the purpose of complexing zirconium, columbium, and uranium so that they can be removed from solutions in which they are contained is described. In the carrier precipitation process for the separation of plutonium from uranium and fission products including zirconium and columbium, the precipitated bismuth phosphate carries some zirconium, columbium, and uranium impurities. According to the invention such impurities can be complexed and removed by dissolving the contaminated carrier precipitate in 10M nitric acid, followed by addition of fluosilicic acid to about 1M, diluting the solution to about 1M in nitric acid, and then adding phosphoric acid to re-precipitate bismuth phosphate carrying plutonium.

DETERMINATION OF URANIUM BY CUPFERRON WITH COMPLEXONE III AS THE MASKING AGENT. A. K. Majumdar and J. B. Ray Chowdhury (Jadavpur Univ., Calcutta). Anal. Chem. Acta 19, 576-7(1958) Dec.

At a pH between 4 and 6, hexavalent uranium is quantitatively precipitated by cupferron in the presence of complexone III and thus can be separated from Bi^{3+}, Cu^{2+}, Fe^{3+}, Cr^{3+}, Al^{3+}, Th^{4+}, Cd^{2+}, Pb^{2+}, Zn^{2+}, Mn^{2+}, Ni^{2+}, Co^{2+}, alkali metals, alkaline earth metals, rare earth metals, VO_3^-, MoO_4^{2-}, and WO_4^{2-}. (auth)

COORDINATION COMPOUND-SOLVENT EXTRACTION PROCESS FOR URANIUM RECOVERY. W. H. Reas (to U. S. Atomic Energy Commission). U. S. Patent 2,877,092. Mar. 10, 1959.

A method is presented for the separation of uranium from aqueous solutions containing a uranyl salt and thorium. The separation is effected by adding to such solutions an organic complexing agent, and then contacting the solution with an organic solvent in which the organic complexing agent is soluble. By use of the proper complexing agent in the proper concentration, uranium will be complexed and subsequently removed in the organic solvent phase, while the thorium remains in the aqueous phase. Mentioned as suitable organic complexing agents are antipyrine, bromoantipyrine, and pyramidon.

UF_6–3NaF COMPLEX FORMATION AND DECOMPOSITION. G. I. Cathers, M. R. Bennett, and R. L. Jolley (Oak Ridge National Lab., Tenn.). Ind. Eng. Chem. 50, 1709-10(1958) Nov.

The reversible absorption of gaseous uranium hexafluoride on solid sodium fluoride is convenient for trapping, handling, and storing this material in chemical processing. The UF_6 is absorbed by the formation of $UF_6 \cdot 3NaF$, a yellow hygroscopic solid complex. Absorption and desorption involve an equilibrium among UF_6, NaF, and $UF_6 \cdot 3NaF$. The vapor pressure of UF_6 over the solid complex conforms to the equation $\log p = 10.88 - 5.09 \times 30^3/T$ from 80° to 360°C. A competing reaction, which interferes with UF_6 desorption, involves decomposition of the U(VI)-NaF complex to a white U(V)-NaF complex. The rate of the decomposition from 250° to 350°C., is expressed as $\log k = 6.09 - 5.22 \times 10^3/T$. By maintaining an excess of fluorine and sweeping UF_6 from the complex as liberated, this reaction is reversed to allow quantitative desorption of uranium. (auth)

6369

RECOVERY OF URANIUM BY SECONDARY XANTHATE COMPLEXING. O. K. Neville (to U. S. Atomic Energy Commission). U. S. Patent 2,902,503. Sept. 1, 1959.

A method is described for separating and recovering uranium values contained in an acidic aqueous solution together with thorium or protactinium values. In accordance with the invention, the acidic solution containing uranium in the uranyl form is contacted with an organic xanthate. The xanthate forms a urano-xanthate complex but is substantially non-reactive with thorium and protactinium. The urano-xanthate complex is recovered by organic solvent extraction.

RECOVERY OF IRRADIATED TH AND U 3.02

COMPLEX FORMATION 3.02.05

URANIUM 3.02.05.02

4442 NP-7088
Polish Academy of Sciences. Inst. of Nuclear Research, Warsaw.
TRENNUNG DES URANS VON DEN BEGLEITMETALLEN VERMITTELS DER CARBONATMETHODE. (Separation of Uranium from Associated Metals by the Carbonate Method.) L. Wodkiewicz. Oct. 1958. 5p.

A method of separating U from associated metals is described. It is based on the complex-forming ability of U with alkali metal carbonates and ammonium carbonate. By this method Fe, Al, Ti, Cr, V, Zn, Mn, and alkaline earths can be separated from U. After separation the U is determined colorimetrically by the peroxide method. The method is suitable for U ores containing from 1 to 0.01% U. (tr-auth)

13864 AECU-3787
Michigan. Univ., Ann Arbor.
POLAROGRAPHIC REDUCTION OF URANIUM(VI) UNDER COMPLEXING AND NONCOMPLEXING CONDITIONS—NATURE OF THE SULFATE COMPLEX. Report No. 34. Philip J. Elving and Alan F. Krivis. July 5, 1958. 19p. Project No. 8. Contract AT(11-1)-70. $3.30(ph OTS); $2.40(mf OTS).

The polarographic reduction of U(VI) to U(V) in acid solution is very sensitive to both type and concentration of anion present. Consequently, the reduction was studied using perchlorate as a noncomplexing anion and sulfate as a complexing anion. In $HClO_4$ solution, increasing the perchlorate concentration shifted $E_{1/2}$ to more positive potentials, which seem to correspond to junction potential effects. Increasing either $HClO_4$ or perchlorate concentrations increased the limiting current slightly, which can be attributed to a higher rate of disproportionation of U(V); other factors, e.g., viscosity of the solution, tend to counteract the effect of the disproportionation. In sulfate media, UO_2^+ is not strongly complexed, the association constant for the U(V)-sulfate complex being 0.15, if UO_2SO_4 is the most stable uranyl sulfate complex present. The effect of acid on the stability of the uranyl sulfate complex confirmed the existence of the complex as an uncharged species. Limiting currents were pseudo diffusion-controlled, e.g., increasing the solution viscosity by increasing the electrolyte content decreased the current; this is due to the maximum disproportionation rate of U(V) having been reached at even the lowest sulfuric acid level investigated; increasing the anion concentration consequently slowed down diffusion. (auth)

COMPLEX FORMATION BETWEEN U^{IV} AND DI-(2 BUTYLOCTYL) + PYROPHOSPHATE. M. Zangen (Atomic Energy Commission, Israel). Bull. Research Council Israel Sect. A, 7A, 153-64(1958) June.

The extraction of uranium(IV) from phosphoric acid by di-(2-butyloctyl)-pyrophosphate (BOPPA), diluted with organic solvents, has been investigated under various conditions in order to establish the parameters of the process. BOPPA in kerosene (SBP 60-90°C) at concentrations ranging from 2.5 to 640 millimoles/l was employed as solvent. The aqueous phase consisted of U^{IV} (0.03-8 millimoles/l) in phosphoric acid. Various volume ratios (2.5-40) of the aqueous and the organic phase were tested in a batch extraction process. It was found that no single simple equilibrium equation governing the extraction process could be set up: the simultaneous formation of at least three complexes—$U(BOPP)_2$ $U(BOPP)(H_2PO_4)_2$ and $U_2(BOPP)(H_2PO_4)_6$—has to be postulated in order to explain the experimental data satisfactorily. Approximate values have been determined for the equilibrium constants governing formation and extraction of the above complexes. (auth)

14407
ISOTOPE FRACTIONATION PROCESS. G. H. Clewett and DeW. A. Lee (to U. S. Atomic Energy Commission). U. S. Patent 2,835,687. May 20, 1958.

A new method is described for isotopic enrichment of uranium. It has been found that when an aqueous acidic solution of ionic tetravalent uranium is contacted with chelate complexed tetravalent uranium, the U^{238} preferentially concentrates in the complexed phase while U^{235} concentrates in the ionic phase. The effect is enhanced when the chelate compound is water insoluble and is dissolved in a water-immiscible organic solvent. Cupferron is one of a number of suitable complexing agents, and chloroform is a suitable organic solvent.

13719
HEMICOLLOIDAL MINERALS. II. Jacques Duclaux and Charlotte Cohn (Institut de Biologie physico-chimique, Paris). Bull. soc. chim. France No. 5, 657-60(1958) May. (In French)

A method is proposed for the preparation of hemicolloidal minerals. It depends on partition between water

RECOVERY OF IRRADIATED TH AND U 3.02

COMPLEX FORMATION 3.02.05

URANIUM 3.02.05.02

and an organic liquid. The colloidal part remains wholly in the aqueous layer while the small molecules distribute between the two. The hydroxides of Th and Zr, prepared by this method or one previously described, show to a great degree the characteristic properties of hemicolloids; high stability, osmotic pressure elevation, and most important of all an exceptionally high solubility. Hemicolloidal solutions with the same characteristics can form just as well in organic solvents such as amyl alcohol. (auth)

9274

EXTRACTION OF URANYL α-NITROSO-β-NAPHTHOLATE AND SEPARATION OF URANIUM FROM VANADIUM AND IRON. I. P. Alimarin and Yu. A. Zolotov (Lomonosov Moscow State Univ.). Zhur. Analit Khim. 12, 176-80(1957) Mar.-Apr. (In Russian)

The quantitative extraction of U^{6+} from aqueous solutions as α-nitroso-β naphtholate was carried out with organic solvents immiscible with water. The separation of U from V and Fe by means of uranium extraction as α-nitroso-β-naphthalate with isoamyl alcohol from neutral solutions after addition of complexone III is described. (tr-auth)

9262

SEPARATION OF URANIUM FROM RARE EARTHS AND SOME OTHER ELEMENTS IN SULPHURIC ACID MEDIUM, USING ANION EXCHANGE RESIN. Milenko V. Šušić. Bull. Inst. Nuclear Sci. "Boris Kidrich" (Belgrade) 7, 35-8(1957) Mar.

The ability of U to form complexes with dilute H_2SO_4 solutions affords a method for the separation of U from rare earths and some other elements by adsorption on anion-exchange resins. From very dilute H_2SO_4 solutions (>1 N H_2SO_4) U is quantitatively adsorbed on the resin, but the other elements pass through column without being adsorbed as they do not form sulfate complexes. (auth)

PROCESS FOR RECOVERY OF URANIUM FROM AQUEOUS SOLUTIONS. J. W. Gates, Jr. and L. J. Andrews (to U. S. Atomic Energy Commission). U. S. Patent 2,780,518. February 5, 1957.

A method is described for the recovery of U from aqueous solutions in which the U is accompanied by F^- The method is particularly useful in connection with organic solvent extraction of U, and is effective in neutral or acidic solutions. It consists of incorporating Al^{3+} in the solution, in order to complex the F^- present. The Al concentration should preferably be sufficient to provide an atomic ratio of Al to F^-, of one to one. (auth)

SPECTROPHOTOMETRIC ESTIMATION OF URANIUM WITH TIRON. B. Sarma and C. P. Savariar (National Chemical Lab. of India, Poona). J. Sci. Ind. Research (India) 16B, 80-2(1957) Feb.

A spectrophotometric method for the estimation of semi-micro quantities of uranium up to 400 ppm has been described. Uranium forms a highly soluble brown complex with tiron (disodium catechol disulphonate). The color of the complex deepens with rise in the pH. The optical density of the complex shows a continuous increase with pH, but an absorption peak is observed at 373 mμ in the range 340 to 750 mμ when the solution is kept at pH 2 to 3.5. Absorbencies were measured at 373 mμ and pH 3. The complex obeys Beer's law perfectly under specified conditions. (auth)

~~6315~~

PREPARATION OF PURE U_3O_8 FROM CRUDE SODIUM DIURNATE BY INTERMEDIATE COMPLEX FORMATION. N. S. Krishnaprasad and V. V. Dadape (Atomic Energy Establishment, Bombay, India). Proc. Indian Acad. Sci. 45, 20-3(1957) Jan.

Preliminary studies of the separation of U from Th. rare earths, and Fe by complexing with KF or NH_4F are reported. (T.R.H.)

10100

STUDIES ON THE FORMATION OF COMPOSITE COMPLEXES BY MEANS OF AN EXTRACTIVE TECHNIQUE. Jan Rydberg (Research Inst. of National Defense, Sunbyberg, Sweden). Rec. trav. chim. 75, 737-42(1956) June.

The method described has been applied to a number of systems. Equations have been applied on the distribution of U^{4+} and Th between several different organic solvents and water in the presence of acetylacetone. Formation constants have been calculated for the complexes. (W.L.H.)

RECOVERY OF IRRADIATED TH AND U 3.02

COMPLEX FORMATION 3.02.05

URANIUM 3.02.05.02

COMPLEXES OF THE 4d- AND 5d-GROUPS. IV. ELECTRON TRANSFER BANDS WITH SPECIAL APPLICATION TO M. DELEPINE'S COMPLEXES AND A TRANSITION FROM IRIDIUM(III) TO PYRIDINE, WITH SOME REMARKS ABOUT INTERMEDIATE COUPLING IN HALIDE COMPLEXES AND THE URANYL ION. Chr. Klixbüll Jorgensen (Univ. of Denmark, Copenhagen). Acta Chem. Scand. 11, No. 1, 166-78(1957).

8088

RE-EXTRACTION OF URANIUM FROM ORGANIC SOLVENTS. F. T. Hagemann, L. I. Katzin, and N. N. Hellman (to U. S. Atomic Energy Commission). U. S. Patent No. 2,743,157, Apr. 24, 1956.

The recovery of UO_2^{2+} values from organic solvent solutions by the use of an aqueous solution of a "complexing" agent is described. The complexing agent may be sulfate, phosphate, oxalate, or fluoride ions. The value of the process lies in the reduction of aqueous recovery solution volume through the addition of the "complexing agent." (auth)

2685 ORNL-1989

Oak Ridge National Lab., Tenn.

THE CHEMISTRY OF URANIUM(VI) ORTHOPHOSPHATE SOLUTIONS. PART IV. A FURTHER SPECTROPHOTOMETRIC INVESTIGATION OF URANYL PHOSPHATE COMPLEX FORMATION IN PERCHLORIC ACID SOLUTION. C. F. Baes, Jr. Oct. 21, 1955. 36p. Contract W-7405-eng-26. $6.30(ph OTS); $3.00(mf OTS).

The spectrophotometric investigation of U^{6+}-orthophosphate complex formation in 1M and 0.1M $HClO_4$ has been continued. Detailed analysis of the results indicates that 1:1 and also 2:1 phosphate-U complexes are formed. The acidity dependence of the formation quotients $K_1 = C_1/C_U C_P$ and $K_2 = C_1/C_U C_P^2$ (in which C_1 and C_2 are the total concentrations of 1:1 and 2:1 complexes, C_U and C_P are the concentrations of uncomplexed U^{6+} and phosphoric acid) can be accounted for in terms of the complex species $UO_2H_2PO_4^+$, $UO_2H_3PO_4^{2+}$, $UO_2(H_2PO_4)_2$, and $UO_2(H_2PO_4)(H_3PO_4)^+$. (auth)

2318 BMI-JDS-163

Battelle Memorial Inst., Columbus, Ohio.

PRECIPITANTS AND COMPLEXING AGENTS FOR URANIUM. Quarterly Report. A. E. Bearse, G. W. Kinzer, G. A. Lutz, R. D. Morin, and R. H. Poirier. Jan. 15, 1949. Decl. Mar. 2, 1956. 48p. Contract AT-30-1-gen-202. $0.35(OTS).

Studies were continued on the precipitation of UO_2^{+2} from H_2SO_4 solutions by organic compounds of P and As. The use of long-chain amines as precipitants was also studied. Sulphonic acid-type resins gave almost quantitative removal of UO_2^{+2} from dilute acid solutions. (C.W.H.)

COSTS 3.02.06

See also, under SURVEYS (GENERAL), COSTS (7.02).

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.)

4574 ORNL-1949

Oak Ridge National Lab., Tenn.

COST OF URANIUM RECOVERY BY THE AMINE EXTRACTION PROCESS. B. B. Klima, H. M. McLeod, Jr., A. D. Ryon, and R. R. Wiethaup—K. O. Johnson, ed. Sept. 8, 1955. Decl. Jan. 10, 1956. 39p. Contract W-7405-eng-26. $0.30(OTS).

The estimated costs of uranium recovery from clarified H_2SO_4 leach liquor by the Amine Extraction Process are outlined. At a production rate of 200 tons of ore/day the estimated costs are $0.67/lb U_3O_8 with no secondary strip and $0.72/lb U_3O_8 with 100% secondary strip. These costs include direct operating costs and amortization but no overhead; they also include the processing steps associated with clarification, solvent extraction, U precipitation, calcination, and product packaging. (C.W.H.)

RECOVERY OF IRRADIATED TH AND U 3.02

COSTS 3.02.06

EFFECT OF FUEL DILUENT ON REPROCESSING COSTS. Archie M. Larson, Jr. (Internuclear Co., Clayton, Mo.) Nucleonics 14, No. 10, 80 & 82(1956)Oct.
There are only two major factors affecting the reprocessing costs for fully enriched fuel elements. The most important of these is the degree by which the enriched U fuel is diluted with alloying and cladding material. The other factor is the type of diluent material used.

5922 IDO-14372(Del.)
Phillips Petroleum Co. Atomic Energy Div., Idaho Falls, Idaho.
EVALUATION OF ICPP FOR DETROIT-EDISON COMPANY FBR II FUEL PROCESSING. H. Schneider. Oct. 18, 1954. Decl. with deletions Feb. 25, 1957. 22p. Contract AT(10-1)-205. (CPP-54-97(Del.)). $4.80(ph OTS); $2.70(mf OTS)
The processing of a 2% Zr–U alloy type fuel is feasible. Recovery of core and blanket can be accomplished at rates compatible with reactor discharge rates with only minor process and equipment modifications and additions. Cost data are summarized. (F.S.)

DISTILLATION 3.02.07

See also, this section,
FLUIDIZED BEDS (3.02.09),
and,
under PROCESS CHEMISTRY AND ENGINEERING,
SPECIAL PROCESSES--Darex (6.17.04),
Fused Salt-Fluoride Volatility (6.17.11),
Hermex (6.17.13),
Metallex (6.17.14).

7572 ORNL-1738
Oak Ridge National Lab., Tenn.
URANIUM HEXAFLUORIDE DISTILLATION: CORRELATIONS FOR ENGINEERING DESIGN. J. T. Long. Aug. 26, 1954. Decl. May 31, 1956. 58p. Contract W-7405-eng-26. $9.30(ph OTS); $3.60(mf OTS).
With the reservations and modifications enumerated, methods of calculation now in the literature may be safely applied in the design of equipment for the fractionation of mixtures of UF_6 and other dense substances. Equipment characteristics treated are flooding, pressure drop, hold-up, and stage efficiency. A design manual for UF_6 fractionating columns is presented, and sample calculations are shown. (auth)

3057
CONCENTRATION OF URANIUM ISOTOPES BY MOLECULAR DISTILLATION OF URANIUM POLY ALKOXIDES. A. K. Brewer, S. L. Madorsky and T. I. Taylor (to U. S. Atomic Energy Commission). U. S. Patent 2,727,000. December 13, 1955.
A method of partial separation concentrating isotopes of U^{238} and U^{235} in a molecular still is reported. Either uranium pentaethoxide or uranium penta normal propoxide may be used although the former is preferred as having greater thermal stability. The distillation is performed under a vacuum of between 10^{-7} and 10^{-3} mm of mercury. For uranium pentaethoxide a temperature of between 85 and 200° C., U^{235} and U^{234} are separated by molecular countercurrent distillation from U^{238}, the latter being the enhanced residue. Uranium penta normal propoxide requires a temperature of between 100 and 210° C., again concentrating U^{238} in the residue and U^{235} with U^{234} in the distillate. (auth)

RECOVERY OF METALS SALTS FROM MIXTURES. W. V. Wirth and D. X. Klein (to U. S. Atomic Energy Commission). U. S. Patent 2,782,092. February 19, 1957.
A method is presented for the separation of UF_6 from certain volatile halogenated hydrocarbons. The separation is accomplished by hydrolyzing the UF_6 to UO_2F_2 in an aqueous medium and then separating the water insoluble fluoro hydrocarbons therefrom by decanting or distillation. Since conducting the hydrolysis in an acid solution results in the formation of corrosive HF it is preferred to carry out the hydrolysis an alkaline or neutral medium. (auth)

RECOVERY OF IRRADIATED TH AND U 3.02

DISTILLATION 3.02.07

6353 ✓
SEPARATION OF FISSION PRODUCTS FROM IRRADIATED URANIUM. (to United Kingdom Atomic Energy Authority). British Patent 817,861. Aug. 6, 1959.

The processing of irradiated U fuel by distillation is described. Heating to 1250°C distills off most fission products and rapid heating to 1400°C removes Pu. (T.R.H.)

9680 MCW-191
Mallinckrodt Chemical Works, St. Louis.
PRODUCTION OF UO_3 FROM SOLID $UO_2(NO_3)_2 \cdot XH_2O$. A. M. Kirby. June 15, 1949. Decl. Feb. 26, 1957. 8p. Contract [W-14-108-Eng-8]. $0.25(OTS).

Two methods are outlined for producing UO_3. The first consisted of heating molten hexafluoride to about 225°C so that a solid product was obtained upon cooling to about 150°C and then decomposing at this temperature in vacuo. The second method consisted of dehydrating $UO_2(NO_3)_2 \cdot 3H_2O$ at 110 to 120°C with a stream of CO_2 containing HNO_3 vapors and then decomposing at about 160°C in vacuo. The behavior of the material in both cases was much the same. The desired product was finally obtained by prolonged heating at 160°C in vacuo using the second method. A diagram of the apparatus for the decomposition of solid $UO_2(NO_3)_2 \cdot XH_2O$ is shown. (D.E.B.)

ELECTROCHEMICAL 3.02.08

See also, this section,

FUSED SALTS AND LIQUID METALS (3.02.11),

and,

under PROCESS CHEMISTRY AND ENGINEERING,

ELECTROCHEMISTRY (6.07),

SPECIAL PROCESSES--Excer (6.17.08),
Flurex (6.17.10).

8411 Y-1285
Union Carbide Nuclear Co. Y-12 Plant, Oak Ridge, Tenn.
REDUCTION CELL WITH VERTICAL ROTATING MERCURY CATHODE. F. A. Schimmel. Jan. 22, 1960. 21p. Contract W-7405-eng-26. OTS.

An electrolytic cell was developed for the reduction of uranyl sulfate in a single-stage continuous operation. (auth)

ANODIC TREATMENT OF URANIUM. Morris Kolodney (to U. S. Atomic Energy Commission). U. S. Patent 2,872,387. Feb. 3, 1959.

A method is presented for effecting electrolytic dissolution of a metallic uranium article at a uniform rate. The uranium is made the anode in an aqueous phosphoric acid solution containing nitrate ions furnished by either ammonium nitrate, lithium nitrate, sodium nitrate, or potassium nitrate. A stainless steel cathode is employed and electrolysis carried out at a current density of about 0.1 to 1 ampere per square inch.

7308 ✓ A/CONF.15/P/1414
OBTAINING URANIUM(IV) FLUORIDES BY ELECTROLYSIS WITH A MERCURY CATHODE. T. Batuecas Rodriguez, M. L. Aceña, F. de la Cruz, and Fernández Cellini (Junta de Energía Nuclear, Madrid). 45p.

The possibilities of reduction of the acid solutions of the uranyl ion with a mercury cathode are tested, along with a study of the most interesting variables of the electrochemical process. The optimal conditions for the separation of the uranium as a fluoride are determined, and a comparative study is presented of the products obtained by precipitation and joint reduction or a precipitation made after the reduction and separate from it, as well as of the factors which affect the yields and composition in each and every case. The degree of hydration is checked, in the products thus obtained, choosing experimental conditions applicable to the preparation of anhydrous fluoride. The method which has been developed applies to concentrated solutions used for the acid digestion of uranium minerals, and the respective yields, compositions, and purities in the products so separated are indicated. (auth)

175 KAPL-1761
Knolls Atomic Power Lab., Schenectady, N. Y.
ELECTROWINNING OF URANIUM FROM ITS OXIDES. I. LABORATORY STUDIES. L. W. Niedrach and B. E. Dearing. Apr. 30, 1957. 54p. Contract W-31-109-Eng-52. $1.50(OTS).

The background work is described for a new continuous process for the production of U by electrolytic reduction of U oxides in fused salt electrolytes. Unlike past electrolytic processes for U production, the present one is operated at temperatures which are above the melting point of U. The effect of current, salt bath composition, and other variables on efficiency and cell operation are discussed. The advantages of the new process are outlined, and the areas requiring additional development work are indicated. (auth)

RECOVERY OF IRRADIATED TH AND U 3.02

ELECTROCHEMICAL 3.02.08

9769

THE PREPARATION OF URANIUM METAL BY THE ELECTROLYTIC REDUCTION OF ITS OXIDES. L. W. Niedrach and B. E. Dearing (General Electric Co., Schenectady, N. Y.). J. Electrochem. Soc. 105, 353-8 (1958) June.

The background work is described for a new continuous process for the production of uranium by electrolytic reduction of its oxides in fused salt electrolytes. Unlike past electrolytic processes for uranium production, the present one is operated at temperatures which are above the melting point of the metal. The effect of current, salt bath composition, and other variables on efficiency and cell operation are discussed. An electrolysis bath containing 20 mole % UF_4 diluted with a 50 : 50 mole % mixture of BaF_2 and MgF_2 was found to be satisfactory. With this bath anode current densities as high as 3.6 amp/cm^2 are feasible without encountering "anode effect." Any of the oxides of uranium can be used as the feed, but UO_3 appears to be preferable. The advantages of the new process are outlined, and the areas requiring additional development work are indicated. (auth)

6367

PURIFICATION OF URANIUM FUELS. L. W. Niedrach and A. C. Glamm (to U. S. Atomic Energy Commission). U. S. Patent 2,902,415. Sept. 1, 1959.

An electrolytic process of refining or decontaminating uranium is presented. The impure uranium is made the anode of an electrolytic cell. The molten salt electrolyte of this cell comprises a uranium halide such as UF_4 or UCl_3 and an alkaline earth metal halide such as $CaCl_2$, BaF_2, or $BaCl_2$. The cathode of the cell is a metal such as Mn, Cr, Co, Fe, or Ni which forms a low melting eutectic with U. The cell is operated at a temperature below the melting point of U. In operation the electrodeposited uranium becomes alloyed with the metal of the cathode, and the low melting alloy thus formed drips from the cathode.

14404

ELECTROCHEMICAL DECONTAMINATION AND RECOVERY OF URANIUM VALUES. J. A. McLaren and J. H. Goode (to U. S. Atomic Energy Commission). U. S. Patent 2,834,722. May 13, 1958.

An electrochemical process is described for separating uranium from fission products. The method comprises subjecting the mass of uranium to anodic dissolution in an electrolytic cell containing aqueous alkali bicarbonate solution as its electrolyte, thereby promoting a settling from the solution of a solid sludge from about the electrodes and separating the resulting electrolyte solution containing the anodically dissolved uranium from the sludge which contains the rare earth fission products.

6374

RECOVERY OF VALUABLE MATERIAL FROM GRAPHITE BODIES. L. W. Fromm, Jr. (to U. S. Atomic Energy Commission). U. S. Patent 2,903,402. Sept. 8, 1959.

An electrolytic process for recovering uranium from a graphite fuel element is described. The uranium-containing graphite body is disposed as the anode of a cell containing a nitric acid electrolyte and a 5 amp/cm^2 current passed to induce a progressive disintegration of the graphite body. The dissolved uranium is quickly and easily separated from the resulting graphite particles by simple mechanical means, such as centrifugation, filtration, and decontamination.

PROCESS OF RECOVERING URANIUM FROM SOLUTION. Robert Q. Boyer and Scott B. Kilner (to U. S. Atomic Energy Commission). U. S. Patent 2,781,303. February 12, 1957.

An electrolytic process is described for recovering U from calutron wash solutions in which it is accompanied by ionized Ni, Fe, Cr and Cu. The process employs a cell with an anode compartment, a cathode compartment, and a mercury cathode. An aqueous acid solution is the catholyte and the U containing solution is the anolyte. Upon electrolysis the U and other metallic ions migrate to the catholyte. The impurities are reduced and amalgamated with the mercury cathode while most of the U remains dissolved. The catholyte is continuously removed and the U recovered therefrom, and the depleted anolyte is continuously withdrawn, concentrated and recycled. (auth)

6078

METAL PRODUCTION BY ELECTROLYSIS. R. A. Noland and C. Marzano (to U. S. Atomic Energy Commission). U. S. Patent 2,739,111, Mar. 20, 1956.

A method of purifying a carbon-containing actinide metal, such as U or Th by electrolysis is presented. The impure actinide metal is made the anode in an electrolytic cell having a cathode of Ta or Mo and containing a fused electrolyte, at a temperature in the range of 385 to 425°C., consisting of 5 to 50% by weight of the actinide tetrafluoride and from 95 to 50% by weight of the LiCl–KCl eutectic mixture. The actinide metal so obtained is substantially spectroscopically pure as to C content, and may be readily freed of small amounts of Li and K by melting in crucible of the corresponding oxide. (auth)

RECOVERY OF IRRADIATED TH AND U 3.02

ELECTROCHEMICAL 3.02.08

Electrorefining for Removing Fission Products from Uranium Fuels, L.W.NIEDRACH, A.C.GLAMM. Indus. and Eng. Chem. 48, 6, June 1956, p 977-81.

Possibility of reducing cost of reprocessing fuel from nuclear reactors by employing process for decontaminating uranium from fission products; feasibility of proposed electrolytic process demonstrated.

1838 SRO-14
Horizons, Inc., Cleveland.
PRODUCTION OF ELECTROLYTIC THORIUM CELL FEED BY A WET CHEMICAL METHOD. Process Report. Charles E. Fisher. Mar. 18, 1955. Decl. Mar. 18, 1957. 13p. Contract AT(30-1)-1335. (HZ-11; DPWR-148). $3.30(ph OTS); $2.40(mf OTS).

In order to produce a substantially oxide-free chloride, a specialized technique, based on the chemical characteristics of the ammonium thorium chloride complexes, has been found optimum. The procedure involves the formation of a solution of thorium chloride and ammonium chloride, evaporation of the solution to form a hydrated ammonium thorium chloride complex, dehydration of the complex by low-temperature drying, and ignition of the complex, after inter-mixture with an alkali chloride, to remove ammonium chloride and to form a thorium alkali chloride mixture for electrolysis. (L.T.W.)

7968 KAPL-1518
Knolls Atomic Power Lab., Schenectady, N. Y.
URANIUM METAL PREPARATION BY ELECTROLYTIC REDUCTION OF OXIDES. L. W. Niedrach and B. E. Dearing. Mar. 12, 1956. Changed from OFFICIAL USE ONLY Dec. 31, 1956. 37p. Contract W-31-109-Eng-52. $0.40(OTS).

A continuous process is outlined for the production of U metal by electrolytic reduction of uranium oxides. The same process is recommended for recovering scrap U from high temperature processing operations on reactor fuel. Laboratory work that has been done towards developing such a process is briefly reviewed. The advantages of such a process are outlined, and the areas requiring additional development work are indicated. Additional development work on the process is recommended. (auth)

DEPOSIT OF UO_2 ON Al BY ELECTROLYSIS. Lydie Koch (Centre d'Etudes nucléaires, Saclay, France). J. Nuclear Energy 2, 110-11(1955) Dec. (In French)

A method is described for the preparation of UO_2 layers strongly adherent to large areas of aluminum sheet. The thickness of the deposits may reach 3 mg/cm^2. (auth)

10236
ELECTROLYTIC PRODUCTION OF URANIUM TETRAFLUORIDE. E. Lofthouse (to U. S. Atomic Energy Commission). U. S. Patent 2,687,995. Aug. 31, 1954.

This patent relates to electrolytic methods for the production of uranium tetrafluoride. According to the present invention a process for the production of uranium tetrafluoride comprises submitting to electrolysis an aqueous solution of uranyl fluoride containing free hydrofluoric acid. Advantageously the aqueous solution of uranyl fluoride is obtained by dissolving uranium hexafluoride in water. On electrolysis, the uranyl ions are reduced to uranous ions at the cathode and immediately combine with the fluoride ions in solution to form the insoluble uranium tetrafluoride which is precipitated.

12419 CF-56-6-158
Oak Ridge National Lab., Tenn.
PURIFICATION OF SCRAP URANIUM BY ELECTROLYSIS. I. R. Higgins. June 29, 1956. Decl. Mar. 19, 1957. 9p. Contract [W-7405-eng-26]. $1.80(ph OTS); $1.80 (mf OTS).

Uranyl fluoride-hydrofluoric acid solution may be electrolytically reduced to hydrated UF_4 with a current efficiency of 90% for a 99% reduction using a Hg electrode. The usual production rate is about 2 a/in^2. or about 2½ lb U/hr-ft^2 of cathode. (L.T.W.)

2992 RMO-2517
Rohm and Haas Co. Research Labs., Philadelphia.
SUMMARY REPORT ON ELECTROLYTIC MEMBRANE CELL WORK AT CLIMAX URANIUM COMPANY [FOR] PERIOD JANUARY 5, 1953-FEBRUARY 12, 1953. Charles T. Dickert. Feb. 26, 1953. Decl. Sept. 23, 1955. 24p. Contract AT(49-1)-535.

Laboratory work on electrolytic precipitation of U from leach liquors has continued. Data are presented on current-density investigation, initial pH, phosphate content, high U and V head liquors, cathode plating of U and V, electrode performance, and membrane performance. Detailed data on experimental observations are included in the appendix. (J.E.D.)

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ELECTROCHEMICAL 3.02.08

2683 KAPL-1154
Knolls Atomic Power Lab., Schenectady, N. Y.
ELECTROREFINING OF URANIUM—A NEW APPROACH.
L. W. Niedrach and A. C. Glamm. Aug. 13, 1954. ~~Decl.~~ Oct. 4, 1955. 40p. Contract W-31-109-Eng-52.

A new approach is presented for the electrorefining of reactor fuels and its corollary, an improved electrowinning process to obtain such fuels from their compounds. Electrorefining is compared with other high-temperature methods of processing reactor fuels. The potential advantage of the method is indicated since it offers a means of separating the fuel material from both more active (chemically) and more noble fission products. Various approaches to electrorefining are then considered and the need for obtaining a molten metal product is stressed. The desirability of a molten metal anode is also discussed. The use of alloying agents to obtain molten electrodes is then considered, and the feasibility of using the alloying agent as the cathode is suggested. This procedure results in a cathode from which the molten alloy drips—a "dripping alloy cathode". This type of cathode offers several advantages over more conventional cathodes. The feasibility of producing molten alloys with a dripping cathode is demonstrated with a uranium–nickel system. Good current efficiencies are demonstrated. and the effect of the common variables of temperature, bath composition, current, etc., are illustrated. The feasibility of prolonged electrolysis with such a system is also indicated. (auth)

10845 K-681
Carbide and Carbon Chemical Div. K-25 Plant, Oak Ridge, Tenn.
ELECTROCHEMICAL PREPARATION OF URANIUM TETRAFLUORIDE. PART II. HIGH TEMPERATURE CELL.
R. W. Anderson, A. L. Allen, and E. W. Powell. Dec. 22, 1950. ~~Decl. Feb. 26, 1957.~~ 47p. Contract W-7405-eng-26. $0.45(OTS).

A continuous high-temperature process was developed for the preparation of UF_4 from UO_3. The process includes: preparation of a UO_2F_2–HF electrolyte by dissolving UO_3 in HF, precipitation of UF_4 hydrate upon reduction of UO_2^{2+} at a Hg cathode, thickening of the UF_4 slurry, filtration, washing of filter cake, and dehydration of UF_4 hydrate in a N_2 stream at elevated temperatures. (W.L.H.)

THE ELECTROLYTIC DISSOLUTION OF METALLIC URANIUM. J. A. McLaren, W. D. Cline, H. S. Clinton, J. J. Finley, J. H. Goode, and J. A. Westbrook. Apr. 17, 1950. ~~Decl. Apr. 10, 1957.~~ 11p. (K-587)

Experiments have shown that U metal can be dissolved in a wide variety of acids, salts, and alkalies by anodic oxidation in an electrolytic cell. U concentrations of 50 to 80 g/l have been obtained with $NaHCO_3$, tartaric acid, or HNO_3 as electrolytes.

8710
THE RECOVERY OF URANIUM FROM LEACH LIQUORS BY ELECTROLYTIC PERMSELECTIVE MEMBRANE PROCESSES. Robert Kunin (Rohm & Haas Co., Philadelphia). pp. 35-44 "Progress in Nuclear Energy. Series III. Process Chemistry. Volume 2."

The techniques of using electrolytic permselective membranes are described. These techniques are combined with electrolytic reduction of uranyl ion and applied to the preparation of solid uranium concentrates from leach liquors encountered in uranium extraction operations. By electrolytic migration through an anion-permeable membrane the pH of an acidic uranium solution may be raised while the uranyl ion is reduced to uranium(IV) at the cathode. A readily filtrable precipitate of hydrated uranium dioxide or uranium(IV) phosphate is produced and the acid recovered in a form suitable for additional leaching operations. An alkaline carbonate leaching process can be combined with electrolytic precipitation of the uranium concentrate and reagent recovery, by use of a cation-permeable membrane. (auth)

13696 K-587
Carbide and Carbon Chemicals Div., K-25 Plant, Oak Ridge, Tenn.
THE ELECTROLYTIC DISSOLUTION OF METALLIC URANIUM. J. A. McLaren, W. D. Cline, H. S. Clinton, J. J. Finley, J. H. Goode, and J. A. Westbrook. Apr. 17, 1950. Decl. Apr. 10, 1957. 11p. Contract W-7405-Eng-26. $1.80(ph OTS); $1.80(mf OTS).

Experiments have shown that U metal can be dissolved in a wide variety of acids, salts, and alkalies by anodic oxidation in an electrolytic cell. Uranium concentrations of 50 to 80 g/l have been obtained with sodium bicarbonate, tartaric acid, or nitric acid as electrolytes. (auth)

RECOVERY OF IRRADIATED TH AND U 3.02

ELECTROCHEMICAL 3.02.08

9751 MITG-A35
Massachusetts Inst. of Tech., Cambridge. Dept. of Metallurgy.
ELECTROLYTIC PRECIPITATION OF URANIUM FROM AQUEOUS SOLUTIONS. Eugene Joseph Michal. Sept. 12, 1947. Decl Apr. 11, 1959. 60p. $0.40(OTS).

The use of electrolysis for recovery of U from pure sulfate solutions was studied with a view toward its application to leach liquors from U ores. These leach liquors are relatively dilute, containing less than one g of U per liter, and precipitation tests described in this thesis were

2358 RL-4.6.315
California. Univ., Berkeley [Radiation Lab.].
THE ELECTROLYTIC REDUCTION-pH CONTROL METHOD OF SEPARATION OF [U] FROM CONTAMINANTS. E. L. Wagner. July 28, 1943. ~~Decl. Sept. 15,~~ 1955. 5p. Contract [W-7405-eng-48]. (CHEM. S-315). $1.80(ph OTS); $1.80(mf OTS).

The electrolytic reduction and the pH control methods of separating U from metallic contaminants are described and interference by Cr is discussed. (T.R.H.)

FLUIDIZED BEDS 3.02.09

See also, under PROCESS CHEMISTRY AND ENGINEERING,

FLUIDIZED BEDS (6.08),

FLUOROX PROCESS (6.17.09).

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.

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.)

13598 TID-10145
New Brunswick Lab., AEC, N. J.
QUARTERLY PROGRESS REPORT FOR THE PERIOD ENDING SEPTEMBER 30, 1954. C. J. Rodden. Dec. 1954. Decl. Apr. 22, 1957. 50p. $0.45(OTS).

Various separate phases of the proposed moving bed reduction of UO_3 to UO_2 have been simulated in the moving bed reactor. The moving bed data indicated that UOx, even where x is as low as 2.35, was thermally dissociated in a steam gas phase with resultant liberation of stoichiometrically equivalent amounts of oxygen. Other sections of the reduction process investigated included the thermal dissociation of UO_3 feeds in the presence of steam, the H_2 reduction of U_3O_8 in the presence of steam, and H_2 reduction of UOx feeds in the presence of steam. The rate of thermal dissociation of $^3/_{16}$ inch UO_3 pellets to U_3O_8 was determined at 350°C. Specific laboratory rates of reduction of $^3/_{16}$ inch U_3O_8 pellets using 3.5 mole % H_2 in steam at 460, 550, 575, and 625°C were found to vary from 0.072% conversion per minute at the lower temperature to 3.08 at the higher temperature. At low concentrations of HF the hydrofluorination of $^3/_{16}$ inch UO_2 pellets in a given gas phase over a wide temperature range indicated that a region of maximum specific reaction rates existed for each gas phase. Setting times of UO_3-H_2O mixtures were studied as a function of setting temperatures and wt. % H_2O. Shear strengths of the resulting $^3/_{16}$ inch pellets were obtained in the hydrated, dehydrated, and reduced states.

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FLUIDIZED BEDS 3.02.09

8720
DEVELOPMENT OF A FLUIDIZED-BED PROCESS FOR THE PRODUCTION OF URANIUM TETRAFLUORIDE. A. A. Jonke and N. M. Levitz (Argonne National Lab., Lemont, Ill.). pp. 122-35 in "Progress in Nuclear Energy. Series III. Process Chemistry. Volume 2."

Experimental work has been carried out to demonstrate the technical feasibility of applying the fluidization technique to the production of uranium tetrafluoride. The results of development studies in equipment ranging in size from bench scale to production scale are described, and the advantages of the fluidized-bed technique over conventional processing methods are pointed out. Development work on the production of uranium trioxide and uranium dioxide, as well as uranium tetrafluoride, is included. The employment of multistage fluidized-bed reactors is described and compared with single-stage reactors. (auth)

6338 A/CONF.15/P/229
CANADIAN DEVELOPMENT WORK WITH MOVING-BED REACTORS FOR REDUCTION OF URANIUM TRIOXIDE AND HYDROFLUORINATION TO URANIUM TETRAFLUORIDE FOR SUBSEQUENT PRODUCTION OF METAL. F. W. Melvanin (Eldorado Mining and Refining Ltd., Ottawa). 13p. (AECL-639). $0.25(AECL).

Moving-bed shaft-type reactors are in use for the reactions $UO_3 + H_2 \rightarrow UO_2 + H_2O$ and $UO_2 + 4\ HF \rightarrow UF_4 + 2H_2O$. Each reaction is carried out in a simple vertical tube where gases enter countercurrently to the solid. The pilot plant producing 45 kg/day at the Port Hope Refinery is described. UO_3 is fed into the first reaction vessel in pellet form. (W.D.M.)

FLUORINATION AND HYDROFLUORINATION 3.02.10

6356
RECOVERY OF URANIUM VALUES FROM RESIDUES. Ward B. Schaap (to U. S. Atomic Energy Commission). U. S. Patent 2,900,226. Aug. 18, 1959.

A process is described for the recovery of uranium from insoluble oxide residues resistant to repeated leaching with mineral acids. The residue is treated with gaseous hydrogen fluoride, then with hydrogen and again with hydrogen fluoride, preferably at 500 to 700°C, prior to the mineral acid leaching.

21004
DRY FLUORINE SEPARATION METHOD. Glenn T. Seaborg, John W. Gofman, and Raymond W. Stoughton (to U. S. Atomic Energy Commission). U. S. Patent 2,887,357. May 19, 1959.

Preparation and separation of U^{233} by irradiation of ThF_4 is described. During the neutron irradiation to produce Pa^{233} a fluorinating agent such as HF, F_2, or $HF + F_2$ is passed through the ThF_4 powder to produce PaF_5. The PaF_5, being more volatile, is removed as a gas and allowed to decay radioactively to U^{233} fluoride. A batch procedure in which ThO_2 or Th metal is irradiated and fluorinated is suggested. Some Pa and U fluoride volatilizes away. Then the remainder is fluorinated with F_2 to produce very volatile UF_6 which is recovered. (T.R.H.)

22061
A NEW METHOD TO OBTAIN URANIUM TETRAFLUORIDE. Jovino D. Pedregal and Francisco Aguilar. Energia nuclear (Madrid) 3, No. 10, 39-49(1959) Apr.-June. (In Spanish)

The production of uranium tetrafluoride by the reaction of a uranium compound with ammonium bifluoride was investigated. Preliminary investigations were made with uranium nitrate and ammonium uranate, and the results showed the possibility of using this method on an industrial scale. The design and construction of reaction vessels for a continuous process production of uranium tetrafluoride and described. The product obtained is of nuclear purity. (J.S.R.)

82 ANL-6054
Argonne National Lab., Lemont, Ill.
FLUID-BED CONVERSION OF URANIUM TETRAFLUORIDE TO URANIUM HEXAFLUORIDE. C. J. Vogel and W. J. Mecham. Oct. 1959. 25p. Contract W-31-109-eng-38. OTS.

Experiments conducted in a 2½-inch diameter reactor have demonstrated that the continuous fluorination of uranium tetrafluoride to hexafluoride with elemental fluorine in a fluidized bed is possible. Practicable fluorine efficiencies and conversion rates were attained with good temperature control in experiments with refined uranium tetrafluoride. Runs made with crude uranium tetrafluoride, derived from the reduction and hydrofluorination of ore concentrates produced by the acid-leach process, indicated that these also can be processed at satisfactory rates and efficiencies. Uranium tetrafluoride derived from carbonate-leached ore concentrate gave a lower production rate and was more difficult to process because of sintering tendencies. (auth)

RECOVERY OF IRRADIATED TH AND U 3.02

FLUORINATION AND HYDROFLUORINATION 3.02.10

PROCESS OF PREPARING A FLUORIDE OF TETRAVLENT URANIUM. Earl J. Wheelwright (to U. S. Atomic Energy Commission). U. S. Patent 2,874,026. Feb. 17, 1959.

A method is described for producing a fluoride salt of tetravalent uranium suitable for bomb reduction to metallic uranium. An aqueous solution of uranyl nitrate is treated with acetic acid and a nitrite-suppressor and then contacted with metallic lead whereby uranium is reduced from the hexavalent to the tetravalent state and soluble lead acetate is formed. Sulfate ions are then added to the solution to precipitate and remove the lead values. Hydrofluoric acid and alkali metal ions are then added causing the formation of an alkali metal uranium double-fluoride in which the uranium is in the tetravalent state. After recovery, this precipitate is suitable for use in the bomb production of metallic uranium.

PROCESS FOR PRODUCTION OF URANIUM. (To U. K. Atomic Energy Authority (U.S.A.).) British Patent 796,589. Nuclear Eng. 4, 48(1959) Jan.

It has been found that close control of the particle size of the uranium tetrafluoride is important in obtaining massive uranium in relatively high yields. The particle size should preferably be such that 90% passes a 60 mesh sieve, 85% an 80 mesh, 75% a 100 mesh, and 50% a 200 mesh sieve. The excess granular calcium used for the reduction at elevated temperature has a particle size not greater than 7 mesh. Very fine calcium should be present for initiating the reaction. The calcium tends to vaporize and intimately mix with the uranium tetrafluoride. Yields range from 80% to 95% of the theoretical yield.

17943

URANIUM FLUORIDE SEPARATION OF PLUTONIUM AND FISSION PRODUCTS FROM IRRADIATED URANIUM. (To Atomic Energy of Canada, Ltd.) British Patent 791,000. Nuclear Eng. 3, 414(1958) Sept.

Uranium tetrafluoride and/or uranium trifluoride is added to neutron irradiated uranium, which is then heated in an inert atmosphere to a temperature of 1,135 to 1,350°C to melt the uranium. The uranium phase and the salt phase are separated by distillation or by gravity separation. For 100 parts of uranium 1 to 50 parts of uranium tetrafluoride may be used.

(See also 3.02.19)

12420 ✓ K-727(Del.)

Carbide and Carbon Chemicals Div. K-25 Plant, Oak Ridge, Tenn.

LIQUID PHASE FLUORINATION OF URANIUM METAL WITH BROMINE TRIFLUORIDE. G. J. Vogel and R. W. Vogel. Mar. 14, 1951. Decl. with deletions Mar. 27, 1957. 21p. Contract W-7405-eng-26. $4.80(ph OTS); $2.70(mf OTS).

Bromine trifluoride of 95 mole % purity has been prepared by reacting gaseous F_2 with liquid Br at 10°C. with a 95% yield based on Br. The reaction rate of U and purified BrF_3 to produce UF_6 is too slow to be feasible. However, with the addition of HF or Br to the BrF_3 reaction rates were observed up to 2 g. UF_6 produced per hour per square centimeter of original surface area at 50°C. To facilitate the separation of UF_6 from the reaction products the Br was first fluorinated by gaseous F_2. The UF_6 was then separated by distillation in a packed column. (auth)

8512

PROCESS OF RECOVERING URANIUM VALUES. (to Union Carbide Corp.). British Patent 824,413. Dec. 2, 1959.

A process is described for recovery of U from alkali metals, alkaline earths, transition elements, and rare earths. The U-containing material is hydrided and then reacted with alkali metal acid fluoride. The resulting mixture is then treated with HF. Then, treatment with F_2 gas causes UF_6 to volatilize and be separated. (T.R.H.)

9267 GAT-L-366

Goodyear Atomic Corp., Portsmouth, Ohio.

FLUORINATION OF RECOVERY OXIDE. G. A. Rampy. June 5, 1957. 18p. Contract [AT(33-2)-1]. $3.30(ph OTS); $2.40(mf OTS).

Due to deterioration of equipment in the conversion of UO_3 to UF_6 by fluorination at 1000°C, investigations were begun on lowering the reaction temperature there by eliminating deterioration. At 530°C ~33% excess F_2 was required to completely convert the UO_3 to UF_6. Using 11% excess F_2, conversion was found to increase rapidly from 200 to 450° and level out slightly above 450°C. (W.L.H.)

RECOVERY OF IRRADIATED TH AND U 3.02

FLUORINATION AND HYDROFLUORINATION 3.02.10

13360 ANL-5959
Argonne National Lab., Lemont, Ill.
CHEMICAL ENGINEERING DIVISION SUMMARY REPORT FOR OCTOBER, NOVEMBER, DECEMBER 1958. Mar. 1959. 171p. Contract W 31 109 eng 38. $3.00 (OTS).

Fluoride Volatilization Separations Process. Work has continued on the development of fluoride volatilization processes for recovery of U and Pu from spent power-reactor fuels. Results are reported on the fluorination rates of U and Pu oxides and fluorides. The conversion rate of UO_2F_2 to UF_6 by elemental fluorine was determined as a function of temperature. Uranium trioxide is fluorinated at a slower rate than the other U compounds investigated. On exposure to fluorine PuO_2 appears to form the PuF_4, which, in turn, is converted to PuF_6. The product of the thermal decomposition of PuF_6 is PuF_4. The fluorination of Ni at 600 to 800°C produces films of a higher density than that of crystalline NiF_2. Fluorination studies on Ti, Pt, Au, and Ag indicated that none of these metals is suitable for use with fluorine at temperatures much above 100°C. Development work continued on a high-temperature fused-salt process for the recovery of enriched U from Zr-matrix fuel alloys. Installation of a pilot-plant dissolver-hydrofluorinator made of graphite is nearly complete. The purification of the Liquid Metal Fuel Reactor (LMFR) core fluid by a fluoride volatility process is being investigated. Fluidization Studies. Investigation of the effect of process variables on the fluid-bed drying and fluid-bed fluorination steps of the ADF process for recovery of U from low-U–Zr fuel alloys continued. Fluorinations of fluid-bed dried material conducted in the 2½-inch diameter fluid-bed fluorinator disclosed a number of factors which affect the degree of U removal. Corrosion results of additional specimens inserted in the fluid-bed fluorinator do not substantiate the excessively high vapor-phase corrosion of Ni observed previously. Additional runs in the fluid-bed waste-calciner, used to convert liquid wastes to granular solids, indicated that Ru decontamination factors from feed through silica gel adsorber of 10^3 to 10^4 can be achieved. Corrosion experiments on sintered stainless steel, using conditions simulating those in the fluid-bed waste calciner, were continued. Reactor Chemistry. The program to clarify the factors governing the pyrophoric characteristics of U, Zr, Th, and Pu has continued with studies of kinetics and mechanism of oxidation. Studies on the oxidation of Zr alloys are reported. Chemical-Metallurgical Processing. No removal of Zr from a Pu-"fissium" alloy was realized by melt refining in either lime-stabilized ZrO_2 or MgO crucibles. In connection with the removal of Zr as the carbide, information has been obtained on the joint solubilities of Zr and C in U at 1400°C. Progress was made in the melting of oxide-coated pins by reduction of the oxide coating, using Mg in a Zn solution.

5332 ANL-5924
Argonne National Lab., Lemont, Ill.
CHEMICAL ENGINEERING DIVISION SUMMARY REPORT [FOR] JULY, AUGUST, SEPTEMBER 1958. Dec. 1958. 184p. Contract W-31-109-eng-38. $3.00(OTS).

Fluoride Volatilization Separation Processes. A new reprocessing scheme which may be applicable to most power reactor fuels is described. The irradiated fuel is dissolved in an appropriate aqueous solution which is then calcined to a mixture of dry oxide. Fluorination of the mixture ultimately produces the hexafluorides of U and Pu, which are volatilized. This process will be called the ADF (aqueous dissolution and fluorination) process. The fluorination rates of UF_4 and PuF_4 were investigated. Since oxides, as well as fluorides, are involved in this scheme, fluorination rate studies were conducted on UO_2. The thermal decomposition of PuF_6 was investigated at 300°C by several methods that showed the reaction products to be fluorine and PuF_4. A brief study was made on the rate of fluorine consumption by Ni vessels at temperatures from 300 to 600°C. Development work has continued on the fused fluoride process for the recovery of enriched U from Zr-matrix fuel alloys. Studies of alternate methods of contacting in the fused salt fluorination step are under way. In one scheme, dropwise fluorination is carried out by spraying molten salt into a fluorine atmosphere. Another scheme involves the use of a frozen salt wall to serve as container of the fused salt during fluorination. Further corrosion data are reported on the behavior of Ni and Ni alloys in fluorinated $NaF-ZrF_4$ systems at 600°C.

7440
METHOD OF PREPARING UF_6. R. Davidson and S. Fried (to U. S. Atomic Energy Commission). U. S. Patent 2,910,344. Oct. 27, 1959.

A method is described of preparing uranium hexafluoride without the use of fluorine gas by reacting uranium tetrafluoride with oxygen gas under rigorously anhydrous conditions at 600 to 1300°K within a pre-fluorinated nickel vessel.

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FLUORINATION AND HYDROFLUORINATION 3.02.10

10858 TID-10160
New Brunswick Lab., AEC, N. J.
QUARTERLY PROGRESS REPORT FOR THE PERIOD ENDING DECEMBER 31, 1953. C. J. Rodden. Mar. 19, 1954. Decl. Mar. 13, 1957. 43p. $0.40(OTS).

Development of the moving-bed hydrofluorination of UO_3 continued with production of a 180-lb. batch containing only 1.01 wt. % $(NH_4)_2C_2O_4$ insolubles and 98.9 wt. % UF_4. Reduction of UO_3 with H_2-N_2 and H_2-H_2O at 550 to 650°C was accomplished. A method of obtaining self-absorption corrections in Ra assay work is described. (T.R.H.)

3335 ✓AECD-3705
Ames Lab., Ames, Iowa.
PILOT PLANT PRODUCTION OF THORIUM FLUORIDE. Norman Barson and Morton Smutz. July 1954. Decl. with deletions Nov. 29, 1955. 52p. W-7405-eng-82. $0.35(OTS).

The pilot-plant production of ThF_4 by a wet process was investigated. The process consisted of dissolving $Th(NO_3)_4 \cdot 4H_2O$ in distilled H_2O, precipitating ThF_4 by addition of aqueous HF, filtering the precipitate, and drying the fluoride to 0.1% moisture content. (auth)

DECONTAMINATION OF IRRADIATED REACTOR FUELS BY FRACTIONAL DISTILLATION PROCESSES USING URANIUM HEXAFLUORIDE. H. H. Hyman, R. C. Vogel, and J. J. Katz. August 1955. 14p. (UN-546)

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

The basic chemical information useful in the development of a UF_6 separation process is discussed. A laboratory investigation of the conversion of U metal fuel elements of UF_6 employing BrF_3 dissolution and the auxiliary problems associated with such an approach to U decontamination are described in detail. The alternative approaches which may be employed for more refractory fuel elements discussed. Those aspects of F technology where progress in recent years has increased the probability of successful application of the chemical principles emphasized in this paper are touched upon. 26 references.

10849√ K-1012
Carbide and Carbon Chemicals Co. K-25 Plant, Oak Ridge, Tenn.
PREPARATION OF URANIUM HEXAFLUORIDE FROM URANIUM TETRAFLUORIDE USING BROMINE TRIFLUORIDE PREPARED IN SITU. E. B. Olszewski. May 7, 1953. Decl. Feb. 26, 1957. 18p. Contract W-7405-eng-26. $0.30(OTS).

A low temperature system for the continuous production of uranium hexafluoride from uranium tetrafluoride and/or the oxides of uranium using bromine trifluoride produced in situ as the fluorinating agent is described. Fluorine was added continuously to regenerate the reacted bromine trifluoride. Complete conversion to uranium hexafluoride was obtained at the maximum obtainable feed rate of 3.3 kg. of uranium tetrafluoride per hour. At feed rates of 0.44 to 2.14 kg. of uranium tetrafluoride per hour, fluorine efficiencies from 98.9 to 99.7 per cent were obtained. A method for the separation of uranium hexafluoride from the reaction products using crystallization in a cold water condenser is described. (auth)

10854 NYO-2040
New Brunswick Lab., AEC, N. J.
SUMMARY REPORT ON THE LONG RANGE PROGRAM FOR THE PERIOD FEBRUARY 16, 1953 TO APRIL 30, 1953. C. J. Rodden. June 24, 1953. Decl. Mar. 11, 1957. 78p. $0.55(OTS).

Additional kinetic studies have been made on the reduction of UO_3 and the hydrofluorination of UO_2 in the presence and absence of steam. Tail end reduction rates and hydrofluorination studies were made using a multiple tube reactor and a thermobalance. Further studies were made on the formation of pellets to be used in the moving bed reactor. A moving bed dehydrator was designed and the unit operated satisfactorily. Operation of the moving bed bench scale reactor was continued with certain modifications to the equipment. Studies on bomb reduction processes and the effect of slagging agents on the production of massive metal were continued. A thermodynamic study of slagging procedures is presented. Experimental work on the separation of powdered U from CaO using various leaching agents

10235 ✓
PROCESS FOR THE PRODUCTION OF URANIUM TETRAFLUORIDE. A. S. Leah and R. B. Mooney (to U. S. Atomic Energy Commission). Patent 2,654,654. Oct 6, 1953.

This patent relates to the manufacture of uranium tetrafluoride from ammonium uranium fluoride, NH_4UF_5. Uranium tetrafluoride is prepared by heating the ammonium uranium fluoride to a temperature at which dissociation occurs with liberation of ammonium fluoride. Preferably the process is carried out under reduced pressure, or in a current of an inert gas.

RECOVERY OF IRRADIATED TH AND U 3.02

FLUORINATION AND HYDROFLUORINATION 3.02.10

86 GAT-252
Goodyear Atomic Corp., Portsmouth, Ohio.
OXIDE FLUORINATION TOWER. L. C. Peoples. Aug. 28, 1959. 25p. Contract AT(33-2)-1. OTS.

A 3-inch-diameter flame tower for the conversion of uranosic oxide to uranium hexafluoride with elemental fluorine was tested for possible use in the fluorination step of the present uranium recovery process. The oxide was fed from a hopper to the tower by a screw feeder. The fluorine and the oxide entered at the top and flowed concurrently down through the tower. The unreacted or partially reacted oxide was collected in an ash receiver at the bottom. Fine solid particles were removed from the gas stream by an electrostatic precipitator and a tube-type filter. The uranium hexafluoride was collected in cold traps. Twenty-five experimental runs were conducted with average oxide feed rates from 3.73 to 19.38 lb/hr. The average fluorine flow rates were from 7.5% below to 448% above the stoichiometric amount of fluorine required. The best operating conditions were at a feed rate of 15 lb of oxide per hour with a minimum fluorine excess of 75% (10.6 lb of fluorine per hr). The material collected in the tower ash receiver represented between 6.0 and 10.0 percent of the total amount of uranium fed during the run. The ash, combined with an equal weight of oxide, can be fed back to the tower. The electrostatic precipitator was capable of collecting up to 92% of the solids in the gas stream. A porous-tube filter worked best for the removal of the remaining particles in the gas stream. The experimental runs have shown that uranosic oxide can be satisfactorily converted to uranium hexafluoride in a 3-in.-diameter flame tower of simple design. The present conversion rate of uranosic oxide, approximately 4 lb per manhour with the tube reactor, could be increased to 10 lb per manhour by using the flame tower. (auth)

6161 AECD-3797
North American Aviation, Inc., Downey, Calif.
THE USE OF BROMINE TRIFLUORIDE IN THE RECOVERY OF URANIUM FROM MIXTURES OF UO_2 AND MgO. F. D. Rosen. June 10, 1953. Decl. with Deletions Dec. 5, 1955. 19p. Contract AT-11-1-GEN-8. $0.20(OTS).

A method is described for recovery of U from $UO_2 \cdot MgO$ ceramic fuel material by the use of BrF_3. Recovery of 99.8% uranium from pressed-sintered $UO_2 \cdot MgO$ was accomplished by grinding the material to 6μ particle size, wetting with Br and boiling with BrF_3 for 3 hours. A study of particle size vs U recovery was made. (auth)

10847 √ K-847
Carbide and Carbon Chemicals Co. K-25 Plant, Oak Ridge, Tenn.
CONVERSION OF URANIUM COMPOUNDS TO URANIUM HEXAFLUORIDE BY MEANS OF CHLORINE TRIFLUORIDE. PART III. FLUORINATION OF THE URANIUM OXIDES AT 60, 100, AND 140°C. R. L. Jarry and W. Davis, Jr. Dec. 21, 1951. Decl. Feb. 26, 1957. 21p. Contract W-7405-eng-26. $0.30(OTS).

Rates of conversion, in a dynamic system, of three uranium oxides (uranic, uranous, and urano-uranic) to uranium hexafluoride by means of chlorine trifluoride have been studied at 60, 100, and 140°C. At 60°C the initial rates for all three oxides were essentially the same, the rate for uranium trioxide falling off rapidly with time. At the higher temperatures this effect for the trioxide was considerably less. Analysis of the residues showed a composition tending toward that of uranyl fluoride in reactions involving uranous and urano-uranic oxide; residues from the uranium trioxide experiments were of the same composition as the original material. Products, other than uranium hexafluoride, were chlorine monofluoride, chlorine, chlorine dioxide, and chlorine oxyfluorides. Distillation analyses of these showed that as the oxygen to uranium ratio in the parent uranium oxide increases: 1) chlorine oxide formation increases; 2) efficiency of chlorine trifluoride utilization decreases. Stoichiometry calculations indicate that for the conversion of one mole of uranium to uranium hexafluoride from uranous oxide, urano-uranic oxide, and uranic oxide, 2.34, 3.03, and 4.84 moles of chlorine trifluoride are utilized. (auth)

10814 TID-10159
New Brunswick Lab., AEC, N. J.
QUARTERLY PROGRESS REPORT FOR THE PERIOD ENDING SEPTEMBER 30, 1953. C. J. Rodden. Mar. 16, 1954. Decl. Mar. 7, 1957. 48p. $0.45(OTS).

Modifications of existing laboratory processes for the extrusion, dehydration and denitration of hydrated UO_3 pellets are discussed. Present start-up procedures for both the reduction and hydrofluorination moving bed reactors are described. A joint operation is described wherein the consecutive reduction and hydrofluorination of dehydrated UO_3 pellets were accomplished in a single multistage reactor. UF_4 pellets were rehydrofluorinated in the moving bed using 150% of the theoretical anhydrous HF. The adiabatic compression of $(CH_3)_2O-BF_3$ complex from 1.0 to 1.25 atm pressure will cause a temperature rise of 7°C (from 128 to 135°). Work was done on a method of isotopic analysis of B in which the B was released in the form of BF_3 from the $(CH_3)_2O-BF_3$ product of an isotope separation column. With the use of a cathode-ray tube densitometer, it is possible to determine B in U at concentrations of approximately 0.02 ppm of B. (T.R.H.)

RECOVERY OF IRRADIATED TH AND U 3.02

FLUORINATION AND HYDROFLUORINATION 3.02.10

LIQUID PHASE FLUORINATION OF URANIUM METAL WITH BROMINE TRIFLUORIDE. G. J. Vogel and R. W. Vogel. Mar. 14, 1951. Decl. with deletions Mar. 27, 1957. 21p. (K-727(Del.))

BrF_3 of 95 mole % purity has been prepared by reacting gaseous F_2 with liquid Br at 10°C with a 95% yield based on Br. The reaction rate of U and purified BrF_3 to produce UF_6 is too slow to be feasible. However, with the addition of HF or Br to the BrF_3 reaction rates were observed up to 2 g. UF_6 produced per hour per square centimeter of original surface area at 50°C. To facilitate the separation of UF_6 from the reaction products the Br was first fluorinated by gaseous F_2. The UF_6 was then separated by distillation in a packed column.

12374 K-725

Carbide and Carbon Chemicals Co. K-25 Plant, Oak Ridge, Tenn.

LABORATORY SCALE DECONTAMINATION OF PILE IRRADIATED URANIUM USING CHLORINE TRIFLUORIDE. R. A. Gustison, S. T. Benton, E. J. Barber, and H. A. Bernhardt. Mar. 1, 1951. Decl. Mar. 27, 1957. 28p. Contract W-7405-eng-26. $0.30(OTS).

A laboratory scale process was developed whereby U irradiated in the ORNL pile for 30 days at a flux of 5×10^{11} neutrons/cm^2/sec and cooled 90 days can be decontaminated to less than 20% of the beta-gamma activity of normal U. The Pu concentration is reduced below the limits of detection. The process involves the fluorination of the irradiated U metal to UF_6, the regeneration of ClF_3 from its reduction product by circulation of the product gases and F_2 over a catalyst and a two-step distillation of the UF_6 to free it from the fluorinating mixture, fission product fluorides, and PuF_4. The Pu appears to be oxidized to the nonvolatile trivalent or tetravalent state. Irradiated U cooled 7, 30, and 60 days was also investigated. (auth)

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9693 NYO-1304

Mallinckrodt Chemical Works, St. Louis.

PILOT PLANT WORK ON UF_4 PRODUCTION BY CONTINUOUS METHODS. Progress Report [for] Period March 6, 1950 to April 21, 1950. E. K. Teter. May 15, 1950. Decl. Feb. 28, 1957. 9p. $0.25(OTS).

UO_2 was fluorinated in a double pass Inconel reactor, the material being conveyed by Inconel conveyors. It was found that the pitch of the deflectors on the conveyors determines whether or not lumps would be formed. A Monel liner was installed in the upper reactor but during a run the conveyor caught on the liner seam and twisted. The Adams poro-carbon filter has filtered the reactor exhaust gases very well. The filter can be easily cleaned by backblowing the tubes with a small amount of air at low pressure. (auth)

6519 A/CONF.15/P/1259

PREPARATION DE TETRAFLUORURE D'URANIUM A PARTIR D'UO_3 OU DE SEL D'URANYLE. Preparation of Tetrafluoride of Uranium from UO_3 or Uranyl Salts.) J. Sanlaville (Societe d'Electrochimie, d'Electrometallurgie et des Acieries Electriques d'Ugine, Paris). 11p.

The results obtained in the investigations on the simultaneous reduction and fluorination of UO_3, U_3O_8, uranyl fluoride, ammonium diuranate, and uranyl oxalate are presented. Hydrazine fluoride and ammonium bifluoride were used as the reducing and fluorination agents. The reagents are mixed with the powdered uranium compounds and heated in a nitrogen atmosphere at 100 to 200°C. The reaction products are collected and decomposed either under vacuum or in a nitrogen atmosphere. The mechanism of the reduction of uranium was not established with any certainty, but the minimum quantity of hydrazine fluoride necessary for complete reduction was 0.7 mole/g-atom of uranium. The UF_4 obtained is very pure, a minimum of 99.5%. It is very reactive. (J.S.R.)

6540 A/CONF.15/P/2253

SUKHOI METOD REGENERATSII OBLUCHENNOGO URANA. (Dry Method of Regeneration of Irradiated Uranium by Gaseous Fluorine.) V. N. Prusakov, N. F. Simonov, and N. M. Trotsenko (U.S.S.R.). 13p.

Studies carried out to develop a method of recovering irradiated uranium by gaseous fluorine are reported.

RECOVERY OF IRRADIATED TH AND U 3.02

FLUORINATION AND HYDROFLUORINATION 3.02.10

6367 ✓ A/CONF.15/P/1418
METHOD FOR DIRECT FLUORIDATION OF URANIUM COMPOUNDS. Jovino D. Pedregal and Francisco Aguilar (Junta de Energia Nuclear, Madrid). 20p.

This method is designed to obtain uranium fluoride by treating some uranium compounds with gaseous ammonium fluoride; the salts used for this process must be such that they contain the uranium as the sole fixed element. The use of ammonium fluoride as the fluoriding and reducing agent affords the possibility of obtaining the uranium fluoride in a single stage, with the advantage that it is totally recoverable as verified by various types of condensers. The uranium compounds used have been, among others, ammonium uranate and uranyl nitrate. Some diagrams were drawn up from the results obtained showing the scheme of the process. Among the various salts used, some offer technological advantages, while others are chemically worth while, since stoichiometric conditions are achieved in less time at the same temperature. All the compounds did not behave in the same manner. A study was conducted of the problem of materials, as raised by the need to use ammonium fluoride at high temperatures. Among the various materials tried, graphite, fluorite, magnesium, and urania, as well as some linings, are listed. The results obtained were good, and nuclearly pure uranium fluoride can be prepared in this fashion. The technological aspect also was dealt with, and various types of ovens were tried: horizontal, vertical, discontinuous, semi-continuous, and continuous, with satisfactory results. (auth)

17288 TID-2501(Del.)(p.173-82)
[Ames Lab.], Ames, Iowa.
PRODUCTION OF THORIUM METAL. PART II. PRODUCTION OF THORIUM OXIDE AND THORIUM FLUORIDE. R. H. Giffen and H. A. Wilhelm. p.173-82 [of] NUCLEAR SCIENCE AND TECHNOLOGY. (EXTRACTS FROM JOURNAL OF METALLURGY AND CERAMICS. ISSUE NOS. 1 TO 6, JULY 1948–JANUARY 1951). 10p. (ISC-34).

Thorium oxide and thorium fluoride are produced at the Iowa State College Institute for Atomic Research as a step in the production of thorium metal. Thorium oxalate of high purity is dried and calcined to the oxide, which is subsequently hydrofluorinated at an elevated temperature to produce anhydrous thorium fluoride. A review is given of both the experimental work leading up to the present pilot-plant process and the current developmental work for improving this process. The operation and equipment of this pilot plant are described in detail. (auth)

12425 MCW-118
Mallinckrodt Chemical Works, St. Louis.
EQUILIBRIUM RATIO OF HYDROGEN FLUORIDE AND WATER IN THE FLUORINATION OF URANIUM DIOXIDE AT HIGH TEMPERATURES. Carl W. Kuhlman. July 15, 1948. Decl. Feb. 26, 1957. 7p. $0.25(OTS).

The concentration of HF in $HF-H_2O$ mixtures in equilibrium with mixtures of UO_2 and UF_4 was measured at various temperatures in the range 400 to 700°C. The results show this equilibrium concentration to increase rapidly with the temperature. Equilibrium constants calculated from these data are fitted to the constant pressure form of the Van't Hoff reaction isochore to give the equation $\log k_p = -\Delta H/2.303\ RT + C$, where $\Delta H = -46{,}680$ calories/mole. This value for the heat of reaction is compared with values calculated from thermodynamic data. (L.T.W.)

FUSED SALTS AND LIQUID METALS 3.02.11

See also, this section,
ELECTROCHEMICAL (3.02.08),
REDUCTION--Thorium (3.02.16.02),
U Fluorides (3.02.16.03),
VOLATILITY METHODS (3.02.18),
ZONE MELTING (3.02.19),
and,
under PROCESS CHEMISTRY AND ENGINEERING,
FUSED SALTS--Surveys and Theory (6.10.01.07),
SPECIAL PROCESSES--Fused Salt-Fluoride
Volatility (6.17.11),
Hermex (6.17.13),
and,
under PLANTS AND EQUIPMENT,
HIGH TEMPERATURE SYSTEMS (5.10).

RECOVERY OF IRRADIATED TH AND U 3.02

FUSED SALTS AND LIQUID METALS 3.02.11

FUSED SALTS 3.02.11.01

METHOD OF SEPARATING FISSION PRODUCTS FROM FUSED BISMUTH-CONTAINING URANIUM. R. H. Wiswall (to Atomic Energy Commission). U. S. Patent 2,840,464. June 24, 1958.

A process is described for removing metal selectively from liquid metal compositions. The method effects separation of fission product metals selectively from dilute solution in fused bismuth, which contains uranium in solution without removal of more than 1% of the uranium. The process comprises contacting the fused bismuth with a fused salt composition consisting of sodium, potassium and lithium chlorides, adding to fused bismuth and molten salt a quantity of bismuth chloride which is stoichiometrically required to convert the fission product metals to be removed to their chlorides which are more stable in the fused salt than in the molten metal and are, therefore, preferentially taken up in the fused salt phase.

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

13865 GAT-230
Goodyear Atomic Corp., Portsmouth, Ohio.
KINETICS OF THE REACTION OF URANIUM HEXAFLUORIDE WITH SODIUM FLUORIDE POWDER, PELLETS, AND CRUSHED PELLETS. F. E. Massoth and W. E. Hensel, Jr. Apr. 24, 1958. 40p. Contract AT(33-2)-1. $1.25(OTS).

A basic study of the kinetics of the reaction between uranium hexafluoride and sodium fluoride powder, pellets, and crushed pellets is presented. The rate of the reaction, which has the complex, $UF_6 \cdot 3NaF$, as its end product, was determined by a weight-change method at constant pressure. The investigation was conducted under static conditions over the temperature range from 24 to 68°C. The reaction of uranium hexafluoride with sodium fluoride powder follows the parabolic rate law, while the reaction with crushed pellets follows the logarithmic rate law initially, and the parabolic law in later stages. The reaction with pellets obeys logarithmic kinetics to about 0.4 mole fraction converted, after which no apparent further reaction occurs. The reaction with the powder and crushed pellets proceeds to completion. Incomplete reaction with pellets is explained by a physical blocking effect. A brief discussion of gas-solid reactions is included. A physical model of the reaction mechanism is advanced; the five essential steps are: surface reaction, adsorption on the complex, build-up of the complex film, volume diffusion through the complex, and reaction at the interface. (auth)

1330
PREPARATION OF URANIUM METAL BY FUSED SALT ELECTROLYSIS. G. Meister and W. C. Lilliendahl (Westinghouse Electric Corp., Bloomfield, N. J.). J. Metals 9, 1445-7(1957) Nov.

Uranium metal with a purity of about 99.9% was produced on a large scale by fused salt electrolysis with a material efficiency of about 90%. The material efficiency depends mainly on bath composition, electrolysis temperature, electrode smothering, and elutriation during washing of the powder. (auth)

RECOVERY OF IRRADIATED TH AND U 3.02

FUSED SALTS AND LIQUID METALS 3.02.11

FUSED SALTS 3.02.11.01

1309

ELECTROLYTIC EXTRACTION OF THORIUM FROM FUSED SALTS. Lothar Abraham, Edward L. Thellmann, and James L. Wyatt (Horizons, Inc., Cleveland, Ohio). J. Electrochem. Soc. 104, 724-6(1957) Dec.

High purity thorium metal powder has been produced by fused salt electrolysis of thorium chloride by a process readily adaptable to continuous production techniques. An electrolytic cell design based on a frozen-salt crucible design concept has been evolved. Heating of the salt bath was accomplished by graphite resistance heaters immersed in the bath. Metal quality, current efficiency, and other operating characteristics of the internally heated electrolytic cell compare favorably with those experienced in smaller scale, externally heated cells. (auth)

1320

PREPARATION OF FEED MATERIALS FOR ELECTROLYTIC PRODUCTION OF THORIUM METAL. Charles E. Fisher and James L. Wyatt (Horizons Inc., Cleveland, Ohio). J. Electrochem. Soc. 104, 672-7(1957) Nov.

A potentially economical method for production of thorium metal is the electrolysis of fused thorium tetrachloride in sodium chloride. Preparation of suitable feed constituents for the electrolyte is described. Thorium nitrate was converted to thorium tetrachloride by aqueous chemical and thermal decomposition techniques; intermediate steps include the formation of an ammonium complex which may be dehydrated. (auth)

306

ELECTROLYTIC PREPARATION OF THORIUM METAL. B. C. Raynes, M. E. Sibert, and M. A. Steinberg (Horizons Inc., Cleveland, Ohio) and J. C. Bleiweiss (Dow Chemical Co., Midland, Mich.). J. Metals 9, 1373-80(1957) Oct.

Thorium chloride dissolved in molten NaCl can be electrolyzed in a continuous process for the production of granular, high purity Th metal powder. The preparation of anhydrous chloride, its electrolysis in an expanded scale cell, and the purity and some mechanical properties of the product Th are described. (auth)

PILOT PLANT FLUORINATION OF URANIUM FUEL ELEMENTS BY BROMINE TRIFLUORIDE. G. Strickland, F. L. Horn, and R. Johnson. July 26, 1957. 31p. (BNL-471)

The term Fluoride Volatility Processes refers to several proposed nonaqueous methods of processing irradiated fuel elements. In each of these methods, the U is fluorinated to UF_6 and then decontaminated by distillation. One of the methods, involving direct fluorination of the U by BrF_3, has been under investigation at BNL since 1950; subsequently a pilot plant was built to study this step of the process. The objectives of the program were to investigate the technical feasibility of continuous dissolution, and to determine the effect of process variables on the capacity of the equipment; namely, the effect of temperature, solution composition, flow rate, and U history on dissolving time as related to fuel elements of the types used at BNL, ORNL, and Hanford. The nominal capacity of the dissolver was five pounds of U per hour, and all process equipment was fabricated of Monel. Special equipment components and numerous safety features were utilized. The major equipment development was a canned-rotor pump used for circulation of the dissolver stream through the heat exchanger. A system for continually charging slugs to the pressurized dissolver was also developed. Operation consisted of batch runs made with unirradiated BNL slugs in order to gain information useful in the planning of continuous runs. Eight runs were made at 250°F (121°C), starting with pure BrF_3 and progressing to solutions containing 4 mole % UF_6. Practical dissolution rates were obtained, and UF_6 was found to increase the dissolution rate. Its effect in reducing slug dissolution time was very marked at concentrations up to 1 mole %, became increasingly less in the 1 to 2% range, and had no effect in the 2 to 4%. This is a revised version of BNL-457. 11 references.

9713 BMI-1074(Del.)

Battelle Memorial Inst., Columbus, Ohio.

REFRACTORIES FOR CONTINUOUS REDUCTION OF URANIUM. Allison K. Smalley, Collin Hyde, Adrian G. Allison, and Winston H. Duckworth. Feb. 27, 1956. Decl. with deletions Feb. 13, 1957. 8p. Contract W-7405-eng-92. $1.80(ph OTS); $1.80(mf OTS).

A magnesia refractory was resistant to both molten NaF and molten U metal at 2200°F in short-time tests. It was attacked moderately by molten AlF_3 at 2100°F and severely by molten MgF_2 and CaF_2 at 2600 and 2700°F, respectively. Uranium metal at 3000°F also attacked it severely. Magnesia refractories appear to warrant further consideration as a lining material in reaction vessels for the continuous reduction of UF_4 by Na, provided that the refractory temperatures can be maintained below 3000°F. Refractories made of alumina, stabilized zirconia, mullite, chrome-magnesite, or alumina-chromite were considered unsuitable for this application, owing to the ease with which they were attacked by NaF, MgF_2, CaF_2, and AlF_3. (auth)

RECOVERY OF IRRADIATED TH AND U 3.02

FUSED SALTS AND LIQUID METALS 3.02.11

FUSED SALTS 3.02.11.01

RECOVERY OF URANIUM FROM STAINLESS STEEL FUEL ELEMENTS. L. W. Niedrach, A. C. Glamm, M. E. Brennan, and B. E. Dearing. July 1, 1957. 16p. (M-6290)

The results of an investigation to provide laboratory and pilot plant data leading to the development of the head end step of a process for the recovery of U from spent UO_2-stainless steel fuel are reported. The head end step demonstrated consisted of the dissolution of the stainless steel cladding in H_2SO_4, the dissolution of the UO_2 in HNO_3, and a single solvent extraction cycle using 10% TBP in a hydrocarbon diluent to separate the U from the stainless steel components. This head end treatment was designed to be compatible with further solvent extraction processing to be carried out in the common chemical processing facilities at the National Reactor Test Station. 9 references.

FUSED-FLUORIDE PROCESSING OF SPENT URANIUM FUEL. Archie G. Buyers (Atomics International, Canoga Park, Calif.). Nucleonics 15, No. 6, 86-9(1957) June.

A fused-fluoride processing experiment at ORNL is described. Uranium irradiated to 95 g Pu/ton of U and cooled for $2\frac{1}{2}$ yr. was melted at 1573°K with UF_4, CaF_2, and LiF. The equilibration results, giving distribution of Pu and fission products in the melt, are tabulated and plotted in a useful form. (T.R.H.)

Fused-Fluoride Processing of Spent Uranium Fuel, A.G. BUYERS. Nucleonics 15, 6, June 1957, p.86-9.

How fused fluorides can remove all but trace of plutonium and 95-100% of fission products (except Ru) from molten uranium; decontamination is less than that obtainable with aqueous processing but would be sufficient for fast breeders; results of investigations of this system.

9254 KAPL-1693
Knolls Atomic Power Lab., Schenectady, N. Y.
RECOVERY OF URANIUM FROM FUSED SALT MELTS BY ELECTRODEPOSITION. L. W. Niedrach and G. R. Fountain. Mar. 29, 1957. 18p. Contract W-31-109-Eng-52. $0.20(OTS).

An electrolytic method is described for recovering U values from waste salts from an electrorefining process for reactor fuels. The behavior of U and representative active metal fission product elements in such a process is described. (auth)

PROCESS FOR FISSION-PRODUCT REMOVAL FROM URANIUM-BISMUTH REACTOR FUELS BY USE OF FUSED-SALT EXTRACTION. O.E. Dwyer (Brookhaven National Lab., Upton, N.Y.) A.I.Ch.E. Journal 2, 163-8 (1956) June.

The Liquid Metal Fuel Reactor under development at the Brookhaven National Laboratory uses a fuel which is a solution of U^{233}, Mg, and Zr in liquid bismuth. For a power breeder thermal reactor, high neutron economy is essential, and this calls for low concentrations of those fission products in the fuel which are high neutron capturers. Roughly 45% by weight of the fission products can be continuously removed from the fuel by salt extraction with alkali and alkaline-earth fused-salt mixtures. These fission products contain the highly "poisonous" rare earths. A discussion of process design considerations and proposed flow sheets is presented. (auth)

ELECTRODEPOSITION OF URANIUM. H. A. Wilhelm and D. H. Ahmann (to U. S. Atomic Energy Commission). U. S. Patent 2,781,304. February 12, 1957.

A process for the electrodeposition of metallic U from fused uranium halide baths is given. It has been found that the U in the bath may be furnished by using an anode of UC_2. Alternatively the UC_2 may be placed in contact with, or adjacent to the anode whereby the UC_2 is broken down and the U dissolved. The electrolyte is a fused uranium alkali halide or a mixture of such halides. (auth)

11693 AEC-tr-3645
METHOD IN THE PREPARATION OF URANIUM METAL. Translated for Mallinckrodt Chemical Works from Swedish Patent No. 157.612. Inventor: J. H. Mogard. [Patent] Granted, Nov. 15, 1956. 12p. $3.30 (ph), $2.40(mf) JCL or LC.

A method for the production of U metal from UF_4 by metallothermal reduction with Mg in a salt fusion bath is described. The process is suited for production of U in large and small quantities, and an open crucible can be used to obtain either solid or liquid U metal. ~~(J.R.D.)~~

(See also 3.02.17.)

RECOVERY OF IRRADIATED TH AND U 3.02

FUSED SALTS AND LIQUID METALS 3.02.11

FUSED SALTS 3.02.11.01

9544
PRODUCTION OF URANIUM. F. H. Spedding, H. A. Wilhelm, and W. H. Keller (to U. S. Atomic Energy Commision). U. S. Patent 2,787,538. Apr. 2, 1957.

A method for the production of massive U metal is described. The metal is produced by reacting UF_4 with Mg. Although this reaction is exothermic, the heat generated is insufficient to make the U fluid enough to coalesce in massive form. To remedy this, additional heat is obtained from a simultaneous auxiliary exothermic reaction. The reaction may be between additional Mg and an oxidant added to the reaction mass and the particular oxidant claimed by this patent is molecular iodine. (auth)

972 SRO-11
Horizons, Inc., Cleveland.
RESEARCH AND DEVELOPMENT IN THE FIELD OF THORIUM CHEMISTRY AND METALLURGY. VOLUME I. PREPARATION OF ELECTROLYTIC CELL FEED FOR PRODUCTION OF THORIUM METAL. Final Report. Charles E. Fisher and James L. Wyatt. June 30, 1956. Decl. June 6, 1957. 140p. Contract AT(30-1)-1335. (HZ-98). $2.75(OTS).

A research and development program in the field of thorium chemistry and metallurgy was conducted. Most of this activity was directed toward the development of techniques for the production of metal by fused salt electrolytic approaches, little effort being proportioned to the preparation of an electrolyte for the process. An aqueous method for the preparation of an anhydrous cell feed was set up and operated for several months. Relatively high operating expense and difficulties with materials of construction prompted the development of a more direct approach for the production of electrolytic cell feed. The system established for cell feed preparation converts thorium nitrate to a basic carbonate via the reaction between the nitrate and sodium carbonate, this precipitation being quantitative. Following a suitable filtration and drying operation, the thorium oxycarbonate is converted directly to the chloride by reaction with carbon and chlorine, forming a cell feed containing between 40 and 45% thorium in a molten matrix of sodium and potassium chlorides. The preparation of cell feed by the described process was carried out on a small-tonnage basis, in 1,000-pound batches. The entire process is capable of being operated on a continuous or semi-continuous basis and presents a commercially feasible approach for the preparation of high quality anhydrous electrolytes suitable for conversion to A.E.C. grade thorium metal by electrolytic techniques. A brief description of small-scale research and development experiments leading to the established system also is included. (auth)

12412 TID-7521(Pt. 1(Del.)(p.13-55))
Horizons, Inc., Cleveland.
THE PREPARATION OF THORIUM METAL POWDER BY FUSED SALT ELECTROCHEMICAL TECHNIQUES. James L. Wyatt. p.13-55 [of] PAPERS FOR THORIUM-URANIUM-233 INFORMATION MEETING HELD AT WILMINGTON, AUGUST 3, 1955. 43p.

Excerpt from SRO-17 (HZ-34).

A review of the program for the preparation of thorium metal powder by fused salt electrolysis is presented. The process which showed greatest promise was a fused salt all-chloride salt bath. The various electrolytic cells used are described. (W.L.H.)

13067 AERE-C/M-229
United Kingdom Atomic Energy Authority. Research Group. Atomic Energy Research Establishment, Harwell, Berks, England.
THE SOLUBILITY OF URANYL COMPOUNDS IN ALKALI NITRATE-NITRITE MELTS. J. R. Findlay and J. N. Gregory. Nov. 29, 1954. Decl. July 24, 1958. 4p. (RCTC-38).

The solubilities of uranyl nitrate hexahydrate and anhydrous uranyl sulfate were measured in $NaNO_3$/$NaNO_2$ and $NaNO_2$/KNO_3 eutectic mixtures at temperatures ranging from 200 to 330°C. It seems that, even at the lowest temperature, both compounds decompose to UO_3 (or a sodium uranate) the solubility of which is negligible (probably less than 0.02% U). (auth)

13096 NYO-3728
Horizons, Inc., Cleveland.
INVESTIGATIONS FOR THE PRODUCTION OF THORIUM METAL. Technical Progress Report [for] Fourth Quarter, February 1 to April 30, 1953. Merle E. Sibert. May 25, 1953. Decl. Mar. 30, 1957. 16p. Contract AT(30-1)-1335. $1.80(ph OTS); $1.80(mf OTS).

A continuation of the investigation of the possibilities for Th production by fused salt electrolysis is presented. The metal produced by the electrolysis of $KThF_5$ and of $NaThCl_5$, was found to be of approximate equivalent quality with respect to purity, hardness, and workability. The chloride process is to be preferred over the fluoride because of its potentiality for continuous operation and its simpler procedure for the preparation of the source electrolyte. (J.E.D.)

RECOVERY OF IRRADIATED TH AND U 3.02

FUSED SALTS AND LIQUID METALS 3.02.11

FUSED SALTS 3.02.11.01

URANIUM PURIFICATION BY ELECTROREFINING. Leonard W. Niedrach and Arthur C. Glamm (Knolls Atomic Power Lab., Schenectady, N. Y.). J. Electrochem. Soc. 103, 521-8(1956) Sept.

The feasibility of a high-temperature electrorefining process for decontamination of U from associated fission products was demonstrated. Low-melting alloys of U with Ni were used to obtain molten products at temperatures below the melting point of pure U. Effects of temperature, current, and salt-bath composition on cell efficiency and cell operation are discussed. (auth)

2385 LA-147
[Los Alamos Scientific Lab., N. Mex.]
PRODUCTION OF URANIUM BY ELECTROLYSIS OF FUSED SALTS. M. Kolodney, H. L. Slatin, and A. S. Covert. Sept. 28, 1944. Decl. Dec. 8, 1955. 18p. Contract [W-7405-eng-36]. $3.30(ph OTS); $2.40(mf OTS).

Electrolytic uranium of high purity has been prepared in crystalline form. The metal is easily handled and melted. The electrolyte consists of 25 to 30% UCl_3 in a

5138
DISTRIBUTION OF ThB BETWEEN THE MELT AND CRYSTALS OF ALKALI METAL HALIDES. V. R. Klokman, G. P. Lovtsyus, and A. A. Mel'nikova. Radiokhimiya 1, 247-52(1959). (In Russian)

The distribution of ThB between the melt and crystals of LiCl, KCl, NaCl, and KBr was studied in the systems LiCl–KCl, NaCl–$LiNO_3$, and KBr–KNO_3. It was observed that during crystallization from the melt the ThB is not captured by the alkali metal halides. It is postulated that the unique behavior of lead in the solution and melt is the result of a strong dependence of the crystallization factor on temperature. (R.V.J.)

8394 CF-59-12-10
Oak Ridge National Lab., Tenn.
RESULTS OF DIFFERENTIAL BED STUDY OF THE KINETICS OF THE ABSORPTION OF UF_6 ON SODIUM FLUORIDE. F. R. Groves, Jr. Jan. 15, 1960. 35p. OTS.

A procedure for studying absorption kinetics involving temperature measurements in an adiabatic differential reactor was developed. The method was tested for the absorption of HF from a nitrogen stream on NaF pellets. The method was applied to a study of the kinetics of the absorption of UF_6 from nitrogen onto NaF. (auth)

6374 A/CONF.15/P/1468
THORIUM METAL PRODUCTION BY A CHLORINATION PROCESS. A. R. Gibson, J. H. Buddery, et al. (Atomic Energy Research Establishment, Harwell, Berks, Eng.) 12p.

The production of thorium metal from thorium oxide via a chloride route is described. The route is based on the formation in situ of anhydrous thorium chloride in an alkali chloride melt, the melt being maintained molten throughout the process. It consists of two stages: chlorination of a thoria–carbon mixture suspended in a fused alkali chloride bath to yield a melt of thorium chloride dissolved in alkali chlorides; and the electrolysis of this melt to produce thorium metal at the cathode. The chlorination stage of the process is more difficult technologically than the electrolysis stage. Basically, it comprises the reaction $ThO_2 + 2C + 2Cl_2 \rightarrow ThCl_4 + 2CO$. The variables affecting this reaction have been extensively studied, and from this study three different ways of carrying out this reaction have been developed. These are: chlorination of thoria suspended in a fused chloride melt by cylinder chlorine, using the

1200
THE PRODUCTION OF URANIUM METAL POWDER BY ELECTROLYSIS IN MOLTEN CHLORIDES. R. G. Canning (Dept. of Mines, Parkside, South Australia). p.115-22 of "Australian Atomic Energy Symposium, 1958."

The conversion of uranium oxides to chlorides in molten chloride medium is described. In the graphite-chlorine reaction employed, the efficiency of chlorine absorption is shown to be dependent on temperature and on the concentration of uranium in the melt. Uranium metal powder is recovered by controlled precipitation with magnesium metal, followed by electro refining on to a molybdenum cathode. Some contamination is derived from graphite and mullite crucibles. A method of preliminary purification of impure oxide is described in which electrolytic UO_2 is produced from a chloride melt containing UO_2Cl_2. (auth)

RECOVERY OF IRRADIATED TH AND U 3.02

FUSED SALTS AND LIQUID METALS 3.02.11

FUSED SALTS 3.02.11.01

PREPARATION OF THORIUM METAL IN A COMPACT STATE BY THE ELECTROLYSIS OF MELTED SALTS. Mints, Rafal'sky, and Bzhesky. Roczniki Chem. 31, No. 2, 741-2(1957). (Translated from Referat. Zhur. Khim. No. 16, 1958, Abstract No. 54598.)

Thorium metal was prepared in a compact state by the electrolysis of a melted mixture of Th, Zn, and Ca salts. The electrolysis was conducted in argon atmosphere at 1000 to 1200°C with a Mo cathode. The metal obtained contains 94.4% thorium, traces of zinc, and other impurities.

GENERAL 3.02.11.02

9458 BNL-483(p.157-63)
Atomics International Div., North American Aviation, Inc., Canoga Park, Calif.
EXPERIMENTS IN LOW DECONTAMINATION PROCESSING OF METALLIC THORIUM–URANIUM FUELS. G. E. Brand, E. W. Murbach, and A. G. Buyers. p.157-63 [of] THORIUM–U^{233} SYMPOSIUM, SPONSORED BY THE UNITED STATES ATOMIC ENERGY COMMISSION AT BROOKHAVEN NATIONAL LABORATORY, JANUARY 9-10, 1958. 7p.

Experiments are being conducted at Atomics International to develop economical methods of reprocessing thorium reactor fuels. Emphasis has been placed on the development of compact low decontamination processes, which yield a relatively small volume of highly radioactive wastes. Although remote fabrication is required for fuel reprocessed by this method, it is felt that this additional cost may be more than offset by savings in the decontamination process. Some of the more promising processes which have been investigated include direct volatilization by arc and induction drip melting, extraction with molten salts, and fused salt electrolysis. One of the difficulties associated with high temperature decontamination of thorium-uranium alloys is containing the molten metal. The experiments were designed to minimize the thorium container contact time at or above the melting point of the alloy. (auth)

Pyroprocessing for Nuclear Fuels, R.C. Reid, D. Duffey, J.E. Vivian. Nucleonics 14, 2, Feb 1956, p 22-5.

Possibilities of alternative to aqueous processing for reactor fuel recovery; one general type is high temperature processing (pyroprocessing), such as molten metal of fused salt extraction and slagging, analogous to methods used successfully in metallurgical industries; some of engineering problems envisioned for pyroprocessing; example of such separation system; advantages offered.

7144 NAA-SR-1872
Atomics International Div., North American Aviation, Inc., Canoga Park, Calif.
SEPARATIONS CHEMISTRY QUARTERLY PROGRESS REPORT [FOR] SEPTEMBER–DECEMBER 1956. E. E. Motta, G. E. Brand, A. G. Buyers, and W. J. Gardner. May 1, 1957. 23p. Contract AT-11-1-GEN-8. $0.25 (OTS).

The hot cave was completed and used for remotely operated kilogram-scale oxide drossing experiments on U fuel. In addition, an experiment using 33 kg of U alloy was conducted to determine the scavenging effect of UC_2 on Zr, Nb, and Mo and to study tilt-pour casting. The decontamination experiments involving fission product evaporation by arc melting have been directed toward the removal of fission products from irradiated Th-U alloys and the determination of the Pa loss from irradiated Th. The radiochemical analyses for the salt bed extraction experiments on irradiated Th–U alloy are nearing completion. The molten arc electrolysis efforts on Th consisted of experiments to determine the properties of container materials, electrolyte composition, and cathode alloy. (For preceding period see NAA-SR-1759.) (T.R.H.)

Oak Ridge National Lab., Tenn.
PROCESSING OF REACTOR FUELS IN MOLTEN $3\ ZrCl_4 \cdot 2\ PoCl_3$. T. A. Gens. Jan. 6, 1958. Decl. Dec. 10, 1958. 7p. Contract [W-7405-eng-26]. $1.80 (ph), $1.80(mf) OTS.

Dissolution rates of approximately 10 mg/min-cm^2 have been obtained with all fuel alloys that have been tried. Uranium recovery of about 97% was achieved in the only run in which a material balance was obtained. A residue, insoluble in nitric acid, was found to contain up to 2.5% of the uranium that had dissolved in the melt. This quantity of uranium cannot be considered a loss unless further studies show that it cannot be dissolved by simple variation in technique. In the first experiment, the nitric acid contained half as much phosphate as uranium, by weight. A simple variation in procedure in the next experiment reduced the phosphate-uranium weight ratio to 1:50. Nichrome V seems to be a suitable and practical material of construction. (auth)

RECOVERY OF IRRADIATED TH AND U 3.02

FUSED SALTS AND LIQUID METALS 3.02.11

GENERAL 3.02.11.02

10829 NAA-SR-1759
Atomics International Div., North American Aviation, Inc., Canoga Park, Calif.
SEPARATIONS CHEMISTRY QUARTERLY PROGRESS REPORT [FOR] JULY–SEPTEMBER 1956. E. E. Motta, A. G. Buyers, and W. J. Gardner. Jan. 1, 1957. Decl. Mar. 13, 1957. 32p. Contract AT-11-1-GEN-8. $0.35 (OTS).

Scale-up efforts for the high temperature processing of U fuels were concerned with relocated hot cell reconstruction and fittings. Heat transfer for induction coil cooling media has been correlated with power output for a 30 kw motor generator power supply. A fundamental, high temperature chemical study of the mechanism of fission product extraction by UO_2 in contact with molten irradiated U has been proposed. High temperature separations studies concerning the removal of fission products from irradiated Th–U alloy have been directed toward the development of evaporative arc melting techniques, salt bed equilibration, and electrorefining from molten salt baths. Refractory slagging of irradiated Th–U alloy using, as dross, solid and liquid oxides and mixtures, represents a completed phase of the development study of the pyrochemical processing of Th fuels. A summary report of this investigation is included. Radiochemical analyses for fission products in solutions containing large amounts of Th and Ca have been improved. (auth)

7550 NAA-SR-1724
Atomics International Div., North American Aviation, Inc., Canoga Park, Calif.
PYROMETALLURGICAL PROCESSES FOR THORIUM–URANIUM FUEL. Semiannual Progress Report [for] July–December 1955. A. G. Buyers, ed. Oct. 1, 1956. Decl. Feb. 28, 1957. 29p. Contract AT-11-1-GEN-8. $0.35 (OTS).

A report of progress is made on pyrometallurgical process for Th–U fuel. Investigation of pyrochemical processing of Th–U fuels has indicated that the de Boer Iodide Refining Process offers promise for fuel decontamination by removing fission products Ru, Cs, Ce, Sr, and rare earths. A two-step process consisting of fractional volatilization of iodides and a de Boer decomposition of condensed iodides in succession, should provide for removal of the fission products mentioned above, Pa, Zr, Nb, Mo, and Te. Studies are continuing on volatilization of fission products from arc-melted Th–U alloy samples and on molten salt slagging of Th–U. Their potential value as indicated by preliminary data warrants further work. Work has been terminated on decontamination by volatilization from Th–U melts contained in a crucible, solid slagging of Th–U, and arc zone melting. These processes, while offering reasonable decontamination from Ce, Sr, Cs, Te, rare earths and in the latter case Ru, did not warrant further investigation. The results from the volatilization experiments which employed hydrided and dehydrided Th at slightly below their melting points, are sufficiently promising to require further consideration. ~~(auth)~~

7548 NAA-SR-1515
Atomics International, North American Aviation, Inc., Canoga Park, Calif.
SEPARATIONS CHEMISTRY QUARTERLY PROGRESS REPORT FOR APRIL–JUNE 1955. E. E. Motta, A. G. Buyers, and S. J. Yosim, eds. Apr. 1, 1956. Decl. Feb. 27, 1957. 46p. Contract AT-11-1-gen-8. $0.45(OTS).

Small-scale experiments involving metal extraction, fused-salt slagging, and oxide scavenging of spent U fuel and irradiated Th–U alloys are reported. Scale-up work on various processes is described. (auth)

LIQUID METALS 3.02.11.03

See also, this section,

RECOVERY OF PLUTONIUM--Fused Salts and Liquid Metals (3.03.07).

267
PURIFICATION OF URANIUM FUELS BY SLAGGING AND FILTRATION. R. F. Taylor and D. L. Banfield (Atomic Energy Research Establishment, Harwell, Berks, Eng.). Reactor Technol. 1, 77-83(1959) Aug.

Using uranium containing 0.1% Ce as a simple model of irradiated fuel, the degree of 'poison' removal by selfslagging methods was found to be comparable with that achieved at very low alloying concentrations. By filtration through oxide beds, removal of 95% Ce was obtained, the speed of processing being limited only by the time required to reach operating temperature. Information is presented on the behavior of inclusions and the mechanism of cerium removal from the alloys. (auth)

RECOVERY OF IRRADIATED TH AND U 3.02

FUSED SALTS AND LIQUID METALS 3.02.11

LIQUID METALS 3.02.11.03

4397
THE MELT REFINING OF IRRADIATED URANIUM: APPLICATION TO EBR-II FAST REACTOR FUEL. I. INTRODUCTION. L. Burris, H. M. Feder, S. Lawroski, W. A. Rodger, and R. C. Vogel (Argonne National Lab., Lemont, Ill.). Nuclear Sci. and Eng. 6, 493-5(1959) Dec.

A process for recycling plutonium and uranium alloy fuels is described; it involves melting and liquating the alloys in ceramic crucibles and casting the partially purified metals. Its particular merit is the rapid turnover of a fuel suitable for a fast reactor. A pilot plant, now under construction, will operate in a closed cycle with the EBR-II, and will permit technical and economic evaluation. (auth)

4398
THE MELT REFINING OF IRRADIATED URANIUM: APPLICATION TO EBR-II FAST REACTOR FUEL. II. EXPERIMENTAL FURNACES. G. J. Bernstein, G. A. Bennett, N. R. Chellew, and V. G. Trice, Jr. (Argonne National Lab., Lemont, Ill.). Nuclear Sci. and Eng. 6, 496-500(1959) Dec.

Six experimental furnaces used for the development of the melt refining process are described. These furnaces permitted uranium or its alloys to be melted without atmospheric contamination while their properties (surface tension, reactions with refractory oxides, decontaminatibility, castability, etc.) were under study. (auth)

1207
THE PREPARATION OF THORIUM CARBIDE AND SOME ASPECTS OF THE HIGH TEMPERATURE DECONTAMINATION OF IRRADIATED CARBIDE FUELS. D. E. Scaife and A. W. Wylie (Commonwealth Scientific and Industrial Research Organization, Melbourne). p.172-81 of "Australian Atomic Energy Symposium, 1958."

A study was made of factors influencing the synthesis of thorium dicarbide from thorium oxide and carbon, and of the volatilization of impurity elements from thorium carbide at high temperatures. Temperature is the most important variable, and under suitable conditions over 99% of thorium oxide can be converted to carbide in 30 minutes at 2130°C. Radiometric and other techniques were used to follow the volatilization of impurity elements from the carbide above 2000°C. The experimental results are correlated with thermodynamic functions, ΔF, for vaporization of these elements, and it is shown that at 2130°C, the ΔF values of yttrium and the rare earths divide a selection of 24 elements into two groups. The first group has ΔF values less than those calculated for the rare earths, and all elements comprising it are substantially volatile at 2130°C. The second group has ΔF values greater than those calculated for the rare earth elements, and all elements comprising it are substantially non-volatile at 2130°C. The thermal decontamination of thorium carbide from fission product poisons is discussed. Isotopes of Cs, Sr, Be, Ba, Sm, Eu, Ag, and Cd can be removed at 2130°C. The possibility of further substantial decontamination at higher temperatures is examined. (auth)

667
SOME PROPERTIES OF URANIUM–FISSION ALLOYS. S. T. Zegler and M. V. Nevitt (Argonne National Lab., Lemont, Ill.). Nuclear Sci. and Eng. 6, 222-28(1959) Sept.

Hardness, density, and thermal expansion data are presented for alloys of uranium with certain fission-product elements. The elements are those expected to remain in a spent fuel from a fast reactor following pyrometallurgical refining. In cast and gamma quenched alloys the retention of the high-temperature gamma phase produces low hardness and low density. Thermal expansion behavior is dependent upon composition and prior thermal history. (auth)

4401
THE MELT REFINING OF IRRADIATED URANIUM: APPLICATION TO EBR-II FAST REACTOR FUEL. V. YIELD OF FISSIONABLE MATERIAL UPON POURING. G. A. Bennett, N. R. Chellew, and D. C. Hampson (Argonne National Lab., Lemont, Ill.). Nuclear Sci. and Eng. 6, 511-13(1959) Dec.

The recovery of metal as cast ingot after melt refining was studied as a function of the size and geometry of the crucible, the time-temperature cycle, the purity of the furnace atmosphere, and the nonmetal impurities present in the charge. The conditions chosen for the pilot plant operation ensure the attainment of the required recovery and recycle of fuel of steady-state composition. (auth)

7474
REMOVAL OF CERTAIN FISSION PRODUCT METALS FROM LIQUID BISMUTH COMPOSITIONS. O. E. Dwyer, H. E. Howe, and E. R. Avrutik (to U. S. Atomic Energy Commission). U. S. Patent 2,914,399. Nov. 24, 1959.

A method is described for purifying a solution of uranium in liquid bismuth containing at least one metal from the group consisting of selenium, tellurium, palladium, ruthenium, rhodium, niobium, and zirconium. The solution is contacted with zinc in an inert atmosphere to form a homogeneous melt, a solid zinc phase is formed, and the zinc phase containing the metal is separated from the melt.

RECOVERY OF IRRADIATED TH AND U 3.02

FUSED SALTS AND LIQUID METALS 3.02.11

LIQUID METALS 3.02.11.03

15179 ✓ NAA-SR-3590
Atomics International Div., North American Aviation, Inc., Canoga Park, Calif.
DECONTAMINATION OF THORIUM–URANIUM ALLOYS BY CONSUMABLE ARC-MELTING. R. E. Johnson and E. W. Murbach. June 15, 1959. 15p. Contract AT-11-1-GEN-8. $0.50(OTS).

A study of the decontamination of thorium–uranium alloy by the consumable-electrode arc-melting process was carried out. Cesium and strontium removals of up to 75% were obtained, whereas cerium and rare earth removals were less than 20%. Metal loss increases with increasing power to the arc. The method is not suitable for a decontamination process but could be utilized as a step in a low-decontamination, reprocessing method. (auth)

13661 ✓
APPARATUS FOR HIGH PURITY METAL RECOVERY. T. T. Magel (to U. S. Atomic Energy Commission). U. S. Patent 2,873,108. Feb. 10, 1959.

An apparatus is described for preparing high purity metal such as uranium, plutonium and the like from an impure mass of the same metal. The apparatus is arranged so that the impure metal is heated and swept by a stream of hydrogen gas bearing a halogen such as iodine. The volatile metal halide formed is carried on to a hot filament where the metal halide is decomposed and the molten high purity metal is collected in a receiver below.

13819 NAA-SR-2543
Atomics International Div., North American Aviation, Inc., Canoga Park, Calif.
RADIATION INTENSITIES ASSOCIATED WITH CHEMICAL PROCESSING OF METALLIC URANIUM FUEL. Sheldon Berger. July 1, 1958. 32p. Contract AT-11-1-GEN-8. $1.00(OTS).

Radiation intensities were calculated for a 50-kg charge of irradiated and unprocessed fuel in the oxide-drossing crucible, a 50-kg processed ingot, an in-cell roughing filter, and the gas-handling system silica-gel adsorption trap for collecting Xe and Kr. The thermal power of β-γ activity in irradiated 10-day cooled, unprocessed fuel is calculated to be 11.7 watts per kg. Over 75% of this thermal power remains in the oxide-drossing crucible as a residue after processing. This residue has a thermal power of 8.2 watts per kg of fuel processed. After successive melts, this continues to build up and the thermal power may exceed 2000 watts after ten melts. For fuel which has undergone a burn-up of 3000 Mwd/t and a cooling period of 10 days, a gamma decontamination factor of two is calculated in processing of metallic uranium. This is due to the fact that a large percentage of the gamma activity is contributed by Zr, Nb, Mo, and Ru which remain with the uranium after processing. (auth)

METHOD OF PURIFYING URANIUM METAL. R. E. Blanco and B. H. Morrison (to U. S. Atomic Energy Commission). U. S. Patent 2,865,737. Dec. 23, 1958.

The removal of impurities from uranium metal can be done by a process consisting of contacting the metal with liquid mercury at 300°C, separating the impurity-containing slag formed, cooling the slag-free liquid substantially below the point at which uranium mercuride solids form, removing the mercury from the solids, and recovering metallic uranium by heating the solids.

6294 ✓ NAA-SR-1734
Atomics International Div., North American Aviation, Inc., Canoga Park, Calif.
SOLID AND LIQUID DROSSING OF THORIUM CONTAINING TRACER LEVEL FISSION PRODUCTS. N. D. Potter. Apr. 15, 1957. 16p. Contract AT-11-1-GEN-8. $0.20 (OTS).

A study has been made of the distribution of the principal fission products, Pa, and U between tracer level Th-(3 wt. %)U and certain oxides, carbides, sulfides and nitrides. The highly volatile elements Cs and Sr are removed from the metal to the extent of 70 to 100%; 30 to 90% of the less volatile elements Ce and other rare earths are removed from the metal. Most of the Zr, Ru, Pa, and U remain in the alloy. (auth)

9543 ✓
METHOD OF PRODUCING METAL. H. A. Wilhelm (to U. S. Atomic Energy Commission). U. S. Patent 2,787,537. Apr. 2, 1957.

A method is given for tapping liquid U from the reduction vessel in which it is produced. The U is tapped immediately after the reaction has subsided so that the U will still be molten and to take advantage of the pressure within the reduction bomb. The actual tapping apparatus consists of a group of valves including one built into the bottom of the reduction crucible itself. These valves are opened by lowering the bomb onto the tapping apparatus. (auth)

RECOVERY OF IRRADIATED TH AND U 3.02

FUSED SALTS AND LIQUID METALS 3.02.11

LIQUID METALS 3.02.11.03

13137 NAA-SR-2628
Atomics International Div., North American Aviation, Inc., Canoga Park, Calif.
DIFFUSION OF CERIUM AND ZIRCONIUM IN MOLTEN URANIUM. T. Smith. July 1, 1958. 26p. Contract AT-11-1-GEN-8. $0.75(OTS).

Coefficients for the diffusion of cerium in molten uranium were measured over the temperature range 1170 to 1480°C. An equation relating the diffusion coefficient and temperature is $D = 0.02e^{-15\ Kcals/RT}$ The diffusion coefficient for cerium at 1200°C is 1.0×10^{-4} cm²/sec. The solubility of cerium in molten uranium was measured and found to vary from 1 to 2% by weight over the temperature range 1140 to 1345°C. Experimental errors involved in measuring diffusion coefficients at high temperatures are discussed. (auth)

PROCESSING-REFABRICATION OF METALLIC-URANIUM FUEL. D. I. Sinizer, K. L. Mattern, J. R. Foltz, and E. G. Kendall (North American Aviation, Inc., Canoga Park, Calif.). Nucleonics 17, No. 1, 50-3(1959) Jan.

Information is presented on the Processing-Refabrication Experiment (PRE) facility for the low-decontamination processing of irradiated uranium fuels. Discussions are included on: equipment development; maintenance and repair; and oxide drossing, a pyrometallurgical method for partial removal of fission products from irradiated metallic-uranium fuel. A schematic is shown of the PRE low-decontamination process. (J.H.M.)

5260 NAA-SR-1793
Atomics International Div., North American Aviation, Inc., Canoga Park, Calif.
EQUILIBRIUM CONSIDERATIONS ON URANIUM DIOXIDE DROSSING OF IRRADIATED URANIUM. A. G. Buyers and E. W. Murbach. Mar. 1, 1957. 15p. Contract AT-11-1-GEN-8. $0.20(OTS).

Equilibrium constants and changes in standard free energies at 1473, 1548, and 1673°K have been calculated for skin and crucible-urania irradiated-molten-uranium contacting. Equilibria postulated included the formation of PuO_2, SrO, and Ce_2O_3 by the reduction of UO_2. Respective equilibrium constants were found to increase with temperature and vary from 1.3 to 3.2 for $\frac{1}{4}Pu \rightarrow \frac{1}{4}PuO_2$, 18.7 to 36.0 for $\frac{1}{2}Sr \rightarrow \frac{1}{2}SrO$, and 32.6 to 52.3 for $\frac{1}{3}Ce \rightarrow \frac{1}{6}Ce_2O_3$. Free energy changes were found to be more negative than those calculated from reported values. The plot of log Ke vs 1/T has indicated positive values for ΔH and ΔS, a situation which was not indicated by calculations for Ce and Sr. It was suggested that this anomaly might be explained by trapping of evaporated metal by the solid oxide layer and failure to attain thermodynamic equilibrium due to interfacial conditions. It is believed that the foregoing interpretation has disclosed a similarity between skin-oxide and crucible-oxide metal contacting, not revealed by the original evaluation of reported distribution data. (auth)

MINERALOGY, METALLURGY, AND CERAMICS

9542
PROCESS FOR MELTING AND REFINING URANIUM. F. H. Spedding and H. A. Wilhelm (to U. S. Atomic Energy Commission). U. S. Patent 2,787,536. Apr. 2, 1957.

A process for melting and casting U without contaminating it with crucible material is described. The metal is melted in a graphite container, but the temperature is not allowed to go above 1650°C. In this manner the UC, which forms by reaction of the U with the crucible walls, is prevented from dissolving in the U and is retained upon its surface as a skin from which the pure U metal may be easily separated. (auth)

PYROMETALLURGICAL PROCESSES FOR THORIUM-URANIUM FUEL. Semiannual Progress Report [for] July-December 1955. A. G. Buyers, ed. Oct. 1, 1956. Decl. Feb. 28, 1957. 29p. (NAA-SR-1724)

A report of progress is made on pyrometallurgical process for Th-U fuel. Investigation of pyrochemical processing of Th-U fuels has indicated that the de Boer Iodide Refining Process offers promise for fuel decontamination by removing fission products Ru, Cs, Ce, Sr, and rare earths. A two-step process consisting of fractional volatilization of iodides and a de Boer decomposition of condensed iodides in succession, should provide for removal of the fission products mentioned above, Pa, Zr, Nb, Mo, and Te. Studies are continuing on volatilization of fission products from arc-melted Th-U alloy samples and on molten salt slagging of Th-U. Their potential value as indicated by preliminary data warrants further work. Work has been terminated on decontamination by volatilization from Th-U melts contained in a crucible, solid slagging of Th-U, and arc zone melting. These processes, while offering reasonable decontamination from Ce, Sr, Cs, Te, rare earths and in the latter case Ru, did not warrant further investigation. The results from the volatilization experiments which employed hydrided and dehydrided Th at slightly below their melting points, are sufficiently promising to require further consideration.

RECOVERY OF IRRADIATED TH AND U 3.02

FUSED SALTS AND LIQUID METALS 3.02.11

LIQUID METALS 3.02.11.03

11911 ✓
THE THERMODYNAMICS OF THE DECARBURISATION OF MOLTEN URANIUM. R. H. Weisser. Sulzer Tech. Rev. (Switz.) 40, No. 3, 31-2(1958).
Three possibilities for removing carbon from molten U are briefly summarized, and it is shown that they cannot be applied in practice. (auth)

11135 ISC-696
Ames Lab., Ames, Iowa.
LIQUID-LIQUID EXTRACTION OF MOLTEN URANIUM WITH SILVER. C. W. Watson and G. H. Beyer. Mar. 1956. 32p. Contract W-7405-eng-82.

Liquid-metal Extraction for Processing of Spent Fuel, A.F. VOIGT, A.H.DAANE, et.al. Am Inst Chem Engrs--J, 2, 2, June 1956, p. 169-73.

Plutonium and fission products can be removed from irradiated uranium by liquid-metal extraction by use of another metal immiscible with uranium; metals studied were silver, cerium and lanthanum; plutonium removal by silver is high, by rare earth metals moderate; in all cases volatile elements, including cesium, strontium and barium, are removed; rare earths are efficiently removed.

13025 ANL-5349
Argonne National Lab., Lemont, Ill.
REFINING OF URANIUM BY MELTING AND LIQUATION. B. Blumenthal. Nov. 1955. Decl. Apr. 4, 1957. 95p. Contract W-31-109-eng-38. $0.60(OTS).
Reactor grade U, both biscuit metal and Hanford fuel slugs, was melted and liquated in a high vacuum in a variety of crucible materials. Resistance and induction heating were used. Liquation in urania crucibles removes N_2 and O_2 to very low limits (less than 10 ppm). Carbon is partially removed by this method. A variety of conditions and reactions was studied to effect a more complete carbon removal. The study comprised the use of MgO crucibles, melting in a H_2 atmosphere, UN, Ta, Ti, and Zr additions, and stirring with a Ta rod. These experiments had but a limited success with respect to carbon removal. Most of the reactions greatly affected the microstructure of U and, therefore, were evaluated metallographically as well as by chemical and spectrochemical analysis. The optimum melting conditions are stated by which the contamination of reactor grade U can be held within 130 to 200 ppm total impurities. An appendix describes a new process for the control of stringers in reactor grade U. (auth)

3345 ✓ BNL-2396
Brookhaven National Lab., Upton, N. Y.
NOTES ON THE EFFECT OF MAGNESIUM ON PROCESSING. J. Weisman. December 7, 1954. Decl. Nov. 16, 1955. 6p. Contract AT-30-2-Gen-16. $0.15(OTS).
Preliminary results are reported from a number of experiments involving Mg. It was found that Mg has a small but appreciable effect on the solubility of U in Bi at 500°C. U and Th oxides are not reduced by Mg, but chlorides of U and Th are. Rare earths may be extracted from rare earth chlorides in molten Bi or Pb by adding Mg to the metal. A proposed flowsheet for Liquid Metal Fuel Reactor fuel processing is presented. (C.W.H.)

10993 ANL-5019
Argonne National Lab., Lemont, Ill.
THE MELTING OF HIGH PURITY URANIUM. B. Blumenthal. Nov. 14, 1952. Decl. Dec. 9, 1955. 56p. Contract W-31-109-Eng-38. $0.40(OTS).
A melting process was developed for the consolidation of high-purity, electrolytic U crystals into sound ingots of high density without serious contamination. The metal is estimated to be better than 99.993 wt. % pure and is metallographically reasonably free from impurities. (auth)

11918
THE IMMISCIBILITY LIMITS OF URANIUM WITH THE RARE-EARTH METALS. J. F. Haefling and A. H. Daane (Iowa State Coll., Ames). Trans. Met. Soc. AIME 215, 336-8(1959) Apr.
The limits of miscibility in some of the uranium-rare earth alloy systems have been determined in the temperature range 1000 to 1250°C. The solubilities of lanthanum and cerium in uranium are greater than those of the remaining rare earths by a factor of more than two. The solubility of uranium is greater in cerium, praseodymium, and neodymium than in the other rare earth metals studied. The values found in this study are in qualitative agreement with those which might be expected if the solubility rules of Hildebrand and Scott are applicable. (auth)

RECOVERY OF IRRADIATED TH AND U 3.02

FUSED SALTS AND LIQUID METALS 3.02.11

LIQUID METALS 3.02.11.03

Remelting May Permit Reclaiming Uranium-Contaminated Metals, R.B. Klevin, W. B. Harris. Nucleonics 14, 4, Apr 1956, p 93-6.

AEC policy permits free disposal of uranium contaminated ferrous steel if certain radioactivity restrictions are met; to obtain information that might further liberalize disposal regulations, study was undertaken covering uranium contaminated nickel, stainless steel, copper, and aluminum scrap; conclusions concerning possibilities of remelting.

HYDRIDING 3.02.12

SEPARATION OF URANIUM FROM OTHER METALS BY HYDRIDING AND EXTRACTION WITH OXIDIZING REAGENTS. J. H. Carter and T. A. Butler (to U. S. Atomic Energy Commission). U. S. Patent 2,784,054. March 5, 1957.

A method is given for separating U from other metals with which the U is alloyed. The method consists in selectively hydriding the U by contacting the alloy with H_2 at a temperature between 200 and 300°C and a pressure of from 3 to 150 mm. The unhydrided metal phase consists mainly of Pu and either Sn, Au, Ag or Cu. The uranium hydride is separated from this phase by treatment with a solvent which selectively converts the uranium hydride to a soluble state. (auth)

SEPARATION OF URANIUM FROM OTHER METALS BY HYDRIDING. T. A. Butler (to U. S. Atomic Energy Commission). U. S. Patent 2,785,046. March 12, 1957.

A method is given for separating U from other metals with which the U is alloyed. The method consists in selectively hydriding the U by contacting the alloy with H_2 at a temperature between 200° and 300°C and a pressure of from 3 to 150 millimeters. The unhydrided metal phase consisting mainly of Pu and either Sn, Au, Ag or Cu is then separated from the U by dissolution in a nonoxidizing mineral acid, e.g. 2N HCl. (auth)

ION EXCHANGE 3.02.13

See also, this section,

COMPLEX FORMATION (3.02.05).

THORIUM 3.02.13.01

See also, under PROCESS CHEMISTRY AND ENGINEERING,

SPECIAL PROCESSES--Thorex (6.17.22).

15990

SEPARATION OF THORIUM FROM RARE EARTHS, ZIRCONIUM, AND OTHER ELEMENTS BY CATION EXCHANGE CHROMATOGRAPHY. F. W. E. Strelow (National Chemical Research Lab., Pretoria). Anal. Chem. 31, 1201-3(1959) July.

Thorium has a high affinity for the Dowex 50 cation exchange resin and it is not eluted from a column of the resin even by relatively concentrated hydrochloric acid. Semiquantitative affinities for AG 50 resins in hydrochloric acid were investigated for 23 cations, and "single element" elution curves were prepared for those with high affinities. A method was developed to separate thorium quantitatively, even in small amounts, from zirconium and the rare earths. (auth)

6364

SEPARATION OF THORIUM FROM URANIUM. R. W. Bane (to U. S. Atomic Energy Commission). U. S. Patent 2,902,338. Sept. 1, 1959.

A description is given for the separation of thorium from uranium by forming an aqueous acidic solution containing ionic species of thorium, uranyl uranium, and hydroxylamine, flowing the solution through a column containing a phenol-formaldehyde type cation exchange resin to selectively adsorb substantially all the thorium values and a portion of the uranium values, flowing a dilute solution of hydrochloric acid through the column to desorb the uranium values, and then flowing a dilute aqueous acidic solution containing an ion, such as bisulfate, which has a complexing effect upon thorium through the column to desorb substantially all of the thorium.

RECOVERY OF IRRADIATED TH AND U 3.02

ION EXCHANGE 3.02.13

THORIUM 3.02.13.01

ADSORPTION PROCEDURE IN PREPARING U^{233}. R. W. Stoughton (to U. S. Atomic Energy Commission). U. S. Patent 2,856,262. Oct. 14, 1958.

A process is presented for the separation of protoactinium and thorium from an aqueous nitric acid solution containing these metals. It comprises contacting the solution with a cation exchange phenol-formaldehyde resin containing sulfonic acid groups, and eluting the adsorbed thorium from the resin by means of aqueous nitric acid. Thereafter the adsorbed protoactinium is eluted from the resin by means of an aqueous solution of ammonium fluoride.

261

INVESTIGATION OF THE SEPARATION PROCESSES OF URANIUM AND THORIUM ON ALGINIC ACID. I. A. Kuzin and V. P. Taushkanov. Izvest. Vysshikh Ucheb. Zavedenii Khim. i Khim. Tekhnol., No. 2, 70-4(1958).

The separation of uranium and thorium was investigated with a weakly acid cationite, alginic acid, and a highly acid "wofatite" KS. A survey of publications indicates that alginic acid represents a mixture of polycarboxylic acids of different degrees of polymerization; it may be used as sorbent for the separation of polyvalent cations from cations of lower valence. The production of alginic acid and the determination of uranium and thorium are described. The sorptive power of alginic acid and of "wofatite" KS are discussed at different pH values. The dependence of the sorptive power of these two substances on the pH value of a solution of the same concentration (per cent by weight) with regard to the ions UO_2^{2+}, Th^{4+}, and Na^+ is shown. The sorption of sodium ions by alginic acid stops at pH 1.8 to 2.0 and of uranyl ions at pH 0.5; at the same time a considerable sorptive power is maintained for thorium ions. Under the same conditions "wofatite" KS remains capable of sorbing all cations. At a pH below 2 thorium is adsorbed by alginic acid and "wofatite" KS; at higher values uranium is adsorbed. Thorium adheres better to either of the sorbents than uranium. The apparatus for separating uranium and thorium on every sorbent and its operation are described. (TCO)

2319 V LA-2245
Los Alamos Scientific Lab., N. Mex.
U^{233} PURIFICATION AND METAL PRODUCTION. J. P. Bertino and J. A. Kircher. Aug. 1958. 46p. Contract W-7405-eng-36. $1.25(OTS).

Two problems are encountered in U^{233} purification and metal production. One is high alpha activity, which requires that all operations be enclosed. The other is beta-gamma activity originating from U^{232} present in U^{233} as a contaminant. Th^{228}, the immediate daughter of U^{232}, produces a chain of strong beta-gamma emitters, which make shielding and thorium separation procedures necessary. Chemically impure solutions of $U^{233}O_2(NO_3)_2$ from Oak Ridge Thorex product or from metal fabrication residues are separated from trace amounts of thorium isotopes by cation resin exchange in shielded equipment. The short-lived thorium daughters are allowed to decay, after which beta-gamma activity is low enough to permit chemical purification in

SEPARATION OF Th, Zr AND Nb FROM SULPHATE MEDIA BY ANION EXCHANGE. Ferenc Molnár and György Tóth. Magyar Tudományos Akad. Központi Fiz. Kutató Intézetének Közleményei 6, 66-70(1958) Jan.-Apr. (In Hungarian)

The anion exchange adsorption rate of Th, Zr, and Nb from sulfate solution was studied. A Hungarian-made quaternary amine type of resin Mykion PA was used for the experiments. Considering the differences between the distribution ratios of Th, Zr, and Nb, the separation of these elements from sulfate solutions by ion exchange was found to be possible, and on the basis of the distribution studies the separations were carried out. (auth)

9459 BNL-483(p.164)
Los Alamos Scientific Lab., N. Mex.
PRESENT STATUS OF U^{233} PURIFICATION AND METAL PRODUCTION AT LASL. J. A. Kircher. p.164 [of] THORIUM-U^{233} SYMPOSIUM, SPONSORED BY THE UNITED STATES ATOMIC ENERGY COMMISSION AT BROOKHAVEN NATIONAL LABORATORY, JANUARY 9-10, 1958. 1p.

Impure U^{233} nitrate solutions originating from Oak Ridge Thorex product or from metal fabrication residues are separated from trace amounts of thorium isotopes with cation resin. After allowing time for decay of thorium daughters, beta-gamma activity is sufficiently reduced to permit conventional processing in unshielded equipment. The U^{233} is chemically purified by one or

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ION EXCHANGE 3.02.13

THORIUM 3.02.13.01

4372
SEPARATION OF THORIUM AND URANIUM NITRATES BY ANION EXCHANGE. D. J. Carswell (Atomic Energy Research Establishment, Harwell, Didcot, Berks). J. Inorg. and Nuclear Chem. 3, 384-7(1957) Jan.

The anionic character of Th in HNO_3 solutions has been investigated and distribution coefficients measured. The results have been employed to effect a separation of Th and U by anion exchange. (auth)

7468 BMI-242
Battelle Memorial Inst., Columbus, Ohio.
ANION-EXCHANGE STUDIES ON THE SEPARATION AND PURIFICATION OF URANIUM AND THORIUM. R. H. Poirier and A. E. Bearse. Aug. 15, 1950. Decl. Feb. 14, 1957. 71p. Contract AT-30-1-Gen-228. $0.55(OTS).

The separation of U from Th in carbonate, HCl, and H_2SO_4 solutions by anion-exchange resins is described. Capacities, influence of extraneous ions, and elution characteristics are discussed for various types of resins. The removal of U and Th from simulated process solutions, similar to those encountered in processing monazite sand, by the application of anion-exchange resins is described. The U–Th separation by selective adsorption of U on anion exchangers was very effective in Na_2CO_3 and HCl solutions, but only a partial separation was obtained in H_2SO_4 solutions. (auth)

9221
ANION-EXCHANGE STUDIES. XXI. Th(IV) AND U(IV) IN HYDROCHLORIC ACID. SEPARATION OF THORIUM, PROTACTINIUM AND URANIUM. Kurt A. Kraus, George E. Moore, and Frederick Nelson (Oak Ridge National Lab., Tenn.). J. Am. Chem. Soc. 78, 2692-5(1956) June 20.

The anion-exchange behavior of Th^{4+} and U^{4+} in HCl solutions was investigated with a strong base quaternary amine resin. Adsorbabilities of these two elements are surprisingly different. Thorium(IV) ion shows no adsorption throughout the HCl concentration range studied (0.1 to 12M); U^{6+} shows negligible adsorption below 5.5M HCl and rapidly increasing adsorbability at higher concentrations. The ion-exchange behavior of these elements is compared with that of other elements of oxidation number four. An anion-exchange method for the separation of Th^{4+}, Pa^{5+}, and U^{6+} has been devised, as well as a method for the separation of U^{4+} and U^{6+}. (auth)

10827 AERE-C/M-230
United Kingdom Atomic Energy Authority. Research Group. Atomic Energy Research Establishment, Harwell, Berks, England.
THE ION EXCHANGE AND ION ADSORPTION PROPERTIES OF THORIA. D. J. O'Connor. Feb. 1, 1955. 22p. (RCTC-P/35).

The ion exchange, ion adsorption, and electrical surface properties of sintered thoria in water, in solutions of acids, alkalis, several potassium salts, hydrogen peroxide, uranyl sulfate, and trisodium polyphosphate were investigated using the streaming current method of electrokinetics. Thoria, sintered at 1700°C, was positively charged in contact with water. The results are best interpreted by assuming that the electrical double layer at the thoria-solution interface arises principally from basic ionization of surface hydroxyl groups, and that in addition to the usual phenomenon of iron adsorption, the surface charge can be considerably influenced by anion exchange involving hydroxyl groups. In suitable circumstances, (e.g., in the presence of added hydroxyl ion), cation exchange of H^+ ions can be stimulated. In most cases, the notable exception being with trisodium polyphosphate, the adsorption of ionic species at the ThO_2-solution interface was completely reversible. The variation of the ζ potential in trisodium polyphosphate solutions indicated that the polyphosphate ion was reacting chemically at the surface (anion exchange). In addition to providing information on the origin of the electrical surface charge at ThO_2-solution interfaces, the streaming current method also provides information on chemical reactions occurring at ThO_2 surfaces. In the present instance, this information may assist in discussing such problems as the stability and deflocculating action of phosphates and other inorganic stabilizing agents on thoria suspensions. (auth)

3575
THE SEPARATION OF THORIUM(IV) AND CERIUM(III) IONS BY USING ALGINATE AS CATION EXCHANGER. Takeo Takahashi and Shingto Miyake (Univ. of Tokyo). Bull. Chem. Soc. Japan 32, 878-9(1959) Aug.

A method for the separation of thorium (IV) and cerium (III) by using alginate as ion exchanger and dilute nitric acid as eluant is described. (C.J.G.)

RECOVERY OF IRRADIATED TH AND U 3.02

ION EXCHANGE 3.02.13

URANIUM 3.02.13.02

See also, under PROCESS CHEMISTRY AND ENGINEERING,

SPECIAL PROCESSES--Excer (6.17.08),
Neptex (6.17.16),
Purex (6.17.17),
25-TBP (6.17.21),
Thorex (6.17.22).

259 Y-1257
Union Carbide Nuclear Co. Y-12 Plant, Oak Ridge, Tenn.
THE RECOVERY OF URANIUM FROM REDUCTION RESIDUES BY SEMICONTINUOUS ION EXCHANGE. N. J. Setter, J. M. Googin, and G. B. Marrow. July 9, 1959. 22p. Contract W-7405-eng-26. OTS.

Laboratory and plant data related to the recovery of uranium from magnesium fluoride slag are presented. The operation of the Higgins semicontinuous ion exchange contactor and the chemical and mechanical characteristics of the process are described. The system has proven satisfactory for processing tonnage quantities of slag waste materials. (auth)

10905 ✓
SOME ASPECTS OF THE ANION EXCHANGE BEHAVIOR OF URANYL NITRATE IN THE PRESENCE OF OTHER INORGANIC NITRATES. J. K. Foreman, I. R. McGowan, and T. D. Smith (United Kingdom Atomic Energy Authority, Seascale, Cumb., Eng. and Royal Coll. of Science and Tech., Glasgow). J. Chem. Soc., 738-42 (1959) Feb.

The addition of inorganic nitrates to aqueous solutions of uranyl nitrate enhances the uptake of uranium on the anion-exchange resin Deacidite FF in the order aluminum > calcium > lithium > ammonium. The predominating cause is considered to be the facilitation of formation of nitrate complexes by the uranyl ion, and of the subsequent ion-exchange, due to the decrease in the effective water content of the system resulting from hydration of the added cation. Visible absorption spectra revealed no new peaks ascribable to an anionic uranyl nitrate complex. (auth)

IMPROVEMENTS RELATING TO THE RECOVERY OF URANIUM FROM SOLUTIONS. Theodore Roger, Ernest Kressman, and John Robert Millar (to The Permutit Co., Ltd.). British Patent 812,815. Apr. 29, 1959.

An ion exchange method for separation of U from solutions is described. It is based on the fact that cation exchange materials containing carboxyl groups have a high affinity for UO_2^{2+} to the exclusion of other mono- and divalent cations. Suitable materials suggested are copolymers of methacrylic acid and divinyl benzene and condensation products of phenol and formaldehyde treated with chloracetic acid. The only limitation on pH for the process is that imposed by the necessity for having the U in the UO_2^{2+} state. The U can be separated from very dilute solutions by continued flow over the ion exchanger for a sufficient time. (T.R.H.)

17000 JENER-57
Joint Establishment for Nuclear Energy Research, Kjeller, Norway.
SEPARATION OF U(VI), Pu(VI) AND Pu(IV) FROM Zr AND Nb ON A COLUMN OF SILICA GEL. D. Cvjetcanin. Aug. 25, 1958. 19p.

The chromatographic method for the separation of U^{6+}, Pu^{6+}, and Pu^{4+} from Zr^{95}–Nb^{95} on a silica gel column is described. It is possible to carry out the separation of these elements from aqueous, as well as from organic solutions. The method is based on the extraction of U^{6+}, Pu^{6+}, and Pu^{4+} nitrates with methyl isobutyl ketone containing HNO_3 in the presence of silica gel. Zr^{95}–Nb^{95} under the same conditions were retained by the silica gel. (auth)

2058 ✓ NP-7031
Polish Academy of Sciences. Inst. of Nuclear Research, Warsaw.
LIQUID IONITES FOR SORPTION OF URANIUM. Report No. 34/IV. W. Korpak, C. Deptula, and S. Goszczyński. Aug. 1958. 3p.

The synthesis and use of liquid ion exchangers were investigated. In the experiments a number of amines were examined, of which one proved suitable for extracting uranium from solutions of sulfuric acid in the presence of vanadium and iron. A solution of the amine in white spirit is a weakly basic liquid ion exchanger. No emulsion developed during either sorption or elution of uranium with weakly acid solutions of sodium chloride or nitrate, and equilibrium between the phases became rapidly established. (auth)

RECOVERY OF IRRADIATED TH AND U 3.02

ION EXCHANGE 3.02.13

URANIUM 3.02.13.02

7473
ELUTION OF URANIUM VALUES FROM ION EXCHANGE RESINS. R. H. Kennedy (to U. S. Atomic Energy Commission). U. S. Patent 2,914,378. Nov. 24, 1959.
A process is described for eluting complex uranium ions absorbed on ion exchange resins. The resin is subjected to the action of an aqueous eluting solution containing sulfuric acid and an alkali metal, ammonium, or magnesium chloride or nitrate, the elution being carried out until the desired amount of the uranium is removed from the resin.

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.)

11612 ✓ CF-54-6-178
Oak Ridge National Lab., Tenn.
URANIUM RECOVERY FROM BOMB LINER AND SCRAP BY ION EXCHANGE. I. R. Higgins. June 8 1954. Decl. Apr. 2, 1957. 9p. Contract [W-7405-eng-26]. $1.80(ph OTS); $1.80(mf OTS).
Three ion exchange processes were studied for the recovery of uranium from acid C—oxide scrap leach liquors. The recovered U product met or was lower than specifications of 0.05% chloride and fluoride content in each case. (auth)

METHOD OF RECOVERING URANIUM FROM AQUEOUS SOLUTIONS. G. A. Lutz (to U. S. Atomic Energy Commission). U. S. Patent 2,780,514. February 5, 1957.
A method for recovering U values contained in dilute aqueous alkali carbonate solutions is described. This recovery is accomplished by contacting the solution with a strong-base quaternary amine anion exchange resin. The U values are absorbed on the resin, the resin is separated from the depleted solution, and the U eluted with a 6% NaOH—0.3% hydrogen peroxide reagent. (auth)

2235 GAT-L-421
Goodyear Atomic Corp., Portsmouth, Ohio.
ION EXCHANGE IN URANIUM ANALYSIS—A LITERATURE SURVEY. C. F. Trivisonno. Nov. 1, 1957. 17p. $3.30 (ph OTS); $2.40(mf OTS).
A literature survey was made on applications of ion exchange in uranium analysis. Topics covered include the absorption of U, the elution of U, and application of ion exchange resins in U analysis. 27 references. (C.H.)

3230
PROCESS FOR RECOVERY OF URANIUM AND THE UPGRADING OF ALKALI-URANIUM FLUORIDE PRECIPITATES. R. H. Bailes et al (to U. S. Atomic Energy Commission). U. S. Patent 2,768,059. Oct. 23, 1956.
A process for recovering U from an acid anion exchange resin eluate containing $NaUO_2^{3+}$, and Cl^- ions is described. Following the addition of HF slightly in excess of the amount required to form UF_4, the eluate is treated with a reducing agent, such as $Na_2S_2O_4$, to reduce the uranyl uranium to the uranous condition and precipitate the same as $NaUF_5$. The $NaUF_5$ after removal may be marked with a dilute HF solution to convert it to UF_4. (auth)

11645 ✓
CONVERSION OF URANYL SULFATE TO NITRATE BY ION EXCHANGE. D. Dolar and I. Berglez. "J. Stefan" Inst. Repts. (Ljubljana) 4, 139-42(1957).
The application of ion exchange columns for the conversion of uranyl sulfate to nitrate is described. The loading and elution curves are given. It is shown that only cation exchange column is suitable for this purpose. (auth)

7459
URANIUM ABSORPTION ON ACTIVATED PHENOLIC GROUPS IN ION-EXCHANGE RESIN SYSTEMS. M. J. Holdoway, E. S. Lane, and J. L. Willians (Atomic Energy Research Establishment, Harwell, Berks, Eng.). Chem. & Ind. (London) No. 15, 483(1959) Apr. 11.
It is reported that activated phenolic groups can participate in uranium absorption to a considerable extent at low pH values in ion-exchange resin systems. The resins were condensed to hard rigid gels and processed without allowing them to dry. A resin prepared from p-cresol sulfonic acid, resorcinol, and formaldehyde was found to have the following exchange capacities: Na^+/H^+, 1.25; Ba^{2+}/H^+, 1.30; and UO_2^{2+}/H^+, 1.9. With some sulfonated resins, the estimated uranium absorbed by the phenolic groups amounted to 50% of the total uranium capacity. (C.J.G.)

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ION EXCHANGE 3.02.13

URANIUM 3.02.13.02

10104
ON THE INFLUENCE OF THE STRUCTURE OF CATION EXCHANGE RESINS IN THE URANIUM–URANIUM X SEPARATION. Roger Lumbroso and Jean Petit. Compt. rend. 243, 145-6(1956) July 9. (In French)

1585 JEN-6
Spain. Junta de Energia Nuclear, Madrid.
ENSAYOS DE INTERCAMBIO ANIONICO CON RESINA AMBERLITA Ir-120. (Experiments on Anion Exchange with Amberlite Ir-120 Resin). R. Fernandez Cellini and J. Vera Palomino. 1956. 13p.

The ion exchange of the uranyl ion on Amberlite Ir-120 resin was studied with different uranyl ion concentrations. Elution with sulfuric acid was investigated and the elution curve for the experimental conditions was determined. From the concentrations of the ions of Cu^{2+}, Ni^{2+}, Fe^{3+}, Cd^{2+}, Mn^{2+}, and Cr^{3+} the maximum exchange capacity was tested and elution curves with 4 N sulfuric acid were obtained. (tr-auth)

12418 CF-56-6-155
Oak Ridge National Lab., Tenn.
RECOVERY OF U FROM RECYCLE WASTES. I. R. Higgins. June 29, 1956. Decl. Mar. 19, 1957. 14p. Contract [W-7405-eng-26]. $3.30(ph OTS); $2.40(mf OTS).

An ion exchange process for recovery of U and HF from MgF_2 scrap is described. If HF is to be recovered, the MgF_2 is leached with H_2SO_4 and retorted to recover HF. The UO_2SO_4 is extracted on anion exchange resin, complexed with brine or 9M HCl, and the UO_2Cl_2 complex is then eluted with water as UO_2Cl_2. If HF is not to be recovered, the MgF_2 is leached with HCl, the UO_2Cl_2 complex is sorbed directly on the resin and eluted with water. A carbonate leach may also be used. (T.R.H.)

9673 CF-56-6-138
Oak Ridge National Lab., Tenn.
RECOVERY AND PURIFICATION OF URANIUM BY CONTINUOUS ION EXCHANGE AND ELECTROLYSIS. I. R. Higgins. June 26, 1956. Decl. Feb. 15, 1957. 19p. Contract [W-7405-eng-26]. $0.30(OTS).

Uranium recovery and purification by continuous ion exchange and electrolysis, either independently or jointly in a single process, are being studied. The function of the ion exchange step is to concentrate, partially purify, and prepare uranium in a suitable chemical form for the subsequent purification step. The function of electrolysis is to provide more purification and reduce UO_2F_2 to $UF_4 \cdot {}^3/_4\ H_2O$. A number of chemical process steps are used for uranium processing, whether from ores to concentrates to pure metal or hexafluoride, or from spent reactor fuels back to purified uranium. The ion exchange and electrolysis steps are proposed as shorter and less expensive routes in this uranium cycle. Even these steps may be complicated by attempts to adapt them to the chemistry of existing processes. (auth)

9228
ION EXCHANGE IN URANIUM PRODUCTION. T. V. Arden. Nuclear Eng. 1, 68-71(1956) May.

Ion exchange has been adapted for U extraction from low-grade ores. The simplicity and cheapness of the process has resulted in the quantity production of U from mines where low concentration previously made the ore unworkable. Clarified acid leach liquor passes through the basic anion exchange resin. When the resin is loaded, U is eluted by acidified metallic salt solution precipitated with an alkali thickened to an oxide cake of about 90% U. (F.S.)

8090
RECOVERY OF URANIUM FROM AQUEOUS SOLUTIONS. G. A. Lutz (to U. S. Atomic Energy Commission). U. S. Patent No. 2,743,159, Apr. 24, 1956.

A process is outlined for separating UO_2^{2+} values from aqueous H_2SO_4 solutions containing impurities usually associated with U and selected from the group consisting of Fe, Al, P, Mg, V, Ti, Cu, Mn, Cr, Ca, K, La, Si, and Sr. The method consists of contacting the solution with an anion exchange resin whereby UO_2^{2+} values are preferentially and some of the contaminants partially adsorbed, treating the resin with an aqueous mineral acid anion-containing solution for the elution of adsorbed values whereby the adsorbed contaminants and the U contaminants are fractionally and selectively eluted and with a H_2O_2-containing solution of NaOH whereby adsorbed Si is removed from the resin, and collecting the eluted solutions in fractions. (auth)

994 WIN-9
National Lead Co., Inc. Raw Materials Development Lab., Winchester, Mass.
MISCELLANEOUS ELUTION STUDIES. A. R. Kazanjian. Mar. 5, 1956. 28p. Contract AT(49-6)-924. $4.80(ph OTS); $2.70(mf OTS).

An investigation was made of the variables involved in a 5M NaCl–H_2O elution scheme. The most promising procedure was used in a cyclic ion exchange system. Studies were also made on the effect of iron-to-phosphate ratios on U ion exchange and the effect of temperature on elution. (auth)

RECOVERY OF IRRADIATED TH AND U 3.02

ION EXCHANGE 3.02.13

URANIUM 3.02.13.02

3053 ✓
CATION EXCHANGE SEPARATION PROCESS. French T. Hagemann and Harry C. Andrews (to U. S. Atomic Energy Commission). U. S. Patent 2,723,901. November 15, 1955.

The cation exchange separation of Ac from aqueous solutions and in particular its separation from a mixture also containing Th, Ra, Pb, Bi and bivalent decay "daughters" of Ra and Ac is described. This is accomplished by forming an aqueous mineral acid solution of the elements to be separated, passing the solution through a cation exchange resin whereby Ac^{3+} and Th^{4+}, are completely adsorbed by the radium and other bivalent ions present are only partly adsorbed. The resin is then treated with a mineral acid, preferably the same acid as that used in the starting solution and also of approximately the same concentration, whereby the "partly" adsorbed elements are washed out, and thereafter using a more concentrated solution of mineral acid whereby the Ac is eluted while the Th remains on the resin. (auth)

3343 ✓ RMO-2530
Rohm and Haas Co. Research Labs., Philadelphia.
USE OF ION EXCHANGE FOR THE SEPARATION OF URANIUM FROM IONS INTERFERING IN ITS COLORIMETRIC DETERMINATION. Sallie Fisher. May 24, 1954. Decl. Sept. 23, 1955. 32p. Contract AT(49-1)-535. $0.25 (OTS).

In sulfate solutions uranium(VI) may be separated from the elements that interfere in its routine colorimetric analysis by absorbing it on an anion exchange resin (Amberlite IRA-400, 40-60 mesh) and subsequently eluting it with perchloric acid. The interference of metals such as Fe and V is eliminated by pre-reduction to forms not retained by the resin. As the procedure serves to concentrate as well as to separate uranium the analysis of very dilute solutions is made possible. (auth)

6529 ✓ A/CONF.15/P/1605
THE ANION EXCHANGE OF METAL COMPLEXES. THE URANYL–PHOSPHATE SYSTEM. Y. Marcus (Israel Atomic Energy Commission). 19p.

The uranyl (M)–phosphate (A) complex system in acid solution was studied by an anion exchange method. Batch equilibrations of Dowex-1 phosphate resin (RA) with phosphoric acid (HA) and tripotassium phosphate-monosodium phosphate (3:1) buffer solutions provided data on uranium distribution and phosphate concentration. The activity of phosphate ions in the solution and in the resin was calculated, and "ideal" distribution values, derived from the data, were used by sweeping and "curve fitting" methods for a calculation of the complexity constants by successive approximations. These constants are illustrated by a "predominance area" diagram. The main species found are: MA^+, Ma_2, $M(HA)_2^{2+}$, MA_3^-, $M(A,2HA)^+$, and $M(HA)_3^{2+}$ in the solution, and RMA_3 in the resin. (auth)

7744 DOW-14, DOW-15, and DOW-16
Dow Chemical Co. Great Western Div., Pittsburg, Calif.
PROGRESS REPORT FOR APRIL, MAY, JUNE 1949. R. H. Bailes. (DOW-14, May 2. Changed from OFFICIAL USE ONLY Mar. 14, 1958; 24p.; DOW-15, May 26. Changed from OFFICIAL USE ONLY Mar. 14, 1958. 38p.; and DOW-16, June 28. Decl. Dec. 28, 1955. 36p.) Contract AT-30-1-GEN-236. (DOW-14, $4.80(ph OTS); $2.70 (mf OTS); DOW-15 and DOW-16, $6.30(ph OTS); $3.00 (mf OTS) ea.)

These three reports were issued separately, but are cataloged as a unit.

Recovery of U from commercial 32% and 30% H_3PO_4 and the recovery of U_3O_8 from commercial 32% H_3PO_4 by means of anion exchange resins is described. The adsorption of V from commercial 32% and 30% H_3PO_4 is presented. Recovery of F_2 from the fluoride precipitate, precipitation of uranous phosphate from synthetic solutions, and the precipitation of U from H_3PO_4 with Sb are discussed. (W.L.H.)

6536 A/CONF.15/P/2122
SEPARATION OF URANIUM IN CHLORIDE–NITRATE MEDIUM. M. Novák and V. Pekárek (Czechoslovakia). 13p.

An extraction of a uranium ore containing calcium carbonate with the aid of a surplus of a mixture of hydrochloric and nitric acids in different concentrations and digestion time was carried out. The separation was carried out by means of Czechoslovak strong basic anex L of the lutidine-formaldehyde type in nitrate cycle. Uranium was separated from the elements of the first and second groups, aluminum, iron, lead, zinc, rare earths, etc. The separation was carried out not only in a mixture of hydrochloric and nitric acids, but also in a medium of corresponding calcium salts. (auth)

RECOVERY OF IRRADIATED TH AND U 3.02

ION EXCHANGE 3.02.13

URANIUM 3.02.13.02

9218
ON THE ION-EXCHANGE OF URANIUM. Tatsujiro Ishimori and Hisateru Okuno. Bull. Chem. Soc. Japan 29, 78-82(1956) Jan.

From the experimental results on the ion exchange of uranium(VI), it was found that acetate solution is excellent for the elution in cation-exchange procedure; and that separation of uranium can be effected with sulfate solution in anion-exchange procedure. (auth)

6507 A/CONF.15/P/468
THE ION-EXCHANGE OF URANIUM AND SOME FISSION PRODUCTS ON TITANIUM AND ZIRCONIUM PHOSPHATES. Ivan J. Gal and Olga S. Gal (Inst. of Nuclear Sciences "Boris Kidric," Belgrade). 17p.

The ion exchange of uranyl ions and some other cations on titanium and zirconium phosphates is investigated. Two inorganic cation exchangers are chosen because of their chemical and thermal stability as well as for their resistance to intense radiation. Due to these remarkable properties they are of special interest in the treatment of radioactive material. The synthesis of the compounds and the preparation of the granular products used are described. First, the capacities of these exchangers and the distribution coefficients for some mono- and polyvalent cations are determined. Further, conditions for sorption and elution in column operation, especially for uranyl ion and ions of some long-lived fission products (Sr^{90}, Zr^{95}, Ru^{106}, Ce^{144}, Cs^{137}), are studied. Special attention is given to mutual separations.

OXIDATION 3.02.14

See also, under PROCESS CHEMISTRY AND ENGINEERING,
SPECIAL PROCESSES--Fluorox (6.17.09),
Redox (6.17.19).

5269
LOW-DECONTAMINATION-PROCESSING OF URANIUM DIOXIDE BY OXIDATION AND REDUCTION. Sydney Strausberg, T. E. Luebben, F. D. Rosen, Jerold Guon, and E. W. Murbach (Atomics International, Canoga Park, Calif.). Ind. Eng. Chem. 52, 45-6(1960) Jan.

Studies were made with uranium dioxide pellets to develop a procedure for low decontamination and preparation of a material suitable for refabrication to reactor fuel shapes. The pellets were pulverized to a finely divided powder by gaseous oxidation. The particle size of the oxidized power was significantly affected by operating temperature and oxidizing media; oxidation in air at about 375°C yielded a satisfactory product. The higher-oxide powder can be reduced back to uranium dioxide by treating with hydrogen at 650°C. The resulting −400 mesh powder is in a suitable form for adding and blending enriched material. Cold-pressed compacts made from this powder can be sintered to high-density fuel shapes and then returned to reactor use. (auth)

7172
RADIOCHEMICAL REACTIONS CATALYZED IN AQUEOUS SOLUTION BY SEMICONDUCTORS AND METALS. Moïse Haïssinsky and Marcelle Duflo. Compt. rend. 246, 1206-9 (1958) Feb. 24. (In French)

The oxidation of uranium sulfate and potassium iodide and the reduction of ferric o-phenanthroline were studied in connection with the catalyzing effects of the oxides of thorium, tantalum, niobium, and titanium, cobalt sulfide, and platinum and palladium black. Gamma or x rays with an average effective energy of 19 kev were used. The solutions contained 3 to 5 mg/cm^3 of the catalyst and were agitated during the irradiation. Thorium oxide and cobalt sulfide, in the presence of air, cause a large increase in the oxidation yield of uranium sulfate; tantalum and titanium oxides also increase the oxidation products, but only a very weak effect was noted with niobium oxide. Thorium oxide causes no detectable effect in the absence of air. Thorium oxide, tantalum oxide, and niobium oxide increase the radio oxidation of potassium iodide. The total yield in the reduction of ferric o-phenanthroline increases in the presence of cobalt sulfide, thorium oxide, platinum black, and palladium black. Thorium oxide caused no effect on degassed solutions. (J.S.R.)

RECOVERY OF IRRADIATED TH AND U 3.02

OXIDATION 3.02.14

6321 NAA-SR-4612
Atomics International Div., North American Aviation, Inc., Canoga Park, Calif.
GASEOUS CHEMICAL TREATMENT OF IRRADIATED URANIUM DIOXIDE. J. Guon and F. D. Rosen. Jan. 15, 1960. 24p. Contract AT-11-1-GEN-8. OTS.

Alternate oxidation and reduction treatments of tracer level irradiated uranium oxide, at 600 to 1250°C, removes most of the cesium, ruthenium, and tellurium. Over 99% of the ruthenium released was either adsorbed on the furnace tube or trapped on in-furnace filters. The release of gaseous activity, probably krypton, occurred instantaneously each time the treatment gas was admitted. The amount of gaseous activity decreased with successive treatments and was greater during oxidation than during reduction. The weight loss of oxide was insignificant. The treatment of uranium dioxide with hydrogen chloride at 1300 and 1400°C reduced the gross activity of the oxide by a factor of two and removed over 90% of the fission product ruthenium, cesium, and tellurium. The weight loss of oxide at 1300°C was low. (auth)

SEPARATION OF URANIUM FROM OTHER METALS BY HYDRIDING AND EXTRACTION WITH OXIDIZING REAGENTS. J. H. Carter and T. A. Butler (to U. S. Atomic Energy Commission). U. S. Patent 2,784,054. March 5, 1957.

A method is given for separating U from other metals with which the U is alloyed. The method consists in selectively hydriding the U by contacting the alloy with H_2 at a temperature between 200 and 300°C and a pressure of from 3 to 150 mm. The unhydrided metal phase consists mainly of Pu and either Sn, Au, Ag or Cu. The uranium hydride is separated from this phase by treatment with a solvent which selectively converts the uranium hydride to a soluble state. (auth)

2392 MITG-261
Massachusetts Inst. of Tech., Watertown, Mass. Mineral Engineering Lab.
AIR OXIDATION OF URANOUS SOLUTIONS. James H. Pannell. Dec. 21, 1950. Decl. Jan. 11, 1956. 23p. Contract AT-30-1-Gen-211. $0.20(OTS).

Rates of oxidation of uranous (U^{4+}) solutions by air under various conditions similar to those prevailing in ore leach liquors were determined. (auth)

3630 Y-353
Carbide and Carbon Chemicals Corp. Y-12 Plant, Oak Ridge, Tenn.
THE DEVELOPMENT OF EQUIPMENT FOR THE CONTINUOUS BURNING AND LEACHING OF GROUND CARBON. P. B. Petretzky, L. P. Twichell, R. D. Williams, and F. B. Waldrop. Apr. 14, 1949. Decl. Dec. 8, 1955. 20p. Contract W-7405-eng-26. $3.30(ph OTS); $2.40 (mf OTS).

Equipment was developed for the continuous burning and leaching of ground C in order to recover U for the proposed cyclotron closed cycle beta process. The burning apparatus consisted of a vertical tube with a grate to support the C. An updraft of air was used to support combustion. The burning rate of the apparatus was 500 g of C per hr; combustion averaged over 98% complete. The leacher consisted of a heated nitric acid oath divided into four compartments through which ground C was pushed by rotating paddles. The acid flowed, by gravity, countercurrent to the C. (auth)

PRECIPITATION 3.02.15

See also, under FISSION PRODUCT, POISON, AND RADIOISOTOPE REMOVAL,
PROTACTINIUM (2.38),
and,
under PROCESS CHEMISTRY AND ENGINEERING,
SPECIAL PROCESSES--Bismuth Phosphate (6.17.02),
Excer (6.17.08),
UAP Process (6.17.23).

RECOVERY OF IRRADIATED TH AND U 3.02

PRECIPITATION 3.02.15

CARBONATE 3.02.15.01

13868 AEC-tr-3324
ADSORPTION OF URANIUM MICRO-AMOUNTS BY IRON HYDROXIDE AND ITS DESORPTION BY THE CARBONATE METHOD. I. E. Starik, F. E. Starik, and A. H. Apollonova. Translated by Oak Ridge National Lab. Library Staff from Zhur. Neorg. Khim. 3, 121-8 (1958). 16p.

It was found that the co-precipitation of micro-amounts of uranium with iron hydroxide takes place by the adsorption capture method. The dependence on the solution pH of the adsorption capacity of iron hydroxide has been established with respect to uranium in ammonium and carbonate media. Conditions for the co-precipitation of micro-amounts of uranium are established with methotitanic acid. The desorption conditions for micro-amounts of uranium ($n \cdot 10^{-5}$ to $n \cdot 10^{-8}$ g) from the surface of iron hydroxide colloids ($U : Fe = 1 : 10^5$) using the carbonate method are established. Conditions for the complete separation of micro amounts of uranium ($n \cdot 10^{-5}$ to $n \cdot 10^{-8}$ g) from solutions by its desorption by iron hydroxide are established. (auth)

9531
PRECIPITATION OF URANIUM FROM ALKALI METAL CARBONATE SOLUTIONS. F. A. Forward and J. Halpern (to U. S. Atomic Energy Commission). U. S. Patent 2,727,806. Dec. 20, 1955.

A method is given for the separation of U^{6+} from carbonate leach solutions. In order to effect this separation, the U is reduced to the U^{4+}, which is insoluble in such carbonate leach solutions. This reduction is accomplished by heating the solution with molecular hydrogen gas under pressure and at the same time contacting the solution with a Ni or Pt hydrogenation catalyst. (auth)

CAUSTIC 3.02.15.02

10827 M-5387
Linde Air Products Co., New York.
CAUSTIC CONSUMPTION IN PRECIPITATION OF URANIUM FROM TRICARBONATE SOLUTIONS. H. C. Givens and R. F. Koontz. Jan. 20, 1947. Decl. Mar. 16, 1957. 10p. $1.80(ph OTS); $1.80(mf OTS).

The amount of NaOH required in the precipitation of $Na_2U_2O_7$ from UO_2CO_3 was determined. (F.S.)

GENERAL 3.02.15.03

6317 AECU-4591
Ionics, Inc., Cambridge, Mass.
PROGRESS REPORT FOR PERIOD COVERING DECEMBER 17, 1959–JANUARY 15, 1960. Jan. 15, 1960. 4p. OTS.

The preparation of synthetic Th solutions of various concentration in 0.1 M H_2SO_4 is described. The precipitation of Th from these synthetic solutions by various reagents was investigated. (W.L.H.)

7470
PROCESS FOR RECOVERY OF URANIUM VALUES FROM IMPURE SOLUTIONS THEREOF. S. B. Kilner (to U. S. Atomic Energy Commission). U. S. Patent 2,911,283. Nov. 3, 1959.

A process is presented for the recovery of uranium values from impure solutions which are obtained, for example, by washing residual uranium salt or uranium metal deposits from stainless steel surfaces using an aqueous or certain acidic aqueous solutions. The solutions include uranyl and oxidized iron, chromium, nickel, and copper ions and may contain manganese, zinc, and silver ions. In accordance with one procedure, the uranyl ions are reduced to the uranous state, and the impurity ions are complexed with cyanide under acidic conditions. The solution is then treated with ammonium hydroxide or alkali metal hydroxide to precipitate uranous hydroxide away from the complexed impurity ions in the solution. Alternatively, an excess of alkali metal cyanide is added to the reduced solution until the solution becomes sufficiently alkaline for the uranous hydroxide to precipitate. An essential feature in operating the process is in maintaining the pH of the solution sufficiently acid during the complexing operation to prevent the precipitation of the impurity metal hydroxides.

PROCESS OF PURIFYING URANIUM. G. T. Seaborg, E. F. Orlemann, and L. H. Jensen (to U. S. Atomic Energy Commission). U. S. Patent 2,865,703. Dec. 23, 1958.

A method of obtaining substantially pure uranium from a uranium composition contaminated with light element impurities such as sodium, magnesium, beryllium, and the like is described. An acidic aqueous solution containing tetravalent uranium is treated with a soluble molybdate to form insoluble uranous molybdate which is removed. This material after washing is dissolved in concentrated nitric acid to obtain a uranyl nitrate solution from which highly purified uranium is obtained by extraction with ether.

RECOVERY OF IRRADIATED TH AND U 3.02

PRECIPITATION 3.02.15

GENERAL 3.02.15.03

WET FLUORIDE SEPARATION METHOD. G. T. Seaborg, J. W. Gofman, and R. W. Stoughton (to U. S. Atomic Energy Commission). U. S. Patent 2,861,866. Nov. 25, 1958.

The separation of U^{233} from thorium, protactinium, and fission products present in neutron-irradiated thorium is accomplished by dissolving the irradiated materials in aqueous nitric acid, adding either a soluble fluoride, iodate, phosphate, or oxalate to precipitate the thorium, separating the precipitate from the solution, and then precipitating uranium and protactinium by alkalizing the solution. The uranium and protactinium precipitate is removed from the solution and dissolved in nitric acid. The uranyl nitrate may then be extracted from the acid solution by means of ether, and the protactinium recovered from the aqueous phase.

CHEMICAL PURIFICATION OF URANIUM COMPOUNDS. J. C. Nevenzel (to U. S. Atomic Energy Commission). U. S. Patent 2,780,516. February 5, 1957.

A method of preparing U compounds of very high purity is described. This purity is achieved by using, in sequence, three separate purification steps. First an organic solvent extraction of $UO_2(NO_3)_2$ solution with diethyl ether is performed. The U is then extracted back into an aqueous phase and the second step, a UO_4 precipitation at low temperature is carried out. The UO_4 precipitate is heated and converted into UO_3 and this is dissolved in dilute H_2SO_4. The third step consists in adding acetone to this solution to precipitate a uranyl sulfate acetone complex. This precipitate may be heated to vaporize the acetone and obtain pure UO_2SO_4, or the precipitate may be calcined to uranium oxide. (auth)

SEPARATING URANIUM CONTAINING SOLIDS SUSPENDED IN A LIQUID. E. C. Creutz and E. P. Wigner (to U. S. Atomic Energy Commission). U. S. Patent 2,833,618. May 6, 1958.

A process is described for separating uranium-containing solids suspended in a liquid. It consists of adding to the liquid a wetting agent for the U-containing solids in an amount sufficient to form a separate liquid phase, the wetting agent having a density greater than the density of the liquid. Ethyl dibromoacetate is noted as one preferred wetting agent.

2321 BMI-JDS-182
Battelle Memorial Inst., Columbus, Ohio.
ORGANIC PRECIPITANTS AND COMPLEXING AGENTS FOR URANIUM. Quarterly Report. A. E. Bearse, G. W. Kinzer, G. A. Lutz, R. D. Morin, and R. H. Poirier. Apr. 15, 1949. Decl. Mar. 7, 1956. 38p. Contract AT-30-1-gen-202. $6.30(ph OTS); $3.00(mf OTS).

PROCESS FOR THE PRODUCTION OF AMMONIUM URANIUM FLUORIDE. A. S. Ellis and R. B. Mooney (to U. S. Atomic Energy Commission). U. S. Patent 2,650,153. Aug. 25, 1953.

This patent relates to the preparation of ammonium uranium fluoride. The process comprises adding a water soluble fluoride to an aqueous solution of a uranous compound containing an ammonium salt, and isolating the resulting precipitate.

7484
PROCESS OF RECOVERING URANIUM. S. B. Kilner (to U. S. Atomic Energy Commission). U. S. Patent 2,919,175. Dec. 29, 1959.

A method is presented for separating and recovering uranium from a complex mixture of impurities. The uranium is dissolved to produce an aqueous acidic solution including various impurities. In accordance with one method, with the uranium in the uranyl state, hydrogen cyanide is introduced into the solution to complex the impurities. Subsequently, ammonia is added to the solution to precipitate the uranium as ammonium diuranate away from the impurities in the solution. Alternatively, the uranium is precipitated by adding an alkaline metal hydroxide. In accordance with the second method, the uranium is reduced to the uranous state in the solution. The reduced solution is then treated with solid alkali metal cyanide sufficient to render the solution about 0.1 to 1.0 N in cyanide ions whereat cyanide complex ions of the metal impurities are produced and the uranium is simultaneously precipitated as uranous hydroxide. Alternatively, hydrogen cyanide may be added to the reduced solution and the uranium precipitated subsequently by adding ammonium hydroxide or an alkali metal hydroxide. Other refinements of the method are also disclosed.

Fe REMOVAL 3.02.15.04

METHOD FOR IMPROVED PRECIPITATION OF URANIUM PEROXIDE. A. J. Miller, B. M. Pitt and P. F. Grieger (to U. S. Atomic Energy Commission). U. S. Patent 2,780,515. February 5, 1957.

An improved method for the recovery of UO_4 from dilute aqueous solutions containing Fe is presented. The presence of Fe^{3+} in such solutions makes difficult the precipitation of UO_4 with H_2O_2. The Fe^{3+} catalytically decomposes the H_2O_2 precipitant, and also co-precipitates with the U, thus affecting the products' purity. The addition of malonate ion to the solutions, before the precipitation step, complexes the Fe^{3+} and prevents its decomposing the H_2O_2 and its precipitation with the U. The malonate is added as malonic acid and the pH of the solution is maintained between 1 and 3. (auth)

6996

RECOVERY OF IRRADIATED TH AND U 3.02

PRECIPITATION 3.02.15

Fe REMOVAL 3.02.15.04

SEPARATION OF THORIUM FROM RARE EARTHS WITH TANNIN. N. I. Vlodavets (Inst. of Mineralogy, Geochemistry and Crystallochemistry of Rare Elements, Academy of Sciences, U.S.S.R., Moscow). Zhur. Anal. Khim. 14, 202-6(1959) Mar.-Apr. (In Russian)

Thorium is quantitatively precipitated with tannin in a 0.005 N solution of nitric or hydrochloric acid. This permits its separation from trivalent rare earths, which are not precipitated with tannin in such relatively weak acid solutions. The accuracy of the determinations is as usual at the gravimetric determinations of macro-quantities of elements. (auth)

PROCESS FOR RECOVERY OF URANIUM FROM AQUEOUS SOLUTIONS. J. W. Gates, Jr. and L. J. Andrews (to U. S. Atomic Energy Commission). U. S. Patent 2,780,518. February 5, 1957.

A method is described for the recovery of U from aqueous solutions in which the U is accompanied by F^- The method is particularly useful in connection with organic solvent extraction of U, and is effective in neutral or acidic solutions. It consists of incorporating Al^{3+} in the solution, in order to complex the F^- present. The Al concentration should preferably be sufficient to provide an atomic ratio of Al to F^-, of one to one. (auth)

PROCESS FOR THE RECOVERY OF U IN THE PRESENCE OF IRON. A. E. Ballard (to U. S. Atomic Energy Commission). U. S. Patent 2,733,128, January 31, 1956.

A method for the quantitative recovery of U from a dilute nitrate solution containing Fe impurity is described. After concentrating to a U content of about 5%, the bulk of the Fe is removed as an easily filtered precipitate in the presence of SO_4^{2-} under controlled pH conditions of about 3.0 to 3.8.

The Fe precipitate is dissolved and the solution obtained is electrolyzed to plate out various metallic contaminants away from the U, the U containing solution from this step may be combined with the filtrate from the Fe removal step and treated to recover the U. (auth)

OXALATE – OXALIC 3.02.15.05

2507

THORIUM OXALATE–URANYL ACETATE COUPLED PROCEDURE FOR THE SEPARATION OF RADIOACTIVE MATERIALS. J. W. Gofman (to U. S. Atomic Energy Commission). U. S. Patent 2,899,452. Aug. 11, 1959.

The recovery of fission products from neutron-irradiated uranium is described. The neutron-irradiated uranium is dissolved in acid and thorium oxalate is precipitated in the solution formed, whereby the fission products are carried on the thorium oxalate. The separated thorium oxalate precipitate is then dissolved in an aqueous oxalate solution and the solution formed is acidified, limiting the excess acidity to a maximum of 2 N, whereby thorium oxalate precipitates and carries lanthanum-rare-earth- and alkaline-earth-metal fission products while the zirconium-fission-product remains in solution. This precipitate, too, is dissolved in an aqueous oxalate solution at elevated temperature, and lanthanum-rare-earth ions are added to the solution whereby lanthanum-rare-earth oxalate forms and the lanthanum-rare-earth-type and alkaline-earth-metal-type fission products are carried on the oxalate. The precipitate is separated from the solution.

SEPARATION OF THORIUM FROM TITANIUM, ZIRCONIUM AND OTHER ACCOMPANYING ELEMENTS BY HOMOGENEOUS PRECIPITATION. F. V. Zaikovskii and L. I. Gerkhardt (All-Union Inst. of Mineral Ray Materials). Zhur. Anal. Khim. 13, 513-18 (1958) Sept.-Oct. (In Russian)

It was established that the precipitation of thorium from a homogeneous solution by acetone dioxalic acid has the advantage over the usual oxalate method: a coarse-crystalline easily filtered precipitate is formed, the co-precipitation of zirconium and titanium is almost absent. Thorium oxalate can be quantitatively precipitated directly, without any additional operations, in the presence of foreign ions. A new method was developed for determining small amounts of thorium (0.002 to 3.0%) in natural materials by homogeneous precipitation with arsenazo I. (tr-auth)

Massachusetts Inst. of Tech., Watertown, Mass. Mineral Engineering Lab.
CARBONATE TREATMENT OF U_3O_8 PRECIPITATES. Gilman Y. Murray and John Dasher. June 21, 1948. Decl. Apr. 11, 1956. 24p. Contract W-7405-eng-85. $0.25(OTS).

PEROXIDE 3.02.15.06

6357

SELECTIVE SEPARATION OF URANIUM FROM THORIUM, PROTACTINIUM AND FISSION PRODUCTS BY PEROXIDE DISSOLUTION METHOD. G. T. Seaborg, J. W. Gofman, and R. W. Stoughton (to U. S. Atomic Energy Commission). U. S. Patent 2,900,228. Aug. 18, 1959.

A method is described for separating U^{233} from thorium and fission products. The separation is effected by forming a thorium–nitric acid solution of about 3 pH, adding hydrogen peroxide to precipitate uranium and thorium peroxide, treating the peroxides with sodium hydroxide to selectively precipitate the uranium peroxide, and reacting the separated solution with nitric acid to re-precipitate the uranium peroxide.

RECOVERY OF IRRADIATED TH AND U 3.02

PRECIPITATION 3.02.15

PEROXIDE 3.02.15.06

15134

FORMATION OF URANIUM PRECIPITATES. John M. Googin, Jr. (to U. S. Atomic Energy Commission). U. S. Patent 2,878,100. Mar. 17, 1959.

A method is described for precipitation of uranium peroxide from uranium-containing solutions so as to obtain larger aggregates which facilitates washing, decantation, filtration, centrifugation, and the like. The desired larger aggregate form is obtained by maintaining the pH of the solution in the approximate range of 1 to 3 and the temperature at about 25°C or below while carrying out the precipitation. Then prior to removal of the precipitate a surface active sulfonated bicarboxyacid, such as di-octyl sodium sulfo-succinate, is incorporated in an amount of the order of 0.01 to 0.05 percent by weight, and the slurry is allowed to ripen for about one-half hour at a temperature below 10°C.

PROCESS OF RECOVERING URANIUM. T. D. Price and N. M. Jeung (to U. S. Atomic Energy Commission). U. S. Patent 2,839,358. June 17, 1958.

An improved precipitation method is described for the recovery of uranium from aqueous solutions. After removal of all but small amounts of Ni or Cu, and after complexing any iron present, the uranium is separated as the peroxide by adding H_2O_2. The improvement lies in the fact that the addition of H_2O_2 and consequent precipitation are carried out at a temperature below the freezing point of the solution, so that minute crystals of solvent are present as seed crystals for the precipitation.

PRECIPITATION OF URANIUM PEROXIDE OF LOW FLUORIDE CONTENT FROM SOLUTIONS CONTAINING FLUORIDES. E. J. King and H. M. Clark (to U. S. Atomic Energy Commission). U. S. Patent 2,847,277. Aug. 12, 1958.

A method is described for the preparation of fluoride free uranium peroxide precipitates, even though the solution from which the precipitation is made is contaminated with fluorides. This is accomplished by adding aluminum ions to the solution, where they complex any fluoride present and prevent its precipitation with the uranium peroxide.

8303 NYO-7928

Aeroprojects, Inc., West Chester, Penna.

APPLICATIONS OF ULTRASONIC ENERGY. Progress Report No. 8 Covering Period from February 1, 1958 to March 31, 1958. Apr. 1958. 59p. Contract AT(30-1)-1836. $9.30(ph OTS); $3.60(mf OTS).

Precipitation of thorium oxalate carried out under ultrasonic influence was investigated using three techniques: addition of solid oxalic acid to thorium nitrate solution; mixing of solutions of oxalic acid and thorium nitrate; and mixing of solid oxalic acid and solid thorium nitrate, with subsequent addition to water under ultrasonic influence. Parameters of reagent concentration, solution temperature, rate of addition of precipitant, duration of ultrasonic treatment, and mechanical agitation are being investigated. The smallest and most uniform thorium oxalate particles to date were produced by fast addition of solid oxalic acid to thorium nitrate solution at low temperature, with short (5 to 7 minutes) ultrasonic treatment time; the particles thus produced were generally less than 1 micron diameter, with a very few as large as 2 microns. Studies were continued in the ultrasonic dissolution or disintegration of fuel-element materials in mercury. A suitable coupler material appears to be heat-treated Allegheny-Ludlum DBL-2 tool steel, which successfully withstood more than 40 hours of ultrasonic application in molten mercury with no evidence of erosion. Ultrasonic dissolution of representative materials (thorium, powdered iron compacts, and Type 304 stainless steel) in mercury at room temperature was found in preliminary tests to be significantly greater in all cases than that achieved without ultrasonic application. Ultrasonic leaching of cesium-bearing alumina waste with 0.01 N nitric acid, using batch-type treatment in a "fixed-bed" cylindrical vessel operating at a frequency of 20 kc and a low (50 watts) power level, was considerably more effective than comparable non-ultrasonic leaching. In a control test, only about 5% of the cesium was removed in 3 hours, while 35 to 50% was removed in the same time under ultrasonic influence. Investigations oriented to ultrasonic agglomeration of submicron radio-active particles in dilute gas streams, using an additive liquid aerosol to aid in coalescence, were continued. A very dilute aerosol of solid potassium chloride particles which could be analyzed by flame photometry was used as a simulant. The dense additive liquid aerosol was a non-evaporating glycerine-water mixture prepared in the optimum size range for the promotion of agglomeration. Preliminary experiments with an 8-foot-long ultrasonic agglomeration chamber through which the aerosol was drawn at low flow rate indicated order of magnitude improvement in fallout of potassium chloride particles when an ultrasonic standing-wave field was imposed upon this aerosol after a dense aerosol of non-evaporating glycerine had been mixed with it. Assembly and preliminary test of an annular agglomeration chamber, utilizing a relatively uniform, high-intensity acoustic pattern produced by a center-mounted, modified Hartmann whistle, indicated the production of annular standing waves and the possibility of highly effective agglomeration. (For preceding period see NYO-7927.) (auth)

RECOVERY OF IRRADIATED TH AND U 3.02

PRECIPITATION 3.02.15

PEROXIDE 3.02.15.06

PRECIPITATION OF FLUORIDE-FREE URANIUM TETRAOXIDE. Louis Spiegler (to U. S. Atomic Energy Commission). U. S. Patent 2,733,127, January 31, 1956.

Uranium (as a peroxide of low fluorine content) can be separated from an aqueous UO_2SO_4 solution containing both Mg^{2+} and F^-, by adding to the solution between about 0.4 and 1 mole of H_3BO_3 for each mole of UO_2SO_4, then adding sufficient H_2O_2 to convert the U to UO_4 while maintaining the solution pH between about 2 and 4 and the temperature between 30 and 65°C, and separating the UO_4 from the resulting mother liquor. (auth)

Carbide and Carbon Chemicals Corp. [K-25 Plant], Oak Ridge, Tenn.

PURIFICATION OF DEPLETED URANIUM OXIDE. J. S. Fox. Nov. 21, 1947. Changed from OFFICIAL USE ONLY Nov. 28, 1956. 25p. Contract [W-7405-eng-26]. $4.80 (ph OTS); $2.70(mf OTS).

The method used for the purification of 11.5 kg of depleted U_3O_8, is described. U_3O_8 with a purity of between 99.98 and 99.99% was obtained using a diethyl ether extraction followed by a hydrogen peroxide precipitation. — Isotopic and chemical uniformity was insured by having a solution of the entire sample in a single container before the final peroxide precipitation. The tests which led to the choice of this method of purification and a gravimetric method of analysis used as a check of the spectrographic analyses are also described. (auth)

PROCESS FOR THE RECOVERY OF URANIUM. G. O. Morris (to U. S. Atomic Energy Commission). U. S. Patent 2,711,361. June 21, 1955.

This patent relates to a process for the recovery of uranium from impure uranium tetrafluoride. The process consists essentially of the steps of dissolving the impure uranium tetrafluoride in excess dilute sulfuric acid in the presence of excess hydrogen peroxide, precipitating ammonium uranate from the solution so formed by adding an excess of aqueous ammonia, dissolving the precipitate in sulfuric acid and adding hydrogen peroxide to precipitate uranium peroxide.

Du Pont de Nemours (E. I.) & Co. Jackson Lab., Wilmington, Del.

CHAMBERS WORKS PROCESS FOR THE MANUFACTURE OF PERURANIC ACID. G. M. Richardson. Nov. 15, 1944. Decl. Mar. 18, 1957. 7p. (BP-13). $1.80(ph OTS); $1.80 (mf OTS).

Directions are given for precipitating peruranic acid by means of hydrogen peroxide from uranyl sulfate solutions which have been clarified by filtration. (F.S.)

PHOSPHATE 3.02.15.07

See also, under PROCESS CHEMISTRY AND ENGINEERING, SPECIAL PROCESSES--UAP (6.17.23).

ANALYTICAL STUDY OF THE PRECIPITATION OF TETRAVALENT URANIUM WITH SODIUM HEXAMETAPHOSPHATE. I. P. Alimarian, E. P. Nikolayeva, and G. I. Malofeeva (Lomonosov Moscow State Univ.). Zhur. Anal. Khim. 13, 464-8(1958) July-Aug. (In Russian)

Tetravalent uranium can be precipitated with sodium hexametaphosphate. The influence of different factors on the uranium precipitation was studied. It was established that uranium is precipitated completely from 3 N solution of perchloric acid. Small amounts of uranium can be completely isolated when co-precipitated with thorium. (tr-auth)

PRODUCTION OF URANIUM. R. P. Linstead, F. M. Burstall and T. V. Arden. (to U. K. Atomic Energy Authority). British Patent 759,900. Nuclear Eng. 2, 130(1957) March.

Selective precipitation of U is effected by adjustment of the pH and control of the anions and the oxidation state of the metals. To an aqueous solution containing U, Cu and/or Al is added a soluble phosphate or arsenate in quantity sufficient to combine with all the U; the pH of the solution is adjusted to 2.5 by an alkali. Where the atomic ratio of Al to U exceeds 1:1, $(NH_4)_3PO_4$ or $(NH_4)_2SO_4$ may be added in quantity sufficient to combine with all the U and Al. The pH is then adjusted by means of an alkali to 1.8 to 2.0 to precipitate U. (auth)

SODIUM HYPOPHOSPHATE AS A REAGENT FOR THE SEPARATION OF THORIUM, ZIRCONIUM AND NIOBIUM. L. Kosta and L. Ravnik (J. Stefan Inst., Ljubljana, Yugoslavia). 11p.

A very selective procedure for the separation of niobium, zirconium, and thorium from most elements as well as from each other has been devised. It is based on the precipitation of these elements with sodium hypophosphate using titanium as carrier. The precipitation from 6 M hydrochloric acid is nearly quantitative. After eliminating other interfering elements, thorium is separated in the form of fluoride. Zirconium is coprecipitated as barium zirconium fluoride from a solution in which niobium was complexed with hydrofluoric acid. Niobium is finally isolated from the hydrofluoric medium as cupferrate. A detailed description of the procedures for the determination of the isolated ions is given. A liquid-liquid extraction system using mesityl oxide as solvent for separating niobium from the hydrochloric–hydrofluoric medium is described, and data for distribution coefficients are included. The advantage of this system over others found in the literature lies in that it requires a low concentration of the hydrofluoric acid.

RECOVERY OF IRRADIATED TH AND U 3.02

PRECIPITATION 3.02.15

PHOSPHATE 3.02.15.07

SEPARATION OF SMALL AMOUNTS OF URANIUM(4) IN THE PRESENCE OF NIOBIUM AND TANTALUM BY MEANS OF A PHOSPHATE METHOD. Yu. V. Morachevskiy and I. A. Tserkovnitskaya. Vestnik. Leningrad Univ. No. 10, 152-4(1957). (Translated from Referat. Zhur., Met. No. 3, 1958).

A method was developed whereby small amounts of U can be deposited in the presence of Nb and Ta. Precipitation is carried out in tartaric acid solutions containing Nb_2O_5, Ta_2O_5, and UO_2Cl_2. The U is first reduced by an amalgam of Zn, a collector is added to it [1 cc of 2 percent solution of $Zr(NO_3)_4$], and both U and the collector are precipitated by a 10 percent solution of $(NH_4)_2HPO_4$. After dissolving the precipitate in a 10 N solution of H_2SO_4, the U is titrated with an 0.01 N solution of NH_4VO_3 with phenyltranyl acid. Final separation is achieved when the concentration of the tartaric acid is 0.5 g or less for every 100 cc of the solution. (TCO)

REDUCTION 3.02.16

See also, this section,

FUSED SALTS AND LIQUID METALS (3.02.11),

and,

under PROCESS CHEMISTRY AND ENGINEERING,

SPECIAL PROCESSES--Druhm (6.17.07),

Excer (6.17.08),

Fluorox (6.17.09),

Metallex (6.17.14),

Redox (6.17.19).

GENERAL 3.02.16.01

22508 ✓

METHOD FOR THE REDUCTION OF URANIUM COMPOUNDS. W. H. Cooke and J. W. C. Crawford (to U. S. Atomic Energy Commission). U. S. Patent 2,886,429. May 12, 1959.

An improved technique of preparing massive metallic uranium by the reaction at elevated temperature between an excess of alkali in alkaline earth metal and a uranium halide, such as uranium tetrafluoride is presented. The improvement comprises employing a reducing atmosphere of hydrogen or the like, such as coal gas, in the vessel during the reduction stage and then replacing the reducing atmosphere with argon gas prior to cooling to ambient temperature.

PREPARATION OF URANIUM COMPOUNDS. (To United Kingdom Atomic Energy Authority.) British Patent 801,380. Platinum Metals Rev. 3, 34(1959) Jan.

A trivalent uranium compound is made by reducing a salt of uranium (valency of uranium higher than 4) by treating a solution of the salt with hydrogen in the presence of a Pt, Pd, or Rh catalyst.

INFLUENCE OF TETRAVALENT VANADIUM ON THE REDUCTION OF URANIUM ON A DROPPING MERCURY ELECTRODE. Yu. V. Morachevskii and A. A. Sakharov (Zhdanov Leningrad State Univ.). Zhur. Anal. Khim. 13, 457-63(1958) July-Aug. (In Russian)

The presence of tetravalent vanadium in the solution results in increasing the limiting reduction current of hexavalent uranium. It is shown by the graphic analysis of polarographic waves that the increase of the wave is caused by the reaction of the oxidation of pentavalent uranium with tetravalent vanadium which occurs at the surface of the dropping electrode. The possibility of such a reaction is proved by amperometric titration and by studying the relation between the limiting current and temperature and hydrogen ions concentration. (tr-auth)

(See also 3.07.09.)

3000 ✓ BMI-550

Battelle Memorial Inst., Columbus, Ohio.

THE THERMODYNAMICS OF THE REDUCTION OF URANIUM COMPOUNDS TO URANIUM METAL. A. W. Lemmon, J. J. Ward, S. M. Fischer, C. J. Geankoplis, and J. W. Clegg. July 21, 1952. Decl. Oct. 6, 1955. 58p. For Catalytic Construction Co. Contract AT(30-1)-1060, Subcontract No. 459.

The thermodynamic study was undertaken to guide the evaluation of new processes for the conversions of uranium compounds to uranium metal. The objective was to show which processes might well justify experimental effort and which processes can be discarded from further consideration on the ground of thermodynamic impracticability. It was necessary to evaluate critically the available experimental thermodynamic information so that the degree of reliance in the conclusions reached may be established. Also, because of the nonexistence of certain experimental values, estimations were necessary. In all cases, these are so identified. (auth)

RECOVERY OF IRRADIATED TH AND U 3.02

REDUCTION 3.02.16

THORIUM 3.02.16.02

SOME METHODS FOR THE EXTRACTION OF THORIUM METAL FROM ITS SALTS. Harold M. Finniston. pp. 393-412 in "Proceeding of the Scientific Congress, Nuclear Sessions, July 2-6, 1956, 3rd. Electronic-Nuclear International Review." Rome, National Committee for Nuclear Research, 1956. 447p.

Three methods for the production of thorium are considered—the calcium reduction of thoria, calcium reduction of thorium fluoride, and the preparation of the metal from its chloride. The purity of the metal obtained from each process is discussed. (J.S.R.)

METHOD OF PREPARING METALS FROM THEIR HALIDES. F. H. Spedding, H. A. Wilhelm and W. H. Keller (to U. S. Atomic Energy Commission). U. S. Patent 2,782,116. February 19, 1957.

A method is given for the production of Th from its halides. The method consists in reducing ThF_4 by reaction with an alkali metal or alkali earth metal, particularly Ca. An additional feature is the admixture with the reaction mass of $ZnCl_2$. The $ZnCl_2$ is reduced by the Ca, and the Zn forms with the Th a metal mixture which melts below the melting point of the Th. The Zn can later be distilled away from the Th, allowing recovery of pure Th. (auth)

Battelle Memorial Inst., Columbus, Ohio.
STUDIES OF THE BOMB REDUCTION OF THORIUM HALIDES. H. A. Saller, J. R. Keeler, and L. J. Cuddy. Mar. 22, 1955. Decl. Mar. 1, 1957. 20p. Contract W-7405-eng-92. $0.30(OTS).

Limited studies were made of the bomb reduction of $ThCl_4$ with Ca and Mg to determine the feasibility of carrying out such reductions in the equipment presently being used for ThF_4 reduction. The bomb reduction of ThF_4 was also studied primarily to determine the feasibility of substituting Mg for all or part of the Ca reductant. Limited investigations of the ThF_4 reduction were also made to determine the effects produced by variation of the amount of $ZnCl_2$ and Ca in the charge, substitution of ZnF_2 for the $ZnCl_2$ booster, and addition of salts to the charge to reduce the viscosity of the slag. (auth)

U FLUORIDES 3.02.16.03

THE PREPARATION OF HIGH-PURITY URANIUM METAL BY THE BOMB REDUCTION OF URANIUM TETRAFLUORIDE WITH CALCIUM. R. W. Kewish and O. E. Fry (Kaiser Aluminum and Chemical Corp., Baton Rouge, La.); R. J. Bard, J. P. Bertino, S. W. Hayter, and A. W. Savage, Jr. (Los Alamos Scientific Lab., N. Mex.); F. J. Hill (Trionics Corp., Madison, Wis.); and B. L. Kelchner (Dow Chemical Corp., Rocky Flats Plant, Colo.). Trans. Met. Soc. AIME 215, 425-9(1959) June.

The preparation of massive uranium metal containing very low concentrations of a number of light elements by bomb reduction of UF_4 with calcium is described. Details of procedures are given for preparing high-purity ingredients for the bomb reduction. The as-reduced uranium metal contained, on the average, less than the following amounts of light element impurities, in ppm: Li, 0.1; Be, 0.1; B, 0.1; C, 25; O, 70; Na, 1; Mg, 3; Al, 2; and Si, 7. Other impurities for which the metal was examined averaged, in ppm: Ca, <10; V, <10; Cr, 2; Mn, 6; Fe, 50; Co, <5; Ni, 8; and Cu, 1. (auth)

THE REDUCTION OF URANIUM HEXAFLUORIDE BY CARBON TETRACHLORIDE. J. S. Nairn, D. A. Collins, and J. C. Taylor (United Kingdom Atomic Energy Authority, Cumb., Eng.). 12p.

A single stage method is described for converting UF_6 to UF . UF_6 dissolves in carbon tetrachloride at ordinary temperatures to form a reasonably stable solution. When heated to temperatures of 150°C and above, however, a rapid reaction takes place which is simply expressed as $UF_6 + CCl_4 \rightarrow UF_4 + Cl_2$ + chlorofluoromethanes. Some excess CCl_4 is required to take the reaction to completion, and a molar proportion of 4 CCl_4:1 UF_6 is satisfactory. Apart from the UF_4, the products of the reaction are volatile even at ordinary temperatures and can be removed in a stream of warm air. The UF_4 product is in a form suitable for direct reduction by calcium or magnesium to uranium metal of high quality suitable for fuel element fabrication.

1573 ✓ K-1379(Del.)
Oak Ridge Gaseous Diffusion Plant, Tenn.
DEVELOPMENT OF THE CONTINUOUS METHOD FOR THE REDUCTION OF URANIUM HEXAFLUORIDE WITH HYDROGEN—PROCESS DEVELOPMENT. HOT WALL REACTOR. S. H. Smiley and D. C. Brater. June 27, 1958. Decl. with deletions Aug. 21, 1959. 18p. OTS.

A continuous process for the reduction of uranium hexafluoride to uranium tetrafluoride was developed and proved on a pilot-plant scale. Complete conversion to uranium tetrafluoride was realized by contacting gaseous uranium hexafluoride with hydrogen in a heated, vertical, open-tube reactor. The purity and density of the solid product met metal grade uranium tetrafluoride specifications. Some difficulty with the accumulation of fused uranium fluorides in the tower was encountered, however, and it was necessary to stop and deslag the unit about every 8 to 24 hours. The reaction of uranium hexafluoride with gaseous trichloroethylene was studied before the tests with hydrogen were made. Although the reduction to uranium tetrafluoride was complete, the solid product was highly contaminated with the organic by-products of the reaction and was quite low in density. Tests of this method were discontinued when promising results were obtained with hydrogen as the reductant. (auth)

RECOVERY OF IRRADIATED TH AND U 3.02

REDUCTION 3.02.16

U FLUORIDES 3.02.16.03

Argonne National Lab., Lemont, Ill.
PRELIMINARY REPORT ON CONVERSION OF URANIUM HEXAFLUORIDE TO URANIUM DIOXIDE IN A ONE-STEP FLUID-BED PROCESS. I. E. Knudsen, N. M. Levitz, and S. Lawroski. Aug. 1959. 15p. Contract W-31-109-eng-38. $0.50(OTS).

A one-step, continuous, fluid-bed process for the conversion of uranium hexafluoride to uranium dioxide was successfully carried out in preliminary experiments in a 1½-inch-diameter Monel reactor. Hydrogen and steam were used for simultaneous reduction and pyrohydrolysis reactions carried out at 600°C, resulting in the formation of a dense coating of dioxide on the starting bed of uranium dioxide. The effects of uranium hexafluoride feed rate and reactant excesses on conversion were investigated. Results of fluid-bed steam pyrohydrolysis runs on uranium tetrafluoride are also presented. (auth)

12729

PRODUCTION OF URANIUM. (to United Kingdom Atomic Energy Authority). British Patent 810,033. Mar. 11, 1959.

Reduction of UF_4 by Mg aided by an auxiliary oxidation reaction of Mg with $KClO_3$ to provide additional heat is described. In one example, where all the heat required is supplied, 440 g of UF_4 powder (75% is −60 mesh, 60% is −80 mesh, 50% is −100 mesh, and 40% is −200 mesh) are mixed with 109 g Mg and 24.5 g $KClO_3$ and packed in a CaO-lined Fe crucible. A 300.2 g ingot of U was obtained, 90% yield. $K_2S_2O_8$ or I_2 may be substituted for $KClO_3$ in another example of the process. The charge is ignited in each case by electrically heating a U wire in the charge. (T.R.H.)

2502

URANIUM RECOVERY PROCESS. J. W. Stevenson and R. G. Werkema (to U. S. Atomic Energy Commission). U. S. Patent 2,897,048. July 28, 1959.

The recovery of uranium from magnesium fluoride slag obtained as a by-product in the production of uranium metal by the bomb reduction process is presented. Generally the recovery is accomplished by finely grinding the slag, roasting the ground slag air, and leaching the roasted slag with a hot, aqueous solution containing an excess of the sodium bicarbonate stoichiometrically required to form soluble uranium carbonate complex. The roasting is preferably carried out at between 425 and 485°C for about three hours. The leaching is preferably done at 70 to 90°C and under pressure. After leaching and filtration the uranium may be recovered from the clear leach liquor by any desired method.

PRODUCTION OF URANIUM. Archie Edwin Ruehle and John Walker Stevenson (to U. S. Atomic Energy Commission). British Patent 809,408. Feb. 25, 1959.

An improved bomb process for reduction of UF_4 to U is offered. The process differs essentially from the conventional process in that Mg is used as reductant and MgF_2 slag is used as bomb liner. The Mg is added to the UF_4 charge in a stoichiometric excess of 3 to 5% along with an additive which forms a film on the Mg to prevent undesirable and premature reactions. The additive material may be UO_3, $UF_4 \cdot 5H_2O$, UO_2F_2, or $NaHF_2$. The additive should be ~0.5 to 2.0% of the UF_4 by weight. Alternatively, the Mg may be treated with the additive prior to mixing with the UF_4. In this alternate procedure the mixture of Mg plus additive is pressed at 10,000 psi into 25 gm wafers. This reduces the firing time without impairing the yield. The MgF_2 slag for the bomb liner is ground so that 75% passes through a 100-mesh screen. The slag powder is then poured into the bomb around a rubber mandrel which is tamped to pack the liner. The rubber mandrel is then lifted out, the charge put in, and the bomb is evacuated for 3½ hours, sealed, and fired. Small metal particles found trapped in the slag are pickled in HNO_3 and recycled. When UO_3 is used as additive, MgO forms in the slag, and as the slag is re-used the MgO content increases. To recondition this slag NaF (~2.5 wt. % of UF_4) is added to the charge. The NaF forms a lower melting slag and gives a shorter reaction time. (T.R.H.)

14403

METHOD OF PRODUCING URANIUM. L. S. Foster and T. T. Magel (to U. S. Atomic Energy Commission). U. S. Patent 2,834,672. May 13, 1958.

A modified process is described for the production of uranium metal by means of a bomb reduction of UF_4. Difficulty is sometimes experienced in obtaining complete separation of the uranium from the slag when the process is carried out on a small scale, i.e., for the production of 10 grams of U or less. Complete separation may be obtained by incorporating in the reaction mixture a quantity of $MnCl_2$, so that this compound is reduced along with the UF_4. As a result a U−Mn alloy is formed which has a melting point lower than that of pure U, and consequently the metal remains molten for a longer period allowing more complete separation from the slag.

RECOVERY OF IRRADIATED TH AND U 3.02

REDUCTION 3.02.16

U FLUORIDES 3.02.16.03

REACTOR NOZZLE ASSEMBLY. F. C. Capuder and J. R. Dearwater (to U. S. Atomic Energy Commission). U. S. Patent 2,873,174. Feb. 10, 1959.

An improved nozzle assembly useful in a process for the direct reduction of uranium hexafluoride to uranium tetrafluoride by means of dissociated ammonia in a heated reaction vessel is described. The nozzle design provides for intimate mixing of the two reactants and at the same time furnishes a layer of dissociated ammonia adjacent to the interior wall of the reaction vessel, thus preventing build-up of the reaction product on the vessel wall.

14731 A/CONF.15/P/523
Oak Ridge Gaseous Diffusion Plant, Tenn.
CONTINUOUS PROCESSES FOR THE DIRECT REDUCTION OF URANIUM HEXAFLUORIDE TO URANIUM TETRAFLUORIDE OF HIGH PURITY AND DENSITY. S. H. Smiley and D. C. Brater. 11p. $0.50(OTS).

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

Contrary to the published literature, the single-step reduction of uranium hexafluoride to uranium tetrafluoride with hydrogen was found to be quantitative; furthermore, a highly efficient, continuous process, both safe and economical, was developed and adapted for large-scale production use. The uranium tetrafluoride product was of high density and purity. The basic hydrogen reduction method is actually uncomplicated; uranium hexafluoride and about 100% excess hydrogen, both preheated to approximately 500°F, are charged into the top of a vertical pipe reactor which is heated to about 1000°F, and complete reduction to uranium tetrafluoride is realized almost instantaneously. The uranium tetrafluoride, having a packed density of 220 lb/ft^3 (3.5 g/cc) and an impurity content of 30 to 50 ppm, is collected in a hopper at the bottom of the pipe. The gaseous products are passed through a metallic filter to remove entrained solids, and pure anhydrous hydrogen fluoride is recovered as a reaction by-product. The only operating difficulty noted is that slow accumulation of fused uranium fluorides, UF_5, U_2F_9, and U_4F_{17}, on the hot reactor walls necessitates periodic deslagging of the unit; however, where large quantities of low assay materials are required and relatively large equipment can be used, the loss in operating time is small. The significance of the development of the hydrogen reduction method is best realized by comparing it to existing wet processing schemes. Feasibility studies showed that the latter methods were both economically and operationally unattractive for handling large quantities of uranium hexafluoride. Bulky equipment built to withstand corrosion from aqueous hydrogen fluoride solutions, as well as expensive precipitators, filters, dryers, and converters for the required multiplicity of operation, made costs prohibitive. Some modification of the "hot wall" method was necessary to process higher assay uranium hexafluoride. Since the equipment must be small to meet criticality dimensional requirements, and opportunities for loss of uranium must be minimized, slagging of the reactor became a serious problem. It was found that the solid deposits could be eliminated entirely if fluorine was added to the uranium hexafluoride stream and the reactor walls were maintained at a low temperature; i.e., about 300°F. The highly exothermic reaction of the fluorine with hydrogen supplied the energy required to initiate and sustain the reduction, and the major portion of the reaction appeared to take place in a narrow flame directly below the gas nozzles. The solid product did not stick to the cool reactor walls, and a reliable correlation between the uranium tetrafluoride density and the amount of fluorine added was established. A process to reduce uranium hexafluoride with gaseous trichloroethylene was also developed. Complete conversion to uranium tetrafluoride was realized, but under most conditions the product was low in density and highly contaminated with the organic by-products; thus, additional treatment was required to make an acceptable material. Pure uranium tetrafluoride could be obtained directly by this method, however, if the stream containing uranium hexafluoride was very dilute, i.e., about 1% uranium hexafluoride in nitrogen. Complete reduction of uranium hexafluoride was also possible with liquid trichloroethylene in a spray column, but it was difficult to separate the solid product from the liquid. (auth)

METHOD OF FORMING CRUCIBLES AND REACTION CHAMBERS FOR PRODUCTION OF URANIUM OF HIGH PURITY. H. A. Wilhelm (to U. S. Atomic Energy Commission). U. S. Patent 2,785,064. March 12, 1957.

A method is given for forming a refractory lining for the reaction chamber or bomb in which alkali earth metal reduction of U halide is carried out. The method comprises inserting a form or mandrel into the chamber such that a uniform narrow space is left between the mandrel and chamber wall on all sides. This space is filled with anhydrous CaO powder. Instead of being tamped or compressed as it is added, the powder and reaction chamber are jolted until the powder has settled into a dense mass.

RECOVERY OF IRRADIATED TH AND U 3.02

REDUCTION 3.02.16

U FLUORIDES 3.02.16.03

National Lead Co. of Ohio, Cincinnati.
PRODUCTION OF LOW ENRICHMENT URANIUM FUEL ELEMENT CORES. F. L. Cuthbert, C. E. Crompton, J. O. Davis, and C. E. Polson. 10p. $0.50(OTS).

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

Reduction of enriched gaseous uranium hexafluoride to uranium tetrafluoride with dissociated ammonia, reduction of uranium tetrafluoride to metal with magnesium metal, vacuum casting of uranium ingots, and subsequent fabrication steps such as swaging and machining are discussed. Also presented are other aspects of production such as: the reclamation and direct recycle of enriched metallic residues by the technique of atmospheric melting under a salt cover, the handling of enriched wastes requiring chemical recovery, and the nuclear safety criteria adopted in a this production and fabrication operation to insure that

Oak Ridge National Lab., Tenn.
REMOVAL OF CARBON FROM UF_4 PELLETS BY OXIDATION. L. M. Ferris. Feb. 29, 1956. Decl. with deletions Feb. 14, 1957. 22p. Contract [W-7405-Eng-26]. $3.30 (ph OTS); $2.40(mf OTS).

All attempts were about 75% effective in removing 0.1 to 0.5 wt. % residual carbon from UF_4 pellets prepared from UO_2 by starch reduction followed by hydrofluorination. Neither dry O_2 nor dry air adequately removed the carbon even at temperatures as high as 600°C, and reaction times of up to 5 hr did not significantly increase the amount of carbon removed over that removed in 1 hr. Combustions carried out much above 400°C resulted in excessive oxidation of the UF_4. Reaction with CO_2 at 750°C did not increase the amount of carbon removed. Calculations showed that CO is the principal carbon-containing product formed from starch in the reduction of UO_2. It was concluded that the bulk of carbon removed was that on the surface of the pellets, having been deposited there when CO converts to CO_2. The remaining carbon was occluded within the pellets and was not easily accessible to a gaseous reactant.

REDUCTION OF URANIUM WITH MAGNESIUM. H. A. Wilhelm (Ames Lab., Ames, Iowa). Metal Progr. 69, No. 3, 81-8(1956) Mar.

A detailed description of the process and equipment used in U production by Mg reduction of UF_4 is presented.

PROCESS FOR PRODUCING URANIUM TETRAFLUORIDE. B. G. Harvey (to U. S. Atomic Energy Commission). U. S. Patent 2,689,165. Sept. 14, 1954.

This patent relates to improvements in the method for producing uranium tetrafluoride by treating an aqueous solution of a uranyl salt at an elevated temperature with a reducing agent effective in acid solution in the presence of hydrofluoric acid. Uranium tetrafluoride produced this way frequently contains impurities in the raw material serving as the source of uranium. Uranium tetrafluoride much less contaminated with impurities than when prepared by the above method can be prepared from materials containing such impurities by first adding a small proportion of reducing agent so as to cause a small fraction, for example 1 to 5% of the uranium tetrafluoride to be precipitated, rejecting such precipitate, and then precipitating and recovering the remainder of the uranium tetrafluoride.

Vitro Corp. of America, New York.
URANIUM RECOVERY FROM SCRAP MATERIAL (23-J). Project Summation [Covering the period] January 1951–January 1952. Job 23. June 16, 1952. Decl. with deletions Feb. 26, 1957. 63p. Contract AT(30-1)-848. $10.80(ph OTS); 3.90(mf OTS).

Methods have been investigated for the recovery of U from various C-Scrap by-product materials resulting from the production of U metal by bomb reduction of UF_4 with Mg. A process developed and demonstrated on a laboratory scale consists of roasting the materials, grinding to 200 mesh, digesting with hot HCl plus added chlorate at ph < 0.5, treating the clarified digest liquor with NH_3 by continuous precipitation procedure and roasting the washed precipitate to form black oxide. The black oxide produced by this process contains over 50% U_3O_8 and less than 0.1% F, and is free of Cl. The by-product insolubles contain less than 0.05% U_3O_8 and the waste liquor is below the U and F tolerance levels. (auth)

10844 ✓ FMPC-467
National Lead Co. of Ohio, Cincinnati.
REDUCTION OF UF_4 TO METAL CONTAINING UP TO 3% U-235. STANDARD OPERATING PROCEDURE FOR PILOT PLANT, METALLURGICAL AREA (SECTION 1.5.2.3). S. L. Reese. Sept. 1, 1954. Decl. Feb. 23, 1957. 13p. Contract AT(30-1)-1156. $0.25(OTS).

A detailed step-by-step description of the plant standard operating procedure is given. (B.J.H.)

RECOVERY OF IRRADIATED TH AND U 3.02

REDUCTION 3.02.16

U FLUORIDES 3.02.16.03

Gt. Brit. Culcheth Labs., Culcheth, Lancs, England. THE MAGNESIUM REDUCTION OF URANIUM TETRAFLUORIDE. W. G. O'Driscoll and E. Calvert. May 1, 1953. 28p. (SFPWP/P-5).

Results obtained for the magnesium reduction of UF_4 in pressurized systems and for reductions by magnesium vapor have indicated that the reduction is initiated as a UF_4 solid/Mg vapor reaction. A solid/solid reaction appears to be possible but this is limited in extent by the true contact area of the reactants. The refractory oxides, lime, alumina, magnesia, thoria, zirconia, and beryllia appear to be unsuitable as reactor lining materials, owing to their reactivity with UF_4 at high temperatures. A proposed mechanism for the reduction is described and recommendations based on it are suggested for the operation of the reduction on a production basis. (auth)

U OXIDES 3.02.16.04

ACTIVATED URANIUM DIOXIDE AND PROCESSES OF PRODUCING THE SAME. (to United Kingdom Atomic Energy Authority). British Patent 812,791. Apr. 29, 1959.

Reduction of UO_3 to UO_2 by CH_4 gas at 400 to 500°C results in UO_2 sufficiently active to oxidize in ordinary air at room temperature. This active UO_2 is readily chlorinated. (T.R.H.)

PRODUCTION OF URANIUM AND PLUTONIUM. R. H. Myers (to U. K. Atomic Energy Authority). British Patent 792,951. Nuclear Eng. 3, 466(1958) Oct.

Metal of high purity, suitable for conversion into shapes without the need for casting, is obtained by a process in which a mixture of a powdered oxide of the metal and particulate alkaline earth metal is heated to produce a reaction product of beads of the uranium or plutonium metal, dispersed in a mass of alkaline earth metal oxide. This is leached with water and/or aqueous nitric acid of 20% concentration. The metal can be compacted and sintered, in vacuo or in a protective atmosphere.

PRODUCTION OF URANIUM. R. H. Myers (to U. K. Atomic Energy Authority). British Patent 792,952. Nuclear Eng. 3, 466(1958) Oct.

When magnesium is used in the process described in B.P. 792,951, for the reduction of uranium dioxide, the metal produced tends to be pyrophoric. This can be avoided by including, in the mixture of uranium dioxide and magnesium, a proportion of alkaline earth metal chloride, afterwards leaching with dilute mineral, or acetic acid.

PRODUCTION OF URANIUM METAL. J. H. Buddery (to U. K. Atomic Energy Authority). British Patent 792,953. Nuclear Eng. 3, 466(1958) Oct.

A modification of B.P. 792,952, uranium dioxide and magnesium metal mixed with magnesium chloride in certain proportions, is heated in a non-reactive vessel to 1200°C in an inert atmosphere. This temperature is maintained for at least one hour to promote growth of the uranium particles, then the reaction product is leached with dilute aqueous acid, the uranium particles are washed and vacuum freeze dried.

3296 AEC-tr-2393
[PERUVIAN CHEMIST SEPARATES U^{235} FROM U^{238}. Report of work of Angel Grayson Fossa.] Translated from La Cronica (1955) Aug. 25-26. 5p.

Uranium isotopes U^{235} have been separated from U^{238} by flotation techniques utilizing mass differences, molecular forces, and osmotic pressures. The uranium solutions were converted to uranoso uranic oxides, which were subsequently reduced to metallic U by reaction with Zn at high temperature. (News report.) (C.W.H.)

12429 NYO-3800
Metal Hydrides Inc. Chemical Research Lab., Beverly, Mass.
SECOND ANNUAL REPORT ON THE PREPARATION OF URANIUM HYDRIDE. Period Covered: July 1, 1953 through June 30, 1954. Sidney Johnson. Aug. 20, 1954. Decl. Feb. 26, 1957. 37p. Contract AT(30-1)-1350. $0.40(OTS).

Uranium metal of density 18.55 g/cm³, or greater than 97% of the theoretical value, has been prepared by hot-pressing UH_3 powder obtained from the direct reduction of UO_2 with CaH_2, and leaching the resulting furnace product with aqueous acid. Massive metal of better quality could not be produced by the process employed because of the interaction between the UH_3 and the aqueous leaching media. Although the initial reaction rate of UH_3 with methanol is smaller by an order of magnitude than the reaction rate of UH_3 with H_2O, methanolic HCl did not prove to be satisfactory leaching medium. Optimum furnacing conditions for the reduction have been determined. Analytical methods for evaluating UH_3 powders were perfected during the course of the investigation. These methods include: (1) direct determination of O_2 in U and UH_3 by reaction with BrF_3; (2) densities of U pellets produced by a small scale hot-pressing technique from UH_3 powders; and (3) U oxide assays by dissolution of UH_3 in a measured excess of $Ce(HSO_4)_4$ solution. (auth)

RECOVERY OF IRRADIATED TH AND U 3.02

REDUCTION 3.02.16

U OXIDES 3.02.16.04

PRODUCTION OF HIGH PURITY URANIUM POWDER.
R. G. Bellamy and J. H. Buddery (A.E.R.E., Harwell). Nuclear Power 2, 175-9(1957) May.

Glove box methods used at Harwell for reduction of uranium oxide with calcium or magnesium are described. Different techniques for large and small scale operations are included. The efficiency and purity of the product are stressed. (M.H.R.)

Metal Hydrides Inc., Beverly, Mass.
REDUCTION OF URANIUM OXIDES. QUARTERLY REPORT NO. 4. Sidney Johnson. Oct. 19, 1953. Decl. Feb. 27, 1957. 14p. Contract AT(30-1)-1350. $0.25(OTS).

Means of reduction of uranium oxides to uranium hydride powder were further studied. On the basis of previous experience, emphasis was upon the reduction of UO_2 by CaH_2, which not only has given some of the best results, but also is considered to be the reaction most readily adaptable to a continuous process. Three methods of evaluation which are considered the most promising are being pursued. These are: (1) a U assay method whereby the UH_3 powder is dissolved directly in a measured excess of $CeSO_4$; (2) the density of U metal prepared by hot-pressing UH_3 powder; and (3) a direct determination of oxygen by displacing this element with BrF_3. In addition, the A. E. C. New Brunswick Laboratories agreed to do a limited number of oxygen determinations by the vacuum fusion technique on U metal produced in this laboratory. It is certain that one or more of these methods will be suitable for a complete evaluation of UH_3 powder. (auth)

Metal Hydrides, Inc. Chemical Research Lab., Beverly, Mass.
SECOND QUARTERLY REPORT ON THE REDUCTIONS OF URANIUM OXIDES. Robert W. Bragdon. Jan. 10, 1953. Decl. Mar. 28, 1957. 15p. Contract AT(30-1)-1350. $3.30(ph OTS); $2.40(mf OTS).

The progress on a program designed to evaluate methods of preparing uranium hydride from the various oxides of uranium is presented. The current portion of the investigation is concerned with the reduction of UO_2, UO_3, and U_3O_8 with calcium hydride in an atmosphere of H_2, and three such reductions are described. Chemical analyses of the products indicate that the reductions are incomplete under the specific conditions employed in these three runs. These conditions are the simplest and are not those which can be considered optimum. An apparently satisfactory standard laboratory leach method for the removal of the by-products has been evaluated, using a synthetic furnace product, and this method is currently being employed in the isolation of actual products. The evaluation of these products was seriously hindered by the difficulty in obtaining a sufficiently pure argon atmosphere for the dry box. The passage of tank argon over titanium sponge at 850 to 900°C has effectively solved this problem. (auth)

Mallinckrodt Chemical Co., St. Louis.
PRODUCTION OF URANIUM METAL FROM URANIUM TETRAFLUORIDE MADE FROM URANIUM TRIOXIDES PRODUCED AT HARSHAW CHEMICAL CO. W. H. Keller. Dec. 27, 1951. Decl. Mar. 2, 1957. 8p. $0.25(OTS).

Uranium(VI) oxide produced batchwise in the thermal decomposition pots was reduced to UO_2, hydrofluorinated to UF_4, reduced with Mg to metal, and cast. This product produced satisfactory intermediates, high metal reduction yields (average 98.2%), and good quality metal in casting. A second type of UO_3 produced on a drum dryer was converted to metal by a similar procedure. This UO_3 was light in weight and of a porous flaky structure. The UF_4 was of low density and the yields in the metal reduction step were considerably lower. (W.L.H.)

Mallinckrodt Chemical Works, St. Louis.
A STUDY OF THE USE OF AMMONIA GAS TO REDUCE URANIUM TRIOXIDE TO URANIUM DIOXIDE. R. W. Shearer. June 12, 1945. Decl. Mar. 8, 1957. 16p. $3.30 (ph OTS); $2.40(mf OTS).

Results of experiments studying the use of NH_3 as a reducing agent in converting UO_3 to UO_2 indicate that cold, raw ammonia gas can be used in the reduction boxes in place of cracked ammonia gas and will reduce the trioxide to dioxide assay of 96.0% or better (gravimetric analysis). Indications are that it will take more raw NH_3 (roughly 20% more) than cracked NH_3 to obtain a given assay. There was no definitely distinguishable difference in reactivity between the UO_3 powder and milled cake. (J.E.D.)

Mallinckrodt Chemical Works, St. Louis.
THE REDUCTION OF ORANGE OXIDE [UO_3] TO BROWN OXIDE [UO_2] BY MEANS OF ARTIFICIAL AND NATURAL GAS. Jan. 6, 1944. Decl. Mar. 28, 1957. 5p. $1.80(ph OTS); $1.80(mf OTS).

It was found that at 800°C, natural and city gas (the latter is a blend of coke-oven and natural gas) will reduce orange oxide to brown and give a product of the desired assay. At temperatures below 700°C, the reduction is slow and is not complete. At 800°C, heavy deposits of carbon formed during this reduction on the metal reaction chamber. The flaking-off of these deposits and the resulting contamination of the brown oxide is sufficient to render the use of gas impractical in the present factory equipment. (auth)

RECOVERY OF IRRADIATED TH AND U 3.02

REDUCTION 3.02.16

URANYLS 3.02.16.05

PRECIPITATION OF URANIUM TETRAFLUORIDE FROM AQUEOUS SOLUTION BY CATALYTIC REDUCTION. R. J. Allen, H. G. Petrow, and P. J. Magno (National Lead Co., Winchester, Mass.). Ind. Eng. Chem. 50, 1748-9(1958) Dec.

While attempting to produce dense, high-purity uranium tetrafluoride from uraniferous ores, a catalytic process was discovered for the reduction of uranyl ion in the presence of fluoride to yield UF_4. Most of the research was conducted with solutions prepared from sulfuric acid leach liquors of uraniferous ores. The solvent extraction process used was essentially the Amex process developed at Oak Ridge, although extensive changes were made in the stripping circuit to accommodate the use of hydrochloric acid. Hydrofluoric acid was added to the concentrated uranium solution, generally in slight excess of the stoichiometric requirement, and various sbustances were examined as possible uranium reductants. Among the reducing agents studied were stannous chloride, cuprous chloride, chromous chloride, titanous chloride, and sulfur dioxide. All but sulfur dioxide were effective reducing agents. The salts obtained were all identified as $UF_4 \cdot {}^3/_4 H_2O$. The quantitative recovery of uranium as pure, dense UF_4 was demonstrated, and at significant cost savings over present techniques. (J.H.M.)

USE OF RONGALITE IN PRODUCTION OF LOW SOLUBILITY TETRAVALENT URANIUM DERIVATIVES. A. A. Greenberg, et al (Leningrad Radium Inst.). Zhur. Analit. Khim. 12. 92-4(1957) Jan.-Feb. (In Russian)

The reduction of uranyl salts by rongalite is described, and the advantages over the reduction with hydrosulfite are shown. The tetravalent uranium has been quantitatively isolated from the solution of oxalic or hydrofluoric acid, and the optimum concentration conditions for U isolation as an oxalate are given. (tr-auth)

Princeton Univ., N. J. Frick Chemical Lab.
THE REDUCTION OF URANYL SOLUTIONS BY SATURATED LIQUID ZINC AMALGAM. N. H. Furman, W. B. Mason, and J. S. Pekola. May 1947. Decl. Oct. 6, 1955. 10p. Contract W-7405-Eng-81. $3.30(ph OTS); $2.40 (mf OTS).

The reduction of uranyl solutions by a saturated Zn – Hg amalgam is accomplished without interference from Ni. Special apparatus, procedures, and conditions are described. (T.R.H.)

6405 ✓ A/CONF.15/P/2067
EKSPERIMENTAL'NYE ISSLEDOVANIYA USLOVII PERENOSSA I OTLOZHENIYA URANA GIDROTERMAL'NYMI RASTVORAMI. (The Experimental Investigation of the Conditions of Uranium Transport and Deposition by Hydrothermal Solutions). R. P. Rafal'skii (U.S.S.R.). 34p.

The investigations were carried out in accordance with the up-to-date concept on the transport of hexavalent uranium under hydrothermal conditions and its precipitation as a result of reduction. The study of the stability of sodium uranyl tricarbonate showed that it is decomposed with precipitation of uranates at a temperature of about 100°C. It has been found that hexavalent uranium is reduced by hydrogen sulfide both in acid sulfate and carbonate solutions at elevated temperatures. In carbonate solutions uranium is reduced even at room temperature. In this case the reaction proceeds in a neutral or even slightly acid medium in the presence of H_2S and CO_2. In excess of H_2S the reaction results practically in complete reduction of hexavalent uranium. At an elevated temperature and pressure uranium is reduced and precipitated from uranyl sulfate solutions by pyrite, chalcopyrite, galena, siderite, hornblende, or organic components of carbonaceous shales. The end product of the reduction is usually crystalline uraninite. The data obtained indicate the possibility of hexavalent uranium being transported by hydrothermal solutions and deposited as a result of reduction process. At the same time the values of solubility of freshly precipitated uranium dioxide at elevated temperatures determined experimentally make it possible to assume that under hydrothermal conditions uranium could migrate in its tetravalent form as well. (auth)

6531 ✓ A/CONF.15/P/1608
A METHOD FOR THE REDUCTION OF URANIUM IN PHOSPHORIC ACID. A. Flaschenberg, J. Lavi, and J. Tulipman (Israel Atomic Energy Commission). 11p.

A new method for the reduction of uranium in phosphoric acid solution was developed and applied on a modest industrial scale. The method employs a galvanic system, an electrode couple having a potential difference of the order of 0.5v, and is used in 40% phosphoric acid. An iron electrode, short-circuited with stainless steel or a copper alloy, is placed in the acid solution against an inert platinum or stainless steel electrode. The speed of the reduction and the slope of the reduction curve depend only on the surface areas of the iron and the stainless steel, per unit volume of acid. In comparison with the method in which the uranium is reduced by dissolving iron, the galvanic reduction permits a considerable saving of iron. (auth)

RECOVERY OF IRRADIATED TH AND U 3.02

SOLVENT EXTRACTION 3.02.17

See also, under PROCESS CHEMISTRY AND ENGINEERING, SOLVENT EXTRACTION (6.16).

GENERAL 3.02.17.01

10977 ✓ CF-58-7-129
Oak Ridge National Lab., Tenn.
SOLVENT EXTRACTION APPLIED TO REACTOR FUEL PROCESSING; FIRST QUARTER CO-OP REPORT.
W. E. Dunn. July 14, 1958. 12p. Contract [W-7405-eng-26]. $3.30(ph), $2.40(mf) OTS.

The solvent extraction of thorium with 42.5% TBP in a Decalin diluent was studied in a compound pulse column. The HETS for the extraction section of the column was 4.7 feet. A discussion of the method of graphical calculations of a compound column is included. (auth)

20918 ✓
METHYLCYCLOHEXANON—NEW SOLVENT FOR THE EXTRACTION OF URANIUM AND FOR THE SEPARATION OF URANIUM FROM THORIUM. K. Seidl and M. Beranek. Chem. listy 52, 337-9(1958). (Translated from Referat. Zhur. Khim. No. 24, 1958, Abstract No. 82208.)

The separation of U is achieved from the water solution without the addition or in the presence of $NaCO_3$ and HNO_3. It was established that the best salting-out reagent is $NaNO_3$, while in the presence of HNO_3 the separation is impaired. The re-extraction of a U salt from methylcyclohexanone with the aid of a saturated caustic solution yielded 99.9% of the original quantity. Advantages of methylcyclohexanone, compared to other reagents employed are listed. (auth)

SOLVENT EXTRACTION PROCESSES FOR ENRICHED URANIUM. C. E. Stevenson. May 1, 1957. p.152-79 of SYMPOSIUM ON THE REPROCESSING OF IRRADIATED FUELS HELD AT BRUSSELS, BELGIUM, MAY 20-5, 1957. 1165p. (TID-7534(Bks. 1-3))

Solvent extraction processes for enriched U are similar to the Purex and Redox processes for natural U except that Pu separation and recovery is not attempted, and the processes generally must provide for the separation of diluent metals in the fuels. Several processes are described for Al, Zr, and stainless steel fuels, and design considerations and typical results are given.

Dow Chemical Co. Western Div., Pittsburg, Calif.
PROGRESS REPORT [FOR] JULY–AUGUST 1956.
Research Dept. R. H. Bailes. Sept. 1, 1956. 28p. Contract AT-30-1-GEN-236. $4.80(ph OTS); $2.70(mf OTS).

Investigations of the possibility of production of high grade U 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)

Technical Information Service Extension, AEC.
SOLVENT EXTRACTION OF URANIUM. A Literature Search. Richard J. Smith, comp. Mar. 1957. 22p. (TID-3502).

A list of 159 references to solvent, or liquid-liquid extraction of U with emphasis on production processes is presented.

Oak Ridge National Lab., Tenn.
URANIUM CHEMISTRY OF RAW MATERIALS SECTION PROGRESS REPORT [FOR] JULY 1, 1952 TO SEPTEMBER 30, 1952. PART II. STUDIES IN URANIUM CHEMISTRY: SOLVENT EXTRACTION. C. A. Blake, J. G. Moore, and W. M. Whaley. Dec. 6, 1952. Decl. May 29, 1956. 68p. Contract W-7405-eng-26. $10.80(ph OTS); $3.90(mf OTS).

Work on U chemistry of raw materials included investigations of the solvent extraction of U from aqueous nitrate, sulfate, phosphate, and fluoride solutions and mixtures through use of organic complexing agents, especially organic P compounds. The screening program for organic P compounds was continued. A new phosphinic acid, di(3-heptyl)-, two new phosphonic acids, 2-ethylhexyl- and 3-heptyl-, and a mixture of mono- and didecylphosphoric acids were prepared. Extraction of U from nitrate, phosphate, and sulfate solutions by these materials are reported. Tri(2-ethylhexyl) phosphine oxide gave good extractions of U. Di(2-ethylhexyl)phosphoric acid showed promise as an extractant for western mill liquors. Use of this reagent in processing a Rifle-type liquor gave high-grade U. Extractions with 2-ethylhexylphosphonic acid from nitrate, phosphate, sulfate, and fluoride solutions are reported. The solubility and distribution of di(2-ethylhexyl)phosphinic acid in water and acid solutions were determined. Several amines were tested for extracting U(VI) from sulfate and sulfate–phosphate liquors. Studies were made of several methods for stripping U from organic solvents. Stripping of U-bearing organic phase with aqueous carbonate or hydroxide solutions is discussed. Detailed description is presented of the preparation of various intermediates, phosphinic acids, phosphonic acids, phosphoric esters, amines, and a phosphine oxide. (See ORNL-1384 for preceding quarter.) (W.L.H.)

RECOVERY OF IRRADIATED TH AND U 3.02

SOLVENT EXTRACTION 3.02.17

GENERAL 3.02.17.01

6346 A/CONF.15/P/474
EXTRACTION OF THORIUM BY A MIXTURE OF ETHYL ACETATE AND ACETONE. Milutin Andjelković and Dragica Rajković (Inst. for Tech. of Mineral Raw Materials, Belgrade). 10p.

A method for extraction of thorium by a mixture of ethyl acetate and acetone, applicable to a wide range of thorium concentrations, was worked out. For this process quantities from 13 to 53 micrograms of thorium were used. The behavior of various cations and anions was examined, and a system for avoiding difficulties caused by zirconium was worked out. The method for determination of thorium is particularly efficient for materials with a low thorium content, using extraction by a mixture of ethyl acetate and acetone as separation method and the Thomason's colorimetric method for final determination. As the material contains larger quantities of thorium, the final determination can be done by the gravimetric method. Besides thorium, only uranium, zirconium, and bismuth were extracted among all the elements analyzed. To avoid difficulties caused by zirconium, the authors exploited its property to form complexes with tartaric acid which are not extracted. Uranium is extracted quantitatively, but during the determination of thorium by thorin it can be present in twenty times greater amounts. Bismuth does not interfere with the final determination. The method is simple, sufficiently rapid, and applicable to routine work. From materials with a complex composition, microgram quantities of thorium can be determined with an error usually below ± 5%. This method has been used for more than one year and has given satisfactory results. (auth)

CARBONATE SYSTEMS 3.02.17.02

URANIUM RECOVERY. F. T. Fitch and A. J. Cruikshank (to U. S. Atomic Energy Commission). U. S. Patent 2,858,187. Oct. 28, 1958.

A process for recovering uranium from a solution of a diethyl dithiocarbamate of uranium in an organic solvent substantially immiscible with water is presented. The process comprises bringing the organic solution into intimate contact with an aqueous solution of ammonium carbonate, whereby the uranium passes to the aqueous carbonate solution as a soluble uranyl carbonate.

252 CF-59-8-45
Oak Ridge National Lab., Tenn.
CHEMICAL TECHNOLOGY DIVISION, CHEMICAL DEVELOPMENT, SECTION C, MONTHLY PROGRESS REPORT, AUGUST 1959. K. B. Brown, K. A. Allen, C. F. Coleman, D. J. Crouse, and A. D. Ryon. Sept. 8, 1959. 37p. Contract [W-7405-eng-26]. OTS.

The solvent extraction of U from carbonate solutions was studied. The prevention of silica stabilized emulsions in the Amex and Dapex processes by solvent-continuous mixing is reported. The recovery of U and Pu from H_2SO_4 decladding solutions by continuous countercurrent extraction with primary amines, HNO_3 stripping from primary amine, U^{6+} extraction by primary amine, and U extraction by dialkylphosphoric acid is being studied. Plutonium was effectively freed from Am contamination by extraction from 2 M HNO_3 with 0.1 M di(2-ethylhexyl)phosphoric acid. (For preceding period see CF-59-7-68). (W.L.H.)

CHLORIDE SYSTEMS 3.02.17.03

THE RATES OF THE ELECTRON EXCHANGE REACTION BETWEEN U(IV) AND U(VI) IONS IN WATER, ETHANOL AND WATER–ETHANOL SOLVENTS. Daniel M. Mathews, Jack D. Hefley, and Edward S. Amis (Univ. of Arkansas, Fayetteville). J. Phys. Chem. 63, 1236–40(1959) Aug.

The rate of the electron exchange reaction between U(IV) and U(VI) ions was studied at 25° in water, ethanol and water–ethanol solvents and at various concentrations of U(IV), U(VI), and hydrochloric acid. The order of the reaction with respect to each of these substances was found to depend strongly on the composition of the solvent. Mechanistic and kinetic expressions are suggested which can account for these orders in solvents up to 60 vol.% ethanol. (auth)

EXTRACTION OF URANIUM TETRACHLORIDE BY TRIBUTYL PHOSPHATE. Eiichi Iwase and Toshiaki Isono. Sci. Papers Inst. Phys. Chem. Research (Tokyo) 53, 13-16(1959) Mar.

The partition coefficient of uranium tetrachloride between 20 volume percent TBP–kerosene and aqueous phases has been determined at 25°C in the presence of either hydrochloric acid, sodium chloride, magnesium chloride, or aluminum chloride. For various amounts of hydrochloric acid, the partition equilibrium of uranium tetrachloride has been measured between organic and aqueous phases. From this measurement it may be concluded that a compound $UCl_4 \cdot 3TBP$ is formed in TBP–kerosene solution. (auth)

RECOVERY OF IRRADIATED TH AND U 3.02

SOLVENT EXTRACTION 3.02.17

CHLORIDE SYSTEMS 3.02.17.03

EXTRACTION OF URANYL CHLORIDE BY TRI-n-OCTYLAMINE. Jean Bizot and Bernard Trémillon (Ecole de Physique et de Chimie, Laboratorie de Chimie Analytique, Paris). Bull. soc. chim. France No. 1, 122-6(1959) Jan. (In French)

The long-chained aliphatic amines are among the organic substances used to extract mineral compounds. The extraction of uranyl chloride with tri-n-octylamine in carbon tetrachloride was studied quantitatively. In the presence of hydrochloric acid the trioctylamine forms a hydrochloride soluble in organic solvent, with the result that the hydrochloric acid is in competition in the extraction of the uranyl salt. The compound extracted has the formula $(R_3NHCl)_2(UO_2Cl_2)$, where R_3N designates the trioctylamine. The extraction equilibrium constants were determined, and the law of mass action was verified. It is only in strongly acid medium that the uranyl chloride can be quantitatively extracted. (tr-auth)

(See also 6.15.05.01)

EXTRACTION OF PERCHLORIC ACID AND URANYL PERCHLORATE WITH TRIBUTYL PHOSPHATE. V. B. Shevchenko, I. V. Shilin, and A. S. Solovkin. Zhur. Neorg. Khim. 3, 225-30(1958). (In Russian)

The distribution of perchloric acid between its aqueous solution and TBP in the solvent extraction of uranyl perchlorate was studied. It was shown that the transition of the uranyl perchlorate and the perchloric acid to TBP is determined by the ionic strength of the water phase, the TBP concentration in the organic phase, the nature of the diluent, and the salting-out properties of the alkali metal ion. It was found that the graphic method of determination of the composition of the complex compounds extracted by the TBP was inapplicable in the case of uranyl perchlorate and perchloric acid. (J.S.R.)

Oak Ridge National Lab., Tenn.
LIQUID-LIQUID EXTRACTION OF URANIUM AND PLUTONIUM FROM HYDROCHLORIC ACID SOLUTION WITH TRI(ISO-OCTYL) AMINE; SEPARATION OF URANIUM AND PLUTONIUM FROM THORIUM AND FISSION PRODUCTS. Fletcher L. Moore. June 18, 1957 21p. Contract [W-7405-eng-26]. $4.80(ph OTS); $2.70 (mf OTS).

A new and rapid method for the liquid-liquid extraction of U and Pu from HCl solution is based on the use of tri(iso-octyl)amine dissolved in xylene or methylisobutylketone. Uranium and/or Pu are separated from Th, alkalis, alkaline earths, rare earths, Zr, Nb, Ru, and other elements which do not form anionic species under the conditions described. The technique may be used for either tracer or macro quantities of U. Several practical applications of the method for the separations chemist are proposed. (auth)

4370

THE MUTUAL SEPARATION OF THORIUM, PROTOACTINIUM, AND URANIUM BY TRIBUTYL PHOSPHATE EXTRACTION FROM HYDROCHLORIC ACID. D. F. Peppard, G. W. Mason, and M. V. Gergel (Argonne National Lab., Lemont, Ill.). J. inorg. and Nuclear Chem. 3, 370-8(1957) Jan.

The distribution behavior of U(VI) and Pa(V) in the system aqueous HCl–TBP (tributyl phosphate) without diluent has been investigated at 22 ± 2°C and the results compared with those reported for Th(IV) in the same system. From the comparison, it is postulated that protoactinium extracts respectively as oxygenated and nonoxygenated entities in the regions of low and high concentration of HCl, the transition range being 4–5 M HCl in the aqueous phase. Although the data indicate the feasibility of mutual separation of Th(IV), Pa(V), and U(VI) in the HCl–TBP system, the separation of Pa(V) from U(VI) is much more readily achieved in the (HCl,HF)–TBP system; since the complexing action of fluoride ion is much more pronounced for Pa(V) than for

6543 A/CONF.15/P/2412

PARTITION OF URANIUM AND THORIUM BETWEEN SOLUTIONS OF NITRIC AND PERCHLORIC ACIDS AND FOUR ORGANIC SOLVENTS. M. Branica and E. Bona (Inst. Ruder Bošković, Zagreb). 12p.

The extraction of uranyl and thorium ion by cyclic ethers is described. The extraction procedures with cyclic ethers are analogous to those of alcohols, ketones, and ethers, where salting agents are required. Extraction equilibria of uranyl and thorium nitrates and perchlorates were determined. The partition was investigated by tetrahydrosylvane (THS), tetrahydropyrane (THP), 2-ethyl-tetrahydrofurane (E-THF), and 2,5-dimethyl-tetrahydrofurane (M-THS) in various concentrations of nitric and perchloric acid. (auth)

RECOVERY OF IRRADIATED TH AND U 3.02

SOLVENT EXTRACTION 3.02.17

CHLORIDE SYSTEMS 3.02.17.03

1000

INTERRELATIONSHIPS IN THE SOLVENT EXTRACTION BEHAVIOUR OF SCANDIUM, THORIUM, AND ZIRCONIUM IN CERTAIN TRIBUTYL PHOSPHATE-MINERAL ACID SYSTEMS. D. F. Peppard, G. W. Mason, and J. L. Maier (Argonne National Lab., Lemont, Ill.). J. Inorg. and Nuclear Chem. 3, 215-28(1956) Oct.

The extraction behavior of Sc and that of Th in the aqueous HCl-TBP (tributyl phosphate) system differ sufficiently that these elements are readily separable. Although the respective extraction curves tend to converge as the HCl concentration is lowered, they do not intersect. In contrast, the been determined, but the Zr extraction curve in the HNO_3-TBP system is shown to cross both the Sc and Th curves. Consequently, in this system, any one of the three elements may be separated as product from the other two as contaminants. In the HCl-TBP system, the TBP dependency of the extraction coefficients for Sc and Th are not readily interpretable. In the HNO_3-TBP system the TBP dependencies are operationally fourth-power at high HNO_3 concentrations and considerably less than fourth-power at intermediate and low HNO_3 concentrations. The mutual separation of a Th-Sc mixture and of a Th-Zr mixture in the HCl-TBP system and the isolation of each of the three elements as product from a Sc-Th-Zr mixture in the HNO_3-TBP system are demonstrated. (auth)

Sc and Th extraction curves in the HNO_3-TBP systems mutually cross. The complete Zr extraction curves have not

NITRATE SYSTEMS 3.02.17.04

See also, under PROCESS CHEMISTRY AND ENGINEERING,

SPECIAL PROCESSES--Darex (6.17.04),
Metal Recovery (6.17.15),
Purex (6.17.17),
Redox (6.17.19),
25-TBP (6.17.21),
Thorex (6.17.22).

NITRATE SYSTEMS 3.02.17.04

1587

SOLVENT EXTRACTION WITH THE DI-ISOAMYL ESTER OF METHYL-PHOSPHINIC ACID. V. B. Shevchenko, I. G. Slepchenko, and V. S. Shmidt. Atomnaya Energ. 7, 236-43 (1959) Sept. (In Russian)

Solvent extraction of uranium with the di-isoamyl ester of methyl-phosphinic acid (DAMPA) is described. A high efficiency for extraction from nitric acid solutions is achieved. Certain regularities in extraction by DAMPA-hydrated kerosene systems are studied. The uranyl nitrate is extracted in the form $[UO_2(NO_3)_2\ (DAMPA)_2]$ with a stability constant 2540 ± 200; HNO_3 is extracted as HNP_3 DAMPA with a stability of 0.30 ± 0.03. (R.V.J.)

22159

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 anine 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.

RECOVERY OF IRRADIATED TH AND U 3.02

SOLVENT EXTRACTION 3.02.17

NITRATE SYSTEMS – dibutyl carbitol 3.02.17.04.01

RECOVERING URANIUM FROM UNIRRADIATED FUEL ELEMENT SCRAP. G. R. Jasny, J. R. Barkman, T. P. Sprague, and R. P. Smith (Union Carbide Nuclear Co., Oak Ridge, Tenn.). Ind. Eng. Chem. 50, 1777-80(1958) Dec.

The following information is presented on the recovery of unirradiated fuel element scrap: solution techniques for U–Zr, U–stainless steel, and U–Al alloy scrap and the recovery of enriched uranium by dibutyl carbitol extraction, with 99.9% over-all efficiency. A summary flowsheet of the over-all enriched uranium scrap dissolving and purification process is shown. Schematics are included of the caustic dissolution process used on U–Al alloy scrap and the dibutyl carbitol process used to remove and purify uranium from various uranium alloy dissolver solutions. (J.H.M.)

7452 Y-1282
Union Carbide Nuclear Co. Y-12 Plant, Oak Ridge, Tenn.
RECOVERY OF URANIUM FROM URANIUM–ZIRCONIUM FUEL SCRAP. T. P. Sprague. Jan. 20, 1958. 31p. Contract W-7405-eng-26. OTS.

Procedures were modified and test runs made for the dissolution of zirconium–uranium alloy scrap and the separation of the uranium from the zirconium by dibutyl carbitol extraction. Uranium containing less than 40 ppm Zr/U_3O_8 can be consistently obtained at increased uranium throughputs. (auth)

2373 AECD-3855
Tennessee Eastman Corp., Oak Ridge, Tenn.
THE ALUMINUM NITRATE PROCESS FOR THE CONVERSION OF TUBALLOY TETRAFLUORIDE [UF_4] TO TUBALLOY PEROXIDE [UO_4]. E. J. Lord, L. J. Andrews, and J. W. Gates, Jr. Aug. 22, 1945. Decl. Dec. 21, 1955. 2p. Contract W-7401-eng-23. (C-1.365.4). $1.80(ph OTS); $1.80(mf OTS).

Quantitative conversion of UF_4 to UO_4 is achieved by dissolution in boiling $Al(NO_3)$ solution and extraction of U from the cool, $Al(NO_3)_3$ saturated, solution with dibutyl carbitol. (T.R.H.)

NITRATE SYSTEMS – ethers 3.02.17.04.02

20008
METHOD OF SEPARATING URANIUM. (to UK Atomic Energy Authority (Canada)). British Patent 805,001. Nuclear Eng. 4, 324(1959) July-Aug.-Sept.

The irradiated uranium metal is dissolved in concentrated nitric acid. The acidity is closely controlled and must not be above stoichiometric neutral in order to avoid extraction of plutonium together with the uranium. A salting out agent such as ammonium nitrate is then added to the solution and extraction is obtained by treating this solution with an organic solvent, preferably ethyl ether, which is sufficiently water-immiscible to form a separate phase and permit extraction of the uranyl nitrate without extracting the plutonium or other fission products at the same time. Details are given of a typical application resulting in 96.9% extraction of the uranyl nitrate with only 1.3% of plutonium and 0.09% of the fission products present in the original solution.

10988
SEPARATION OF URANIUM FROM THORIUM. U. K. Atomic Energy Authority (U.S.A.). British Patent 800,791. Nuclear Eng. 4, 188(1959) Apr.

On exposure of thorium to slow neutrons a small amount of uranium is formed in the irradiated material. From the irradiation product an aqueous solution is prepared of thorium and uranium nitrates, the proportion of nitrate: water ranging from 0.7:1 to 1.4:1 and the proportion of uranium: thorium not exceeding 1:50. This solution is treated with diethyl ether resulting in preferential extraction of the uranium.

RECOVERY OF IRRADIATED TH AND U 3.02

SOLVENT EXTRACTION 3.02.17

NITRATE SYSTEMS – ethers 3.02.17.04.02

13369

EXTRACTION OF URANYL NITRATE FROM THE WATER–NITRIC ACID–AMMONIUM NITRATE SYSTEM WITH DIETHYLETHER. J. Čepelák, J. Malý, and V. Macháček (Tschechoslowakische Akademie der Wissenschaften, Prague). Collection Czechoslov. Chem. Communs. 23, 1509-14(1958) Aug. (In German)

The equilibrium distribution of uranyl nitrate between aqueous nitric acid and diethyl ether in the presence of ammonium nitrate was determined. In the extraction of uranyl nitrate from 1N and 5N nitric acid, a different distribution between both phases was observed. On the basis of these data, the absorption spectra of both phases and the melting point curve of the aqueous phase, it was assumed that in the extraction of uranyl nitrate with diethyl ether from 5N HNO_3 the uranium in the aqueous phase is present as $U_2O_5^{2+}$ and in the organic phase as a complex with the $UO_2(NO_3)_3$ ion. (tr-auth)

5135

ON THE DISTRIBUTION OF URANYL NITRATES BETWEEN AQUEOUS SOLUTIONS AND SOME SIMPLE AND COMPLEX ETHERS. V. M. Vdovenko and E. A. Smirnova Radiokhimiya 1, 43-51(1959). (In Russian)

The distribution of uranyl nitrate between aqueous solutions and a series of simple and complex ethers follows the formulas $UO_2(NO_3)_2 \cdot hH_2O \rightleftharpoons UO_2^{2+} + 2NO^- + hH_2O$ and $\log m\gamma a_w^{h/3} = \frac{1}{3} \log K_1 + \frac{1}{3} \log m_\mu$. The equilibrium constants, activity factors, and molar volumes were determined. (R.V.J.)

URANIUM SEPARATION PROCESS. R. M. Hainer and E. C. Evers (to U. S. Atomic Energy Commission). U. S. Patent 2,780,532. February 5, 1957.

A process is described for separating U from aqueous solutions in which it is accompanied by other metals, chiefly Fe, Ni and Cu. A dialkyl ether of a polyethylene glycol is used as an extracting agent after large quantities of NO_3^- have been added to the solution to affect salting out of $UO_2(NO_3)_2$. Under these conditions it has been found that the organic phase will extract approximately 98% of the U but will not extract appreciable amounts of any of the other metals present. $Ca(NO_3)_2$ and $NH_4(NO_3)$ are mentioned as being effective salting out agents. (auth)

CHEMICAL PURIFICATION OF URANIUM COMPOUNDS. J. C. Nevenzel (to U. S. Atomic Energy Commission). U. S. Patent 2,780,516. February 5, 1957.

A method of preparing U compounds of very high purity is described. This purity is achieved by using, in sequence, three separate purification steps. First an organic solvent extraction of $UO_2(NO_3)_2$ solution with diethyl ether is performed. The U is then extracted back into an aqueous phase and the second step, a UO_4 precipitation at low temperature is carried out. The UO_4 precipitate is heated and converted into UO_3 and this is dissolved in dilute H_2SO_4. The third step consists in adding acetone to this solution to precipitate a uranyl sulfate acetone complex. This precipitate may be heated to vaporize the acetone and obtain pure UO_2SO_4, or the precipitate may be calcined to uranium oxide. (auth)

EXTRACTION OF URANYL NITRATE BY DIETHYL ETHER FROM WATER–NITRIC ACID–AMMONIUM NITRATE SOLUTIONS. Josef Cepelák, Jaromír Malý, and Vojtech Machácek. Chem. listy 51, No. 2, 2195-9 (1957). (Translated from Referat. Zhur. Khim. No. 22, 1958, Abstract No. 73264.)

The equilibrium distribution of uranyl nitrate (I) between aqueous HNO_3 solution and diethyl ether (II) in the presence of NH_4NO_3 was established. The distribution was different at HNO_3 concentrations of 1 and 5 N. From the study of the equilibrium of absorption spectra of the two phases and of the course of the curve of the liquid-phase melting, it is assumed that at the extraction of I with II from 5 N HNO_3, uranium is in the aqueous phase as $U_2O_5^{2+}$, and in the organic phase it is as a complex with a $UO_2(NO_3)_3^-$ ion. The exact equation of the equilibrium reaction on the dividing interface of both the phases could not be determined because of the difficulty in the determination of HNO_3 bound with II and with I in the organic phase.

RECOVERY OF IRRADIATED TH AND U 3.02

SOLVENT EXTRACTION 3.02.17

NITRATE SYSTEMS – ethers 3.02.17.04.02

1584 JEN-3
Spain. Junta de Energia Nuclear, Madrid.
SEPARACION DE NITRATO DE URANILO DE HIERRO(III), TITANO(IV) Y ALUMINIO(III) POR EXTRACCIÓN CON ETER. (Separation of the Uranyl Nitrates of Iron(III), Titanium(IV), and Aluminum(III) by Extraction with Ether). R. Fernandez Cellini and F. de la Cruz Castillo. 1956. 19p.

The theoretical and experimental data on the extraction of uranyl nitrate with ether, factors which affect the process, and analytical methods developed using this method are discussed. A modification to the extraction methods published is described and applied to the separation of uranyl nitrate from iron, titanium, and aluminum. The solvent partition of uranyl nitrate in the ether is determined. The method is applicable to the determination of uranium in minerals and other materials. (tr-auth)

1326
EXTRACTION OF URANYL NITRATE WITH ETHER FROM AQUEOUS SOLUTIONS IN A SPRAY COLUMN. F. Oltra Oltra, A. Pérez Luiña, and L. Gutiérrez Jodra. Anales real soc. españ. fís. y quim. (Madrid), Ser. B 51, 581-91 (1955) Sept.-Oct. (In Spanish)

The extraction of uranyl nitrate by diethylether from aqueous solutions in a spray column has been studied. The direction of extraction is opposite that one used before. The variables studied are the rates of flow of the phases and continuous phase. The transfer coefficients have been calculated and correlations of these constants are given. A comparison with previous results and a discussion of the values obtained are included. (auth)

2324 A-2710
Purdue Univ., Lafayette, Ind.
THE SEPARATION OF URANIUM FROM OTHER ELEMENTS BY THE ETHYL ETHER EXTRACTION OF A NITRATE SOLUTION. A. M. Ribley, C. V. St. John, H. Hunt, T. DeVries, and M. G. Mellon. Mar. 8, 1946. Decl. Dec. 8, 1955. 9p. Contract W-7405-eng-74. $1.80(ph OTS); $1.80(mf OTS).

A method is described for extracting U from a nitrate solution with ethyl ether. The sample is evaporated to the point of incipient crystal formation, then the concentrate is transferred to the extractor using a solution ¾ saturated with $Ca(NO_3)_2$ and 5 to 10% HNO_3 by volume. Small amounts of other ions are also extracted. The amount of Fe extracted is diminished by removal of chloride ion from the solution before extraction. The amounts of Cr and V extracted are decreased by treatment of the sample with Na bisulfite prior to extraction. At least 99.8% of the U is extracted under the conditions described. (auth)

9756 CN-2491
[Chicago. Univ. Metallurgical Lab.]
EQUILIBRIUM DISTRIBUTION AND PHYSICAL PROPERTIES FOR THE SYSTEM URANYL NITRATE–DIETHYL ETHER–WATER. Progress Report. Marion Monet. Dec. 18, 1944. Decl. Feb. 14, 1956. 36p. Contract W-7401-eng-37. $6.30(ph OTS); $3.00(mf OTS).

Physical and chemical properties and a critical evaluation of the equilibrium distribution of the uranyl nitrate–diethyl ether–water system are given. (F.S.)

2480
SEPARATION OF URANIUM FROM THORIUM. N. N. Hellman (to U. S. Atomic Energy Commission). U. S. Patent 2,893,823. July 7, 1959.

A process is presented for separating uranium from thorium wherein the ratio of thorium to uranium is between 100 to 10,000. According to the invention the thorium-uranium mixture is dissolved in nitric acid, and the solution is prepared so as to obtain the desired concentration within a critical range of from 4 to 8 N with regard to the total nitrate due to thorium nitrate, with or without nitric acid or any nitrate salting out agent. The solution is then contacted with an ether, such as diethyl ether, whereby uranium is extracted into the organic phase while thorium remains in the aqueous phase.

RECOVERY OF IRRADIATED TH AND U 3.02

SOLVENT EXTRACTION 3.02.17

NITRATE SYSTEMS – general 3.02.17.04.03

See also, under PROCESS CHEMISTRY AND ENGINEERING,

SOLVENT EXTRACTION--Surveys and Theory (6.16.06).

11606 LS-19

Israel. Atomic Energy Commission, Tel-Aviv. LITERATURE SURVEY ON THE ELEMENTS: CERIUM, EUROPIUM, MOLYBDENUM, PROMETHIUM, TECHNETIUM, ZIRCONIUM. 1. THEIR CHEMISTRY IN NITRIC ACID SOLUTIONS. 2. THEIR VOLATILE COMPOUNDS AND VOLATILIZATION METHODS. Oct. 1958 21p.

This survey includes 148 references from the report and published literature on the elements Ce, Eu, Mo, Pm, Tc, and Zr. Chemical Abstracts, Jan. 1923 through Sept. 1958, and Nuclear Science Abstracts, Jan. 1948 through Aug. 1958, were the sources. (J.E.D.)

20931

THERMODYNAMICS FOR THE EXTRACTION OF URANYL NITRATE AND NITRIC ACID BY ESTERS OF THE TYPES $(RO)_3P = O$ AND $(RO)_2RP = O$. T. H. Siddall, III (E. I. du Pont de Nemours & Co., Aiken, S.C.). J. Am. Chem. Soc. 81, 4176-80(1959) Aug. 20.

The thermodynamic quantities, ΔH, ΔF and ΔS, were obtained for the extraction of uranyl nitrate and of nitric acid by twenty-one compounds of the types $(RO)_3P = O$ and $(RO)_2RP = O$. The values of ΔS suggest that a very open structure is retained by the extractant molecules when they are bound to uranyl nitrate. The strength of the bonds formed with the uranyl ion is substantially altered as substituents on the phosphorus atom are replaced by various radicals. A partial correlation with Kharasch's electronegativity scale was obtained for the substituents. When ΔF for the extraction of uranyl nitrate was plotted against ΔF for the extraction of nitric acid, a straight line was obtained. However, the fit of the data to the straight line is only approximate. Evidence was obtained that an extra order–disorder transition accompanies extraction of uranyl nitrate by certain extractants, but not with others. (auth)

22168

SEPARATION OF URANIUM FROM THORIUM AND PROTACTINIUM. W. K. R. Musgrave (to U. S. Atomic Energy Commission). U. S. Patent 2,892,677. June 30, 1959.

This patent relates to the separation of uranium from thorium and protactinium; such mixtures of elements usually being obtained by neutron irradiation of thorium. The method of separating the constituents has been first to dissolve the mixture of elements in concentrated nitric acid and then to remove the protactinium by absorption on manganese dioxide and the uranium by solvent extraction with ether. Prior to now, comparatively large amounts of thorium were extracted with the uranium. According to the invention this is completely prevented by adding sodium diethyldithiocarbamate to the mixture of soluble nitrate salts. The organic salt has the effect of reacting only with the uranyl nitrate to form the corresponding uranyl salt which can then be selectively extracted from the mixture with amyl acetate.

SOLVENT EXTRACTION OF URANIUM VALUES. H. M. Feder, M. Ader, and L. E. Ross (to U. S. Atomic Energy Commission). U. S. Patent 2,872,285. Feb. 3, 1959.

A process is presented for extracting uranium salt from aqueous acidic solutions by organic solvent extraction. It consists in contacting the uranium bearing solution with a water immiscible dialkylacetamide having at least 8 carbon atoms in the molecule. Mentioned as a preferred extractant is dibutylacetamide. The organic solvent is usually used with a diluent such as kerosene or CCl_4.

METHYL CYCLOHEXANONE AS SOLVENT FOR THE EXTRACTION OF URANIUM AS WELL AS FOR THE SEPARATION OF URANIUM FROM THORIUM. K. Seidl and M. Beránek (Technische Hochschule für Chemie, Prague). Collection Czechoslov. Chem. Communs. 24, 298-301(1959) Jan. (In German)

The characteristics of a solvent for the extraction of uranium from thorium salts are described, and methyl cyclohexanone was investigated as a possible solvent. The distribution coefficients of uranyl nitrate and thorium nitrate between 2-methyl cyclohexanone and water or aqueous solution of various salting out reagents were determined. The results showed that the solvent extracts uranium from a solution with sodium nitrate as salting out agent with a yield of 99.9%, but that thorium is not extracted at all. (J.S.R.)

RECOVERY OF IRRADIATED TH AND U 3.02

SOLVENT EXTRACTION 3.02.17

NITRATE SYSTEMS – general 3.02.17.04.03

14412
U-EXTRACTION—IMPROVEMENTS IN ELIMINATION OF Mo BY USE OF FERRIC ION. H. M. Clark and D. Duffey (to U. S. Atomic Energy Commission). U. S. Patent 2,838,367. June 10, 1958.

An improved solvent extraction process is described whereby U may be extracted by a water immiscible organic solvent from an aqueous solution of uranyl nitrate. It has been found that Mo in the presence of phosphate ions appears to form a complex with the phosphate which extracts along with the U. This extraction of Mo may be suppressed by providing ferric ion in the solution prior to the extraction step. The ferric ion is preferably provided in the form of ferric nitrate.

THE MECHANISM OF MASS TRANSFER OF SOLUTES ACROSS LIQUID-LIQUID INTERFACES. III. THE TRANSFER OF URANYL NITRATE BETWEEN SOLVENT AND AQUEOUS PHASES. J. B. Lewis (Atomic Energy Research Establishment, Harwell, Berks, Eng.). Chem. Eng. Sci. 8, 295-308(1958) June.

Over-all transfer coefficients were determined for the transfer of uranyl nitrate between water and the three solvents dibutoxy diethyl ether (D.B.C.), methyl iso-butyl ketone (hexone), and a 20 per cent solution of tri n-butyl phosphate (T.B.P.) in odorless kerosene (O.K.), benzene, or carbon tetrachloride. In some experiments with D.B.C. the aqueous phase was 3N nitric acid in equilibrium with D.B.C. at 1.5N in nitric acid. Some systems gave larger transfer rates than predicted, and in all of these interfacial turbulence was observed. In the other systems the initial values of the calculated and observed coefficients were usually in good agreement, but the observed rate tended to diminish with time apparently due to the building up of an interfacial barrier. The formation of this barrier appears to depend solely on the duration of the experiment, being independent of uranyl nitrate concentration and all other experimental variables. The transfer of nitric acid alone between water and D.B.C. was investigated. No interfacial resistance was found when the transfer was from water to D.B.C., the transfer in the other direction was accelerated by interfacial turbulence. (auth)

12518 ✓ CEA-tr-R-618
DIAGRAMMES DE SOLUBILITE DES NITRATES D'URANYLE ET DE THORIUM AVEC LES RELARGUANTS. (Solubility Diagrams of Uranyl and Thorium Nitrates with Salting out Agents.) A. G. Kurnakova and A. V. Nikolaev. Translated into French from Zhur. Neorg. Khim. 3, 1028-36(1958). 22p.

This paper was previously abstracted from the original language and appears in NSA, Vol. 12, abstract No. 15345.

THE ABSORPTION SPECTRA OF URANYL NITRATE IN SOME ORGANIC SOLVENTS. B. Jezowska-Trzebiatowska and A. Bartecki (Wrocław Univ., Poland and Polish Academy of Sciences). Bull. acad. polon. sci. Sér. sci. Chim. géol. et géograph. 6, 567-74(1958).

The absorption spectra of uranyl nitrate in acetone, methyl ethyl ketone, methyl isobutyl ketone, acetylacetone, 1,4-dioxace, acetonitrile, and formamide were investigated within the visible and near ultra-violet ranges of 360 to 500 mμ, using a Zeisa universal spectrophotometer. For sake of comparison the spectrum of uranyl nitrate was also made in water. The absorption data are discussed and tabulated. (J.H.M.)

HYDRATION OF URANYL NITRATE IN ORGANIC SOLVENTS. H. A. C. McKay (Atomic Energy Research Establishment, Harwell, Didcot, Berks). J. Inorg. and Nuclear Chem. 4, 375-6(1957)

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.

RECOVERY OF IRRADIATED TH AND U 3.02

SOLVENT EXTRACTION 3.02.17

NITRATE SYSTEMS – general 3.02.17.04.03

16093 SRS-94
Gt. Brit. Springfields Works, Springfields, Lancs, England.
THE SOLVENT EXTRACTION OF URANYL NITRATE.
H. A. C. McKay. Feb. 15, 1954. 6p.

A general review of work on the solvent extraction of uranyl nitrate is presented. (J.E.D.)

2333 UCRL-764
California. Univ., Berkeley. Radiation Lab.
THE DISTRIBUTION OF TRACE URANIUM(VI) IN NITRIC ACID—TTA—ORGANIC SOLVENT MIXTURES. D. L. Heisig and H. W. Crandall. June 30, 1950. Decl. Dec. 8, 1955. 31p. Contract W-7405-eng-48. $4.80(ph OTS); $2.70(mf OTS).

The distribution of U^{6+} in systems of HNO_3, TTA, and hexone, cyclohexanone, or pentaether has been shown to depend upon three specific reactions. The values of the equilibrium constants in pentaether and cyclohexanone when activity effects are neglected have been estimated at various nitric acid concentrations. These constants can be used to calculate the distribution coefficient of U^{6+} with an accuracy of 15% in pentaether–HNO_3 mixtures containing up to 1.42M total HNO_3 and 0.6M TTA and in cyclohexanone–HNO_3 mixtures containing up to 1.0M total HNO_3 and 0.5M TTA. (auth)

8807 M-1048
Brown Univ., Providence.
INVESTIGATIONS ON THE DISTRIBUTION OF $RO_2(NO_3)_2$ [$UO_2(NO_3)_2$] BETWEEN H_2O AND ORGANIC SOLVENTS. Monthly Report No. 8. Charles A. Kraus. May 24, 1944. Decl. Jan. 27, 1956. 4p. (BM-8). $1.80(ph OTS); $1.80(mf OTS).

The distribution of $UO_2(NO_3)_2$ between diethyl cellosolve and H_2O was investigated. The addition of salts (e.g., NH_3NO_3, $Ca(NO_3)_2$, and $Cu(NO_3)_2$ caused the distribution to shift in favor of the organic solvent. (C.W.H.)

8748
PHYSICAL PROPERTIES OF THE URANYL NITRATE, ALUMINIUM NITRATE, NITRIC ACID AND WATER SYSTEM. C. M. Slansky (Phillips Petroleum Co., Idaho Falls, Idaho). pp. 535-43 in "Progress in Nuclear Energy. Series III. Process Chemistry. Volume 2."

Physical properties are summarized for aqueous and organic solutions pertinent to several major solvent-extraction recovery systems for nuclear fuel. The data include freezing points, densities, viscosity, pH, mutual solubilities, boiling points, specific heats, refractive index, and uranium extraction coefficients. (auth)

NITRATE SYSTEMS – methyl isobutyl ketone 3.02.17.04.04

See also, under PROCESS CHEMISTRY AND ENGINEERING, SPECIAL PROCESSES--Redox (6.17.19).

URANIUM SEPARATION PROCESS. William H. McVey and William H. Reas (to U. S. Atomic Energy Commission). U. S. Patent 2,877,087. Mar. 10, 1959.

The separation of uranium from an aqueous solution containing a water soluble uranyl salt is described. The process involves adding an alkali thiocyanate to the aqueous solution, contacting the resulting solution with methyl isobutyl ketone and separating the resulting aqueous and organic phase. The uranium is extracted in the organic phase as $UO_2(SCN)_2$.

SEPARATION OF URANIUM FROM DIVERSE IONS. METHYL ISOBUTYL KETONE LIQUID-LIQUID EXTRACTION SYSTEM. William J. Maeck, Glenn L. Booman, Maxine C. Elliott, and James E. Rein (Phillips Petroleum Co., Idaho Falls, Idaho). Anal. Chem. 30, 1902-7(1958) Dec.

A liquid-liquid extraction procedure for the separation of greater than 99.8% of uranium(VI) from complex mixtures with one-batch contact is described. It is based on the extraction of tetrapropylammonium uranyl trinitrate with methyl isobutyl ketone from an acid-deficient aluminum nitrate solution. Because 1-year-cooled fission products extract less than 1%, this procedure is especially suitable for separation of uranium from reactor fuel element reprocessing solutions prior to its analytical determination. Per cent extraction for 49 cations at mole ratios of 10 to 1, 1 to 1, and 0.1 to 1 to uranium have been determined. The effects of 30 anions on the extraction of both uranium and fission products have been established. (auth)

RECOVERY OF IRRADIATED TH AND U 3.02

SOLVENT EXTRACTION 3.02.17

NITRATE SYSTEMS – methyl isobutyl ketone 3.02.17.04.04

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)

7482
URANIUM DECONTAMINATION. J. S. Buckingham and J. L. Carroll (to U. S. Atomic Energy Commission). U. S. Patent 2,918,350. Dec. 22, 1959.

A process is described for reducing the extractability of ruthenium, zirconium, and niobium values into hexone contained in an aqueous nitric acid uranium-containing solution. The solution is made acid-deficient, heated to between 55 and 70°C, and at that temperature a water-soluble inorganic thiosulfate is added. By this, a precipitate is formed which carries the bulk of the ruthenium, and the remainder of the ruthenium as well as the zirconium and niobium are converted to a hexone-nonextractable form. The ruthenium-containing precipitate can either be removed from the solution or it can be dissolved as a hexone-non-extractable compound by the addition of sodium dichromate prior to hexone extraction.

EXTRACTION OF URANYL NITRATE WITH ORGANIC SOLVENTS. A. Cacciari, R. De Leone, C. Fizzotti, and M. Gabaglio (Laboratori CISE, Milan). Energia nucleare (Milan) 3, 176-81(1956) June 15. (In Italian)

A study was made of the influence of the "salting" and of the presence of HNO_3 on the extraction of uranyl nitrate with methyl isobutyl ketone. In the same conditions, the extraction of the ferric nitrate was estimated. A determination was also made of the effect of the presence in the extraction solutions of sulfates, phosphates and chlorides. The results are reported in graphs which show the variation of the distribution coefficient in each case examined. (tr-auth)

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)

2496
URANIUM SEPARATION PROCESS. E. K. Hyde, L. I. Katzin, and M. J. Wolf (to U. S. Atomic Energy Commission). U. S. Patent 2,894,827. July 14, 1959.

The separation of uranium from a mixture of uranium and thorium by organic solvent extraction from an aqueous solution is described. The uranium is separated from an aqueous mixture of uranium and thorium nitrates 3 N in nitric acid and containing salting out agents such as ammonium nitrate, so as to bring the total nitrate ion concentration to a maximum of about 8 N by contacting the mixture with an immiscible aliphatic oxygen containing organic solvent such as diethyl carbinol, hexone, n-amyl acetate and the like. The uranium values may be recovered from the organic phase by back extraction with water.

2479
SEPARATION OF URANIUM FROM OTHER METALS. H. H. Hyman (to U. S. Atomic Energy Commission). U. S. Patent 2,893,822. July 7, 1959.

The separation of uranium from other elements, such as ruthenium, zirconium, niobium, cerium, and other rare earth metals is described. According to the invention, this is accomplished by adding hydrazine to an acid aqueous solution containing salts of uranium, preferably hexavalent uranium, and then treating the mixture with a substantially water immiscible ketone, such as hexone. A reaction takes place between the ketone and the hydrazine whereby a complex, a ketazine, is formed; this complex has a greater power of extraction for uranium than the ketone by itself. When contaminating elements are present, they substantially remain in the aqueous solution.

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2500
SEPARATION OF THORIUM FROM URANIUM BY EXTRACTION. E. G. Bohlmann (to U. S. Atomic Energy Commission). U. S. Patent 2,897,046. July 28, 1959.

A method is presented for the recovery and separation of uranium and thorium values contained in an aqueous nitric acid solution which is more than 3 M in nitric acid. The uranium and thorium containing solution preferable about 7 M in nitric acid is contacted with tributyl phosphate-kerosene mixture. Both U and Th are extracted by the immiscible organic. After phase separation the Th is selectively back extracted by contacting with an aqueous nitric acid solution preferably between 0.1 to 1.5 M in nitric acid. The uranium which is still in the organic extractant phase may be recovered by contacting with water.

1489
EXTRACTION OF THORIUM FROM AQUEOUS SOLUTION. Arnold Audsley and Walter Ryan (to United Kingdom Atomic Energy Authority). British Patent 821,973. Oct. 14, 1959.

A method of extracting Th from Fe-containing aqueous solutions is described. The Th is extracted into an amine phase such as 2-amino-2:4:4-trimethylpentane with kerosene diluent. The Th can then be separated into an HNO_3 solution. (T.R.H.)

22047
SOLVENT EXTRACTION OF THORIUM VALUES FROM AQUEOUS SOLUTIONS. J. C. Warf (to U. S. Atomic Energy Commission). U. S. Patent 2,883,264. Apr. 21, 1959.

The separation of thorium values from rare earth metals contained in aqueous solutions by means of extraction with a water immiscible alkyl phosphate diluted with a hydrocarbon such as hexane is described. While the extraction according to this invention may be carried out from any aqueous salt solution, it is preferred to use solutions containing free mineral acid. Hydrochloric acid and in particular nitric acid are suitable in a concentration ranging from 0.1 to 7 normal. The higher acid concentration results in higher extraction values.

Rio de Janeiro. Centro Brasileiro de Pesquisas Físicas.
DETERMINATION OF THE STABILITY CONSTANTS OF THORIUM NITRATE COMPLEXES WITH ANION-EXCHANGE RESINS. J. Danon. 1959. 16p. (Notas de Física Vol. V, No. 6.)

The determination of successive stability constants of thorium nitrate complexes by solvent extraction methods was investigated. (J.E.D.)

1123 ORNL-2627
Oak Ridge National Lab., Tenn.
THE USE OF TRI-n-OCTYLPHOSPHINE OXIDE IN THE SOLVENT EXTRACTION OF THORIUM FROM ACIDIC SOLUTIONS. W. J. Ross and J. C. White. Nov. 20, 1958. 33p. Contract W-7405-eng-26. $1.00(OTS).

Thorium is readily extracted by a solution of tri-n-octylphosphine oxide in cyclohexane from either acidic nitrate or chloride solutions. The maximum extraction coefficient in a nitrate medium is 380 as compared to 1300 for a chloride solution. In nitrate media the extraction coefficient is relatively unaffected by changes in the anion concentration. In chloride media the coefficient varies directly with increasing chloride concentration, i.e., from about 0.4 in one molar chloride to the maximum in 7 to 10 molar. The acid concentration should be at least one molar. Thorium is not appreciably extracted from sulfate solutions. The extraction coef-

~~11501~~
SOLVENT EXTRACTION PROCESS FOR PURIFICATION OF THORIUM. F. H. Spedding and A. Kant (to U. S. Atomic Energy Commission). U. S. Patent 2,796,320. June 18, 1957.

The separation and purification of Th are described. This purification of Th can be made by extracting an aqueous Th salt solution by means of a liquid organic solvent. Satisfactory organic solvents include ethers, esters, ketones, alcohols and alkyl phosphates. The extraction process also calls for the use of very water-soluble nitrate or chloride as a salting-out agent. (auth)

~~11125~~
TRI-n-BUTYL PHOSPHATE AS AN EXTRACTING SOLVENT FOR INORGANIC NITRATES. IV. THORIUM NITRATE. E. Hesford, H. A. C. McKay, and D. Scargill (Atomic Energy Research Establishment, Harwell, Didcot, Berks). J. Inorg. and Nuclear Chem. 4, 321-5(1957).

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Thorium nitrate is more readily extracted by tri-n-butyl phosphate (TBP) than are the rare earths, but it is less extractable than $Pu(NO_3)_4$ by a factor of about 10. It extracts as a di-solvate, $Th(NO_3)_4 \cdot 2TBP$, according to solubility measurements and to partition data at varying TBP concentrations. It extracts more readily from slightly acidified $NaNO_3$ than from HNO_3 of similar concentration, but if the pH rises above about 3 the partition coefficient falls again owing to Th hydrolysis. (auth)

90
THE RELATIONS OF THORIUM NITRATE AND CERTAIN ORGANIC SOLVENTS. THE COÖRDINATION NUMBER OF Th^{4+}. Leonard I. Katzin, John R. Ferraro, W. W. Wendlandt, and R. L. McBeth (Argonne National Lab., Lemont, Ill.). J. Am. Chem. Soc. 78, 5139-44(1956) Oct. 20.

At constant H_2O and $Th(NO_3)_4$ activities (saturated aqueous $Th(NO_3)_4$, the $Th(NO_3)_4$ dissolving in isobutyl alcohol, methyl isobutyl ketone, diethyl ether, ethylene glycol diethyl ether and diethylene glycol diethyl ether is accompanied by 6 to 7 moles of water, and this ratio is not greatly affected by dilution with inert solvent such as carbon tetrachloride. With the dibutyl ethers of ethylene glycol and diethylene glycol the water-thorium ratio is 5. These are consistent with coördination number 8 for the Th^{4+} cation, nitrate ions being partially displaced from the coördination sphere. With tributyl phosphate and dibutyl butylphosphonate the thorium appears essentially without accompanying water, and with 2 to 3 molecules of organic phosphate per thorium. It is demonstrated that the solute is monodisperse, and that there is probably a single bond between the organic component and thorium, which leads to a coördination number less than 8, probably 6. (auth)

EXTRACTION OF THORIUM NITRATE FROM NITRIC ACID BY TBP—"ULTRASENE." Thomas H. Siddall, III. Oct. 1956. 22p. (DP-181)

Equilibrium data are presented for the distribution of $Th(NO_3)_4$ and HNO_3 between aqueous solutions and TBP diluted with kerosene.

4453 AERE-Lib/Trans-802(Papers 1&2)
PAPER 1. DETERMINATION OF STABILITY CONSTANTS OF $Th(NO_3)_x^{4-x}$ IONS BY MEANS OF TRIBUTYL PHOSPHATE EXTRACTION. V. V. Fomin and E. P. Maiorova. Translated by S. Botcharsky (U.K.A.E.A. Atomic Energy Research Establishment) from Zhur. Neorg. Khim. 1, 1703-12(1956). PAPER 2. DETERMINATION OF STABILITY CONSTANTS $Th(NO_3)_j^{4-j}$ IONS. Ibid., 2749-52. 19p.

The distribution of nitric acid between an aqueous solution and TBP in benzene and the distribution of trace amounts of thorium between the aqueous solution of nitric acid and TBP in benzene were investigated. The nitric acid distribution was determined so that known conditions could be effected for the experiments to determine the composition and stability of thorium nitrate. To verify the constants previously established, experiments on extraction of weighable amounts of thorium from solutions of a given strength in equilibrium solutions were carried out. (J.R.D.)

12359 V BMI-955
Battelle Memorial Inst., Columbus, Ohio.
REFINING OF THORIUM BY SOLVENT EXTRACTION. R. A. Ewing, S. J. Kiehl, Jr., and A. E. Bearse. Oct. 19, 1954. Decl. Mar. 1, 1957. 70p. Contract W-7405-eng-92. $0.50(OTS).

Thorium extractions up to 99.99 + % were obtained by extraction of mantle-grade $Th(NO_3)_4$ with 30% TBP-70% Solvesso 100, using nitric acid salting. Rare earths were undetectable in the purified thorium. Data are presented on the re-extraction of thorium from organic extracts containing from 0.1N to 0.4N free HNO_3. Critical thorium concentrations for phase separation have been determined at 25°C for TBP-kerosene mixtures containing from 10 to 50% TBP; the concentration is approximated by the ratio 4 TBP: + $Th(NO_3)_4$ + $\frac{1}{6}$ HNO_3. The critical concentration decreases with increasing acidity, and increases with increasing temperature. Data are also presented on critical thorium concentrations for 30 and 40% TBP at 45

11626 ISC-415
Ames Lab., Ames, Iowa.
PURIFICATION OF THORIUM BY SOLVENT EXTRACTION. M. E. Whatley, Morton Smutz, and G. L. Bridger. July 1953. Decl. Feb. 26, 1957. 92p. Contract W-7405-eng-82. $0.60(OTS).

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Investigations were made of the solvent extraction of thorium nitrate with tributyl phosphate from aqueous solutions containing phosphate, sulfate, and nitric acid. It was the specific objective to develop a process to produce pure thorium from a thorium concentrate obtained by the selective precipitation of thorium phosphate from a monazite-sulfuric acid solution. A study of the effect of a diluent in the solvent mixture led to the choice of 80% TBP–20% inert diluent. The effects of phosphate, sulfate, and nitric acid on the thorium distribution coefficient were studied in detail. The thorium distribution coefficient was greatly decreased by the presence of phosphate, decreased to a lesser extent by the presence of sulfate, and greatly increased by the presence of large amounts of nitric acid. It was found that, by the use of high concentrations of nitric acid, the detrimental effect of phosphate and sulfate could be overcome and thorium extracted from solutions containing up to 20 g/l phosphate (P_2O_5) and/or sulfate. Because the use of 80% TBP afforded only a small difference in density between the solvent and aqueous phases, it was necessary to develop a mixer-settler with exceptionally good interface level control. The principle of operation and the design of both a laboratory glass unit and a box-type unit adaptable to industrial use are discussed. A pilot plant extractor was assembled and a continuous countercurrent extraction run was made. The capacity of the unit was 0.39 lb of thorium per hr. The feed, containing about equal parts thorium and rare earths, was prepared from a thorium phosphate concentrate from the Ames monazite process. There were seven stages in the extraction section, nine stages in the scrub section, and five stages in the strip section. The recovery of thorium was 92% and the product thorium contained less than four parts per million total rare earths. The selection of operating conditions for the run and the interpretation of the data are discussed. (auth)

12986 ✓ BMI-262
Battelle Memorial Inst., Columbus, Ohio.
PURIFICATION OF THORIUM NITRATE BY SOLVENT EXTRACTION WITH TRIBUTYL PHOSPHATE. Final Report. R. A. Ewing, J. B. Fishel, S. J. Kiehl, Jr., R. E. Sharpe, and A. E. Bearse. July 31, 1952. Decl. Feb. 14, 1957. 90p. Contract AT-(30-1)-gen-228. $0.60(OTS).

Purification of mantle-grade thorium nitrate by solvent extraction with 30% tributyl phosphate–70% Solvesso 100 was intensively investigated on a laboratory scale. Nitric acid was found to be a satisfactory salting agent. Batch equilibrium data are presented, covering the effects on thorium distribution of thorium concentration, TBP concentration, acidity, temperature, type of diluent, and the presence of sulfate and phosphate. Batch equilibrium data are also included on the intereffects of thorium and uranium. Solubility, viscosity, and density data are given for aqueous and organic solutions of thorium nitrate. The results of seven batch countercurrent extraction tests and two selective stripping tests are presented. Rare-earth assays of thorium products are included. (auth)

5250 ✓ CRDC-674
Atomic Energy of Canada Ltd. Chalk River Project, Chalk River, Ont.
THE PROCESSING OF IRRADIATED THORIUM FUELS USING TRIBUTYL PHOSPHATE-DECALIN. PART I. PARTITION DATA AND CALCULATIONS. W. W. Morgan. Dec. 1955. 30p. (AECL-508). AECL.

Partition data for Th, U, and HNO_3 between aqueous solution and 50% TBP-decalin (v/v) were obtained. McCabe-Thiele calculations were made which show that Th and U can be separated and purified by extracting into 50% TBP-decalin and partitioning with 0.25 M HNO_3. By utilizing decalin as the TBP diluent greater operating leeway results because the three phase problem no longer exists. (auth)

CF-58-7-129
Oak Ridge National Lab., Tenn.
SOLVENT EXTRACTION APPLIED TO REACTOR FUEL PROCESSING;FIRST QUARTER CO-OP REPORT.
W. E. Dunn. July 14, 1958. 12p. Contract [W-7405-eng-26].

The solvent extraction of thorium with 42.5% TBP in a Decalin diluent was studied in a compound pulse column. The HETS for the extraction section of the column was 4.7 feet. A discussion of the method of graphical calculations of a compound column is included.

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NITRATE SYSTEMS - tbp 3.02.17.04.06

See also, under PROCESS CHEMISTRY AND ENGINEERING, SOLVENTS--TBP - General (6.16.05.03).

5067 ✓ IDO-14490
Phillips Petroleum Co. Atomic Energy Div., Idaho Falls, Idaho.
EXCHANGE STUDIES OF THE URANIUM-TRIBUTYL PHOSPHATE REACTION. Bernard Keisch. Nov. 10, 1959. 23p. Contract AT(10-1)-205. OTS.
The kinetics and mechanism of the interfacial reaction of uranium with tributyl phosphate have been investigated by means of a heterogeneous exchange technique in a vessel of special design. At 25°C the rate law was determined to be Rate = $k(UO_2^{++})_{aq}(NO_3^-)_{aq}^{1.2}(TBP)_{org}^{1.8}$. The dominant reaction path varies with temperature at 6 to 45°C. Mechanisms were proposed to fit the rate laws determined. (auth)

5279 ✓ IDO-14454
Phillips Petroleum Co. Atomic Energy Div., Idaho Falls, Idaho.
THE PARTITION OF URANIUM(VI) BETWEEN TRIBUTYL PHOSPHATE AND BASIC ALUMINUM NITRATE SOLUTIONS. James W. Codding, Jr. Dec. 12, 1958. 33p. Contract AT(10-1)-205. $1.00(OTS).
The extraction coefficients (organic/aqueous) for tracer uranyl nitrate between various solutions of basic aluminum nitrate ("DIBAN") and 5 per cent TBP have been investigated, and the data are presented and correlated. A correction to the extraction data for uranyl hydrolysis and nitrate complexing is included. Through the application of activity coefficients a system of correlation is developed which brings the data into line with the generally accepted mass action law for the extraction of the species $UO_2(NO_3)_2 \cdot 2TBP$. The thermodynamic equilibrium constant for this extraction system is evaluated as $K_E = 50.8\ M_{TBP}^{-2}\ m_{NO_3}^{-2}$, with the activity coefficient vs. ionic strength relationship appearing in good agreement with that of similar solvent extraction systems. A method is outlined by which the activity coefficient values can be used to extrapolate uranium distribution data to provide X-Y diagrams for process design in the acid-deficient aluminum nitrate-TBP solvent extraction system. (auth)

17007
THE BACK-EXTRACTION OF URANYL NITRATE FROM TRIBUTYL PHOSPHATE SOLUTION. Taichi Sato (Government Chemical Industrial Research Inst., Tokyo). J. Inorg. & Nuclear Chem. 7, 147-9(1958) Aug.-Sept.
Sulfuric, hydrochloric, acetic, and oxalic acids, their ammonium and sodium salts, and ammonium and sodium carbonate were used as back-extraction agents for uranyl nitrate from TBP solution, and the results are tabulated and an evaluation made. (J.R.D.)

PROCESS OF SEPARATING URANIUM FROM AQUEOUS SOLUTION BY SOLVENT EXTRACTION. J. C. Warf (to U. S. Atomic Energy Commission). U. S. Patent 2,848,300. Aug. 19, 1958.
A process is described for separating uranium values from aqueous uranyl nitrate solutions. The process consists in contacting the uranium bearing solution with an organic solvent, tributyl phosphate, preferably diluted with a less viscous organic liquid, whereby the uranyl nitrate is extracted into the organic solvent phase. The uranyl nitrate may be recovered from the solvent phase by back extracting with an aqueous medium.

13831
THE EXTRACTION OF URANYL NITRATE FROM NITRIC ACID SOLUTIONS BY TRIBUTYL PHOSPHATE. Taichi Sato (Gov't. Chemical Industrial Research Inst., Tokyo). J. Inorg. & Nuclear Chem. 6, 334-7(1958) July.
The effects of nitric acid concentration, uranyl nitrate concentration, and temperature were investigated in order to determine the optimum conditions for the extraction of uranyl nitrate from nitric acid solutions by TBP. It is apparent from the results that greater than 97% extraction is obtained when uranyl nitrate solution of less than 10 g/l concentration and containing 6 M HNO_3 is extracted with 19% TBP in kerosene at a temperature of less than 20°C. (auth)

12318 ✓
KINETIC EFFECTS IN THE PARTITIONING OF URANIUM AND PLUTONIUM IN THE PROCESSING OF IRRADIATED REACTOR FUELS BY EXTRACTION WITH TRIBUTYL PHOSPHATE. Presented at Nuclear Engineering and Science Conference, held at Chicago, March 17 to 21, 1958. Preprint 168, Session 37. W. W.

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Morgan, W. G. Mathers, and R. G. Hart (Atomic Energy of Canada, Ltd., Chalk River, Ont.). New York, American Institute of Chemical Engineers, 1958. 26p.

A study of the chemical variables in the operation of contactor II of the TBP (tributyl phosphate) process for irradiated uranium has been carried out. Conditions under which small amounts of plutonium are precipitated in this contactor have been observed, and various methods of avoiding this precipitation have been discovered. A study of the effect of trace quantities of dibutyl phosphate on the operation of this contactor have clearly shown it to have a detrimental effect on the decontamination of uranium from plutonium. By allowing time for the uranium in the solvent to compete with the plutonium for the dibutyl phosphate it has been found possible to eliminate this interference. By this technique a uranium product stream was obtained with a uranium to plutonium weight ratio of 3.9×10^8. Under the best operating conditions found in this work, 0.1 M hydroxyl ammonium nitrate proved to be as effective a stripping agent as ferrous ion. (auth)

9261

SEPARATION OF URANIUM FROM RARE EARTHS AND SOME OTHER ELEMENTS BY SOLVENT EXTRACTION WITH TRI-BUTYL PHOSPHATE. Milenko V. Šušić and Nikola Jelić. Bull. Inst. Nuclear Sci. "Boris Kidrich" (Belgrade) 7, 29-33(1957) Mar.

The use of 20% TBP in kerosene as an extracting agent for the separation of U from rare earths and other elements, from 2 N nitric acid solution was studied. The dependence of the distribution coefficients of U, rare earths and other elements on their respective concentrations in solution was investigated, as also the influence of the concentration of TBP in kerosene on the distribution coefficient of U. For the investigations with rare earths, radioactive isotopes of Ce^{144} and Eu^{152} were employed. (auth)

9264

INFLUENCE OF TEMPERATURE ON THE EXTRACTION OF URANYL NITRATE WITH TRIBUTYL PHOSPHATE AT VARYING CONCENTRATIONS OF NITRIC ACID. Zdenko I. Dizdar, Olga S. Gal, and Jelena K. Rajnvajn. Bull. Inst. Nuclear Sci. "Boris Kidrich" (Belgrade) 7, 43-8(1957) Mar.

The dependence of the mass partition coefficient (μ) of $UO_2(NO_3)_2$ on temperature (1° to 80°C) is determined for the system: aqueous solution of HNO_3 – 30% TBP (diluted with dibutyl ether), at different HNO_3 concentrations. It is found that μ decreases with increasing temperatures at all acid concentrations. For a definite concentration of HNO_3, μ is logarithmically related to the temperature. Change in volume and optical transmittancy and decrease in the acidity of the aqueous phase show that the two phases chemically interact to an extent which rises with temperature and acid concentration. The limit of stability at 20°C is at 6M HNO_3, but changes to 4M HNO_3 at 80°C. (auth)

5272

ON THE INFLUENCE OF THE DILUENT ON URANYL NITRATE EXTRACTION WITH TRIBUTYL PHOSPHATE. V. B. Shevchenko, A. S. Solovkin, I. V. Shilin, L. M. Kirillov, A. V. Rodionov, and V. V. Balandina. Radiokhimiya 1, 257-69(1959). (In Russian)

The influence of diluent on uranyl nitrate distribution between nitric acid and tributyl phosphate was investigated. The dipole moment of $UO_2(NO_3)_2 \cdot 2TBP$ was equal to $(3.1 \pm 0.02) \times 10^{-18}$. (R.V.J.)

19997 CEA-tr-X-144

EXTRACTION DU NITRATE D'URANYLE AU MOYEN DU TBP (TRIBUTYL PHOSPHATE). EXTRACTION DES SELS MINÉRAUX CONTENUS DANS LA SOLUTION D'ACIDE NITRIQUE. (Extraction of Uranyl Nitrate with TBP (Tributyl Phosphate). Extraction of Mineral Salts Contained in the Nitric Acid Solution.) Taichi Sato and Totaro Goto. Translated into French by E. Hara from Kōgyō Kagaku Zasshi 60, 1444-7(1957). 17p.

A study was made to determine the best conditions for the extraction of uranyl nitrate with TBP. Nitric acid was used as the salting-out agent. The effects of diluents for the TBP on the extraction process was first determined. The variation of the extraction rate was determined as a function of the TBP concentration with respect to the quantity of diluent added. The experimental results showed that extraction efficiency is best when kerosene is used to dilute the TBP and when the ratio of kerosene to TBP is 8:2. Under these conditions the solubility of water in the solvent is quite small. The effects of the nitric acid concentration, uranyl nitrate concentration, and extraction temperature on the ex-

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traction coefficient were determined. The effects of coexistent ions were also studied. An aqueous solution of uranyl nitrate was extracted with 20% TBP and 6M nitric acid at a temperature less than 20°C. The uranyl nitrate concentration was below 10 g/l, and an extraction coefficient of more than 97% was obtained. The extraction mechanism for these conditions is discussed. (J.S.R.)

2868
EXTRACTION OF URANYL NITRATE WITH ORGANIC SOLVENTS. II. WITH TRI-N-BUTYL PHOSPHATE.
A. Cacciari, R. De Leone, C. Fizzotti and M. Gabaglio (Laboratori CISE, Milano). Energia Nucleare (Milan) 3, 368-75(1956) Oct. 15. (In Italian)

Studying the extraction of uranyl nitrate with tributylphosphate diluted with white spirit, the dependence from tributylphosphate concentration is determined and the equilibrium curve for a 40% concentration in volume of tributylphosphate is drawn. The influence of nitric acid and ammonium nitrate under these very conditions is examined. Besides the equations for the equilibrium curves, the extraction of nitric acid in presence of uranyl nitrate and ammonium nitrate and the influence of phosphates are considered. Also the partition coefficients of iron and copper are measured. (auth)

212 IDO-14381
Phillips Petroleum Co. Atomic Energy Div., Idaho Falls, Idaho.
DISTRIBUTION OF URANIUM AT LOW ACID–LOW URANIUM CONCENTRATIONS INTO 4½ PERCENT TBP–AMSCO. R. L. Andelin, E. L. Anderson, and W. H. McVey. Aug. 23, 1956. 26p. Contract AT(10-1)-205. $0.35(OTS).

Acid free $UO_2(NO_3)_2 \cdot 4H_2O$ was prepared and its distribution into 4½% TBP–Amsco 125-90W in the aqueous concentration range 1 gram per liter to 30 grams per liter was measured. Acid concentrations were varied from 0.00M added acid to 0.06M added acid. Uranium distribution was found to be strongly dependent on HNO_3 and U concentrations as expected. Hydrolysis effects under these conditions were studied. No attempt was made to measure HNO_3 distribution. A value of 171.8 was found for the thermodynamic equilibrium constant in the reaction for complexing of $UO_2(NO_3)_2$ by TBP. (auth)

7484 IGR-TM/W-055
United Kingdom Atomic Energy Authority. Industrial Group. Windscale Works, Sellafield, Cumb., England.
URANIUM PARTITION COEFFICIENTS AT LOW URANIUM CONCENTRATIONS IN NITRIC ACID–20% TBP/OK SYSTEMS. A. Duncan and G. A. Holburt. May 30, 1956. 8p. (IGC-DCPWP/P-66).

Factorial regression analysis of the results show that at the low uranium concentrations the uranium partition coefficient K_u is not affected by change of uranium concentration; K_u, however, changes markedly with change of nitric acid, particularly in the region 0 to 0.5N. A nomogram was constructed of K_u versus nitric acid concentration. To check the nomogram, partition coefficients of uranium at 1.0 and 3N acidity were determined. (auth)

581 CF-54-7-203
Oak Ridge National Lab., Tenn.
THE EFFECT OF SOLVENT TRIBUTYL PHOSPHATE CONCENTRATION ON THE DECONTAMINATION OF URANIUM EXTRACTED FROM ALUMINUM NITRATE SOLUTIONS. G. I. Cathers. July 26, 1954. Decl. Sept. 26, 1958. 21p. Contract [W-7405-eng-26]. $4.80 (ph OTS); $2.70(mf OTS).

The results are reported of a study made to determine the effect of solvent TBP concentration on fission product decontamination of U extracted from $Al(NO_3)_3$ solutions. (auth)

2376 ✓ AECD-3805
Hanford Works, Richland, Wash.
THE DENSITIES OF TRIBUTYL PHOSPHATE-CARBON TETRACHLORIDE SOLUTIONS CONTAINING URANIUM AND NITRIC ACID. Robert Lee Moore. Dec. 20, 1949. Decl. with deletions Dec. 2, 1955. 15p. Contract W-31-109-Eng-52. $3.30(ph OTS); $2.40(mf OTS).

Linear equations have been derived which correlate the densities of TBP–CCl_4 solutions containing U and HNO_3, with the limits of experimental error, as functions of U concentration and solvent composition. The differences between the densities of equilibrated aqueous and organic phases are tabulated for 20% TBP–CCl_4 as a function of UNH and HNO_3 concentrations. (auth)

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6505 A/CONF.15/P/179
REFINING OF URANIUM CONCENTRATE AND PRODUCTION OF URANIUM OXIDE AND METAL.
R. Gelin, H. Mogard, and B. Nelson (AB Atomenergi, Stockholm). 9p.

The processes used in Sweden for the production of uranium oxide and metal from uranous phosphate concentrate are presented. The refining process involves digestion of the uranous phosphate with caustic soda, conversion of the uranous hydroxide to ammonium uranyl carbonate, dissolution in nitric acid, and purification by solvent extraction using hexone. Ammonium uranate is recovered from the pure uranyl nitrate solution by homogeneous precipitation with urea. After calcination the UO_3 product is reduced to UO_2, converted to UF_4 in a "dryway" hydrofluorination process, and reduced by calcium to U metal, which subsequently is remelted under vacuum. (auth)

PHOSPHATE SYSTEMS 3.02.17.05

See also, under PROCESS CHEMISTRY AND ENGINEERING, SPECIAL PROCESSES--UAP Process (6.17.23).

URANIUM EXTRACTION PROCESS. W. H. Baldwin and C. E. Higgins (to U. S. Atomic Energy Commission). U. S. Patent 2,864,668. Dec. 16, 1958.

A process is described for recovering uranium values from acidic aqueous solutions containing hexavalent uranium by contacting the solution with an organic solution comprised of a substantially water-immiscible organic diluent and an organic phosphate to extract the uranium values into the organic phase. Carbon tetrachloride and a petroleum hydrocarbon fraction, such as kerosene, are suitable diluents to be used in combination with organic phosphates such as dibutyl butylphosphonate, tributyl phosphine oxide, and tributyl phosphate.

PHOSPHATE SYSTEMS 3.02.17.05

ALKYL PYROPHOSPHATE METAL SOLVENT EXTRACTANTS AND PROCESS. R. L. Long (to U. S. Atomic Energy Commission). U. S. Patent 2.866.680. Sept. 30, 1958.

A process is presented for the recovery of uranium from aqueous mineral acidic solutions by solvent extraction. The extractant is a symmetrical dialkyl pyrophosphate in which the alkyl substituents have a chain length of from 4 to 17 carbon atoms. Mentioned as a preferred extractant is dioctyl pyrophosphate. The uranium is precipitated from the organic extractant phase with an agent such as HF, fluoride salts, alcohol, or ammonia.

7142 DOW-157
Dow Chemical Co. Western Div., Pittsburgh, Calif.
PROGRESS REPORT [FOR] APRIL 1957. Research Dept. R. H. Bailes. May 1, 1957. 19p. Contract AT-30-1-GEN-236. $3.30(ph OTS); $2.40(mf OTS).

A study of the preparation of high-grade U products by solvent extraction is presented. Tests of several alternative alkyl phosphate extractants for U from H_3PO_4 are reported. (auth)

2054 AECD-3746
Los Alamos Scientific Lab., N. Mex.
THE EXTRACTION OF URANIUM FROM PHOSPHATE SOLUTIONS. H. R. Baxman. [1956?]. Decl. May 21, 1956. 9p. Contract [W-7405-eng-36]. $1.80(ph OTS); $1.80(mf OTS).

A process for the extraction and depoisoning of uranium in strong phosphoric acid solution with tributyl phosphate (TBP) is described. The formation of a complex between uranyl and phosphate ions necessitates the use of a salting agent which is capable of destroying this complex and releasing the uranyl ion into an organic extractable species. Of various salting agents tried, ferric nitrate was the most successful. It not only provides nitrate ion for the formation of uranyl nitrate, but also forms a very stable ferric-phosphate complex, thus completely tying up the phosphate ion when added in equimolar quantities. Stripping of the enriched solvent is accomplished with phosphoric acid. (auth)

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SOLVENT EXTRACTION 3.02.17

PHOSPHATE SYSTEMS 3.02.17.05

3912 (RMO-2030)
International Minerals and Chemical Corp., Chicago.
URANIUM EXTRACTANTS—PREPARATION AND USE IN PHOSPHATE SYSTEM. Henry M. Heidt, Donald E. Tynan, Robert F. McCullough, and Roger Bart. Sept. 13, 1954. Decl. Oct. 27, 1955. 121p. Contract AT(49-1)-545. (IMCC-2165). $0.60(OTS).

Research accomplishments on the preparation of organic phosphate esters for the extraction of uranium from monocalcium phosphate, phosphoric acid and other acidic phosphate solutions using liquid-liquid extraction techniques are presented. Optimum combination of variables studied permit production of uranium extractants superior to those previously reported, or in use today. Pyro and/or ortho phosphoric acid esters of various alcohols were prepared by reaction of alcohol with phosphorus pentoxide in presence of kerosene as an extender. (auth)

SULFATE SYSTEMS 3.02.17.06

See also, under PROCESS CHEMISTRY AND ENGINEERING,

SOLVENTS--Amines (6.16.05.01),

TBP - General (6.16.05.08).

2835 ORNL-2615
Oak Ridge National Lab., Tenn.
THE RECOVERY OF URANIUM FROM SULFATE SOLUTIONS BY SOLVENT EXTRACTION WITH TRIBUTYL PHOSPHATE. J. R. Flanary and J. H. Goode. Dec. 3, 1958. 15p. Contract W-7405-eng-26. $0.50(OTS).

Laboratory data and a process chemical flowsheet are given for the recovery of uranium from aqueous sulfate solutions by solvent extraction with tributyl phosphate. Uranium may be recovered from solutions containing as much as 2.5 moles of sulfate per mole of uranium by adding sufficient nitrate-ion salting strength, in the form of aluminum nitrate and nitric acid, to overcome the complexing effect of the sulfate. The use of 15 moles of nitrate per mole of sulfate permitted essentially complete extraction of uranium in about 2.5 theoretical stages. Gross β and gross γ decontamination factors of 1×10^5 and 2.43×10^3, respectively, were obtained during processing of the uranyl sulfate core of the Raleigh Research Reactor; uranium losses were 0.008%. (auth)

17032
EXTRACTION OF URANYL SULFATE WITH AMINES. Ch. Boirie (Commissariat a l'Energie Atomique, Gif-sur-Yvette, Saclay, France). Bull. soc. chim. France No. 8-9, 1088-93(1958) Aug.-Sept. (In French)

The extraction of uranyl sulfate with long-chain amines was studied. The formulas of the compounds formed with different amines in solutions of carbon tetrachloride were established. The effect of several factors were controlled. Di-n-decylamine was chosen to study the amine sulfate/uranyl sulfate equilibrium and to determine its dissociation constant. (tr-auth)

16305
THE URANYL SULFATE COMPLEXES FROM TRI-n-OCTYLAMINE EXTRACTION EQUILIBRIA. Kenneth A. Allen (Oak Ridge National Lab., Tenn.). J. Am. Chem. Soc. 80, 4133-7(1958) Aug. 20.

The extraction of uranium from acidic sulfate aqueous systems by benzene solutions of tri-n-octylamine sulfate was measured under various conditions of uranium loading, acid activity, sulfate ion concentration, and amine concentration. An equation for the reaction between the amine sulfate-bisulfate mixture and the uranium is proposed which is consistent with the results. Under conditions of constant acid activity, amine concentration, and low uranium loading, the distributions observed at varying aqueous sulfate levels are shown to lead to estimates of the formation constants for the aqueous uranium mono- and disulfate complexes which are in reasonably good agreement with those reported by Ahrland. The present evidence suggests only very low or negligible proportions of the trisulfate complex at sulfate molarities below one. (auth)

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SOLVENT EXTRACTION 3.02.17

SULFATE SYSTEMS 3.02.17.06

13832
URANIUM EXTRACTION BY DI-n-DECYLAMINE SULFATE. W. J. McDowell and C. F. Baes, Jr. (Oak Ridge National Lab., Tenn.). J. Phys. Chem. 62, 777-83(1958) July.

The extraction of uranium from sulfuric acid–sodium sulfate solutions by di-n-decylamine sulfate in benzene is examined as a function of uranium, amine, acid, and sulfate ion concentration. The stoichiometry of the principal organic complex is uranium : sulfate : amine = 1 : 4 : 6. The dependence of uranium extraction on total uranium concentration and on extractant concentration is discussed in terms of the extensive association of di-n-decylamine sulfate in the organic phase. Data for variation of sulfate ion concentration are shown to be consistent with formation quotients of aqueous UO_2SO_4 and $UO_2(SO_4)_2^{=}$ to be found in the literature. No evidence was found for the formation of a $UO_2(SO_4)_3^{-4}$ complex. (auth)

14945 ORNL-2709
Oak Ridge National Lab., Tenn.
URANIUM EXTRACTION BY TRI-n-OCTYLAMINE SULFATE. Kenneth A. Allen. June 4, 1959. 23p. Contract W-7405-eng-26. $0.75(OTS).

The extraction of uranium from acidic sulfate aqueous systems by benzene solutions of tri-n-octylamine sulfate was studied under various conditions of uranium loading, acid activity, sulfate ion concentration, and amine concentration. The data are consistent with the reaction $nxRH_2SO_4 + n(1-x)/2\ R_2H_2SO_4 + UO_2SO_4 = R_nH_nUO_2(SO_4)_{n/2+1} + nx/2\ H_2SO_4$, where $R = (C_8H_{17})_3N$, $x = [RH_2SO_4]/([RH_2SO_4] + 2[R_2H_2SO_4])$, and n is the number of equivalents of amine per mole of uranium in the complex. Under conditions of constant acid activity, amine concentration, and low uranium loading, the distributions observed at varying aqueous sulfate levels lead to the following estimates of the formation quotients for the aqueous complexes UO_2SO_4 (K_1) and $UO_2(SO_4)_2^{=}$ (K_2) at unit ionic strength: $K_1 = 34(17-135)$, $K_2 = 200(100-760)$, $K_2/K_1 = 6.0 \pm 1.0$. (auth)

AN ATTEMPT TO EXTRACT URANIUM FROM SULFATE SOLUTIONS WITH TRIBUTYL PHOSPHATE. U. Veereswararao. Bull. Inst. Nuclear Sci. "Boris Kidrich" (Belgrade) 8, 75-83(1958) Mar.

Distribution of uranyl sulfate between 30% TBP(v/v) in kerosene and different aqueous H_2SO_4 solutions with and without the addition of NaCl was studied. The partition of the other elements that usually accompany uranium was also investigated under the same conditions as those for uranium in the presence of sodium chloride. A method for the elimination of the interference of Fe in the extraction of uranium is suggested. (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)

975 AECD-4145
Oak Ridge National Lab., Tenn.
FURTHER STUDIES OF AMINES AS EXTRACTANTS FOR URANIUM FROM ACID SULFATE SOLUTIONS. J. G. Moore, K. B. Brown, and C. F. Coleman. June 24, 1955. Decl. with deletions Nov. 11, 1955. 96p. Contract W-7405-eng-26. $15.30(ph OTS); $5.40(mf OTS).

Since the first report on the use of amines as U extractants over eighty additional organo-nitrogen compounds have been examined for their ability to extract U from aqueous solutions, particularly sulfate solutions, of the types usually encountered in U ore processing. As before, the more promising of these (as well as other compounds previously reported) have been examined further with respect to other characteristics essential to practical application, i.e., reagent loss to the aqueous phase, compatibility with practicable diluents, influence of commonly present anions, reagent stability, sufficient selectivity for

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11136 RMO-2533
Rohm and Haas Co. Research Labs., Philadelphia.
THE SOLVENT-SOLVENT EXTRACTION OF URANIUM FROM SULFURIC ACID SOLUTIONS WITH OIL SOLUBLE AMINES. Al Preuss and Jean Saunders. Apr. 14, 1955. Decl. Dec. 14, 1955. 39p. Contract AT(49-1)-535.

Fifty oil soluble amines were screened for possible use as extractants for U from H_2SO_4 solutions. The most promising of these were studied further to obtain the optimum conditions for operation of such a process. Kerosene was used as the diluent. (auth)

9740 AECD-4142
Oak Ridge National Lab., Tenn.
THE USE OF AMINES AS EXTRACTANTS FOR URANIUM FROM ACIDIC SULFATE LIQUORS. A Preliminary Report. K. B. Brown, C. F. Coleman, D. J. Crouse, J. O. Denis, and J. G. Moore. May 27, 1954. Decl. with deletions Jan. 11, 1956. 118p. Contract W-7405-eng-26. $0.60(OTS).

Over 100 different organonitrogen compounds have been examined for their ability to extract U from aqueous solutions, particularly sulfate solutions, of the types usually encountered in U ore processing. The more promising of these have been examined further with respect to other characteristics essential to practical application, especially selectivity for U, reagent loss to the aqueous phase, compatibility with practicable diluents, maintenance of adequate extraction power over a range of

OTHER SYSTEMS 3.02.17.07

13247 AEC-tr-3655
EXTRACTION OF PERCHLORIC ACID AND URANYL PERCHLORATE WITH TRIBUTYL PHOSPHATE.
V. B. Shevchenko, I. V. Shilin, and A. S. Solovkin.
Translated for Los Alamos Scientific Lab. from Zhur. Neorg. Khim. 3, 225-30(1958). 7p. $1.80(ph), $1.80(mf) JCL or LC.

This paper was previously abstracted from the original language and appears in NSA, Volume 12, as abstract No. 13879.

OTHER SYSTEMS 3.02.17.07

6363
SALICYLATE PROCESS FOR THORIUM SEPARATION FROM RARE EARTHS. G. A. Cowan (to U. S. Atomic Energy Commission). U. S. Patent 2,901,496. Aug. 25, 1959.

The separation of thorium from rare earths is accomplished by forming an aqueous solution of salts of thorium and rare earths and sufficient acetate buffer to provide a pH of between 2 and 5, adding an ammonium salicylate to the aqueous buffered solution, contacting the resultant solution with a substantially water-immiscible organic solvent mixture of an ether and an ester, and separating the solvent extract phase containing thorium salicylate from the aqueous phase containing the rare earths.

SYNERGISTIC REAGENTS 3.02.17.08

See also, under PROCESS CHEMISTRY AND ENGINEERING, SOLVENTS--Organophosphorous (6.16.05.05).

8345 ORNL-2259
Oak Ridge National Lab., Tenn.
SYNERGISTIC URANIUM EXTRACTANTS: COMBINATION OF NEUTRAL ORGANOPHOSPHORUS COMPOUNDS WITH DIALKYLPHOSPHORIC ACIDS. C. A. Blake, D. E. Horner, and J. M. Schmitt. Feb. 25, 1959. 42p. Contract W-7405-eng-26. $1.25(OTS).

Dialkylphosphoric acids in combination with trialkylphosphates, dialkyl alkylphosphonates, trialkylphosphine oxides, and other neutral organophosphorus compounds have uranium extraction abilities as much as two orders of magnitude stronger than the cumulative abilities of the individual reagents. With the possible exception of plutonium, other metals tested, e.g., iron, aluminum, vanadium, molybdenum, titanium, show no synergistic enhancement of extraction coefficient. Of the acids tested, only dialkylphosphoric acids showed a synergistic enhancement. Extractions can be achieved from sulfate and phosphate solutions in addition to ni-

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SYNERGISTIC REAGENTS 3.02.17.08

trate solutions. Effects of anion concentration, pH and dialkylphosphoric acid concentration are parallel with and without the neutral additive. High neutral additive concentrations decrease uranium extraction. The reagents allow high uranium concentration from very low-grade liquors and are usable with liquors usually difficult to extract. Several useful reagent combinations can be prepared from commercial reagents. (auth)

7473 AERE-C/M-369
United Kingdom Atomic Energy Authority. Research Group. Atomic Energy Research Establishment, Harwell, Berks, England.
SYNERGISM IN THE SOLVENT EXTRACTION OF URANIUM: A TENTATIVE EXPLANATION. J. Kennedy. Dec. 1958. 4p. $0.16(BIS).
Suggestions were made to account for the enhanced extraction of uranium by the dimeric acidic dialkyl phosphates $(HX)_2$ in the presence of nonionic phosphorylated reagents. Extraction which in the absence of the nonionic reagents occurs via the mechanism. $UO_2^{2+} + 2(HX)_2 \rightleftharpoons UO_2X_2 \cdot 2HX + 2H^+$ may be enhanced in their presence as a result of obviating the necessity of monomerizing a mole of the dimeric extractant. With TBP the suggested extraction mechanism is $UO_2^{2+} + (HX)_2 + 2TBP \rightleftharpoons UO_2X_2 \cdot 2TBP + 2H^+$. (auth)

URANIUM EXTRACTION PROCESS USING SYNERGISTIC REAGENTS. J. M. Schmitt, C. A. Blake, Jr., K. B. Brown, and C. F. Coleman (to U. S. Atomic Energy Commission). U. S. Patent 2,859.094. Nov. 4, 1958.
Improved methods are presented for recovering uranium values from aqueous solutions by organic solvent extraction. The improvement lies in the use, in combination, of two classes of organic compounds so that their extracting properties are enhanced synergistically. The two classes of organic compounds are dialkylphosphoric acid and certain neutral organophosphorus compounds such as trialkylphosphates, trialkylphosphonates, trialkylphosphinates and trialkylphosphine oxides.

VOLATILITY METHODS 3.02.18

See also, this section,

RECOVERY OF IRRADIATED TH AND U
--Distillation (3.02.07),
--Fluorination and Hydrofluorination (3.02.10),
--Fused Salts and Liquid Metals (3.02.11),

and,

under PLANTS AND EQUIPMENT,

DISTILLATION (5.07),
HIGH PRESSURE SYSTEMS--Vessels (5.09.03),

and,

under PROCESS CHEMISTRY AND ENGINEERING,
SPECIAL PROCESSES--DeBoer (6.17.05).

FLUORIDE VOLATILITY 3.02.18.01

See also, under PROCESS CHEMISTRY AND ENGINEERING,

DISTILLATION AND CONDENSATION (6.06),

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

119 MCW-1418
Mallinckrodt Chemical Works, St. Louis.
URANIUM RECOVERY FROM SLAG BY FLUORIDE VOLATILIZATION. A PROCESS PROPOSAL. R. M. Edwards, J. A. Kennelley, and E. F. Sanders. Sept. 15, 1958. 28p. $1.00(OTS).
Laboratory and pilot plant studies are presented which indicate the technical feasibility of a uranium recovery process based on the reaction: $U_{(s)} + 3F_{2(g)} \rightarrow UF_{6(g)}$. Residual uranium in the fluorinated slag was in the range 0.14 to 0.20%. Reaction conditions were: feed rate: 100 pounds per hour; slag residence time: one hour; fluorine use: 1.4 times theoretical; temperature: 435 to 545°C. Economic analyses of the process are presented. (auth)

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FLUORIDE VOLATILITY 3.02.18.01

14393

FLUORIDE VOLATILITY PROCESS FOR THE RECOVERY OF URANIUM. Joseph J. Katz, H. H. Hyman, and Irving Sheft (to U. S. Atomic Energy Commission). U. S. Patent 2,830,873. Apr. 15, 1958.

The separation and recovery of uranium from contaminants introduced by neutron irradiation by a halogenation and volatilization method are described. The irradiated uranium is dissolved in bromine trifluoride in

13070 NLCO-743

National Lead Co. of Ohio, Cincinnati.
THE PREPARATION OF URANIUM HEXAFLUORIDE FROM URANIUM ORES AND CONCENTRATES BY FLUORIDE VOLATILITY PROCESSES—AN ANNOTATED BIBLIOGRAPHY. Earl W. Mautz and Charles J. Fetner. Feb. 26, 1958. 16p. Contract AT(30-1)-1156. $0.75(OTS).

An annotated bibliography of publications relating to the preparation of uranium hexafluoride from uranium ores and concentrates by fluoride volatility processes is presented. (auth)

14713 A/CONF.15/P/2388

Argonne National Lab., Lemont, Ill.
FLUORIDE VOLATILITY PROCESSES FOR SPENT REACTOR FUELS. (A Survey of Recent Work in the United States.) R. K. Steunenberg, and R. C. Vogel. 29p. $0.50(OTS).

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

Work has continued at various sites on the development of uranium hexafluoride distillation processes for the recovery of fissionable values from spent reactor fuels. Metallic uranium fuels can be dissolved in bromine trifluoride or chlorine trifluoride, producing uranium hexafluoride. The hexafluoride is purified and decontaminated by fractional distillation. In an advanced version of this process, plutonium in the fuel is also converted to the hexafluoride which is purified by volatilization then decomposed to a nonvolatile product. The

10239

PROCESS FOR TREATING VOLATILE METAL FLUORIDES. A. J. Rudge and A. J. Lowe (to U. S. Atomic Energy Commission). U. S. Patent 2,808,312. Oct. 1, 1957.

This patent relates to the purification of uranium hexafluoride, made by reacting the metal or its tetrafluoride with fluorine, from the frequently contained traces of hydrofluoric acid. According to the present process, UF_6 containing as an impurity a small amount of hydrofluoric acid, is treated to remove such impurity by contact with an anhydrous alkali metal fluoride such as sodium fluoride. In this way a non-volatile complex containing hydrofluoric acid and the alkali metal fluoride is formed, and the volatile UF_6 may then be removed by distillation.

FLUORIDE VOLATILITY PROCESSES FOR LOW ALLOY FUELS. R. C. Vogel and R. K. Steunenberg. May 1, 1957. p.498-559 of SYMPOSIUM ON THE REPROCESSING OF IRRADIATED FUELS HELD AT BRUSSELS, BELGIUM, MAY 20-5, 1957. 1165p. (TID-7534(Bks. 1-3))

In a fluoride volatility separations process the species in the irradiated fuel are all converted to the fluorides. The UF_6 can then be separated by distillation from the fission product fluorides. It is believed that the conversion of the fission products and U to the fluorides leads to a relatively easy separations problem in decontaminating the U. The problems involved in the separation of uranium hexafluoride from the fission product fluorides are depicted graphically. 11 references.

9270 NLCO-671

National Lead Co. of Ohio, Cincinnati.
THE SUBLIMATION OF URANYL FLUORIDE IN THE PRESENCE OF HYDROGEN FLUORIDE. Richard E. DeMarco, Robert C. Abbott, DuWane C. Bonfer, and Paul A. Cook. Mar. 26, 1957. 15p. Contract AT(30-1)-1156. $0.20(OTS).

Uranyl fluoride was found to sublime in the presence of HF at temperatures greater than 1000°F. The rate of sublimation was found to be 1.35 order with respect to HF and to possess an activation energy of 23 ± 1 kcal/mole. The chemical composition of the volatile compound formed in sublimation was not established. (auth)

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FLUORIDE VOLATILITY 3.02.18.01

5288 ✓
URANIUM RECOVERY FROM AQUEOUS WASTES. R. J. Clouse, J. Dykstra, and B. H. Thompson (Union Carbide Nuclear Co., Oak Ridge, Tenn.). Chem. Eng. Progr. 53, 65F-9F(1957) Feb.

The solutions collected from equipment cleaning at the Gaseous Diffusion Plant contain isotopically enriched U. The facility for recovering this U, processing it to the oxide, and fluorinating the oxide to UF_6 is discussed. (W.L.H.)

5269 ✓
DECONTAMINATION OF IRRADIATED URANIUM BY A FLUORIDE VOLATILITY PROCESS. William J. Mecham, Robert C. Liimatainen, Robert W. Kessie, and Waldemar B. Seefeldt (Argonne National Lab., Lemont, Ill.). Chem. Eng. Progr. 53, 72F-7F(1957) Feb.

A series of six pilot plant runs was made on 10-kg lots of U. The process steps, corrosion of equipment, process equipment, flowsheets and application of the process to enriched fuels are discussed in detail. (W.L.H.)

RECOVERY OF URANIUM FROM RECYCLE WASTES BY FLUORIDE VOLATILITY. G. I. Cathers and D. E. Ferguson. June 29, 1956. Decl. Mar. 19, 1957. 13p. (CF-56-6-159)

The present method of recovering U from the scrap obtained in the production of reactor fuel elements consists of obtaining a nitrate solution by dissolving or leaching, solvent extraction, and, finally, precipitation. An alternate method that has been investigated for scrap processing is fluoride volatility. It was found that reaction of MgF_2 slag with F_2 at 400°C volatilized 99% of the contained U, provided that good gas-solid contact was obtained. Based on these laboratory results, a process which consists of grinding, screening, fluorinating with F_2 at 400°C in an agitated reactor, and collecting the product UF_6 in a cold trap appears to be an attractive method for treating the scrap from the UF_6 and UF_4 reduction steps. The oxide and metal scrap and HNO_3 pickling liquor would require chemical conversion to UF_4 before fluorination, and for this reason the volatility method does not appear to be greatly simpler than the present aqueous method for this type of scrap.

7559 ANL-5593
Argonne National Lab., Lemont, Ill.
CHEMICAL AND RADIOCHEMICAL STUDIES OF TELLURIUM IN FLUORIDE VOLATILITY STUDIES. J. Fischer and R. K. Steunenberg. June 1956. 19p. Contract W-31-109-eng-38. $0.30(OTS).

Chemical and radiochemical studies were made to clarify the behavior of fission product Te in fluoride volatility processes for the recovery of U from spent reactor fuels. The chemical nature of mixtures of TeF_6 and BrF_3 was investigated in the gas phase at 75 and 100°C, and in the liquid phase at 25°C. The gas phase behavior of TeF_6 in BrF_5 was studied at 75°C, and liquid mixtures of TeF_6 and Br_2 were examined at 25°C. In none of these systems was there evidence for a chemical reaction which would produce Te compounds of low volatility. Quantitative radiochemical observations were made on the partial reduction of TeF_6 and the separation of daughter isomers due to the internal conversion of gamma rays of Te^{127m} and Te^{129m}.

4752 ANL-4709(Del.)
Argonne National Lab., Lemont, Ill.
THE ARGONNE FLUORIDE VOLATILITY PROCESS, STATUS AS OF DECEMBER 31, 1951. H. H. Hyman and J. J. Katz. Feb. 10, 1952. Decl. with deletions Feb. 13, 1957. 207p. Contract W-31-109-eng-38. $30.30(ph OTS); $8.70(mf OTS).

The development of a chemically feasible fluoride volatility process is summarized. The Argonne fluoride volatility process is described, and flowsheets are given. Experimental programs involved in demonstrating the separation of UF_6 and fission product fluorides and separation of U and Pu are summarized. Discussions are given of waste disposal and by-product recovery problems associated with this type of process, economic aspects of the process, and analytical problems. A bibliography of 238 references is included. (W.L.H.)

RECOVERY OF URANIUM FROM HANFORD WASTE BY PRECIPITATION AS URANIUM TETRAFLUORIDE AND SUBSEQUENT FLUORINATION. H. A. Bernhardt, S. Bernstein, and F. D. Rosen. June 10, 1949. Decl. Dec. 27, 1955. 16p. (K-416; KLO-107)

U may be recovered from Hanford waste by precipitation as UF_4 and subsequent fluorination to UF_6. This treatment has been used to obtain separation of all the fission products, except Ru^{106}, from the U. Activity due to the Ru was reduced to a value of 1 mc/kg of U.

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VOLATILITY METHODS 3.02.18

GENERAL 3.02.18.02

9639 ✓ NAA-SR-1374
Atomics International Div., North American Aviation, Inc., Canoga Park, Calif.
INVESTIGATION OF PYROCHEMICAL PROCESSING OF Th–U FUEL. Semi-Annual Report [for] September 1954–February 1955. F. J. Keneshea, Jr., A. G. Buyers, and E. E. Motta. Jan. 15, 1957. Decl. Mar. 14, 1957. 18p. Contract AT-11-1-GEN-8. $0.30(OTS).

Separation of Ce, rare earths, Cs, Sr, and Te from irradiated Th–U was accomplished by volatilization from a Ta crucible, leaving Pa and Ru. With a TiC crucible the same thing occurred. In a ThO_2 crucible 50% removal of Ce and rare earths occurred, and in ZrO_2 70% separation occurred. In a five-pass arc-zone-melting run Cs and Sr were completely volatilized leaving Ce and the rare earths. (T.R.H.)

12487 ✓ NAA-SR-1680
Atomics International Div., North American Aviation, Inc., Canoga Park, Calif.
THE VOLATILIZATION OF FISSION PRODUCTS FROM MOLTEN AND SOLID THORIUM–URANIUM ALLOY. T. A. Milne and C. T. Young. Dec. 15, 1956. ~~Decl. Mar. 14, 1957.~~ 19p. Contract AT-11-1-GEN-8. $0.30(OTS).

The volatilization of fission products from molten and solid irradiated Th-(3 wt. %) U has been investigated. The effects of temperature, time, and area-to-weight ratio have been studied. Ninety-five per cent or more of the Cs, Sr, and Te were volatilized from solid or liquid Th with ease. Cerium and other rare earths were more difficult to remove but might be volatilized from liquid or solid Th by employing a sufficient time or large surface areas. It does not appear feasible to remove U from Th by volatilization. (auth)

PROCESS OF VACUUM REFINING URANIUM. G. Meister (to U. S. Atomic Energy Commission). U. S. Patent No. 2,756,138, July 24, 1956.

An impure U slug is supported upon a strainer which is placed in a crucible with a hole in its bottom. Under the crucible is a mold, and the entire assembly is enclosed in a vycor glass envelope. The envelope is evacuated and an induction coil, wrapped around the outside of the envelope, is used to heat and melt the U. As the U melts, the volatile impurities vaporize and are condensed on the inner surface of the glass envelope. The non-volatile oxide and fluoride impurities form a slag which is retained on the strainer when the melting U flows through it. The molten U passes through the hole in the crucible and is cast in the mold below as pure metal. (auth)

11653 ✓ CF-52-4-64
Oak Ridge National Lab., Tenn.
APPLICATION OF VOLATILITY PROCESSES TO URANIUM RECOVERY. R. E. Leuze. Apr. 14, 1952. Decl. Feb. 16, 1957. 12p. $3.30(ph OTS); $2.40(mf OTS).

The production of U from ores and decontamination of reactor fuels are suggested as applications of volatilization processes for U recovery. (C.W.H.)

6353 ✓ A/CONF.15/P/1098
A CARBIDE-IODIDE PROCESS FOR HIGH PURITY THORIUM. D. E. Scaife and A. W. Wylie (Commonwealth Scientific and Industrial Research Organization, Melbourne). 58p.

Thorium carbide can be used as a feed material for production of thorium metal filaments by a Van Arkel–de Boer-type process. Factors influencing the synthesis of carbide from oxide and the growth of thorium metal from carbide were studied and optimum conditions of growth established. Metal having a carbon, nitrogen, and oxygen content lower than the best material obtainable from a conventional metal feed can be produced at satisfactory rates and temperatures. A thorium iodide decomposition unit with carbide feed material involves processes which are highly efficient at separating from thorium all major metallic impurities except zirconium, hafnium, and uranium. A large group of these impurities

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VOLATILITY METHODS 3.02.18

FLUORIDE VOLATILITY 3.02.18.01

1203 K-945
Carbide and Carbon Chemicals Co. K-25 Plant, Oak Ridge, Tenn.
STUDIES OF THE RATE OF REACTION BETWEEN METALLIC URANIUM AND SOLUTIONS OF CHLORINE TRIFLUORIDE AND HYDROGEN FLUORIDE. R. A. Gustison and E. J. Barber. Oct. 8, 1952. Decl. Oct. 29, 1957. 16p. Contract W-7405-eng-26. $3.30(ph OTS); $2.40(mf OTS).

The temperature dependence of the rate of reaction between metallic uranium and a solution containing about 0.3 moles of chlorine trifluoride per mole of hydrogen fluoride was determined between 10 and 80°C. The data indicate that at 75°C about 29 hrs would be required to dissolve a Hanford slug and that at 80°C about 18 hrs would be required. The dependence of the rate of reaction on temperature underwent an abrupt change at about 48°C, becoming more dependent above this temperature. The reaction proceeded smoothly at all temperatures investigated. The experimental activation energy of the high temperature reaction was found to be 18.5 kcal $mole^{-1}$ and the entropy of activation and free energy were calculated to be about −33 cal. $mole^{-1}$ $degree^{-1}$ and 30 kcal $mole^{-1}$ at 373°K, respectively. Similar data were collected for the reaction of metallic uranium with liquid and gaseous chlorine trifluoride. (auth)

GENERAL 3.02.18.02

See also, under FISSION PRODUCT, POISON AND RADIOISOTOPE REMOVAL,
DISTILLATION (2.15),
and,
under PROCESS CHEMISTRY AND ENGINEERING,
DISTILLATION AND CONDENSATION (6.06).

DEPOSITION OF METAL ON NONMETAL FILAMENT. T. T. Magel (to U. S. Atomic Energy Commission). U. S. Patent 2,873,185. Feb. 10, 1959.

A method is described for purifying metallic uranium by passing a halogen vapor continuously over the impure uranium to form uranium halide vapor and immediately passing the halide vapor into contact with a nonmetallic refractory surface which is at a temperature above the melting point of uranium metal. The halide is decomposed at the heated surface depositing molten metal, which collects and falls into a receiver below.

See, under Process Chemistry and Engineering, De Boer Process

THERMAL DECOMPOSITION OF URANIUM COMPOUNDS. T. T. Magel and L. Brewer (to U. S. Atomic Energy Commission). U. S. Patent 2,873,184. Feb. 10, 1959.

A method is presented of preparing uranium metal of high purity consisting contacting impure U metal with halogen vapor at between 450 and 550°C to form uranium halide vapor, contacting the uranium halide vapor in the presence of H_2 with a refractory surface at about 1400°C to thermally decompose the uranium halides and deposit molten U on the refractory surface and collecting the molten U dripping from the surface. The entire operation is carried on at a sub-atmospheric pressure of below 1 mm mercury.

PROCESS FOR PREPARING URANIUM METAL. C. H. Prescott, Jr. and F. L. Reynolds (to U. S. Atomic Energy Commission). U. S. Patent 2,868,636. Jan. 13, 1959.

A process is presented for producing oxygen-free uranium metal comprising contacting iodine vapor with crude uranium in a reaction zone maintained at 400 to 800°C to produce a vaporous mixture of UI_4 and iodine. Also disposed within the reaction zone is a tungsten filament which is heated to about 1600°C. The UI_4, upon contacting the hot filament, is decomposed to molten uranium substantially free of oxygen.

cross ref to De Boer

RECOVERY OF IRRADIATED TH AND U 3.02

VOLATILITY METHODS 3.02.18

GENERAL 3.02.18.02

VOLATILE CHLORIDE PROCESS FOR THE RECOVERY OF METAL VALUES. W. R. Hanley (to U. S. Atomic Energy Commission). U. S. Patent 2,867,501. Jan. 6, 1959.

A process is presented for recovering uranium, iron, and aluminum from certain shale type ores which contain uranium in minute quantities. The ore is heated with a chlorinating agent, such as chlorine, to form a volatilized stream of metal chlorides. The chloride stream is then passed through granular alumina which preferentially absorbs the volatile uranium chloride and from which the uranium may later be recovered. The remaining volatilized chlorides, chiefly those of iron and aluminum, are further treated to recover chlorine gas for recycle, and to recover ferric oxide and aluminum oxide as valuable by-products.

13364 ✓ NAA-SR-3219

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

LOW DECONTAMINATION REPROCESSING OF THORIUM-URANIUM ALLOYS BY INDUCTION DRIP MELTING. F. W. Dodge, E. W. Murbach, and L. A. Hanson. Dec. 15, 1958. 20p. Contract AT-11-1-GEN-8. $0.75(OTS).

The feasibility of low decontamination of thorium-uranium alloys by drip melting under vacuum in a high frequency induction field was investigated experimentally. A substantial portion of the rare earths as well as the more volatile fission products were removed and no detectable vaporization of thorium or uranium was found. Fission product removal was improved by extending the heating period, that is, by decreasing the drip rate. A satisfactory, completely remote handling system for in-cave experiments with irradiated fuel was developed. The process shows promise for reprocessing of thorium-uranium fuels if 100% decontamination is not required. The method's simplicity makes it readily adaptable to remote handling techniques, and power requirements are not excessive. (auth)

ENGINEERING AND ECONOMIC CONSIDERATIONS OF VOLATILITY PROCESSES. PART I. ENGINEERING. R. W. Kessie, S. Lawroski, M. Levenson, R. C. Lumatainen, W. J. Mecham, W. A. Rodger, W. B. Seefeldt, G. J. Vogel, and G. E. Goring. May 1, 1957. p.576-613 of SYMPOSIUM ON THE REPROCESSING OF IRRADIATED FUELS HELD AT BRUSSELS, BELGIUM, MAY 20-5, 1957. 1165p. (TID-7534(Bks. 1-3))

Engineering considerations are presented for two types of fluoride volatility processes, high temperature and low temperature. Flow-sheets, corrosion studies, dissolution methods, safety, process chemistry, plant equipment, and pilot plants are discussed. 26 references.

ENGINEERING AND ECONOMIC CONSIDERATIONS OF VOLATILITY PROCESSES. PART II-ECONOMICS. O. J. DuTemple, W. J. Mecham, and G. J. Vogel. May 1, 1957. p.613-66 of SYMPOSIUM ON THE REPROCESSING OF IRRADIATED FUELS HELD AT BRUSSELS, BELGIUM, MAY 20-5, 1957. 1165p. (TID-7534(Bks. 1-3))

An attempt is made to present the probable economic position of volatility processes. Two hypothetical plants (using a low and a high temperature process) are postulated and the capital and operating costs of each are estimated. It should be emphasized that these are hypothetical plants and the costs are calculated without benefit of data from operating plants of this type. 11 references.

PREPARATION OF URANIUM METAL. C. H. Prescott, Jr., and J. A. Holmes (to U. S. Atomic Energy Commission). U. S. Patent 2,785,972. March 19, 1957

A method is given for production of high purity U by decomposing volatile UI_4 on a hot refractory surface. Iodine vapors are passed over solid UI_3 and the UI_4 thus formed is passed over a series of tungsten filaments heated to a temperature above 800°C but below the melting point of U. The UI_4 decomposes depositing pure U on the hot tungsten filaments. The process is carried out in a sealed and evacuated apparatus. (auth)

(See also 6.16.04.)

RECOVERY OF IRRADIATED TH AND U 3.02

ZONE MELTING 3.02.19

See also, this section,

FUSED SALTS AND LIQUID METALS (3.02.11),

and,

under FISSION PRODUCT, POISON, AND RADIOISOTOPE REMOVAL,

DISTILLATION (2.15),

and,

under PROCESS CHEMISTRY AND ENGINEERING,

ZONE MELTING (6.21).

11901 ✓

THE ZONE MELTING OF REFRACTORY METALS INCLUDING RHENIUM AND TUNGSTEN. G. A. Geach and F. O. Jones (Associated Electrical Industries, Ltd., Aldermaston, Berks, Eng.). J. Less-Common Metals 1, 56-9(1959) Feb.

Zone melting as a method of preparation for refractory metals is discussed. In purification of reactive refractory metals arc melting on a water-cooled hearth or electron bombardment floating zone methods are best. The arc zone melting furnace is described along with the electron bombardment floating zone furnace. Techniques of operation are described, and the single crystal growth examined. (J.R.D.)

6295 ✓ NAA-SR-1735

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

FISSION PRODUCT SEPARATION FROM THORIUM–URANIUM ALLOY BY ARC-ZONE MELTING. R. D. Burch and C. T. Young. Apr. 15, 1957. 23p. Contract AT-11-1-GEN-8. $0.25(OTS).

An experimental investigation was made to determine the feasibility of removing fission products from irradiated Th–U alloys by using a zone-melting process. This process utilizes an electric arc to maintain the molten zone. Radiochemical analyses of the processed alloys indicated that U, Te, Zr, and Ru were successfully moved in the direction of zone travel, and that some of the fission products were removed during processing by volatilization. (auth)

6182 ✓

ZONE MELTING OF URANIUM. C. I. Whitman, V. Compton, and R. B. Holden (Sylvania Electric Products Inc., Bayside, N. Y.). J. Electrochem Soc. 104, 240-4 (1957) April.

A study was made of the possible application of the zone melting technique to the separation of uranium from fission products and other impurities. Bar specimens of U having various alloying elements were zone purified in thoria-coated alumina boats. Uranium bars previously irradiated in the Brookhaven pile were also zone melted. Zone melting has potential application in the removal of such impurities as B, Fe, Si, Ni, and Co from ordinary U. In the reprocessing of irradiated U, zone melting offers a method for removing such fission products as Zr, Nb, Ru. However, as employed here, it shows little promise as a method for complete decontamination of irradiated U from fission products, primarily because of concurrent slagging effects. (auth)

INVESTIGATION OF PYROCHEMICAL PROCESSING OF Th–U FUEL. Semi-Annual Report [for] September 1954–February 1955. F. J. Keneshea, Jr., A. G. Buyers, and E. E. Motta. Jan. 15, 1957. Decl. Mar. 14, 1957. 18p. (NAA-SR-1374)

Separation of Ce, rare earths, Cs, Sr, and Te from irradiated Th–U was accomplished by volatilization from a Ta crucible, leaving Pa and Ru. With a TiC crucible the same thing occurred. In a ThO_2 crucible 50% removal of Ce and rare earths occurred, and in ZrO_2 70% separation occurred. In a five-pass arc-zone-melting run Cs and Sr were completely volatilized leaving Ce and the rare earths.

4446 AERE-C/M-243

Gt. Brit. Atomic Energy Research Establishment, Harwell, Berks, England.

STUDY OF THE RADIOCHEMICAL TECHNIQUE EMPLOYED FOR DETERMINING THE BEHAVIOUR OF FISSION PRODUCT RUTHENIUM IN THE PURIFICATION OF IRRADIATED URANIUM BY ZONE MELTING. R. S. Forsyth and D. F. C. Morris. Mar. 1955. ~~Decl. Nov. 9, 1956.~~ 6p.

Some anomalous results have been obtained in an investigation of the behavior of fission products in the purification of irradiated U by zone melting, especially in the case of Ru. The radiochemical technique employed for determining the behavior of Ru has therefore been studied. It is concluded that non uniform pile neutron irradiation of the U rods used in the experiments, is responsible for the apparently anomalous behavior of the fission products which has been observed. (auth)

RECOVERY OF IRRADIATED TH AND U 3.02

ZONE MELTING 3.02.19

ZONE MELTING OF URANIUM. J. E. Antill, E. Barnes, and M. Gardner (Atomic Energy Research Establishment, Harwell, Berks, Eng.). p.9-18 in "Progress in Nuclear Energy. Series V. Metallurgy and Fuels. Volume 2."

Zone melting has been considered as a purification process for small amounts of uranium for experimental purposes. Experiments in a vertical furnace have shown that density differences between an impurity and uranium may be harnessed to increase the purification. Slow zone speeds (1/4 in/hr) are necessary to remove soluble metallic impurities while the non-metallic elements, oxygen and carbon, separate at much faster speeds (3 in/hr). Zone melting applied to the removal of fission products from uranium shows most promise as a modified self-slagging process. (auth)

RECOVERY OF PLUTONIUM 3.03

See also, under PROCESS CHEMISTRY AND ENGINEERING,

SPECIAL PROCESSES--Butex (6.17.03).

ADSORPTION 3.03.01

See also, this section,

ION EXCHANGE (3.03.09),

and,

under PROCESS CHEMISTRY AND ENGINEERING,

EXCHANGERS--Non-resins (6.12.02.02),

Resins-General (6.12.02.04).

ZIRCONIUM PHOSPHATE ADSORPTION METHOD. E. R. Russell, A. S. Adamson, J. Schubert, and G. E. Boyd (to U. S. Atomic Energy Commission). U. S. Patent 2,859,093. Nov. 4, 1958.

A method is presented for separating plutonium values from fission product values in aqueous acidic solution. ~~This is accomplished~~ by flowing the solution ~~containing such values~~ through a bed of zirconium orthophosphate. Any fission products adsorbed can subsequently be eluted by washing the column with a solution of 2N HNO_3 and 0.1N H_3PO_4. Plutonium ~~values~~ may subsequently be desorbed by contacting the column with a solution of 7N HNO_3.

21005

SEPARATION BY ADSORPTION. Charles S. Lowe (to U. S. Atomic Energy Commission). U. S. Patent 2,890,932. June 16, 1959.

Separation of Pu from fission products by adsorption on hydrous aluminum silicate is described. The Pu in a HNO_3 solution is oxidized to the hexavalent state and contacted with the silicate which adsorbs fission products. (T.R.H.)

9757

A METHOD FOR THE SEPARATION OF PLUTONIUM FROM URANIUM. E. Haeffner and A. Hultgren (AB Atomenergi, Stockholm). Nuclear Sci. and Eng. 3, 471-81(1958) May.

A chromatographic method with possibilities for use on a larger scale has been developed for the separation of plutonium from uranium. Some decontamination from extractable fission products is also obtained. The separation from nonextractable fission products is attained in a primary extraction step. (auth)

RECOVERY OF PLUTONIUM 3.03

ADSORPTION 3.03.01

9744 ✓ JENER-54
Joint Establishment for Nuclear Energy Research, Kjeller, Norway.
SEPARATION OF U AND Pu FROM Zr, Nb, Ru, AND Cs ON A COLUMN OF MANGANESE DIOXIDE.
D. Cvjeticanin and N. Cvjeticanin. Apr. 11, 1958. 10p.
The adsorption of U, Pu, Zr, Nb, Ru, Sr–Y, and Cs was examined to provide a good separation of uranium and plutonium from the fission products mentioned. It was established that it is quantitatively possible to wash UO_2^{2+} and PuO_2^{2+} from a column of manganese dioxide with 0.1 N HNO_3. Under the same conditions, 99.2% of Zr^{95}, Nb^{95}, Ru^{106}, and Cs^{137} was adsorbed. (auth)

6227
INVESTIGATIONS ON THE ADSORPTION OF RADIOELEMENTS IN SOLUTION. II. ADSORPTION OF Pu(IV) IN NITRIC MEDIUM. M. Haissinsky and Y. Paiss (Laboratoire Curie, Paris). J. chim. phys. 56, 915-19(1959) Nov.-Dec. (In French)
The adsorption of tetravalent plutonium on platinum, gold, stainless steel, and polyvinyl chloride from nitric solution increases with decrease in acidity. It is shown that in the case of platinum the adsorption increases with the decrease in H^+ and NO_3^- concentration. It follows the Langmuir isotherm, whereas the adsorption in steel and polyvinyl chloride varies linearly with the concentration. An attempt to interpret the processes on metal surfaces is given by using the constitution mechanism of the double layer at the metal/solution interface. (tr-auth)

8513
PROCESS FOR THE PURIFICATION OF PLUTONIUM SOLUTIONS. (to United Kingdom Atomic Energy Authority). British Patent 826,910. Jan. 27, 1960.
A process is presented whereby Pu solutions are purified by adsorption on $Zr_3(PO_4)_4$, $BaIO_3$, or zeolite, followed by elution. In the example given, the Pu solution was passed at 33 ml/min through 4 g of $Zr_3(PO_4)_4$. The desorbing solution to remove fission products was 3.3 l of 0.1 M H_3PO_4 and 2 M HNO_3. The Pu was removed in 5.8 l of 7 M HNO_3. (T.R.H.)

7001
SEPARATION OF PLUTONIUM FROM URANIUM AND FISSION PRODUCTS BY ADSORPTION. G. T. Seaborg and J. E. Willard (to U. S. Atomic Energy Commission). U. S. Patent 2,819,144. Jan. 7, 1958.

A method is presented for the separation of plutonium from solutions containing that element in a valence state not higher than 4^+ together with uranium ions and fission products. This separation is accomplished by contacting the solutions with diatomaceous earth which preferentially adsorbs the plutonium present. Also mentioned as effective for this adsorbtive separation are silica gel, fuller's earth and alumina.

11506
SEPARATION PROCESS BY ADSORPTION. W. W. Schulz et al. (to U. S. Atomic Energy Commission). U. S. Patent 2,796,424. June 18, 1957.
The process of this invention is applicable to the separation of Ru and Zr and/or Nb values from aqueous raffinate solutions containing U obtained when neutron-irradiated U is processed to recover Pu. This separation is accomplished by adding to an aqueous solution containing mineral acid salts of U, Ru, Zr or Nb, an organic-sulfo compound such as a mercaptan, theioether or alkyl disulfide, whereby the Ru salt is complexed, and passing the solution over silica gel whereby the Ru complex, the Nb and Zr salts are adsorbed on said silica gel. (auth)

10782 HW-24115
[Hanford Works, Richland, Wash.]
THE MECHANISM OF CARRYING Pu(III) ON LANTHANUM FLUORIDE (thesis). Delebert Lloyd Ralphs. Apr. 15, 1952. Decl. Feb. 1, 1956. 53p. Contract [W-31-109-Eng-52].
Thesis submitted to Oregon State Coll. $9.30(ph OTS); $3.60(mf OTS).

11619 ✓ CN-508
[Chicago. Univ. Metallurgical Lab.]
DECONTAMINATION OF PRODUCT BY ADSORPTION ON TAILOR-MADE INORGANIC ADSORBENTS. E. R. Russell, A. W. Adamson, J. Schubert, and G. E. Boyd. Mar. 6, 1943. Decl. Mar. 21, 1957. 23p. (A-653). $4.80(ph OTS); $2.70(mf OTS).

RECOVERY OF PLUTONIUM 3.03

ADSORPTION 3.03.01

The surface properties of various substances known to carry Pu were studied in order to find a method of separating Pu from a large number of adsorbed fission products. Of these substances, zirconium phosphate seemed to offer promise as a specific adsorbent for Pu. A number of exploratory experiments were performed, and the results are summarized. (B.J.H.)

6504 A/CONF.15/P/144
SEPARATION OF PLUTONIUM FROM URANIUM BY PARTITION BETWEEN AN ORGANIC SOLVENT AND A STATIONARY WATER PHASE ADSORBED ON SILICA GEL. Å. Hultgren and E. Haeffner (AB Atomenergi, Stockholm). 11p.

A chromatographic method for the separation of plutonium from uranium was developed. A certain decontamination from extractable fission products is also obtained. Non-extractable fission products are separated in a primary extraction step which is carried out countercurrently in an extraction column. The conditions are adjusted so as to reach as quantitative an extraction of uranium and plutonium as possible and to give the highest possible decontamination of these elements from the fission products. At the same time the acidity of the organic product stream must not exceed a predetermined limit. The organic product stream is then passed downwards through a silica gel column, in which the silica gel has been packed in water or dilute nitric acid and the free water removed using pure organic solvent. Because of different partition coefficients, the uranium leaves the column quickly while the plutonium is practically fixed in the stationary water phase of the column. After a certain volume of primary organic solution has passed the column, the uranium left in the column is eluted with pure solvent, to which some nitric acid may be added. The plutonium is then eluted separately, either with a more acid portion of the organic solvent, or with nitric acid in water. The β-decontamination is better if the elution is carried out with organic solvent. It is possible to increase the capacity in each run considerably by loading the stationary water phase with a reducing agent to reduce the plutonium to non-extractable Pu(III) and thus fix it more effectively in the water phase. In this case, however, the plutonium can be eluted only with nitric acid in water.

2491
COLUMBIC OXIDE ADSORPTION PROCESS FOR SEPARATING URANIUM AND PLUTONIUM IONS. R. H. Beaton (to U. S. Atomic Energy Commission). U. S. Patent 2,894,810. July 14, 1959.

A process is described for separating plutonium ions from a solution of neutron irradiated uranium in which columbic oxide is used as an adsorbent. According to the invention the plutonium ion is selectively adsorbed by passing a solution containing the plutonium in a valence state not higher than 4 through a porous bed or column of granules of hydrated columbic oxide. The adsorbed plutonium is then desorbed by elution with 3 N nitric acid.

CHELATION 3.03.02

See also, this section,
ION EXCHANGE (3.03.09),
SOLVENT EXTRACTION (3.03.14),
and,
under PROCESS CHEMISTRY AND ENGINEERING,
CHELATION (6.02),
SPECIAL PROCESSES--Bismuth Phosphate (6.17.02).

10990
SEPARATION OF PLUTONIUM. U. K. Atomic Energy Authority (U.S.A.). British Patent 801,520. Nuclear Eng. 4, 188(1959) Apr.

The separation of plutonium from fission products in aqueous solutions is effected by forming a water-insoluble complex of plutonium with cupferron. The complex is probably chelate. The method can be employed in an advanced phase of the process of isolating plutonium from neutron irradiated uranium.

SEPARATION OF PLUTONIUM FROM LANTHANUM BY CHELATION-EXTRACTION. R. A. James and S. G. Thompson (to U. S. Atomic Energy Commission). U. S. Patent 2,863,892. Dec. 9, 1958.

Plutonium can be separated from a mixture of plutonium and lanthanum in which the lanthanum to plutonium

RECOVERY OF PLUTONIUM 3.03

CHELATION 3.03.02

THE FEASIBILITY OF CONTINUOUS CHELATION. H. H. Willis. Apr. 29, 1949. ~~Decl. Mar. 28, 1957~~. 52p. (KLX-44)

Recovery of Pu from $BiPO_4$ waste solutions by continuous chelation appears feasible with either of the 3 feed solutions: (a) solutions of Na diuranate ppt., (b) the IAW stream of the Redox process with a Pu reducing agent present, and, (c) IAW solution with $Al(NO_3)_3$ removed.

2367 ISC-14
Ames Lab., Ames Iowa.
THE CHELATE COMPOUNDS OF PLUTONIUM (thesis). Frederick John Wolter. May 25, 1946. Decl. Jan. 6, 1956. 104p. Contract W-7405-eng-82. $16.80 (ph OTS); $5.70 (mf OTS).

CHLORINATION 3.03.03

See also, under PROCESS CHEMISTRY AND ENGINEERING, CHLORINATION (6.03).

METHOD OF SEPARATING URANIUM VALUES, PLUTONIUM VALUES AND FISSION PRODUCTS BY CHLORINATION. H. S. Brown and G. T. Seaborg (to U. S. Atomic Energy Commission). U. S. Patent 2,875,021. Feb. 24, 1959.

The separation of plutonium and uranium from each other and from other substances is described. In general, the method comprises the steps of contacting the uranium with chlorine in the presence of a holdback material selected from the group consisting of lanthanum oxide and thorium oxide to form a uranium chloride higher than uranium tetrachloride, and thereafter heating the uranium chloride thus formed to a temperature at which the uranium chloride is volatilized off but below the volatilization temperature of plutonium chloride.

THE EXTRACTION OF PLUTONIUM FROM NEUTRON-IRRADIATED URANIUM BY URANIUM TRICHLORIDE AND BY MAGNESIUM CHLORIDE. D. E. McKenzie, W. L. Elsdon, and J. W. Fletcher (Atomic Energy of Canada Ltd., Chalk River, Ont., Can.). Can. J. Chem. 36, 1233-40 (1958) Sept.

The extraction of plutonium from neutron-irradiated uranium by uranium trichloride and by magnesium chloride was examined in the temperature range 1150 to 1250°C. The results of the uranium trichloride experiments were used to evaluate the standard free-energy change of the equilibrium reaction $UCl_3 + Pu \rightleftharpoons PuCl_3 + U$. These free-energy values were used to predict the plutonium extraction in the magnesium chloride experiments on the assumption that the uranium trichloride, continuously formed from the non-equilibrium reaction $\frac{3}{2} MgCl_2 + U \rightarrow UCl_3 + \frac{3}{2} Mg$, rapidly established the above equilibrium with the plutonium in the uranium. Agreement between predicted and experimental results supports this assumption. A preliminary study of the kinetics of the $MgCl_2$–U reaction showed that the initial rate of reaction is controlled by the distillation of magnesium metal from the reaction zone. (auth)

PROCESS OF FORMING PLUTONIUM SALTS FROM PLUTONIUM OXALATES. C. S. Garner (to U. S. Atomic Energy Commission). U. S. Patent 2,875,025. Feb. 24, 1959.

A process is presented for converting plutonium oxalate to other plutonium compounds by a dry conversion method. According to the process, lower valence plutonium oxalate is heated in the presence of a vapor of a volatile non-oxygenated monobasic acid, such as HCl or HF. For example, in order to produce plutonium chloride, the pure plutonium oxalate is heated to about 700°C in a slow stream of hydrogen plus HCl. By the proper selection of an oxidizing or reducing atmosphere, the plutonium halide product can be obtained in either the plus 3 or plus 4 valence state.

RECOVERY OF PLUTONIUM 3.03

CHELATION 3.03.02

molal ratio is at least five by adding the ammonium salt of N-nitrosoarylhydroxylamine to an aqueous solution having a pH between about 3 and 0.2 and containing the plutonium in a valence state of at least +3, to form a plutonium chelate compound of N-nitrosoarylhydroxylamine. The plutonium chelate compound may be recovered from the solution by extracting with an immiscible organic solvent such as chloroform.

SEPARATION PROCESS FOR TRANSURANIC ELEMENT AND COMPOUNDS THEREOF. M. Calvin (to U. S. Atomic Energy Commission). U. S. Patent 2,856,418. Oct. 14, 1958.

A process is presented for the separation of plutonium from uranium and fission products in an aqueous acidic solution by use of a chelating agent. The plutonium is maintained in the tetravalent state and the uranium in the hexavalent state, and the acidic concentration is adjusted to about 1 N. The aqueous solution is then contacted with a water-immiscible organic solvent solution and the chelating agent. The chelating agents covered by this invention comprise a group of compounds characterized as fluorinated beta-diketones.

14410
SEPARATION PROCESS USING COMPLEXING AND ADSORPTION. J. H. Spedding and J. A. Ayers (to U. S. Atomic Energy Commission). U. S. Patent 2,837,548. June 3, 1958.

An adsorption process is described for separating plutonium from a solution of neutron-irradiated uranium containing ions of a compound of plutonium and other cations. The method consists of forming a chelate complex compound with plutonium ions in the solution by adding a derivative of 8-hydroxyquinoline, which derivative contains a sulfonic acid group, and adsorbing the remaining cations from the solution on a cation exchange resin, while the complexed plutonium remains in the solution.

2332 UCRL-126
California. Univ., Berkeley. Radiation Lab.
THE CHELATE PROCESS III. PRELIMINARY CONTINUOUS-OPERATION STUDIES. B. Rubin and T. E. Hicks. [nd]. Decl. Dec. 6, 1955. 26p. Contract [W-7405-eng-48]. $4.80(ph OTS); $2.70(mf OTS).

The rate of extraction of Pu^{IV} in either direction between benzene-TTA and aqueous HNO_3 solutions is shown in the present study to be fast enough so that application of the chelate process to continuous operation should be feasible. This conclusion is based on the two primary considerations that the mechanism limiting the rate of extraction appears to be diffusion-and not chemical-reaction-limiting, and that the extraction may be controlled by adjustment of the system conditions to give almost any desired specifications of rate and/or equilibrium. A continuous process involving multiple extraction stages with a controlled pH gradient is proposed for obtaining maximum decontamination and plutonium recovery. (auth)

12407 UCRL-400(Rev.)
California. Univ., Berkeley. Radiation Lab.
THE CHELATE PROCESS. V. INVESTIGATION IN HORIZONTAL EXTRACTORS. T. E. Hicks, B. Rubin, and T. Vermeulen. July 29, 1949. Decl. Mar. 18, 1957. 47p. Contract W-7405-eng-48. $7.80(ph OTS); $3.30(mf OTS).

The recovery of plutonium has been demonstrated in a laboratory countercurrent horizontal extractor using the TTA process. Using three extractive stages and two washing stages in each step, a recovery of 91% has been obtained in the chelation step and a recovery of 99.9+ percent in the de-chelation step. With five extractive stages, recoveries of 94 to 98% have been found for the chelation step. A calculation method has been developed in order to correlate these continuous-flow results with rate data for batch experiments. This correlation utilizes dynamic extraction coefficients that are calculated from the rate data and the equilibrium extraction coefficients. Values for column recovery of plutonium are predicted that are in agreement with the observed results. On the basis of this agreement, recovery and decontamination factors are predicted for a pair of center-fed extraction columns of seven stages each.

RECOVERY OF PLUTONIUM 3.03

COMPLEX FORMATION 3.03.04

3573 IGO-R/W-2
United Kingdom Atomic Energy Authority. Industrial Group. Windscale Works, Sellafield, Cumb., England.
COMPLEXES OF PLUTONIUM (IV) WITH PHENYL-ARSONIC ACID DERIVATIVES. PART II. 2-(o-ARSONOPHENYLAZO)-1:8-DIHYDROXY-3:6-NAPHTHALENEDISULPHONIC ACID (URANOL), AND OTHERS. D. W. Ockenden. Feb. 1, 1956. 37p. (IGC-ARDC/P-150).

It is shown that tetravalent plutonium and uranol form a violet blue complex in 0.1M nitric acid solution. The complex was formed quantitatively and immediately only over the pH range 0.95 to 1.35. The dissociation constant of the complex is approximately determined, and the behavior of other ions, e.g., UO_2^{2+}, Fe^{3+}, Cr^{3+}, Al^{3+}, La^{3+}, and Th^{4+}, with the reagent was studied. Unfortunately, owing to slight fading, this reagent is no better for the absorptiometric determination of plutonium than thoronol (1-(o-arsonophenylazo)-2-naphthol-3:6-disulphonic acid). Many other reagents of similar structure have been investigated for this purpose, but few show any appreciable reaction with tetravalent plutonium even in the ultra-violet region. (auth)

1763

CHLORIDE COMPLEXING AND DISPROPORTIONATION OF PU(IV) IN HYDROCHLORIC ACID. S. W. Rabideau and H. D. Cowan (Los Alamos Scientific Lab., N. Mex.). J. Am. Chem. Soc. 77, 6145-8(1955) Dec. 5.

It has been shown from measurements of the formal potentials of the $Pu^{3+}-Pu^{4+}$ couple in $HCl-HClO_4$ solutions at constant total acidity and ionic strength that the complex ion $PuCl^{3+}$ is formed. The dissociation quotient of this complex at 25° has been found to be 1.77 ± 0.10 at unit ionic strength and 1.70 ± 0.03 at an ionic strength of two. At 25° in molar hydrochloric acid the thermodynamic quantities for the reaction $PuCl^{3+} \rightleftharpoons Pu^{4+} + Cl^-$ are $\Delta F = -0.34$ kcal/mole, $\Delta H = -1.9$ kcal/mole and $\Delta S = -5$ eu. Equilibrium and rate measurements were made in hydrochloric acid solutions at 6, 25, 35, and 45° in a study of the disproportionation reaction $3Pu^{4+} + 2H_2O \rightleftharpoons 2Pu^{3+} + PuO_2^{2+} + 4H^+$. In accord with this reaction, a fourth power hydrogen ion dependence of the equilibrium quotient was observed. The disproportionation reaction rate constant exhibited an inverse third power dependence upon the hydrogen ion concentration. Values of 39 kcal/mole and 53 eu. were obtained for the heat of activation and for the entropy of activation, respectively, for the disproportionation reaction in molar hydrochloric acid. The change in the heat content at 25° for the reaction $Pu^{3+} + H^+ \rightleftharpoons Pu^{4+} + \frac{1}{2}H_2$ was calculated to be 14.32 kcal/mole in molar hydrochloric acid and 13.63 kcal/mole in molar perchloric acid from measurements of the formal potential of the $Pu^{3+}-Pu^{4+}$ couple as a function of temperature. The $Pu^{3+}-PuO_2^{2+}$ potential in molar hydrochloric acid was calculated from the measured $Pu^{3+}-Pu^{4+}$ formal potential and from the disproportionation equilibrium quotient at each temperature. For the reaction $Pu^{3+} + 2H_2O \rightleftharpoons PuO_2^{2+} + H^+ + 3/2H_2$ in molar hydrochloric acid at 25°, $\Delta F = 70.8$ kcal/mole, $\Delta H = 78.4$ kcal/mole and $\Delta S = 25.4$ eu. The ionic entropies of PuO_2^{2+} and Pu^{4+} have been calculated to be −28.9 and −87 eu., respectively. (auth)

7170 ✓ ANL-4573
Argonne National Lab., Lemont, Ill.
SPECTROPHOTOMETRIC STUDIES OF PLUTONIUM(IV) AND (VI) NITRATES IN METHYL ISOBUTYL KETONE. Michael G. Berkman and Louis Kaplan. Jan. 1951. Decl. Jan. 5, 1956. 21p. Contract W-31-109-eng-38. $0.25(OTS).

Changes in the spectra of plutonium(IV) solutions in methyl isobutyl ketone (Hexone) upon the addition of tetrabutylammonium nitrate are caused by the formation of the stable hexanitrate complex $Pu(NO_3)_6^=$. The similarity of the spectrum of this complex to that of plutonium(IV) in 11M aqueous nitric acid confirms the existence of the hexanitrate in the latter solution. Nitric acid is much less effective than is tetrabutylammonium nitrate with respect to

RECOVERY OF PLUTONIUM 3.03

COMPLEX FORMATION 3.03.04

See also, this section,
ION EXCHANGE (3.03.09),
PRECIPITATION (3.03.12),
and,
under CHEMISTRY AND PHYSICS OF IMPORTANT ELEMENTS,
PLUTONIUM--Complex Formation (1.28.06.01),
and,
under PROCESS CHEMISTRY AND ENGINEERING,
COMPLEX FORMATION (6.05).

14397

PROCESS FOR SEPARATION OF HEAVY METALS. R. B. Duffield (to U. S. Atomic Energy Commission). U. S. Patent 2,832,793. Apr. 29, 1958.

A method is described for separating plutonium from aqueous acidic solutions of neutron-irradiated uranium and the impurities associated therewith. The separation is effected by adding, to the solution containing hexavalent uranium and plutonium, acetate ions and the ions of an alkali metal and those of a divalent metal and thus forming a complex plutonium acetate salt which is carried by the corresponding complex of uranium, such as sodium magnesium uranyl acetate. The plutonium may be separated from the precipitated salt by taking the same back into solution, reducing the plutonium to a lower valent state on reprecipitating the sodium magnesium uranyl salt, removing the latter, and then carrying the plutonium from the solution by means of lanthanum fluoride.

13376

PLUTONIUM-CUPFERRON COMPLEX AND METHOD OF REMOVING PLUTONIUM FROM SOLUTION. H. A. Potratz (to U. S. Atomic Energy Commission). U. S. Patent 2,868,817. Jan. 13, 1959.

A method is presented for separating plutonium from fission products present in solutions of neutron-irradiated uranium. The process consists in treating such acidic solutions with cupferron so that the cupferron reacts with the plutonium present to form an insoluble complex. This plutonium cupferride precipitates and may then be separated from the solution.

8376

DETERMINATION OF THE COMPOSITION AND DISSOCIATION CONSTANT FOR OXALATE COMPLEXES OF Pu^{+3} COMPOUNDS BY ION EXCHANGE. A. D. Gelman, N. N. Matorina, and A. I. Moskvin. Atomnaya Energiya 4, 52-6(1958) Jan. (In Russian)

The method of ionic exchange was used in the investigation of the Pu^{3+} complex formation in oxalate solutions in the interval pH 1.4 to 3.0. It has been shown that the complex ions $Pu(C_2O_4)_2^-$ and $Pu(HC_2O_4)_2^-$ with the instability constants $K_1 = 7.1 \times 10^{-10}$ and $K_2 = 1.1 \times 10^{-11}$ are formed in the given pH range. The magnitudes of $Pu(C_2O_4)_2^-$ instability constants coincide satisfactorily with the instability constants of the corresponding ionic complex obtained by the solubility method. (tr-auth)

SPECTROSCOPIC STUDIES OF Pu^{+3} COMPLEX FORMATION AND STABILITY. A. D. Gel'man and A. I. Moskin. Atomnaya Energiya 3, No. 10, 314-16(1957).

The complex formation of Pu^{3+} with $C_2O_4^{2-}$, $C_2O_3^{2-}$, and $C_6H_5O_7^{3-}$ ions as well as with trylon B is proved by means of the spectrograph. The absorption spectra were measured, and their characteristic maxima were found at the following wave lengths: 565, 605, 665, 780 to 790. 905 to 910, 1090 mμ. (TCO)

3228

PLUTONIUM RECOVERY PROCESS. O. F. Hill and S. G. Thompson (to U. S. Atomic Energy Commission). U. S. Patent 2,767,044. Oct. 16, 1956.

In order to separate Pu from an aqueous solution of U and fission product salts a cycle of carrier precipitations is employed. First, the Pu is oxidized to the hexavalent state, and carbonate ion is added to the solution to complex the Pu. By addition of an aqueous solution of a La salt, a basic lanthanum carbonate precipitate is formed, which removes much of the Ba, Zr and Nb by carrying but does not affect hexavalent Pu. The filtered solution is then treated with a reducing agent which will reduce Pu but not the uranyl ion. The Pu is then removed from the solution by formation of a bismuth hydroxide carrier precipitate. (auth)

RECOVERY OF PLUTONIUM 3.03

ELECTROCHEMICAL 3.03.05

See also, this section,

FUSED SALTS AND LIQUID METALS (3.03.07),

and,

under PROCESS CHEMISTRY AND ENGINEERING,

ELECTROCHEMISTRY (6.07),

FUSED SALTS--Electrochemistry (6.10.01.02).

255 DP-388
Du Pont de Nemours (E. I.) & Co., Savannah River Lab., Aiken, S. C.
ELECTRODEPOSITION OF PLUTONIUM AT A MERCURY CATHODE. John A. Porter. June 1959. 14p. Contract AT(07-2)-1. OTS.

Plutonium was electrodeposited at a mercury cathode from an ethanol solution of plutonium trichloride. The electrodeposition process was studied, using cerium(III) as a stand-in for plutonium(III). (auth)

14395
PROCESS OF SEPARATING PLUTONIUM VALUES BY ELECTRODEPOSITION. A. C. Whal (to U. S. Atomic Energy Commission). U. S. Patent 2,830,939. Apr. 15, 1958.

A process is described of separating plutonium values from an aqueous solution by electrodeposition. The process consists of subjecting an aqueous 0.1 to 1.0 N nitric acid solution containing plutonium ions to electrolysis between inert metallic electrodes. A current density of one milliampere to one ampere per square centimeter of cathode surface and a temperature between 10 and 60°C are maintained. Plutonium is electrodeposited on the cathode surface and recovered.

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. (KAPL-1692)

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, Pu 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.

6211 ✓ HW-32673
Hanford Atomic Products Operation, Richland, Wash.
THE ELECTRODEPOSITION OF PLUTONIUM FROM ACID SOLUTION. R. Ko. Sept. 7, 1954. Decl. Jan. 6, 1956. 11p. Contract W-31-109-eng-52. $0.15(OTS).

Plutonium in the tetravalent state is electrodeposited from an initially acid solution as $Pu(OH)_4$ by electrolytic base formation. Base is formed at the cathode by reduction of hydrogen and nitrate ions. The formation of acid at the anode is prevented in part by the oxidation of oxalate ion at that electrode. Plutonium is electrodeposited at 320 ma from a solution of 0.25M HNO_3, 0.10M $(NH_4)_2C_2O_4$ at stainless steel and Al cathodes with a Pt disc anode placed within 10 mm of the cathode and rotated at 200 rpm. More than 95% of the plutonium added to the cell (0.001 μg to mg) is deposited in one hour. (auth)

Refer also to abstract 6131.

8507 DP-389
Du Pont de Nemours (E. I.) & Co. Savannah River Lab., Aiken, S. C.
A SURVEY OF ORGANIC SOLVENTS FOR THE ELECTRODEPOSITION OF PLUTONIUM. John A. Porter. July 1959. 12p. Contract AT(07-2)-1. OTS.

The solubility and conductivity of cerium(III) salts (as stand-ins for plutonium(III) salts) in dimethylsulfoxide, acetonitrile, ethylenediamine, and dimethylformamide were investigated to evaluate these systems as possible electroplating baths. The system dimethylsulfoxide–cerium chloride is the most promising for electrodeposition of cerium at a mercury cathode. (auth)

RECOVERY OF PLUTONIUM 3.03

FLUORINATION 3.03.06

13378
RECOVERY OF Pu VALUES BY FLUORINATION AND FRACTIONATION. H. S. Brown and D. S. Webster (to U. S. Atomic Energy Commission). U. S. Patent 2,869,982. Jan. 20, 1959.

A method is presented for the concentration and recovery of plutonium by fluorination and fractionation. A metallic mass containing uranium and plutonium is heated to 250°C and contacted with a stream of elemental fluorine. After fluorination of the metallic mass, the reaction products are withdrawn and subjected to a distillation treatment to separate the fluorination products of uranium and to obtain a residue containing the fluorination products of plutonium.

52 ANL-5875
Argonne National Lab., Lemont, Ill.
THE FLUORINATION OF PLUTONIUM TETRAFLUORIDE. M. J. Steindler, D. V. Steidl, and R. K. Steunenberg. June 1958. 29p. Contract W-31-109-eng-38. $1.00(OTS).

The fluorination of plutonium tetrafluoride by elemental fluorine to form plutonium hexafluoride was studied in a flow system at temperatures between 100 and 600°C. Partial pressures of fluorine from 0.25 to 1.0 atmosphere were used. The plutonium tetrafluoride was obtained from several sources, including the dissolution of uranium-plutonium alloys in bromine trifluoride. Activation energies between 10 and 12 kcal/mole were obtained. Details are included on the techniques and precautions employed in handling powdered plutonium compounds and plutonium hexafluoride. (auth)

3447
SEPARATION OF PLUTONIUM AND URANIUM FROM FISSION PRODUCTS. J. K. Dawson and A. E. Truswell (to U. K. Atomic Energy Authority). British Patent 778,051. Nuclear Eng. 2, 494(1957) Nov.

Production of plutonium and recovery of uranium from neutron irradiated uranium by organic solvent extraction of the nitric acid solution of the irradiated uranium in a number of stages does not readily eliminate all fission products from the uranium and plutonium. Some products, particularly ruthenium, present difficult problems. A simple method of separating plutonium and/or uranium, especially from ruthenium and rhodium, consists in treating the metal or compound with a gaseous fluorinating agent (hydrogen fluoride; chlorine trifluoride; bromide trifluoride) in admixture with 2 to 10% of a gaseous oxidizing agent (air; oxygen). (auth)

87 KAPL-M-NJH-2
Knolls Atomic Power Lab., Schenectady, N. Y.
PROCESS OF THE STORAGE OF PLUTONIUM HEXAFLUORIDE. Norval J. Hawkins. [1957]. Decl. Oct. 15, 1959. 9p. Contract W-31-109-eng-52. OTS.

Freshly prepared PuF_6 may be stored by dissolving in liquid carbon tetrafluoride (CF_4) in quartz or glass vessels maintained slightly warmer than liquid nitrogen temperature (−195°C) to be certain that the solvent is initially in the liquid state. The solution, of predetermined composition, is maintained at liquid nitrogen temperature for the period under storage. Storage vessels in a supply of liquid nitrogen can be designed which are quite efficient as far as liquid nitrogen evaporation is concerned. To obtain the PuF_6 again in a pure state, the solvent is removed by distillation. This can easily be accomplished by replacing the liquid nitrogen bath by a dry ice−trichloroethylene bath (−78°C) and pumping off the solvent, CF_4, as well as any F_2 and C_2F_6 produced. Very little PuF_6 should be lost by such a procedure. (auth)

7301 HW-26022
Hanford Works, Richland, Wash.
REPORT OF INVENTION ON THE PREPARATION OF PLUTONIUM TRIFLUORIDE. L. L. Burger and W. E. Roake. Oct. 22, 1952. Decl. Jan. 28, 1960. 3p. OTS.

A method is described for the preparation of PuF_3 by means of a solid phase-gas reaction between Pu(IV) oxalate or Pu(III) oxalate and Freon-type compounds such as CCl_2F_2. (auth)

RECOVERY OF PLUTONIUM 3.03

FLUORINATION 3.03.06

See also, this section,
FUSED SALTS AND LIQUID METALS (3.03.07),

and,

under PROCESS CHEMISTRY AND ENGINEERING,
FLUORINATION (6.09).

6360
SEPARATION OF PLUTONIUM. A. G. Maddock and F. Smith (to U. S. Atomic Energy Commission). U. S. Patent 2,901,313. Aug. 25, 1959.

A method is described for separating plutonium from uranium and fission products by treating a nitrate solution of fission products, uranium, and hexavalent plutonium with a relatively water-insoluble fluoride to adsorb fission products on the fluoride, treating the residual solution with a reducing agent for plutonium to reduce its valence to four and less, treating the reduced plutonium solution with a relatively insoluble fluoride to adsorb the plutonium on the fluoride, removing the solution, and subsequently treating the fluoride with its adsorbed plutonium with a concentrated aqueous solution of at least one of a group consisting of aluminum nitrate, ferric nitrate, and manganous nitrate to remove the plutonium from the fluoride.

1321 ANL-5796
Argonne National Lab., Lemont, Ill.
THE TRANSFER OF PLUTONIUM HEXAFLUORIDE IN THE VAPOR PHASE. M. D. Adams, R. K. Steunenberg, and R. C. Vogel. Oct. 1957. 14p. Contract W-31-109-eng-38. $0.50(OTS).

Plutonium hexafluoride is decomposed by heat, by its alpha radiation, and by reactions with most materials of construction. Laboratory experiments were performed to investigate means by which plutonium losses due to these reactions can be minimized during vapor-phase transfers of the compound. The experiments showed that plutonium hexafluoride vapor could be transferred satisfactorily through well fluorinated nickel equipment in a stream of fluorine or helium. The decomposition product of plutonium hexafluoride was refluorinated at 250°C by fluorine. (auth)

4379
THE FLUORINATION OF PLUTONIUM TETRAFLUORIDE AND PLUTONIUM DIOXIDE BY FLUORINE. M. J. Steindler, D. V. Steidl, and R. K. Steunenberg (Argonne National Lab., Lemont, Ill.). Nuclear Sci. and Eng. 6, 333-40(1959) Oct.

The conversion of plutonium tetrafluoride and plutonium dioxide to the hexafluoride by elemental fluorine was investigated in a flow system at temperatures between 100 and 600°C. The partial pressure of fluorine used was varied from 0.25 to 1.0 atmosphere. The rates of fluorination were dependent on the source of the starting material, probably due to differences in particle size. Activation energies of the reaction were between 10 and 12 kcal per mole. The reactions are believed to show promise for process application in the recovery of plutonium from nuclear reactor fuels. (auth)

2391
PLATINUM HEXAFLUORIDE AND METHOD OF FLUORINATING PLUTONIUM CONTAINING MIXTURES THEREWITH. J. G. Malm, B. Weinstock, and H. H. Claassen (to U. S. Atomic Energy Commission). U. S. Patent 2,893,826. July 7, 1959.

The preparation of platinum hexafluoride and its use as a fluorinating agent in a process for separating plutonium from fission products is presented. According to the invention, platinum is reacted with fluorine gas at from 900 to 1100°C to form platinum hexafluoride. The platinum hexafluoride is then contacted with the plutonium containing mixture at room temperature to form plutonium hexafluoride which is more volatile than the fission products fluorides and therefore can be isolated by distillation.

13387
RECOVERY OF Pu FROM CERIUM TRIFLUORIDE BY FLUORINATION. H. S. Brown and E. G. Bohlmann (to U. S. Atomic Energy Commission). U. S. Patent 2,873,168. Feb. 10, 1959.

An improved process is presented for selectively recovering plutonium from a solution containing fission products comprising precipitating cerium trifluoride in the solution for effecting carrier precipitation of plutonium. The resulting carrier precipitate is dried and subjected to fluorination at about 600°C. The plutonium forms a volatile fluoride and is so separated from the nonvolatile cerium fluoride.

RECOVERY OF PLUTONIUM 3.03

FUSED SALTS AND LIQUID METALS 3.03.07

See also, this section,

FLUORINATION (3.03.06),

ION EXCHANGE (3.03.09),

MAGNESIUM EXTRACTION (3.03.10).

22170
PYROCHEMICAL DECONTAMINATION METHOD FOR REACTOR FUEL. A. G. Buyers (to U. S. Atomic Energy Commission). U. S. Patent 2,892,701. June 30, 1959.

A pyro-chemical method is presented for decontaminating neutron irradiated uranium and separating plutonium therefrom by contact in the molten state with a metal chloride salt. Uranium trichloride and uranium tetrachloride either alone or in admixture with alkaline metal and alkaline earth metal fluorides under specified temperature and specified phase ratio conditions extract substantially all of the uranium from the irradiated uranium fuel together with certain fission products. The phases are then separated leaving purified uranium metal. The uranium and plutonium in the salt phase can be reduced to form a highly decontaminated uranium-plutonium alloy. The present method possesses advantages for economically decontaminating irradiated nuclear fuel elements since irradiated fuel may be processed immediately after withdrawal from the reactor and the uranium need not be dissolved and later reduced to the metallic form. Accordingly, the uranium may be economically refabricated and reinserted into the reactor.

14809 ✓ A/CONF.15/P/517
Ames Lab., Ames, Iowa and Iowa State Coll., Ames. Inst. for Atomic Research.
PYROMETALLURGICAL PROCESSING. Premo Chiotti and Adolf F. Voigt. 17p. (ISC-1018). $0.50(OTS).

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 plutonium from uranium and the separation of uranium from thorium. The use of thermodynamic data in estimating the effectiveness of various pyrometallurgical separations is considered. Those fission products which extract into silver, including the alkaline earths, rare earths, and others, can readily be removed from the silver layer by melting the latter with sodium chloride containing a small amount of silver chloride. The silver is thus recovered in good condition for reuse and the tated as thorium dihydride was investigated. The major part of the thorium can be precipitated at 675°C and one atmosphere hydrogen pressure. Under these conditions the residual thorium content in the magnesium was found to be 8.1 wt. %. More complete separation can be obtained by lowering the temperature and/or increasing the hydrogen pressure. (auth)

2392
PREPARATION OF PLUTONIUM. M. Kolodney (to U. S. Atomic Energy Commission). U. S. Patent 2,893,928. July 7, 1959.

Methods are presented for the electro-deposition of plutonium from fused mixtures of plutonium halides and halides of the alkali metals and alkaline earth metals. The salts, preferably chlorides and with the plutonium preferably in the trivalent state, are placed in a refractory crucible such as tantalum or molybdenum and heated in a non-oxidizing atmosphere to 600 to 850°C, the higher temperatures being used to obtain massive plutonium and the lower for the powder form. Electrodes of graphite or non-reactive refractory metals are used, the crucible serving as the cathode in one apparatus described in the patent.

22507
NON-AQUEOUS DISSOLUTION OF MASSIVE PLUTONIUM. J. G. Reavis, J. A. Leary, and K. A. Walsh (to U. S. Atomic Energy Commission). U. S. Patent 2,886,410. May 12, 1959.

A method is presented for obtaining non-aqueous solutions of plutonium from massive forms of the metal. In the present invention massive plutonium is added to a salt melt consisting of 10 to 40 weight per cent of sodium chloride and the balance zinc chloride. The plutonium reacts at about 800°C with the zinc chloride to form a salt bath of plutonium trichloride, sodium chloride, and metallic zinc. The zinc is separated from the salt melt by forcing the molten mixture through a Pyrex filter.

RECOVERY OF PLUTONIUM 3.03

FUSED SALTS AND LIQUID METALS 3.03.07

found to be −5 kcal per equivalent instead of the −8 kcal in the literature. The distribution of the fission products was essentially as predicted from thermodynamic data assuming that UF_4 in the salt was the oxidizing agent. Cerium, rare earths, Sr, and Cs activities appeared predominantly in the salt. Ruthenium did not concentrate in the salt-phase, but remained in the metal after contacting. Tellurium was removed in an erratic fashion which was not unexpected considering its volatility and instability as the fluoride. (auth)

4121 TID-5189
Ames Lab., Ames, Iowa.
HIGH TEMPERATURE FUEL PROCESSING CONFERENCE HELD AT AMES LABORATORY, NOVEMBER 10-11, 1953. Oct. 6, 1954. Decl. Mar. 4, 1957. 44p. Contract W-7405-eng-82. $1.25(OTS).

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 extrusion, electrolytic refining, C slagging coupled with UCl_3 extraction, fluorine extraction, and liquid metal extraction. (C.H.)

3348 NAA-SR-926
North American Aviation, Inc. Downey, Calif.
THE DISTRIBUTION OF TRACER PLUTONIUM AND FISSION PRODUCTS BETWEEN MOLTEN URANIUM METAL AND MOLTEN URANIUM TETRAFLUORIDE. A. G. Buyers, F. J. Keneshea, Jr., and R. A. Barney. June 1, 1954. Decl. Dec. 12, 1955. 26p. Contract AT-11-1-Gen-8. $0.25(OTS).

A study has been made of the distribution of Pu and the principal fission products between molten U and fused U tetrafluoride. Plutonium and those fission products such as the rare earth metals and Cs, whose fluorides have free energies of formation more negative than UF_4, were extracted into the salt. Fission products whose fluorides have free energies of formation more positive than that of UF_4, such as Zr, Nb, and Ru remained largely in the U. (auth)

GENERAL 3.03.08

260
THE DEVELOPMENT OF PLUTONIUM SEPARATION PROCESSES. F. R. Paulsen. Chem. & Process Eng. 40, 271-3(1959) Aug.

The basic separation processes for plutonium, precipitation methods, solvent extraction, and dry techniques, are discussed. (C.J.G.)

3577
DESIGN OF PLUTONIUM PROCESSING PLANTS. B. F. Judson (Hanford Atomic Products Operation, Richland, Wash.). Chem. Eng. Progr. 55, Symposium Ser. No. 22, 33-6(1959).

A design philosophy for plutonium processing plants is presented. Concepts of the design include the use of contamination barriers made up of sealed hoods and directionalized air flow, partial separation of operating and maintenance functions in the physical layout, inclusion of multicomponent processing systems in single large hoods, and the considerations necessary for critical mass control. (auth)

PRIMARY SEPARATION PLANT AT THE WINDSCALE WORKS. H. G. Davey. Nuclear Power 1, 53-61(1956) June.

Plutonium is manufactured in Britain in the two air-cooled, graphite-moderated piles of the Windscale works of the United Kingdom Atomic Energy Authority. After removal from the piles the irradiated fuel rods are first stored under water for several weeks. They are then dissolved in nitric acid and the solution passed through three towers from which streams of fission products, plutonium and uranium are obtained by solvent extraction with dibutyl carbitol. The plutonium and uranium streams undergo further purification while the fission products are concentrated and stored

European Company for the Chemical Processing of Irradiated Fuels, Mol, Belgium.
FINAL PURIFICATION OF PLUTONIUM: PROCEDURE TO BE USED IN THE EUROCHEMIC PLANT. Technical Report No. 33. M. Zifferero, M. Lung, and A. Redon. Feb. 18, 1959. 16p. $3.30(ph), $2.40(mf) OTS.

RECOVERY OF PLUTONIUM 3.03

FUSED SALTS AND LIQUID METALS 3.03.07

7480
SEPARATION OF PLUTONIUM FROM URANIUM. H. M. Feder and R. L. Nuttall (to U. S. Atomic Energy Commission). U. S. Patent 2,917,382. Dec. 15, 1959.

A process is described for extracting plutonium from powdered neutron-irradiated uranium metal by contacting the latter, while maintaining it in the solid form, with molten magnesium which takes up the plutonium and separating the molten magnesium from the solid uranium.

14811 A/CONF.15/P/529
Los Alamos Scientific Lab., N. Mex.
PYROMETALLURGICAL PURIFICATION OF PLUTONIUM REACTOR FUELS. J. A. Leary, R. Benz, D. F. Bowersox, C. W. Bjorklund, K. W. R. Johnson, W. J. Maraman, L. J. Mullins, and J. G. Reavis. 14p. $0.50(OTS).

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

Pyrometallurgical methods studied are liquation, self-drossing and filtration, slagging by the addition of oxide, carbide, or halide, liquid metal extraction, complete conversion to halide, followed by filtration and selective reduction of the plutonium, and electrorefining. Experimental techniques and results are presented for each method. (auth)

14708 A/CONF.15/P/1830
Los Alamos Scientific Lab., N. Mex.
RECENT DEVELOPMENTS IN PLUTONIUM PROCESSING IN THE UNITED STATES. R. D. Baker and J. A. Leary. 10p. $0.50(OTS).

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

Recent developments in various fields of plutonium processing are discussed. Aqueous and pyrometallurgical processing methods are considered. (W.D.M.)

PYROMETALLURGICAL PURIFICATION OF PLUTONIUM REACTOR FUELS. J. A. Leary, R. Benz, D. F. Bowersox, C. W. Bjorklund, K. W. R. Johnson, W. J. Maraman, L. J. Mullins, and J. G. Reavis. 14p. (A/CONF.15/P/529)

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

Pyrometallurgical methods studied are liquation, self-drossing and filtration, slagging by the addition of oxide, carbide, or halide, liquid metal extraction, complete conversion to halide, following by filtration and selective reduction of the Pu, and electrorefining. Experimental techniques and results are presented for each method.

6308
THE DISTRIBUTION OF PLUTONIUM AND FISSION PRODUCTS BETWEEN MOLTEN URANIUM AND MOLTEN URANIUM TRIFLUORIDE – BARIUM HALIDE MIXTURES. F. S. Martin and E. W. Hooper (Atomic Energy Research Establishment, Harwell, Didcot, Berks). J. Inorg. and Nuclear Chem. 4, 93-9(1957) Mar.

The distribution of Pu between molten U and mixtures of UF_3 with $BaCl_2$ or fluoride has been measured at temperatures of 1200 to 1400°C. The equilibrium constant for the reaction $Pu + UF_3 = PuF_3 + U$ is found to be 72 ± 50%, calculated on a mole fraction basis. This corresponds to a standard free energy change for the reaction at 1200°C of approximately – 13 kcals, which agrees well with the estimated value. On the 20-g-of-U scale, at least 60-min contact between phases is required to establish equilibrium. Fission-product removals from the metal are greater than 99% under a variety of conditions, but owing to the compli-

THE DISTRIBUTION OF PLUTONIUM AND OF THE FISSION PRODUCTS IN A MIXTURE OF MOLTEN URANIUM AND UF_3–$BaCl_2(F_3)$. V. P. (P. V.). Atomnaya Energiya 3, No. 8, 175(1957).

At a temperature of 1200 to 1400°C plutonium and fission products are extracted from irradiated uranium on the basis of transition from the metal to the salt phase. It is necessary to wait at least 1 hour before equilibrium is established. For the extraction of plutonium the equilibrium constant is given. The fission products are separated up to 99%. Determination of the equilibrium constant was not possible because of the disturbing influence of the process of oxidation. ~~(TCO)~~

(See also 6.09 01.04)

RECOVERY OF PLUTONIUM 3.03

FUSED SALTS AND LIQUID METALS 3.03.07

153 RCTC/P-82
Gt. Brit. Atomic Energy Research Establishment, Harwell, Berks, England.
THE SELECTION OF A HIGH TEMPERATURE PROCESS FOR EXTRACTING PLUTONIUM FROM IRRADIATED URANIUM. K. M. Towers and F. Roberts. Nov. 1956. 14p.
A comparative study of pyrometallurgical methods for extracting Pu from U metal points to the use of molten Mg metal. The Pu is recovered by distilling away the Mg. (auth)

(See also 3.03.09.)

6600
LIQUID-METAL EXTRACTION FOR PROCESSING OF SPENT FUEL. A. F. Voigt, A. H. Daane, E. H. Dewell, R. G. Clark, J. E. Gonser, J. F. Haefling, and K. L. Maiaby (Iowa State Coll., Ames). A.I.Ch.E. Journal 2, 169-73 (1956) June.
Plutonium and the fission products can be removed from irradiated uranium by liquid-metal extraction by use of another metal immiscible with uranium. Metals studied have been silver, cerium, and lanthanum. Plutonium removal by silver is high, by the rare-earth metals moderate. In all cases volatile elements, including cesium, strontium, and barium, are removed. Rare earths are efficiently removed. Ruthenium and molybdenum are largely unaffected. Experiments with synthetic fuels corresponding to long burn-up periods show improved removal of most elements. Repeated batch extractions indicate that a continuous process separating the fuel into uranium, plutonium, and fission-product fractions could be developed. (auth)

PYRO-MET PLUTONIUM FLOWSHEET. M. Levenson. Mar. 5, 1956. Decl. Mar. 13, 1957. 2p. (ANL-ML-SL-1025)
A preliminary flowsheet for a pyrometallurgical process for Pu breeder cores is given.

2871
THE PURIFICATION OF URANIUM REACTOR FUEL BY LIQUID—METAL EXTRACTION. A. F. Voigt (Iowa State College, Ames). Prog. Nuclear Energy 1, 322-8(1956).
Plutonium can be satisfactorily removed from molten U by extraction with molten Ag. Other extractants, e.g., Ce, and La, are less promising with respect to Pu. Fission product data are inconclusive although no extractant was effective in removing molybdenum or ruthenium. (auth)

7547 NAA-SR-1435
North American Aviation, Inc., Downey, Calif.
THE PYROCHEMICAL RECYCLING OF PLUTONIUM FROM SPENT TO FRESH URANIUM FUEL USING MOLTEN UF_4 AS TRANSFER MEDIUM. Archie G. Buyers. Oct. 1, 1955. Decl. Feb. 27, 1957. 31p. Contract AT-11-1-GEN-8. $4.80(ph OTS); $2.70(mf OTS).
The pyrochemical transfer of Pu from spent to fresh U using molten UF_4 as transfer agent is demonstrated. The resultant U–Pu alloy contained fission products in small fractions of the amounts originally present in the spent fuel. Distribution of Pu and fission products throughout recycling is correlated with thermodynamic data. Application of this method for control of Pu concentration in a Na graphite reactor fuel cycle and fast breeder reactors is suggested. ~~(auth)~~

(See also 3.03.06.00.03.)

3911 NAA-SR-1157
North American Aviation, Inc. Downey, Calif.
PYROCHEMICAL SEPARATIONS METHODS: I. THE DISTRIBUTION OF PLUTONIUM AND LONG-LIVED FISSION PRODUCTS BETWEEN MOLTEN IRRADIATED URANIUM FUEL AND FUSED INORGANIC FLUORIDES. A~~rchie~~ G. Buyers. Mar. 15, 1955. Decl. Dec. 14, 1955. 32p. Contract AT-11-1-GEN-8. $0.25(OTS).
Distribution of Pu and fission products between molten U and fused inorganic fluorides, after equilibration at 1300°C, has been measured. Salt scrubs used included: UF_4, CaF_2, LiF, mixtures of these three, and mixtures of Mg and Li fluorides. Plutonium was found in the salt phases to the extent of 75 to 95% after one equilibration. Where three successive contactings were made with fresh portions of salt all but a negligible trace of the Pu was extracted into the salt. The standard free energy change for the reaction: $\frac{1}{3}$ Pu + $\frac{1}{4}$ UF_4 $\rightleftharpoons$ $\frac{1}{3}$ PuF_3 + $\frac{1}{4}$ U was

RECOVERY OF PLUTONIUM 3.03

GENERAL 3.03.08

Detailed development of the final Eurochemic process flowsheet is presented. Proposed apparatus arrangement is included, and reasons for modifications of the preliminary operational flowsheet are given. In addition, new aspects of the purification of Pu are considered for the first time, especially the general plan, the problems of protection against gamma and alpha radiation, and diagrams of the adsorption cycle. (J.R.D.)

AERE-C/M-88 (Rev.)
United Kingdom Atomic Energy Authority. Research Group. Atomic Energy Research Establishment, Harwell, Berks, England,
THE FLUORIDE-HYDROXIDE, PEROXIDE AND IODATE CYCLES IN THE PURIFICATION OF PLUTONIUM. G. R. Hall and R. Hurst. 1957. Date of MS. Oct. 1950 6p.

The results of the purification of a batch of plutonium by a potassium plutonium fluoride-plutonium hydroxide cycle followed by a plutonium peroxide and plutonium iodate cycle are reported. Good decontamination of uranium, chromium, and iron has been obtained and the final plutonium solution has a purity of 101± 3%. (auth)

16999 BNL-511
Brookhaven National Lab., Upton, N. Y.
REPROCESSING OF POWER REACTOR FUELS. THE ENRICO FERMI FAST BREEDER REACTOR FUEL PROGRESS REPORT NO. 1. C. W. Pierce and B. Manowitz. June 1958. 9p. $0.50(OTS).
Studies on the recovery of U and Pu from the Enrico Fermi Fast Breeder Reactor fuel are presented. (W.L.H.)

European Company for the Chemical Processing of Irradiated Fuels, Mol, Belgium.
BIBLIOGRAPHY OF REPORTS ON PLUTONIUM PROCESSING. Technical Report No. 3. A. Norström. Mar. 24, 1958. 24p. $4.80(ph), $2.70(mf) OTS.

A bibliography on Pu processing is presented. References concerning various aspects of processing such as head-end treatment, chemical processing, buildings, equipment, instrumentation, public health aspects, economy, and analysis are included. About 400 references. (J.R.D.)

Oak Ridge National Lab., Tenn.
EUROCHEMIC ASSISTANCE: ANALYTICAL—ANSWERS TO SPECIFIC QUESTIONS. L. T. Corbin. July 14, 1959. 7p. Contract W-7405-eng-26. $1.80 (ph), $1.80(mf) OTS.
The spectrochemical, carrier distillation, liquid-liquid extraction, and ion exchange of plutonium are discussed. (W.L.H.)

AQUEOUS DECONTAMINATION OF PLUTONIUM FROM FISSION PRODUCT ELEMENTS. R. S. Winchester and W. J. Maraman. 8p. (A/CONF.15/P/530)
Prepared for the Second U. N. International Conference on the Peaceful Uses of Atomic Energy, 1958.
A survey of aqueous Pu recovery and purification methods as applied to Pu-rich reactor fuels has been made. These methods consist of precipitating Pu-fluoride, -oxalate, and -peroxide, sorbing on anion exchange resins, and solvent-extracting with tri-n-butyl phosphate. The standard fluoride, oxalate, and peroxide precipitation methods gave decontamination factors of Pu from the typical fission product elements Zr, Mo, Ru, and Ce ranging from 0.002 for Ce to 140 for Mo. Sorption of Pu on anion exchanger resins from 8 M HNO_3 solutions and the subsequent elution resulted in decontamination factors of 16 for Ru to 52 for Zr. Solvent extraction of Pu in 2.7 M HNO_3 by TBP gave decontamination factors ranging from 1.3 for Ru to greater than 100 for Mo. Extraction of Pu from 8 M HNO_3 solution by primary, secondary, tertiary, and quaternary amines gave greater over-all decontamination. The secondary amine was selected for recovery processing. Pu is recovered and purified from pyrometallurgical and analytical wastes on a 300-gram scale using secondary amine solvent extraction followed by oxalate precipitation. A flowsheet for this process is discussed.

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GENERAL 3.03.08

6314
EXTRACTION OF PLUTONIUM AND URANIUM FROM NUCLEAR FUEL. N. Isaac. Industrie chim. belge 22, 139-52(1957) Feb. (In French)

The different methods used for the extraction of Pu from nuclear fuel, irradiated natural U, are reviewed. Two separate groups are considered: (a) processes call ing for dissolution of the fuel in aqueous media; (b) meth ods characterized by the use of high temperature in the absence of water. The advantages and disadvantages of the most common processes are discussed and some puri fication flowsheets are given. Stress is particularly laid on the solvent extraction method and the fractional distillation of fluorinated derivatives. (auth)

RECENT DEVELOPMENTS IN PLUTONIUM PROCESSING IN THE UNITED STATES. R. D. Baker and J. A. Leary. 10p. (A/CONF.15/P/1830)

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

Recent developments in various fields of Pu processing are discussed. Aqueous and pyrometallurgical processing methods are considered.

ION EXCHANGE 3.03.09

See also, this section,

COMPLEX FORMATION (3.03.04),

and,

under PROCESS CHEMISTRY AND ENGINEERING,

SPECIAL PROCESSES--Purex (6.17.17).

22147 HW-55893(Del.)
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
THE RECOVERY, PURIFICATION, AND CONCENTRATION OF PLUTONIUM BY ANION EXCHANGE IN NITRIC ACID. J. L. Ryan and E. J. Wheelwright. Jan. 2, 1959. Decl. with deletions June 25, 1959. 104p. Contract W-31-109-Eng-52. $16.80(ph), $5.70(mf) OTS.

A detailed experimental study has shown absorption of plutonium(IV) from 7 M HNO_3 solutions by strong-base (quaternary amine) anion exchange resins to be a very powerful and versatile processing method for plutonium. The simple expedient of operating at 50 to 60°C relieves most of the objections of former anion exchange processes for plutonium in that it permits adequate processing rates and efficiency to be obtained with four per cent cross-linked resins. Commercially available strong-base anion exchange resins Dowex-1, X-4 (50 to 100 mesh), among 50 to 100 mesh resins, and Permutit SK (20 to 50 mesh), among 20 to 50 mesh resins, are markedly superior for most process applications. The anion exchange process consists of an absorption or loading step in which plutonium is absorbed out of a feed solution by the resin, a washing step in which impurities are removed by washing the plutonium-laden resin with an appropriate wash solution, and an elution step in which plutonium is stripped off the resin in an appropriate aqueous solution. The effects of the pertinent variables in each of these steps have been studied in some detail, yielding information which can be readily extrapolated to new processing applications. (auth)

10989
RECOVERY OF PLUTONIUM. U. K. Atomic Energy Authority (U.S.A.). British Patent 801,379. Nuclear Eng. 4, 188(1959) Apr.

If an aqueous solution of fission products, in which plutonium may be present, is brought into contact with diatomaceous earth, silicon gel, or magnesium silicate, the plutonium will be adsorbed preferentially to uranium or other fission materials. The adsorbed plutonium may be eluted with 6N nitric acid.

RECOVERY AND PURIFICATION OF PLUTONIUM BY ANION EXCHANGE. J. L. Ryan and E. J. Wheelwright (General Electric Co., Richland, Wash.). Ind. Eng. Chem. 51, 60-5(1959) Jan.

Results to date indicate that plutonium can be absorbed at a reasonably rapid rate on any of several commercially available anion exchange resins from 7M nitric acid at 60°C. When the plutonium-loaded resin is then washed with 7M nitric acid, very high factors are obtained for separation of plutonium from uranium, fission products, and common ionic and nonionic

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ION EXCHANGE 3.03.09

impurities. The degree of separation depends on the extent of the washing cycle. Fission product separation factors as high as 3×10^7 have been obtained in two cycles of anion exchange. Uranium separation factors of comparable magnitude are possible with moderate

9841 CRDC-818
Atomic Energy of Canada Ltd., Chalk River, Ont.
THE CHEMISTRY OF THE ANION EXCHANGE PROCESS FOR THE RECOVERY OF PLUTONIUM. R. G. Hart, J. A. Brothers, and I. W. Allam. Dec. 1958. 24p. (AECL-755). $0.50(AECL).

A semi-theoretical study was made of the chemistry of the Anion Exchange Process for the recovery of plutonium. Pseudo-equilibrium constants were obtained for the systems plutonium(III)–Dowex 50, plutonium(III)–Dowex 50-W, and plutonium(IV)–Dowex 1, in nitric acid solution. Exchange height equations were obtained for the plutonium(III)–Dowex 50 and the plutonium(IV)–Dowex 1 systems. Elution equations are given for the systems plutonium(III)–Dowex 50 and plutonium(III)–Dowex 50-W. The equilibrium plutonium(IV)–Dowex 1 in the presence of uranium is also briefly discussed. (auth)

METHOD OF SEPARATION. G. E. Boyd (to U. S. Atomic Energy Commission). U. S. Patent 2,849,282. Aug. 26, 1958.

A process is presented for separating uranium, plutonium, and fission products ions from uranyl nitrate solutions having a pH value between 1 and 3 obtained by dissolving neutron irradiated uranium. The method consists in passing such solutions through a bed of cation exchange resin, which may be a sulfonated phenol formaldehyde type. Following the adsorption step the resin is first treated with a solution of 0.2M to 0.3M sulfuric acid to desorb the uranium. Fission product ions are then desorbed by treating the resin in phosphoric acid and 1M in nitric acid. Lastly, the plutonium may be desorbed by treating the resin with a solution approximately 0.8M in phosphoric acid and 1M in nitric acid.

14418
SEPARATION OF INORGANIC SALTS FROM ORGANIC SOLUTIONS. L. I. Katzin and J. C. Sullivan (to U. S. Atomic Energy Commission). U. S. Patent 2,840,451. June 24, 1958.

A process is described for recovering the nitrates of uranium and plutonium from solution in oxygen-containing organic solvents such as ketones or ethers. The solution of such salts dissolved in an oxygen-containing organic compound is contacted with an ion exchange resin whereby sorption of the entire salt on the resin takes place and then the salt-depleted liquid and the resin are separated from each other. The reaction seems to be based on an anion formation of the entire salt by complexing with the anion of the resin. Strong base or quaternary ammonium type resins can be used successfully in this process.

13044 JENER-55
Joint Establishment for Nuclear Energy Research, Kjeller, Norway.
A 3-STAGED METHOD FOR THE RECOVERY OF PLUTONIUM FROM IRRADIATED FUELS. Rolf O. Lingjaerde. June 24, 1958. 11p.

A method is described for the recovery of Pu from spent fuel elements. The process involves 3 stages: 1) Extraction of U + Pu with TBP, 2) separation of Zr/Nb from the TBP phase on a silica gel column, and 3) separating Pu from U–still in the TBP phase–on a Dowex-50 column. The experimental procedure is given, and the mechanisms for the changes occurring on the silica gel and the Dowex-50 columns are discussed. The final Pu product is not completely free from U, and further purification is necessary. (auth)

14408
SEPARATION OF PLUTONIUM FROM AQUEOUS SOLUTIONS BY ION-EXCHANGE. Jack Schubert (to U. S. Atomic Energy Commission). U. S. Patent 2,837,401. June 3, 1958.

A process is described for the separation of plutonium from an aqueous solution of a plutonium salt, which comprises adding to the solution an acid of the group consisting of sulfuric acid, phosphoric acid, and oxalic acid, and mixtures thereof to provide an acid concentration between 0.0001 and 1 M, contacting the resultant solution with a synthetic organic anion exchange resin, and separating the aqueous phase and the resin which contains the plutonium.

RECOVERY OF PLUTONIUM 3.03

ION EXCHANGE 3.03.09

211
THE PREPARATION AND PROPERTIES OF SOME PLUTONIUM COMPOUNDS. PART V. COLLOIDAL QUADRIVALENT PLUTONIUM. D. W. Ockenden and G. A. Welch (U. K. Atomic Energy Authority, Windscale Works, Cumb., England). J. Chem. Soc., 3358-63(1956) Sept.

Colloidal, or polymeric, quadrivalent Pu has been separated from the ionic species of Pu by ion exchange. The absorption spectrum of the colloid is distinctive, quite different from those of other species of Pu and varies slightly with the method of preparation. The rate of formation of the polymer in dilute HNO_3, its stability in more concentrated acid, its adsorption on certain surfaces, and other properties have been studied. The minimum and maximum molecular weights of the colloid have been calculated from diffusion experiments and from work with nuclear emulsion plates. (auth)

Refer to abstract 769.

986 JENER-44
Joint Establishment for Nuclear Energy Research, Kjeller, Norway.
ANION EXCHANGE STUDIES OF PLUTONIUM. T. Sikkeland and J. Juul. Aug. 27, 1956. 10p.

The adsorption of Pu on Dowex-1 resin is studied. Regardless of concentration in HCl, Pu^{3+} is not adsorbed. Above 2.5 M HCl Pu^{4+} is adsorbed but is unstable in the 4 to 7 M range; Pu^{4+} is stabilized by $NaNO_2$. Above 3.6 M HCl, PuO_2^{2+} is adsorbed, however it reduces to Pu^{3+}, and attempts to stabilize it with Cl_2 or $K_2Cr_2O_7$ were unsuccessful. (T.R.H.)

1319 JENER-38
Joint Establishment For Nuclear Energy Research, Kjeller, Norway.
THE SEPARATION OF PLUTONIUM FROM URANIUM AND FISSION PRODUCTS WITH ION EXCHANGE COLUMNS. T. Sikkeland. 4p.

Tetravalent Pu is quantitatively adsorbed from a U solution by passing this through a column with a cation exchange resin. The U and most of the fission products are eluted with 2M HCl and Pu and the remaining fission products with 5M HCl. The final separation of Pu from the fission products is obtained with an anion exchange column. The recovery of radiochemically pure Pu by the whole procedure is 99%. (auth)

8736
THE CONCENTRATION AND PURIFICATION OF URANIUM AND PLUTONIUM BY ION EXCHANGE. F. R. Bruce (Oak Ridge National Lab., Tenn.). pp. 363-76 in "Progress in Nuclear Energy. Series III. Process Chemistry. Volume 2."

Cation exchange may be used to concentrate the dilute uranium-233 or plutonium products which are obtained from solvent-extraction processes. Losses of fissionable material are less than 0.01%, and some separation of the products from fission and corrosion products is achieved. (auth)

MAGNESIUM EXTRACTION 3.03.10

See also, this section,
FUSED SALTS AND LIQUID METALS (3.03.07),
PRECIPITATION (3.03.12),
REDUCTION (3.03.13),
and, under FISSION PRODUCT, POISON AND RADIOISOTOPE REMOVAL,
PROTACTINIUM (2.38).

16202
PROCESS FOR THE PURIFICATION OF SOLUTIONS OF PLUTONIUM NITRATE AND PREPARATION OF THE METAL. S. Chambers and T.-G. Hughes. Energie nucléaire 2, 202-6(1958) July-Sept. (In French)

The solution of plutonium nitrate, separated by extraction with dibutyl glycol from the solution of fuel elements, is concentrated by evaporation and purified by extraction with a mixture of tributyl phosphate and kerosene. The residual impurities coming from the reagents and the apparatus can be eliminated by a second purification in glass apparatus. The nitrate

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16885 ✓ NP-7661
European Company for the Chemical Processing of Irradiated Fuels, Mol, Belgium.
TAIL-END TREATMENTS FOR URANIUM AND PLUTONIUM CONCENTRATION AND PURIFICATION. Technical Report No. 4. M. Zifferero. May 16, 1958. 28p. $4.80(ph), $2.70(mf) OTS.

Uranium and plutonium products from a Purex-type flowsheet to be tail-end treated are discussed. A survey is presented of the methods so far applied or proposed in the field of tail-end treatments with the emphasis on silica gel U purification, and ion exchange Pu concentration and purification. Also a program for testing procedures and materials is outlined. (J.R.D.)

14711 ✓ A/CONF 15/P/1915
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
APPLICATION OF ANION EXCHANGE TO THE REPROCESSING OF PLUTONIUM. J. L. Ryan and E. J. Wheelwright. 21p. $0.50(OTS).

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

It has been demonstrated that plutonium (IV) can be absorbed from nitric acid solution by anion exchange resins. Anion exchange offers a marked advantage over cation exchange for plutonium processing because of the ease of decontaminating the plutonium from all normally encountered impurities. Anion exchange has been applied as a primary method for the separation of plutonium from irradiated uranium, but processing rates and product concentrations were low By operating at elevated temperature (50 to 60°C) and by proper choice of anion exchange resins, throughputs may be increased considerably, product concentration is increased, decontamination improved, and the use of reagents other than

11358 CRCE-743
Atomic Energy of Canada Ltd. Chalk River Project, Chalk River, Ont.
STUDY OF THE PROCESS VARIABLES IN THE ANION EXCHANGE PLUTONIUM SEPARATION PROCESS. D. T. Nishimura. Nov. 1957. 34p. (AECL-556). $1.00(AECL).

A study of the process variables in the Anion Exchange Process Pilot Plant for the separation of plutonium from irradiated uranium is discussed. Variables associated with the feed, wash, and elution cycles were studied with the aim of improving the quality of the final plutonium product, reduce cycling time and reagent requirements and also to obtain data for prediction of resin column behavior under various feed conditions. A cation resin column and a silica gel column were installed in the system and these were studied for plutonium recovery and product quality. The product obtained from the plant was acceptable in all the impurities except the associated gamma activity which was too high for easy product handling. (auth)

5271 ✓
ION EXCHANGE RECOVERS PLUTONIUM FROM IRRADIATED FUELS. A. M. Aikin (Atomic Energy of Canada Ltd., Chalk River, Ontario). Chem Eng. Progr. 53, 82F-5F (1957) Feb.

A pilot plant operation is presented for the separation of irradiated Pu from U by ion exchange. An advantage claimed for this process is that a small ion exchange unit can be put on a competitive basis with large solvent extraction units. (W.L.H.)

7592 ✓ DP-192
Du Pont de Nemours (E. I.) & Co. Savannah River Lab., Augusta, Ga.
CONCENTRATION OF PLUTONIUM BY CATION EXCHANGE. PART I. NEW ELUTRIANTS. Oscar D. Bonner, Glenn A. Burney, and Frank W. Tober. Dec. 1956. Decl. Jan. 14, 1959. 16p. Contract AT(07-2)-1. $3.30(ph), $2.40(mf) OTS.

Over 99% of the total plutonium absorbed on a cation resin was eluted at an average concentration of 43.8 grams per liter by a solution of ammonium lactate plus ammonium sulfamate. This elutriant does not evolve gases in the resin bed. (auth)

(See also C.11.04.)

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OXIDATION 3.03.11

3502✓ CK-1529
Chicago. Univ. Metallurgical Lab.
THE DISTRIBUTION OF PLUTONIUM TRACER BETWEEN METALS AND THEIR MOLTEN HALIDES. VI. DISTRIBUTION OF PLUTONIUM AND URANIUM TRACERS IN THE MAGNESIUM METAL-HALIDE SYSTEM. B. J. Fontana. Feb. 21, 1944. Decl. Dec. 13, 1955. 15p. (A-2207). $3.30(ph OTS); $2.40(mf OTS).

Additional studies on the electrochemical behavior of Pu and U in $Mg-MgX_2$ (where X = halide) suggest that Pu is more electropositive than U or Mg. (See also CK-942.) (C.W.H.)

7429
METHOD OF OXIDIZING PLUTONIUM ION WITH BISMUTHATE ION. C. S. Garner (to U. S. Atomic Energy Commission). U. S. Patent 2,917,362. Dec. 15, 1959.

A method is presented for oxidizing plutonium from the tetravalent state to the hexavalent state by means of bismuthate oxidizing agents.

PRECIPITATION 3.03.12

See also, this section,
ELECTROCHEMICAL (3.03.05),
MAGNESIUM EXTRACTION (3.03.10),
and,
under PROCESS CHEMISTRY AND ENGINEERING,
SPECIAL PROCESSES--Bismuth Phosphate (6.17.02).

ACETATE 3.03.12.01

RECOVERY OF PLUTONIUM. (to United Kingdom Atomic Energy Authority). British Patent 810,541. Mar. 18, 1959.

Separation of Pu from U by precipitation as a PuO_2^{2+} complex with a metal acetate is described. The metal acetate may be Na, Li, Co, Cu, Mn, Ni, Mg, Zn, or Cd. The U and Pu in a HNO_3 solution containing fission products are oxidized by $K_2Cr_2O_7$ and then precipitated by adding CH_3COOMg and CH_3COONa. This precipitate is filtered, washed, and redissolved in HNO_3. The Pu is then selectively reduced by $NaHSO_3$, and the UO_2^{2+} is precipitated out by a second CH_3COONa and CH_3COOMg treatment. Then the filtrate containing Pu is acidified, oxidized, and $1/20$ of the amount of U initially present is added as carrier for another precipitation of Pu by acetate. This precipitation cycle is repeated until the Pu is obtained almost free of U and fission products. Np which accompanies Pu throughout this process later decays to Pu. (T.R.H.)

PROCESS FOR THE SEPARATION OF HEAVY METALS. J. W. Gofman, R. E. Connick, and A. C. Wahl (to U. S. Atomic Energy Commission). U. S. Patent 2,871,251. Jan. 27, 1959.

A method is presented for the separation of plutonium from uranium and the fission products with which it is associated. The method is based on the fact that hexavalent plutonium forms an insoluble complex precipitate with sodium acetate, as does the uranyl ion, while reduced plutonium is not precipitated by sodium acetate. Several embodiments are shown, e.g., a solution containing plutonium and uranium in the hexavalent state may be contacted with sodium acetate causing the formation of a sodium uranyl acetate precipitate which carries the plutonium values while the fission products remain in solution. If the original solution is treated with a reducing agent, so that the plutonium is reduced while the uranium remains in the hexavalent state, and sodium and acetate ions are added, the uranium will precipitate while the plutonium remains in solution effecting separation of the Pu from uranium.

LANTHANUM 3.03.12.02

22158
METHOD OF FORMING PLUTONIUM-BEARING CARRIER PRECIPITATES AND WASHING SAME. B. F. Faris (to U. S. Atomic Energy Commission). U. S. Patent 2,875,022. Feb. 24, 1959.

An improvement of the lanthanum fluoride carrier precipitation process for the recovery of plutonium is presented. In this process the plutonium is first

RECOVERY OF PLUTONIUM 3.03

MAGNESIUM EXTRACTION 3.03.10

solution is concentrated. The nitrate is transformed to the oxide, then to the fluoride, and finally to the metal by Ca or Mg reduction. The impurities, which represent less than 1% by weight, are iron, nickel, chromium, and uranium. The construction of an installation for this process is briefly described. (tr-auth)

(See also 3.03.3.)

16387 ANL-5749
Argonne National Lab., Lemont, Ill.
PROPERTIES AND HANDLING PRACTICES FOR MAGNESIUM: A LITERATURE SURVEY. M. Breederman, G. A. Bennett, L. Burris, Jr., I. G. Dillon, I. O. Winsch, M. W. Nathans, E. Greenberg, and J. Wolkoff. Mar. 1958. 132p. Contract W-31-109-eng-38. $2.75(OTS).

A survey of Mg including the cost and availability of various high-purity grades, safe handling considerations, methods of distillation and production, physical and thermodynamic properties, and the binary and ternary alloys is presented. A bibliography is included with 157 references. (J.R.D.)

7973
MAGNESIUM EXTRACTION PROCESS FOR PLUTONIUM SEPARATION FROM URANIUM. Irvin O. Winsch and Leslie Burris, Jr. (Argonne National Lab., Lemont, Ill.). Chem. Eng. Progr. 53, 237-42(1957) May.

A high-temperature extraction process for the separation of plutonium from nuclear reactor core and blanket materials is described and data are presented. Molten Mg, which is immiscible with molten U, may be used to extract Pu from U. Plutonium distributes between Mg and U or the lower melting U alloys such as U–Cr and U–Fe with a distribution coefficient of about 2 (weight basis) in favor of the Mg. The Pu may be separated from the Mg by volatilization of the Mg. Auxiliary operations such as molten-metal transfer, phase separation, and sampling are also discussed. (auth)

OXIDATION 3.03.11

See also, under PROCESS CHEMISTRY AND ENGINEERING, SPECIAL PROCESSES--Bismuth Phosphate (6.17.02), Redox (6.17.19).

6245
PROCESS OF OXIDIZING PLUTONIUM. C. D. Coryell (to U. S. Atomic Energy Commission). U. S. Patent 2,901,315. Aug. 25, 1959.

The oxidation of plutonium to the plus six valence state is described. The oxidation is accomplished by treating the plutonium in aqueous solution with a solution above 0.01 molar in argentic ion, above 1.1 molar in nitric acid, and above 0.02 molar in argentous ion.

OXIDATION OF TRANSURANIC ELEMENTS. R. L. Moore (to U. S. Atomic Energy Commission). U. S. Patent 2,874,025. Feb. 17, 1959.

A method is reported for oxidizing neptunium or plutonium in the presence of cerous values without also oxidizing the cerous values. The method consists in treating an aqueous 1N nitric acid solution, containing such cerous values together with the trivalent transuranic elements, with a quantity of hydrogen peroxide stoichiometrically sufficient to oxidize the transuranic values to the hexavalent state, and digesting the solution at room temperature.

PLUTONIUM SEPARATION METHOD. L. J. Beaufait, Jr., F. R. Stevenson, and G. K. Rollefson (to U. S. Atomic Energy Commission). U. S. Patent 2,860,949. Nov. 18, 1958.

The recovery of plutonium ions from neutron-irradiated uranium can be accomplished by buffering an aqueous solution of the irradiated materials containing tetravalent plutonium to a pH of 4 to 7, adding sufficient acetate to the solution to complex the uranyl present, adding ferric nitrate to form a colloid of ferric hydroxide, plutonium, and associated fission products, removing and dissolving the colloid in aqueous nitric acid, oxidizing the plutonium to the hexavalent state by adding permanganate or dichromate, treating the resultant solution with ferric nitrate to form a colloid of ferric hydroxide and associated fission products, and separating the colloid from the plutonium left in solution.

See also (3.02.06.)

RECOVERY OF PLUTONIUM 3.03

OXIDATION 3.03.11

14415
METHOD OF SEPARATING PLUTONIUM FROM LANTHANUM FLUORIDE CARRIER. G. W. Watt and R. H. Goeckermann (to U. S. Atomic Energy Commission). U. S. Patent 2,838,371. June 10, 1958.

An improvement in oxidation-reduction type methods of separating plutonium from elements associated with it in a neutron-irradiated uranium solution is described. The method relates to the separating of plutonium from lanthanum ions in an aqueous 0.5 to 2.5 N nitric acid solution by treating the solution, at room temperature, with ammonium sulfite in an amount sufficient to reduce the hexavalent plutonium present to a lower valence state, and then treating the solution with H_2O_2 thereby forming a tetravalent plutonium peroxide precipitate.

(See also 6.16.08.)

1322 CRDC-697
Atomic Energy of Canada Ltd. Chalk River Project, Chalk River, Ontario.
OXIDATION OF Pu(IV) TO Pu(VI) IN SOLUTIONS OF NITRIC ACID, ALUMINIUM NITRATES, SODIUM NITRATE, AND URANYL NITRATE. I. H. Crocker. May 1957. 57p. (AECL-488).

The effects of temperature, Pu concentration, HNO_3 concentration, and NO_3^- concentration on the oxidation of Pu^{4+} to Pu^{6+} have been studied. The rate of oxidation increases as temperature and Pu concentration increase, decreases as the HNO_3 concentration increases, and, in general, decreases as the NO_3^- concentration increases, particularly when the ion is supplied by a highly ionized nitrate salt, such as $NaNO_3$ or $Al(NO_3)_3$. $UO_2(NO_3)_2$, which is strongly complexed in HNO_3 solution, inhibits Pu^{6+} formation when present in high concentration, but enhances its formation when present in low concentration. Quantitative data were obtained over a range of acid–salt concentrations, that should enable prediction of the oxidation state of Pu in most process solutions. (auth)

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.)

(See also 6.16.08.)

[illegible] HW-17266
Hanford Works, Richland, Wash.
THE OXIDATION OF PLUTONIUM TO THE PLUTONYL STATE. H. W. Miller and R. J. Brouns. Mar. 22, 1950. ~~Decl. Dec. 5, 1955.~~ 10p. Contract W-31-109-Eng-52. $1.80(ph OTS); $1.80(mf OTS). (HW-17266)

Several methods of oxidizing Pu to the plutonyl ion in an acid solution were investigated and an ozonization procedure was selected as the most satisfactory. Oxygen gas containing ozone is bubbled through a 2N HCl solution of the Pu for 2 to 8 hr. the time depending upon the amount of Pu in the sample. The ozone was prepared by passing acid scrubbed, alkali scrubbed, and dried oxygen through a modified Siemen's ozonator. Moisture must be scrupulously excluded from the ozonator. The advantages of the ozonization procedure for plutonium oxidation are: the oxidation is quantitative; no foreign substances are added; the apparatus and procedure are simple and convenient; and the overall time consumed is relatively small. The plutonyl solutions in 2N HCl were stable; no appreciable reduction, in the absence of impurities, was observed in 20 days. (auth)

3503 ✓ CK-1712
[California. Univ., Berkeley. Radiation Lab.]
THE DISTRIBUTION OF PLUTONIUM TRACER BETWEEN METALS AND THEIR MOLTEN HALIDES. VII. DISTRIBUTION OF PLUTONIUM TRACER IN THE STRONTIUM—STRONTIUM BROMIDE AND THE BARIUM—BARIUM BROMIDE SYSTEMS. B. J. Fontana. May 16, 1944. Decl. Dec. 15, 1955. 11p. (A-2437). $3.30(ph OTS); $2.40 (mf OTS).

Investigations on the high-temperature oxidation–reduction relations of Pu in $Sr-SrBr_2$ and $Ba-BaBr_2$ systems indicate that Sr and Ba are more electropositive than Pu. (See also CK-942.) (C.W.H.)

RECOVERY OF PLUTONIUM 3.03

PRECIPITATION 3.03.12

LANTHANUM 3.03.12.02

segregated in the LaF_3 precipitate and this precipitate is later dissolved and the plutonium reprecipitated as the peroxide. It has been found that the loss of plutonium by its remaining in the supernatant liquid associated with the peroxide precipitate is greatly reduced if, before dissolution, the LaF_3 precipitate is subjected to a novel washing step which constitutes the improvement of this patent. The step consists in intimately contacting the LaF_3 precipitate with a 4 to 10 percent solution of sodium hydrogen sulfate at a temperature between 10 and 95°C for ½ to 3 hours.

METHOD OF IMPROVING THE CARRIER PRECIPITATION OF PLUTONIUM. H. J. Kamack and J. H. Balthis (to U. S. Atomic Energy Commission). U. S. Patent 2,863,719. Dec. 9, 1958.

Plutonium values can be recovered from acidic solutions by adding lead nitrate, hydrogen fluoride, lanthanum nitrate, and sulfuric acid to the solution to form a carrier precipitate. The lead sulfate formed improves the separation characteristics of the lanthanum fluoride carrier precipitate.

PROCESS USING POTASSIUM LANTHANUM SULFATE FOR FORMING A CARRIER PRECIPITATE FOR PLUTONIUM VALUES. A. A. Angerman (to U. S. Atomic Energy Commission). U. S. Patent 2,857,241. Oct. 21, 1958.

A process is presented for recovering plutonium values in an oxidation state not greater than +4 from fluoride-soluble fission products. The process consists of adding to an aqueous acidic solution of such plutonium values a crystalline potassium lanthanum sulfate precipitate which carries the plutonium values from the solution.

METHOD OF SEPARATING PLUTONIUM FROM CONTAMINANTS. H. S. Brown and O. F. Hill (to U. S. Atomic Energy Commission). U. S. Patent 2,785,047. March 12, 1957.

A method is presented for separating Pu from fission products, and for obtaining the Pu in a concentrated form. Irradiated U is dissolved in HNO_3, and a LaF_3 precipitate, which carries the Pu and the fluoride soluble fission products, is formed in the solution. The present invention comprises reacting this dried precipitate with HF whereby fission products which form relatively volatile fluorides are volatilized away from the Pu-containing LaF_3 mass. The mass is then treated with anhydrous F_2 at elevated temperatures whereby the Pu is oxidized and volatilized as its higher fluorides, and is thus separated from the LaF_3 and other less volatile constituents of the mass. (auth)

3228

PLUTONIUM RECOVERY PROCESS. O. F. Hill and S. G. Thompson (to U. S. Atomic Energy Commission). U. S. Patent 2,767,044. Oct. 16, 1956.

In order to separate Pu from an aqueous solution of U and fission product salts a cycle of carrier precipitations is employed. First, the Pu is oxidized to the hexavalent state, and carbonate ion is added to the solution to complex the Pu. By addition of an aqueous solution of a La salt, a basic lanthanum carbonate precipitate is formed, which removes much of the Ba, Zr and Nb by carrying but does not affect hexavalent Pu. The filtered solution is then treated with a reducing agent which will reduce Pu but not the uranyl ion. The Pu is then removed from the solution by formation of a bismuth hydroxide carrier precipitate. (auth)

2492

DISSOLUTION OF PLUTONIUM CONTAINING CARRIER PRECIPITATE BY CARBONATE METATHESIS AND SEPARATION OF SULFIDE IMPURITIES THEREFROM BY SULFIDE PRECIPITATION. R. B. Duffield (to U. S. Atomic Energy Commission). U. S. Patent 2,894,812. July 14, 1959.

A process is described for recovering plutonium from foreign products wherein a carrier precipitate of lanthanum fluoride containing plutonium is obtained and includes the steps of dissolving the carrier precipitate in an alkali metal carbonate solution, adding a soluble sulfide, separating the sulfide precipitate, adding an alkali metal hydroxide, separating the resulting precipitate, washing, and dissolving in a strong acid.

RECOVERY OF PLUTONIUM 3.03

PRECIPITATION 3.03.12

PHOSPHATE 3.03.12.05

($Na_4P_2O_7$) whereby a carrier precipitate of thorium pyrophosphate is formed in the solution, separating, and redissolving the thorium pyrophosphate precipitate together with the tetravalent plutonium carried with the precipitate, treating the solution with hydroxylamine to reduce tetravalent plutonium into the trivalent state, and reprecipitating and separating the thorium pyrophosphate leaving the trivalent plutonium in the solution in a substantially pure state.

6373

SEPARATION OF FISSION PRODUCTS FROM PLUTONIUM BY PRECIPITATION. G. T. Seaborg, S. G. Thompson, and N. R. Davidson (to U. S. Atomic Energy Commission). U. S. Patent 2,903,335. Sept. 8, 1959.

Fission product separation from hexavalent plutonium by bismuth phosphate precipitation of the fission products is described. The precipitation, according to this invention, is improved by coprecipitating ceric and zirconium phosphates (0.05 to 2.5 grams/liter) with the bismuth phosphate.

PROCESS USING BISMUTH PHOSPHATE AS A CARRIER PRECIPITATE FOR FISSION PRODUCTS AND PLUTONIUM VALUES. T. G. Finzel (to U. S. Atomic Energy Commission). U. S. Patent 2,877,090. Mar. 10, 1959.

A process is described for separating plutonium from fission products carried therewith when plutonium in the reduced oxidation state is removed from a nitric acid solution of irradiated uranium by means of bismuth phosphate as a carrier precipitate. The bismuth phosphate carrier precipitate is dissolved by treatment with nitric acid and the plutonium therein is oxidized to the hexavalent oxidation state by means of potassium dichromate. Separation of the plutonium from the fission products is accomplished by again precipitating bismuth phosphate and removing the precipitate which now carries the fission products and a small percentage of the plutonium present. The amount of plutonium carried in this last step may be minimized by addition of sodium fluoride, so as to make the solution 0.03N in NaF, prior to the oxidation and precipitation step.

13375

PROCESS FOR THE RECOVERY OF PLUTONIUM. D. M. Ritter (to U. S. Atomic Energy Commission). U. S. Patent 2,868,619. Jan. 13, 1959.

An improvement is presented in the process for recovery and decontamination of plutonium. The carrier precipitate containing plutonium is dissolved and treated with an oxidizing agent to place the plutonium in a hexavalent oxidation state. A lanthanum fluoride precipitate is then formed in and removed from the solution to carry undesired fission products. The fluoride ions in the remaining solution are complexed by addition of a borate such as boric acid, sodium metaborate or the like. The plutonium is then reduced and carried from the solution by the formation of a bismuth phosphate precipitate. This process effects a better separation from unwanted fission products along with concentration of the plutonium by using a smaller amount of carrier.

METHOD FOR REMOVING CONTAMINATION FROM PRECIPITATES. G. W. Stahl (to U. S. Atomic Energy Commission). U. S. Patent 2,867,506. Jan. 6, 1959.

An improvement in the bismuth phosphate carrier precipitation process is presented for the recovery and purification of plutonium. When plutonium, in the tetravalent state, is carried on a bismuth phosphate precipitate, amounts of certain of the fission products are carried along with the plutonium. The improvement consists in washing such fission product contaminated precipitates with an aqueous solution of ammonium hydrogen fluoride, since this solution has been found to be uniquely effective in washing fission production contamination from the bismuth phosphate precipitate.

IMPROVEMENT UPON THE CARRIER PRECIPITATION OF PLUTONIUM IONS FROM NITRIC ACID SOLUTIONS. R. A. James and S. G. Thompson (to U. S. Atomic Energy Commission). U. S. Patent 2,865,705. Dec. 23, 1958.

A process is reported for improving the removal of plutonium by carrier precipitation by the addition of nitrite ions to a nitric acid solution of neutron-irradiated uranium so as to destroy any hydrazine that may be present in the solution since the hydrazine tends to complex the tetravalent plutonium and prevents removal by the carrier precipitate, such as bismuth phosphate.

RECOVERY OF PLUTONIUM 3.03

PRECIPITATION 3.03.12

OXALATE 3.03.12.03

13374
OXALATE PROCESS FOR SEPARATING ELEMENT 94. J. W. Gofman (to U. S. Atomic Energy Commission). U. S. Patent 2,867,640. Jan. 6, 1959.
A process is presented for separating plutonium values in the tetravalent state from hexavalent uranium with which it is associated in aqueous solutions. This separation is accomplished by forming a thorium oxalate precipitate in order to carry only the plutonium. By carefully regulating the acidity of the solution and the oxalate ion concentration, the precipitation of uranium oxalate may be avoided.

8379 ✓
PROBLEMS AT THE MARCOULE PLUTONIUM PLANT AND THEIR SOLUTIONS. R. Galley. Energie nucléaire 2, 2-20(1958) Jan.-Mar. (In French)
The ~~industrial~~ problems of plutonium extraction are connected with the low plutonium content of the uranium bars and with the high activity of the bars which necessitates stringent protective measures. The general production scheme used at Marcoule is indicated. The concentration of the separated fission products is made in two evaporation stages. Plutonium, concentrated by precipitation and selective fixation of resins. is precipitated as the oxalate and then reduced to the metal. (tr-auth)

(See also 3.03-12)

1886 LA-1991
Los Alamos Scientific Lab., N. Mex.
PURIFICATION AND CONCENTRATION OF SOLVENT EXTRACTED PLUTONIUM BY OXALATE PRECIPITATION. W. J. Maraman, A. J. Beaumont, R. L. Nance, J. C. Roybal and R. S. Winchester. Jan. 1956. Decl. Apr. 1, 1957. 12p. Contract [W-7405-eng-36]. $0.25(OTS).
A production-scale procedure for purifying and concentrating Pu contained in the stripping solution from a TBP extraction process is described. Conclusions from exploratory tests on variables affecting the efficiency are included. (auth)

1294 LA-1692
Los Alamos Scientific Lab., N. Mex.
CALCIUM OXALATE CARRIER PRECIPITATION OF Pu. W. J. Maraman, A. J. Beaumont, E. L. Christensen, A. V. Henrickson, J. A. Hermann, K. W. R. Johnson, L. J. Mullins, and R. S. Winchester. Feb. 25, 1954. Decl. Oct. 28, 1957. 9p. Contract W-7405-Eng-36. $1.80(ph OTS); $1.80(mf OTS).
A plant-scale procedure for concentrating Pu and Am in slag and crucible (metal reduction residues) and other recovery solutions by a calcium oxalate carrier precipitation is described. Conclusions from exploratory laboratory tests on the variables affecting the efficiency are included. (auth)

PEROXIDE 3.03.12.04

13370 ✓
THE PREPARATION OF HIGH-PURITY PLUTONIUM METAL. K. W. R. Johnson (Los Alamos Scientific Lab., N. Mex.). J. Inorg. & Nuclear Chem. 9, 200-3(1959) Mar.
High-purity α-phase plutonium metal was produced by a process which consisted of two successive peroxide precipitations, direct hydrofluorination with gaseous hydrogen fluoride, and reduction to metal with calcium and iodine "booster." The total detectable impurities in the metal produced varied from 94 to 276 ppm. (auth)

BASIC PEROXIDE PRECIPITATION METHOD OF SEPARATING PLUTONIUM FROM CONTAMINANTS. G. T. Seaborg and I. Perlman (to U. S. Atomic Energy Commission). U. S. Patent 2,873,169. Feb. 10, 1959.
A process is described for the separation from each other of uranyl values, tetravalent plutonium values and fission products contained in an aqueous acidic solution. First the pH of the solution is adjusted to between 2.5 and 8 and hydrogen peroxide is then added to the solution causing precipitation of uranium peroxide which carries any plutonium values present, while the fission products remain in solution. Separation of the uranium and plutonium values is then effected by dissolving the peroxide precipitate in an acidic solution and incorporating a second carrier precipitate, selective for plutonium. The plutonium values are thus carried from the solution while the uranium remains dissolved. The second carrier precipitate may be selected from among the group consisting of rare earth fluorides and oxalates, zirconium phosphate, and bismuth phosphate.

RECOVERY OF PLUTONIUM 3.03

PRECIPITATION 3.03.12

PEROXIDE 3.03.12.04

PLUTONIUM PEROXIDE PRECIPITATION. J. A. Leary, A. N. Morgan, and W. J. Maraman (Los Alamos Scientific Lab., N. Mex.). Ind. Eng. Chem. 51, 27-31 (1959) Jan.

Precipitation of the hexagonal structure of plutonium peroxide is an excellent concentration and purification step in plutonium metal preparation. Formation of an undesirable cubic structure is avoided by proper control of process variables. (auth)

PEROXIDE PROCESS FOR SEPARATION OF RADIOACTIVE MATERIALS. G. T. Seaborg and I. Perlman (to U. S. Atomic Energy Commission). U. S. Patent 2,852,336. Sept. 16, 1958.

A method is described of separating plutonium, in the reduced state, from hexavalent uranium. It consists in treating an aqueous solution containing such uranium and plutonium ions with sulfate ions in order to form a soluble uranium sulfate complex and then treating the solution with a soluble thorium compound and a soluble peroxide compound in order to form a thorium peroxide carrier precipitate which carries down with it the plutonium peroxide present. During this treatment the pH of the solution must be maintained between 2 and 3.

(See also 3.03 11.05)

PHOSPHATE 3.03.12.05

See also, under PROCESS CHEMISTRY AND ENGINEERING, SPECIAL PROCESSES--Bismuth Phosphate (6.17.02).

6365

RECOVERY OF PLUTONIUM FROM AQUEOUS SOLUTIONS. E. J. Reber (to U. S. Atomic Energy Commission). U. S. Patent 2,902,339. Sept. 1, 1959.

A process is described for recovering plutonium values from aqueous solutions by precipitation on bismuth phosphate. The plutonium is secured in its tetravalent state, bismuth salt is added to the solution, and an excess of phosphoric acid anions is added to the solution in two approximately equal installments. The rate of addition of the first installment is about two to three times as high as the rate of addition of the second installment, whereby a precipitate of bismuth phosphate forms, the precipitate carrying the plutonium values. The precipitate is separated from the solution.

22164.

TREATMENT OF PLUTONIUM SOLUTION WITH NO OR NO_2 PRIOR TO REMOVAL OF TETRAVALENT PLUTONIUM FROM SAID SOLUTION BY CARRIER PRECIPITATION. G. E. Moore (to U. S. Atomic Energy Commission). U. S. Patent 2,886,407. May 12, 1959.

A process is presented for the separation of plutonium from fission products carried therewith when plutonium in the reduced oxidation state is removed from a nitric acid solution of irradiated uranium by means of bismuth or lanthanum phosphate as a carrier percipitate. It is known that in plutonium containing solutions prepared by dissolving neutron irradiated uranium in nitric acid the plutonium is at least partially converted to a non-carriable state apparently by the presence of some intermediate reduction product produced by the reduction of nitric acid, such as for example hydrazine, which tends to form a soluble complex therewith. According to the invention such a complex can be avoided by introducing oxides of nitrogen such as nitrogen dioxide into the nitrogen acid solution of neutron irradiated uranium preferably at elevated temperatures as for example 50 to 85% C, prior to the removal of plutonium by the use of a carrier. By this procedure plutonium can be recovered in amounts which represent about 90% when the resulting solution is treated in accordance with known carrier methods referred to above.

22162

PLUTONIUM PURIFICATION PROCESS EMPLOYING THORIUM PYROPHOSPHATE CARRIER. Edward L. King (to U. S. Atomic Energy Commission). U. S. Patent 2,884,305. Apr. 28, 1959.

The separation and purification of plutonium from the radioactive elements of lower atomic weight is described. The process of this invention comprises forming a 0.5 to 2 M aqueous acidic solution containing plutonium ions in the tetravalent state and elements with which it is normally contaminated in neutron irradiated uranium, treating the solution with a double thorium compound and a soluble pyrophosphate compound

RECOVERY OF PLUTONIUM 3.03

PRECIPITATION 3.03.12

PHOSPHATE 3.03.12.05

14401
RECOVERY OF TETRAVALENT CATIONS FROM AQUEOUS SOLUTIONS. R. L. Moore (to U. S. Atomic Energy Commission). U. S. Patent 2,833,799. May 6, 1958.

The recovery of plutonium, zirconium, and tetravalent cerium values from aqueous solutions is described. It consists of adding an alkyl phosphate to a mineral acid aqueous solution containing the metal to be recovered, whereby a precipitate forms with the tetravalent values, and separating the precipitate from the solution. All alkyl phosphates, if water-soluble, are suitable for the process; however, monobutyl phosphate has been found best.

7469
METHOD OF RECOVERING PLUTONIUM VALUES FROM AQUEOUS SOLUTIONS BY CARRIER PRECIPITATION. R. A. James and S. G. Thompson (to U. S. Atomic Energy Commission). U. S. Patent 2,911,282. Nov. 3, 1959.

A process is presented for pretreating aqueous nitric acid–plutonium solutions containing a small quantity of hydrazine that has formed as a decomposition product during the dissolution of neutron-bombarded uranium in nitric acid and that impairs the precipitation of plutonium on bismuth phosphate. The solution is digested with alkali metal dichromate or potassium permanganate at between 75 and 100°C; sulfuric acid at approximately 75°C and sodium nitrate, oxalic acid plus manganous nitrate, or hydroxylamine are added to the solution to secure the plutonium in the tetravalent state and make it suitable for precipitation on $BiPO_4$.

11516
METHOD OF SEPARATING PLUTONIUM. F. Morgan (to U. S. Atomic Energy Commission). U. S. Patent 2,799,555. July 16, 1957.

A method is given for separating Pu from U and fission products. The method consists in dissolving the mixture of Pu, U and fission products in HNO_3 adding $Bi(NO_3)_3$ and $Zr(NO_3)_4$ as carrier compounds, precipitating insoluble phosphates of Pu, Zr, Bi and Nb by adding an aqueous solution of a soluble phosphate, and treating the precipitated phosphates with $H_2C_2O_4$, whereby the compounds of Zr and Nb are made soluble and an insoluble residue of bismuth and plutonium oxalates is formed. The separation of the Pu from the bismuth may be effected by converting the oxalates to chlorides by treatment with HCl. Bismuth is then precipitated by treatment with H_2S, and the residual solution evaporated to obtain plutonium chloride. (auth)

11515
METHOD OF SEPARATING PLUTONIUM. F. Morgan (to U. S. Atomic Energy Commission). U. S. Patent 2,799,554. July 16, 1957.

A method is given for separating Pu from U and fission products. The mixture of Pu with U and fission products is dissolved in HNO_3; $Bi(NO_3)_3$ and $Zr(NO_3)_4$ are added as carriers, and insoluble phosphates of Pu, Zr, Bi and Nb are precipitated by adding an aqueous solution of a soluble phosphate. This mixed phosphate precipitate is treated first with dilute HCl to dissolve $BiPO_4$, and then with HCl of a strength greater than 7 N to dissolve plutonium phosphate, which is subsequently recovered from this HCl solution. (auth)

11514
PHOSPHATE METHOD FOR SEPARATION OF RADIOACTIVE ELEMENTS. S. G. Thompson and G. T. Seaborg (to U. S. Atomic Energy Commission). U. S. Patent 2,799,553. July 16, 1957.

A method is given for separating Pu from U, certain fission products and Np. Neutron irradiated U containing Pu, U, Np and fission products is treated with HNO_3 to obtain an aqueous solution which is adjusted to have a UNH concentration of about 22%. Sulfuric acid is added to the solution to give an acidity of about 0.85 to 1.24N based on sulfuric and nitric acids present. A soluble Bi compound is added to give a Bi ion concentration of about 1 to 2.5 grams per liter of solution. The addition of a soluble phosphate then precipitates $BiPO_4$ carrying substantially all the Pu and leaving the bulk of the U, Np and fission products in solution. (auth)

7478
SEPARATION OF FISSION PRODUCT VALUES FROM THE HEXAVALENT PLUTONIUM BY CARRIER PRECIPITATION. T. H. Davies (to U. S. Atomic Energy Commission). U. S. Patent 2,917,359. Dec. 15, 1959.

RECOVERY OF PLUTONIUM 3.03

PRECIPITATION 3.03.12

PHOSPHATE 3.03.12.05

An improved precipitation of fission products on bismuth phosphate from an aqueous mineral acid solution also containing hexavalent plutonium by incorporating, prior to bismuth phosphate precipitation, from 0.05 to 2.5 grams/liter of zirconium phosphate, niobium oxide, and/or lanthanum fluoride is described. The plutonium remains in solution.

6583
METHOD OF SEPARATING PLUTONIUM. (To U. K. Atomic Energy Authority (Canada).) British Patent 765,924. Nuclear Eng. 2, 172(1957) Apr.

The Pu is to be separated from a mixture containing U and fission products, including Zr and Nb. This mixture is dissolved in HNO_3, then $Bi(NO_3)_3$ and $Zr(NO_3)_4$ are added as carrier compounds; insoluble phosphates of Pu, Zr, Bi, and Nb are precipitated from the solution by adding an aqueous solution of a phosphate. This mixed phosphate precipitate is now subjected to a treatment based on the known fact that $Bi(PO_4)_3$ is easily soluble in dilute HCl and on the discovery that, in such dilute HCl, plutonium phosphate and niobium phosphate are absorbed on zirconium phosphate but that plutonium phosphate may be leached from these other phosphates in HCl of a strength greater than 7N. (auth)

IMPROVEMENTS IN OR RELATING TO METHODS OF SEPARATING PLUTONIUM. Frank Morgan. British Patent 754,651. Aug. 8, 1956.

A method is described for separating Pu, U, and mixed fission products including Zr and Nb from irradiated fuel elements. The rods are dissolved in HNO_3 with the addition of nitrates of Zr and Nb as carrier compounds. An aqueous solution of a soluble phosphate is added which causes the precipitation of insoluble phosphates of Pu, Zr, Bi, and Nb. These are treated with oxalic acids and Zr and Nb are rendered soluble leaving an insoluble residue of Zr and Bi oxalates. These may be separated by several methods. ~~(F.S.)~~

(see also 3.03.11.02)

1762
TWO METHODS FOR THE ISOLATION OF TRACER AMOUNTS OF PLUTONIUM. Jan Rydberg (Research Inst. of National Defence, Sundbyberg, Sweden). Acta Chem. Scand. 9, No. 8, 1252-60(1955).

A modified bismuth phosphate carrier method and a sulfide precipitation method are outlined for the separation of trace quantities of Pu from neutron-irradiated U (irradiation time—1 yr, cooling time—3 mo.). The relative yields and activities of fission products produced in the U samples are tabulated. (C.W.H.)

SULFATE OR SULFIDE 3.03.12.06

PRECIPITATION METHOD OF SEPARATING PLUTONIUM FROM CONTAMINATING ELEMENTS. R. B. Duffield (to U. S. Atomic Energy Commission). U. S. Patent 2,875,026. Feb. 24, 1959.

A method is described for separating plutonium, in a valence state of less than five, from an aqueous solution in which it is dissolved. The method consists in adding potassium and sulfate ions to such a solution while maintaining the solution at a pH of less than 7.1, and isolating the precipitate of potassium plutonium sulfate thus formed.

SULFIDE METHOD PLUTONIUM SEPARATION. R. B. Duffield (to U. S. Atomic Energy Commission). U. S. Patent 2,847,274. Aug. 12, 1958.

A process is described for the recovery of plutonium from neutron irradiated uranium solutions. Such a solution is first treated with a soluble sulfide, causing precipitation of the plutonium and uranium values present, along with those impurities which form insoluble sulfides. The precipitate is then treated with a solution of carbonate ions, which will dissolve the uranium and plutonium present while the fission product sulfides remain unaffected. After separation from the residue, this solution may then be treated by any of the usual methods, such as formation of a lanthanum fluoride precipitate, to effect separation of plutonium from uranium.

RECOVERY OF PLUTONIUM 3.03

PRECIPITATION 3.03.12

SULFATE OR SULFIDE 3.03.12.06

11496 ✓

CHEMICAL TREATMENT OF PLUTONIUM HEXAFLUORIDE AND URANIUM HEXAFLUORIDE. R. Hurst and H. K. Ray (to U. K. Atomic Energy Authority). British Patent 772,617. Nuclear Eng. 2, 350(1957) Aug.

If PuF_6 or UF_6 vapor is passed into strong H_2SO_4, a sulfate of plutonium or uranium becomes deposited as small crystals from the H_2SO_4. The process may be used in connection with a homogeneous nuclear reactor with a solution of UO_2SO_4 and a ThO_2 blanket. Uranium hexafluoride extracted by means of fluoride from the blanket can then be converted into UO_2SO_4 for the reactor core. (auth)

(S also 8 03.14)

GENERAL 3.03.12,07

CARBONATE METHOD OF SEPARATION OF TETRAVALENT PLUTONIUM FROM FISSION PRODUCT VALUES. R. B. Duffield and R. W. Stoughton (to U. S. Atomic Energy Commission). U. S. Patent 2,872,288. Feb. 3, 1959.

It has been found that plutonium forms an insoluble precipitate with carbonate ion when the carbonate ion is present in stoichiometric proportions, while an excess of the carbonate ion complexes plutonium and renders it soluble. A method for separating tetravalent plutonium from lanthanum-group rare earths has been based on this discovery, since these rare earths form insoluble carbonates in approximately neutral solutions. According to the process the pH is adjusted to between 5 and 7, and approximately stoichiometric amounts of carbonate ion are added to the solution causing the formation of a precipitate of plutonium carbonate and the lanthanum-group rare earth carbonates. The precipitate is then separated from the solution and contacted with a carbonate solution of a concentration between 1 M and 3 M to complex and redissolve the plutonium precipitate, and thus separate it from the insoluble rare earth precipitate.

13382 ✓

METHOD OF SEPARATING TETRAVALENT PLUTONIUM VALUES FROM CERIUM SUB-GROUP RARE EARTH VALUES. R. B. Duffield and R. W. Stoughton (to U. S. Atomic Energy Commission). U. S. Patent 2,872,287. Feb. 3, 1959.

A method is presented for separating plutonium from the cerium sub-group of rare earths when both are present in an aqueous solution. The method consists in adding an excess of alkali metal carbonate to the solution, which causes the formation of a soluble plutonium carbonate precipitate and at the same time forms an insoluble cerium-group rare earth carbonate. The pH value must be adjusted to between 5.5 and 7.5, and prior to the precipitation step the plutonium must be reduced to the tetravalent state since only tetravalent plutonium will form the soluble carbonate complex.

POLYMERIZATION AND PRECIPITATION OF PLUTONIUM(IV) IN NITRIC ACID. Arthur Brunstad (General Electric Co., Richland, Wash.). Ind. Eng. Chem. 51, 38-40(1959) Jan. 1.

The formation of colloidal plutonium(IV) hydroxide from plutonium(IV) nitrate solutions was determined as a function of acid and plutonium concentration at several temperatures. Hydrolysis, polymerization, and precipitation take place in unstable solutions with a resulting increase in acidity to a concentration sufficient for a stable true solution. Except for colloidal sols formed from solutions of a few grams per liter, the acidities become too high for the plutonium to remain as a stable colloid. (auth)

6375

METHOD OF SEPARATION OF PLUTONIUM FROM CARRIER PRECIPITATES. L. R. Dawson (to U. S. Atomic Energy Commission). U. S. Patent 2,905,525. Sept. 22, 1959.

The recovery of plutonium from fluoride carrier precipitates is described. The precipitate is dissolved in zirconyl nitrate, ferric nitrate, aluminum nitrate, or a mixture of these complexing agents, and the plutonium is then extracted from the aqueous solution ~~formed~~ with a water-immiscible organic solvent.

RECOVERY OF PLUTONIUM 3.03

PRECIPITATION 3.03.12

GENERAL 3.03.12.07

7471
PROCESSES FOR SEPARATING AND RECOVERING CONSTITUENTS OF NEUTRON IRRADIATED URANIUM. R. E. Connick, J. W. Gofman, and G. C. Pimentel (to U. S. Atomic Energy Commission). U. S. Patent 2,912,302. Nov. 10, 1959.

Processes are described for preparing plutonium, particularly processes of separating plutonium from uranium and fission products in neutron-irradiated uranium-containing matter. Specifically, plutonium solutions containing uranium, fission products and other impurities are contacted with reducing agents such as sulfur dioxide, uranous ion, hydroxyl ammonium chloride, hydrogen peroxide, and ferrous ion whereby the plutonium is reduced to its fluoride-insoluble state. The reduced plutonium is then carried out of solution by precipitating niobic oxide therein. Uranium and certain fission products remain behind in the solution. Certain other fission products precipitate along with the plutonium. Subsequently, the plutonium and fission product precipitates are redissolved, and the solution is oxidized with oxidizing agents such as chlorine, peroxydisulfate ion in the presence of silver ion, permanganate ion, dichromate ion, ceric ion, and a bromate ion, whereby plutonium is oxidized to the fluoride-soluble state. The oxidized solution is once again treated with niobic oxide, thus precipitating the contaminant fission products along with the niobic oxide while the oxidized plutonium remains in solution. Plutonium is then recovered from the decontaminated solution.

7477 ✓
URANOUS IODATE AS A CARRIER FOR PLUTONIUM. D. R. Miller, G. T. Seaborg, and S. G. Thompson (to U. S. Atomic Energy Commission). U. S. Patent 2,917,358. Dec. 15, 1959.

A process is described for precipitating plutonium on a uranous iodate carrier from an aqueous acid solution containing the plutonium in a valence state not greater than +4. A plutonium solution more concentrated than the original solution can then be obtained by oxidizing the uranium to the hexavalent state and dissolving the precipitate, after separating the latter from the original solution, by means of warm nitric acid.

PROCESS OF SEPARATING PLUTONIUM FROM URANIUM. H. S. Brown and O. F. Hill (to U. S. Atomic Energy Commission). U. S. Patent 2,851,333. Sept. 9, 1958.

A process is presented for recovering plutonium values from aqueous solutions. It comprises forming a uranous hydroxide precipitate in such a plutonium bearing solution, at a pH of at least 5. The plutonium values are precipitated with and carried by the uranium hydroxide. The carrier precipitate is then redissolved in acid solution and the pH is adjusted to about 2.5, causing precipitation of the uranous hydroxide but leaving the still soluble plutonium values in solution.

SEPARATION OF PLUTONYL IONS. R. E. Connick and Wm. H. McVey (to U. S. Atomic Energy Commission). U. S. Patent 2,843,453. July 15, 1958.

A process is described for separating plutonyl ions from the acetate ions with which they are associated in certain carrier precipitation methods of concentrating plutonium. The method consists in adding alkaline earth metal ions and subsequently alkalizing the solution, causing formation of an alkaline earth plutonate precipitate. Barium hydroxide is used in a preferred embodiment since it provides alkaline earth metal ion and alkalizes the solution in one step forming insoluble barium plutonate.

10244 ✓
PRECIPITATION METHOD OF SEPARATING PLUTONIUM FROM CONTAMINATING ELEMENTS. J. B. Sutton (to U. S. Atomic Energy Commission). U. S. Patent 2,823,978. Feb. 18, 1958.

This patent relates to an improved method for the decontamination of plutonium. The process consists broadly in an improvement in a method for recovering plutonium from radioactive uranium fission products in aqueous solutions by decontamination steps including by-product carrier precipitation comprising the step of introducing a preformed aqueous slurry of a hydroxide of a metal of group IV B into an aqueous acidic solution which contains the plutonium in the hexavalent state, radioactive uranium fission products contaminant and a by-product carrier precipitate and separating the metal hydroxide and by-product precipitate from the solution. The process of this invention is especially useful in the separation of plutonium from radioactive zirconium and columbium fission products.

RECOVERY OF PLUTONIUM 3.03

PRECIPITATION 3.03.12

GENERAL 3.03.12.07

7000
PLUTONIUM SEPARATION METHOD. J. L. Dreher and S. G. Thompson (to U. S. Atomic Energy Commission). U. S. Patent 2,819,143. Jan. 7, 1958.
An improved method is described for separating plutonium from fission product contaminants. The process consists in treating a cerium group trifluoride carrier precipitate containing reduced plutonium with an acidic solution containing ceric ion which causes the precipitate to be dissolved and oxidizes the plutonium to the hexavalent state. The plutonium containing solution is then contacted with a soluble alkali metal compound and a soluble fluoride which precipitates the rare earth and contaminant ions present but leaves the plutonium in solution. The precipitate is then removed leaving contaminant free plutonium bearing solution.

SOME EXPERIMENTS ON THE PRECIPITATION OF PLUTONIUM COMPOUNDS FROM TRIBUTYL PHOSPHATE SOLUTIONS. W. J. Maraman and L. J. Mullins. June 11, 1956. Decl. Apr. 2, 1957. 8p. (LA-2050)
A survey of the feasibility of precipitating Pu^{4+} compounds from 35% TBP in Gulf BT solution has been made. In general, the precipitations were made using an anhydrous salt, a saturated aqueous solution, and a dilute aqueous solution. The anhydrous precipitants which showed promise for further study were Na_2O_2 and NaF. Promising aqueous solutions were H_2O_2, NaF, HF, and K_2SO_4. The oxalate, hydroxide, iodate, phosphate, sulfite, and sulfide systems either gave precipitates of poor physical characteristics or resulted in high Pu solubilities in the organic phase. The precipitation of Pu^{3+} in the same solvent was not studied because of the relatively rapid oxidation of Pu^{3+} to Pu^{4+} in TBP solution containing HNO_3.

7173 ✓ LA-1139
Los Alamos Scientific Lab., N. Mex.
REMOVAL OF IRON FROM PLUTONIUM SOLUTION BY BASIC ACETATE PRECIPITATION. L. J. Mullins. June 1, 1950. Decl. Dec. 7, 1955. 14p. Contract W-7405-eng-36. $0.15(OTS).
The separation of Pu from Fe by means of a "basic acetate" precipitation was studied. The Pu was oxidized to PuO_2^{2+} with $KMnO_4$ and the Fe precipitated by the addition of $NH_4C_2H_3O_2$. The following results were obtained: 96 to 98% of the Pu may be separated from Fe by the acetate method if acetate is >1.9M and the Pu is oxidized to Pu^{6+}. The carrying of PuO_2^{2+} by the Fe precipitate is primarily a function of acetate concentration. No difference in the carrying of PuO_2^{2+} by the Fe precipitate was noted when Fe was present initially in the concentration range 0.005M to 0.1M. UO_2^{2+} acts effectively as a holdback carrier for Pu when Pu is present in tracer quantities. The effect is negligible for macro concentrations of Pu. (auth)

6191 ✓ N-2205
[Chicago. Univ. Metallurgical Lab.]
SUMMARY OF PLUTONIUM CARRYING AGENTS. B. B. Cunningham. Jan. 16, 1946. Decl. Jan. 16, 1956. (MUC-GTS-2148). $7.80(ph OTS); $3.30(mf OTS).

REDUCTION 3.03.13

See also, under PROCESS CHEMISTRY AND ENGINEERING,
SPECIAL PROCESSES--Purex (6.17.17),
Redox (6.17.19).

6376
REDUCTION IN Pu RECOVERY PROCESSES. D. M. Ritter and R. P. S. Black (to U. S. Atomic Energy Commission). U. S. Patent 2,906,597. Sept. 29, 1959.
A method is described for reducing plutonium from the hexavalent to the tetravalent state in a carrier precipitation process for separating plutonium and nuclear fission products. In accordance with the invention oxalate ions are incorporated in the hexavalent plutonium-containing solution prior to a step of precipitating lanthanum fluoride in the solution.

21003 ✓
PRODUCTION OF PLUTONIUM FROM PLUTONIUM FLUORIDE. Richard D. Baker (to U. S. Atomic Energy Commission). U. S. Patent 2,890,110. June 9, 1959.
Reduction of PuF_4 to metal is described. In the example given, PuF_4 is mixed with 0.3 mole I_2 per mole

RECOVERY OF PLUTONIUM 3.03

REDUCTION 3.03.13

of Pu and Ca powder 25% in excess of that required for reduction of the Pu salt, and I_2 is added. The mixture is charged to a magnesia-lined steel bomb which is heated until reacted in a furnace. The Pu is reduced to metal and recovered as a slug after the bomb is cooled and opened. About 90% yield is obtained. (T.R.H.)

6366
A CHEMICAL METHOD OF TREATING FISSIONABLE MATERIAL. Carl M. Olson (to U. S. Atomic Energy Commission). U. S. Patent 2,902,340. Sept. 1, 1959.

One step of a process for separating plutonium from uranium and fission products is presented. A nitric acid solution containing these constituents is treated with formic acid to reduce simultaneously the plutonium to a valence state of not greater than +4 and destroy and eliminate the excess nitric acid.

7463
METHOD OF REDUCING PLUTONIUM WITH FERROUS IONS. J. L. Dreher, D. E. Koshland, S. G. Thompson, and J. E. Willard (to U. S. Atomic Energy Commission). U. S. Patent 2,907,631. Oct. 6, 1959.

A process is presented for separating hexavalent plutonium from fission product values. To a nitric acid solution containing the values, ferrous ions are added and the solution is heated and held at elevated temperature to convert the plutonium to the tetravalent state via the trivalent state, and the plutonium is then selectively precipitated on a $BiPO_4$ or LaF_3 carrier. The tetravalent plutonium formed is optionally complexed with fluoride, oxalate, or phosphate anion prior to carrier precipitation.

21002
REDUCTION OF PLUTONIUM VALUES IN AN ACIDIC AQUEOUS SOLUTION WITH FORMALDEHYDE. Carl M. Olson (to U. S. Atomic Energy Commission). U. S. Patent 2,890,098. June 9, 1959.

A method is given for reducing Pu to the tetravalent state and lowering the high acidity of dissolver solutions containing U and Pu. Formaldehyde is added to the HNO_3 solution of U and Pu to effect a formaldehyde to HNO_3 molar ratio of 0.375:1 to 1.5:1. The Pu can then be removed from the solution by carrier precipitation using $BiPO_4$ or by ion exchange. (T.R.H.)

22163
RECOVERY OF PLUTONIUM AND NEPTUNIUM FROM AQUEOUS SOLUTIONS. Glenn T. Seaborg, Roy C. Thompson, and Frederic W. Albaugh (to U. S. Atomic Energy Commission). U. S. Patent 2,886,406. May 12, 1959.

A method is presented of reducing neptunium and plutonium to a +4 state of oxidation so that plutonium may be carried with bismuth phosphate from a solution containing uranyl ion and fission products. The reduction process consists of treating a nitric acid solution of neutron irradiated uranium with oxalate ions, manganous ions, and fluoride ions. The presence of any sodium nitrite is advantageous since it destroys any hydrazine formed in the reduction of nitric acid and assures the more complete carrying of plutonium. The reduction is carried out for 1 hour at 15°C in the presence of 0.09 molar oxalic acid, 0.0075 to 0.01 molar Mn^{++} ions and 0.02 molar $(NH_4)_2SiF_6$. Sodium nitrite to the extent of 0.1 molar may also be present under these conditions. 98% of the plutonium is carried by the bismuth phosphate precipitate.

2504
SOLUBILIZATION OF ACTINIDE METAL-CONTAINING SLAG. H. H. Hopkins, Jr. (to U. S. Atomic Energy Commission). U. S. Patent 2,898,186. Aug. 4, 1959.

This patent relates to solubilization of the actinide rare earths valves contained in the slag materials resulting from the reduction of actinide salts, such as plutonium tetrafluoride. According to the invention the slag is subjected to a high temperature chloridizing roast, preferably at about 700°C with gaseous hydrogen chloride, until the actinides within the slag are substantially converted to the chlorides. The resultant chlorinated actinides are then leached from the cooled roasted mass by treating with aqueous 0.01 M nitric acid.

12539 HW-59283(Del.)
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
THE USE OF TETRAVALENT URANIUM AND HYDRAZINE AS PARTITIONING AGENTS IN SOLVENT EXTRACTION PROCESS FOR PLUTONIUM AND URANIUM. J. S. Buckingham, C. A. Colvin, and C. A. Goodall. Feb. 1, 1959. Decl. with deletions Mar. 23, 1959. 14p. Contract W-31-109-Eng-52. $3.30(ph), $2.40(mf) OTS.

RECOVERY OF PLUTONIUM 3.03

REDUCTION 3.03.13

A study of the oxidation-reduction potentials for plutonium and various reducing agents indicates that hydrazine, tetravalent uranium, and ferrous ion have the potential to reduce all valence states of plutonium. It was demonstrated that tetravalent uranium nitrate can be used to reduce plutonium to the trivalent state ~~and~~ so (hence) ~~hence~~ can be separated from uranium by solvent extraction. (J.E.D.) the plutonium

2504

SOLUBILIZATION OF ACTINIDE METAL-CONTAINING SLAG. H. H. Hopkins, Jr. (to U. S. Atomic Energy Commission). U. S. Patent 2,898,186. Aug. 4, 1959.

This patent relates to solubilization of the actinide rare earths valves contained in the slag materials resulting from the reduction of actinide salts, such as plutonium tetrafluoride. According to the invention the slag is subjected to a high temperature chloridizing roast, preferably at about 700°C with gaseous hydrogen chloride, until the actinides within the slag are substantially converted to the chlorides. The resultant chlorinated actinides are then leached from the cooled roasted mass by treating with aqueous 0.01 M nitric acid.

PLUTONIUM CONCENTRATION AND DECONTAMINATION METHOD. D. C. Overholt and F. W. Tober (to U. S. Atomic Energy Commission). U. S. Patent 2,863,718. Dec. 9, 1958.

Plutonium can be removed from a solution containing uranium-irradiated materials by adjusting the solution to fractional-normal nitric acid concentration while reducing the plutonium ion to the tetravalent state by adding hydrazine, ferrous sulfamate, or hydroxylamine sulfate, contacting the solution with a cation exchanger such as $Na_2O \cdot Al_2O_3 \times SiO_2 \cdot yH_2O$, and then selectively eluting the absorbed plutonium with 4 molar nitric acid. If the cation exchanger contains sulfonic acid, uranium is absorbed and selectively eluted with dilute sulfuric acid.

PROCESS OF SECURING PLUTONIUM IN NITRIC ACID SOLUTIONS IN ITS TRIVALENT OXIDATION STATE. J. R. Thomas (to U. S. Atomic Energy Commission). U. S. Patent 2,849,277. Aug. 26, 1958.

Various processes for the recovery of plutonium require that the plutonium be obtained and maintained in the reduced or trivalent state in solution. Ferrous ions are commonly used as the reducing agent for this purpose, but it is difficult to maintain the plutonium in a reduced state in nitric acid solutions due to the oxidizing effects of the acid. It has been found that the addition of a stabilizing or holding reductant to such solution prevents reoxidation of the plutonium. Sulfamate ions have been found to be ideally suitable as such a stabilizer even in the presence of nitric acid.

SEPARATION OF PLUTONIUM HYDROXIDE FROM BISMUTH HYDROXIDE. G. W. Watt (to U. S. Atomic Energy Commission). U. S. Patent 2,848,301. Aug. 19, 1958.

An improved method is described for separating plutonium hydroxide from bismuth hydroxide. The end product of the bismuth phosphate processes for the separation and concentration of plutonium is a mixture of bismuth hydroxide and plutonium hydroxide. It has been found that these compounds can be advantageously separated by treatment with a reducing agent having a potential sufficient to reduce bismuth hydroxide to metallic bismuth but not sufficient to reduce the plutonium present. The resulting mixture of metallic bismuth and plutonium hydroxide can then be separated by treatment with a material which will dissolve plutonium hydroxide but not metallic bismuth. Sodium stannite is mentioned as a preferred reducing agent, and dilute nitric acid may be used as the separatory solvent.

14409

METHOD OF REDUCING PLUTONIUM COMPOUNDS. I. B. Johns (to U. S. Atomic Energy Commission). U. S. Patent 2,837,402. June 3, 1958.

A method is described for reducing plutonium compounds in aqueous solution from a higher to a lower valence state. This reduction of valence is achieved by treating the aqueous solution of higher valence plutonium compounds with hydrogen in contact with an activated platinum catalyst.

RECOVERY OF PLUTONIUM 3.03

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14402 ✓
PROCESS FOR PURIFYING PLUTONIUM. D. F. Mastick and E. P. Wigner (to U. S. Atomic Energy Commission). U. S. Patent 2,833,800. May 6, 1958.

A method is described of separating plutonium from small amounts of uranium and other contaminants. An acidic aqueous solution of higher valent plutonium and hexavalent uranium is treated with a soluble iodide to obtain the plutonium in the plus three oxidation state while leaving the uranium in the hexavalent state, adding a soluble oxalate such as oxalic acid, and then separating the insoluble plus the plutonium trioxalate from the solution.

10240
METHOD OF SEPARATING PLUTONIUM. H. S. Brown and O. F. Hill (to U. S. Atomic Energy Commission). U. S. Patent 2,822,239. Feb. 4, 1958.

Plutonium hexafluoride is a satisfactory fluorinating agent and may be reacted with various materials capable of forming fluorides, such as copper, iron, zinc, etc., with consequent formation of the metal fluoride and reduction of the plutonium to the form of a lower fluoride. In accordance with the present invention, it has been found that the reactivity of plutonium hexafluoride with other fluoridizable materials is so great that the process may be used as a method of separating plutonium from mixtures containing plutonium hexafluoride and other vaporized fluorides even though the plutonium is present in but minute quantities. This process may be carried out by treating a mixture of fluoride vapors comprising plutonium hexafluoride and fluoride of uranium to selectively reduce the plutonium hexafluoride and convert it to a less volatile fluoride, and then recovering said less volatile fluoride from the vapor by condensation.

1265
THE REDUCTION OF PLUTONYL SULFATE BY GAMMA RAYS. Monique Pagès, Christiane Ferradini, and Moïse Haïssinsky. Compt. rend. 245, 1128-9(1957) Sept. 30. (In French)

THE REDUCTION OF PLUTONIUM BY TETRAVALENT URANIUM. Jan Rydberg (Research Inst. of National Defence, Sundbyberg, Sweden). Acta Chem. Scand. 11, No. 1, 201-2(1957).

9235
ISOTOPIC EXCHANGE BETWEEN Pu(III) AND Pu(IV). Thomas K. Keenan (Los Alamos Scientific Lab., N. Mex.). J. Am. Chem. Soc. 78, 2339(1956) May 20.

Studies were initiated on the exchange reaction between Pu^{3+} and Pu^{4+} in $HClO_4$ solutions at 0°C. The rate of exchange in 0.5 formal is approximated by the equation, $R = k^1 (III) (IV)$, where $k^1 = 1.10 \pm 0.10 \times 10^4$ liter/mole/min. (C.W.H.)

13856 LA-473
Los Alamos Scientific Lab., N. Mex.
PREPARATION OF PLUTONIUM METAL BY THE BOMB METHOD. R. D. Baker. June 25, 1946. Decl. Aug. 8, 1958. 63p. Contract [W-7405-eng-36]. $10.80 (ph OTS); $3.90(mf OTS).

The preparation of Pu by bomb reduction of $PuCl_3$ and PuF_4 with Ca in MgO crucibles is described. Good Pu yields were obtained with both chloride and fluoride. (C.W.H.)

SOLVENT EXTRACTION 3.03.14

See also, this section,
CHELATION (3.03.02),
and,
under PROCESS CHEMISTRY AND ENGINEERING,
SOLVENTS--Dibutyl Carbitol (6.16.05.02),
SPECIAL PROCESSES--Butex (6.17.03),
Metal Recovery (6.17.15),
Purex (6.17.17),
Redox (6.17.19).

RECOVERY OF PLUTONIUM 3.03

SOLVENT EXTRACTION 3.03.14

TBP 3.03.14.01

See also, under PROCESS CHEMISTRY AND ENGINEERING, SPECIAL PROCESSES--Purex (6.17.17).

PROCESSING OF IRRADIATED REACTOR FUELS. EFFECT OF DIBUTYL PHOSPHATE IN THE TRIBUTYL PHOSPHATE PROCESSING SYSTEM. W. W. Morgan, W. G. Mathers, and R. G. Hart (Atomic Energy of Canada, Ltd., Chalk River, Ont.). Ind. Eng. Chem. 51, 817-20(1959) July.

Separation of Pu and U by use of a contactor which takes advantage of the partition differences of Pu(IV) and U(IV) in dilute HNO_3, or by selectively reducing Pu(IV) to Pu(III) and taking advantage of the gross partition differences of Pu(III) and U(IV) is discussed. A study to achieve this separation in a single contactor using a Pu stripping stream which introduces no foreign materials such as ferrous ions to the product stream was conducted. Trace quantities of dibutyl phosphates have deterimental effects on separation. Practical methods were evolved for overcoming difficulties and producing, in a single cycle, a U product with a U to Pu wt. ratio of 10^8 to 1. (J.R.D.)

14390 ✓

EXTRACTION OF PLUTONIUM AND URANIUM. U. K. Atomic Energy Authority (Canada). British Patent 801,743. Nuclear Eng. 4, 238(1959) May.

An aqueous nitric acid solution, containing fission products in addition to plutonium in the tetravalent or higher state and uranium in the hexavalent state is treated with a water-immiscible phase which includes tributyl phosphate.

THE STABILITY CONSTANTS OF THE COMPLEX NITRATE OF TRIVALENT PLUTONIUM IN AQUEOUS AND TRIBUTYL PHOSPHATE SOLUTIONS. V. B. Shevchenko, V. G. Timoshev, and A. A. Voikov. Atomnaya Energ. 6, 426-30(1959) Apr. (In Russian)

The extraction of the complex nitrate of trivalent plutonium from various aqueous phase concentrations was analyzed. The extraction by tributyl phosphate solution from nitric acid solutions was achieved in the form of $Pu(NO_3)_3 \cdot TBP$. The trivalent plutonium distribution coefficient is only slightly dependent on the hydrogen ion concentration. The stability constants were determined for the complexes $Pu(NO_3)_3 \cdot 3TBP$, $Pu(NO_3)_3$, $Pu(NO_3)_2^+$, and $Pu(NO_3)^{2+}$ which are equal to 0.75 ± 0.1, 14.4 ± 0.8, 14.3 ± 0.8, and 5.9 ± 0.5, respectively. (tr-auth)

THE EFFECTS OF MONO- AND DIBUTYL PHOSPHATES ON THE TRIBUTYL PHOSPHATE EXTRACTION OF Pu. V. B. Shevchenko and V. S. Smelov. Atomnaya Energ. 5, 542-5(1958). (In Russian)

Coefficients of mono- and dibutyl phosphate distributions between the organic and aqueous phases were determined. It is shown that monobutyl phosphate concentrations below 0.001 mole/liter (balanced concentration) and dibutyl phosphates below 0.0001 mole/liter have no effect on the plutonium extraction by tributyl phosphates. (tr-auth)

2267 HW-51958

General Electric Co. Hanford Atomic Products Operation, Richland, Wash.

THE EXTRACTION OF NEPTUNIUM(IV) AND PLUTONIUM (IV) FROM NITRIC ACID SOLUTION WITH TRI-N-OCTYL AMINE. John C. Sheppard. Aug. 22, 1957. Decl. Oct. 23, 1957. 13p. Contract [W-31-109-Eng-52]. $3.30 (ph OTS); $2.40(mf OTS).

Tri-n-octylamine conveniently extracts neptunium(IV) and plutonium(IV) from nitric acid solutions. Preliminary experiments to demonstrate the utility of this type of reagent have been performed; in particular the extraction of neptunium or plutonium from Purex waste streams has been demonstrated. Experiments have also been performed to determine what may be expected in the way of fission product decontamination and radiation stability of tri-n-octylamine. Fission product decontamination is not appreciably altered by the organic phase being highly irradiated. This report describes preliminary work designed to determine the solvent extraction characteristics

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SOLVENT EXTRACTION 3.03.14

TBP 3.03.14.01

7141 CRDC-630
Atomic Energy of Canada Ltd. Chalk River Project, Chalk River, Ont.
TRIBUTYL PHOSPHATE PROCESSING OF PLUTONIUM-ALUMINUM ALLOY FUELS. R. G. Hart. Mar. 1957. 27p. (AECL-429). $1.00(AECL).

Partition coefficients pertinent to the processing of Pu-Al alloy fuels by TBP extraction have been obtained. McCabe-Thiele calculations have been made to predict behavior under various conditions in a continuous system. The calculations show that a system of extraction using 5% TBP-soltrol should give good Pu recovery and satisfactory fission product decontamination. A system of extraction using 20% TBP-soltrol would give good Pu recovery but poor fission product decontamination. If sufficient U to saturate the solvent is added to the Pu-Al dissolver solution, the fuel could be processed using 20% TBP-soltrol in the same manner and in the same plant as irradiated U. Good Pu recovery and good fission product decontamination should be obtained with this system. (auth)

11136 ✓
TRI-n-BUTYL PHOSPHATE AS AN EXTRACTING SOLVENT FOR INORGANIC NITRATES. III. THE PLUTONIUM NITRATES. G. F. Best, H. A. C. McKay, and P. R. Woodgate (Atomic Energy Research Establishment, Harwell, Didcot, Berks). J. Inorg. and Nuclear Chem. 4, 315-20 (1957).

Plutonium(III) nitrate is relatively inextractable by tri-n-butyl phosphate (TBP), but plutonium^{4+} and $PuO_2(NO_3)_2$ are freely extractable. Plutonyl nitrate is, however, less extractable than $UO_2(NO_3)_2$ by a factor of about 10. Experiments in which the TBP concentration was varied indicated the formation of $Pu(NO_3)_4 \cdot 2TBP$ and $PuO_2(NO_3)_2 \cdot 2TBP$. In the case of $Pu(NO_3)_4$ it was shown that the temperature coefficient of extraction is small, that salting-out by $NaNO_3$ causes a striking increase in extraction, and that $UO_2(NO_3)_2$ depresses the extraction. Increase in the $Pu(NO_3)_4$ concentration may either raise or lower the partition coefficient according to the conditions. (auth)

1477 JENER-45
Joint Establishment for Nuclear Energy Research, Kjeller, Norway.
ON THE ISOLATION OF PLUTONIUM BY A SOLVENT EXTRACTION PROCEDURE. Period Covered: October 1955-May 1956. D. H. W. den Boer and Z. I. Dizdar. Oct. 1956. 20p.

A procedure for the separation of Pu from neutron-irradiated U by solvent extraction is presented. The starting material was U rods from JEEP containing ~5 mg Pu per kg U. The Pu and U were extracted from the fission products with a 30 vol. % TBP-70 vol. % kerosene solution. The Pu was reduced and stripped into the aqueous phase by aqueous hydrazine solutions. A final washing with TBP-kerosene removed most of the U. A complete separation of the layers was accomplished by centrifugation. (W.L.H.)

2666 ✓
A SOLVENT PROCESS FOR THE EXTRACTION OF PLUTONIUM FROM URANIUM IRRADIATED IN REACTORS. Bertrand Goldschmidt and Isabelle Prevot. Bull. soc. chim. France, No. I, 103-6(1956) Jan. (In French)

A description is given for the conditions of the separation of Pu, fission products, and U by means of a selective extraction of the nitrates by tributyl phosphate diluted with a paraffin hydrocarbon. The Pu and U are first extracted simultaneously, and then they are successively re-extracted by reduction. Since the decontamination of γ emitters is inadequate in the first cycle, the complementary treatments of U and Pu, in order to obtain amounts of the desired purity, are also indicated. Also, a description is given for a process of nitric dissolution of active U, without liberating vapors, allowing the reclamation of radioactive gases. The final concentration of fission products in a solution is described. (tr-auth)

PLANT-SCALE CONCENTRATION OF Am AND Pu(III) USING TRIBUTYL PHOSPHATE. W. J. Maraman, A. J. Beaumont, R. S. Day, and R. S. Winchester. Aug. 2, 1954. 13p. Decl. Nov. 6, 1957. (LA-1699)

Recovery of Am^{3+} and Pu^{3+} on a plant scale by extraction with TBP from slag and crucible and other recovery solutions is described. Process losses were 0.03% Am and 0.01% Pu. The product stream contained 0.08 g Am/l and 0.15 g Pu/l. The final solution cleanup to allow discard (total alpha activity equivalent to less than 10^{-4} g Pu/l) was accomplished by TBP extraction of Pu^{3+} under conditions of low acidity and high nitrate.

RECOVERY OF PLUTONIUM 3.03

SOLVENT EXTRACTION 3.03.14

TBP 3.03.14,01

18644 AERE-R-2933
United Kingdom Atomic Energy Authority. Research Group. Atomic Energy Research Establishment, Harwell, Berks, England.
SOME OBSERVATIONS ON THE EXTRACTION OF NITRIC ACID, URANIUM AND PLUTONIUM BY TRI-ISONONYLAMINE. U. Bertocci. May 1959. 17p.
The extraction of nitric acid, nitrous acid, and U and Pu nitrates from acidic solutions using tri-isononylamine (TNA) in xylene as extractant was studied. Some experiments were also done on the back extraction of Pu from the organic phase by reduction to the trivalent state. (auth)

15195
SOLVENT EXTRACTION PROCESS FOR PLUTONIUM. G. T. Seaborg (to U. S. Atomic Energy Commission). U. S. Patent 2,882,124. Apr. 14, 1959.
The separation of plutonium from aqueous inorganic acid solutions by the use of a water immiscible organic extractant liquid is described. The plutonium must be in the oxidized state, and the solvents covered by the patent include nitromethane, nitroethane, nitropropane, and nitrobenzene. The use of a salting out agent, such as ammonium nitrate in the case of an aqueous nitric acid solution is advantageous. After contacting the aqueous solution with the organic extractant, the resulting extract and raffinate phases are separated. The plutonium may be recovered by any suitable method.

THE PROPERTIES OF INORGANIC COMPOUNDS IN ORGANIC SOLVENTS. II. THE ION EXCHANGE REACTIONS OF QUADRIVALENT PLUTONIUM IN DIETHYLENE GLYCOL DIBUTYL ETHER. D. G. Tuck and G. A. Welch (United Kingdom Atomic Energy Authority, Sellafield, Cumb., Eng.). J. Inorg. & Nuclear Chem. 9, 302-10(1959) Mar.
The ion exchange reactions of plutonium(IV) nitrate extracted into diethylene glycol dibutyl ether (butex) from aqueous nitric acid have been investigated. For both cation and anion resins considerable sorption of plutonium occurred; in both systems, maximum values of K_d were found at nitric acid concentrations in the solvent of about 0·4 M. The effect of nitric acid on the distribution coefficients has been correlated with the plutonium species present; in strong nitric acid plutonium(IV) appears to exist as solvated $H_2Pu(NO_3)_6$, $Pu(NO_3)_4$ having little stability in this solvent. The ion exchange behavior has also been studied in solutions of perchloric acid in butex. (auth)

22154
MECHANISM OF THE PLUTONIUM NITRATE EXTRACTION BY MONO- AND DIBUTYL PHOSPHATES. V. B. Shevchenko and V. S. Smelov. Atomnaya Energ. 6, 140-4(1959) Feb. (In Russian)
The mechanism of plutonium nitrate extraction by mono- and dibutyl phosphates (MBP and DBP) is described. It is shown that plutonium is extracted as PuK_4. Where K is $[(C_4H_9)_2PO_4]^-$ or $[C_4H_9HPO]_4^-$. The equilibrium constant with MBP is $(1.5 \pm 0.25) \times 10^3$ and with DBP is $(6.15 \pm 0.85) \times 10^3$. (tr-auth)

PROCESS FOR THE RECOVERY OF PLUTONIUM. H. A. Potratz (to U. S. Atomic Energy Commission). U. S. Patent 2,864,841. Dec. 16, 1958
A process for the separation of plutonium from uranium and other associated radioactive fission products is described consisting of contacting an acid solution containing plutonium in the tetravalent state and uranium in the hexavalent state with enough ammonium carbonate to form an alkaline solution, adding cupferron to selectively form plutonium cupferride, then recovering the plutonium cupferride by extraction with a water immiscible organic solvent such as chloroform.

SEPARATION OF URANIUM, PLUTONIUM, AND FISSION PRODUCTS. R. Spence and M. W. Lister (to U. S. Atomic Energy Commission). U. S. Patent 2,864,664. Dec. 16, 1958.
Uranium and plutonium can be separated from neutron-irradiated uranium by a process consisting of dissolving the irradiated material in nitric acid, saturating the solution with a nitrate salt such as ammonium nitrate, rendering the solution substantially neutral with a base such as ammonia, adding a reducing agent such as hydroxylamine to change plutonium to the trivalent state, treating the solution with a substantially water immiscible organic solvent such as dibutoxy di-

RECOVERY OF PLUTONIUM 3.03

SOLVENT EXTRACTION 3.03.14

TBP 3.03.14.01

6542 A/CONF.15/P/2346
PURIFICATION ET CONCENTRATION FINALES DU PLUTONIUM EN FIN DE TRAITEMENT DE L'URANIUM IRRADIE A L'USINE-PILOTE DE FONTENAY-AUX-ROSES. (Purification and Final Concentration of Plutonium at the End of the Treatment of Irradiated Uranium at the Pilot Plant of Fontenay-aux-Roses.) I. Prevot, J. Corpel, and P. Regnaut (Commissariat à l'Énergie Atomique, Paris). 38p.

A method is given for the concentration and purification of plutonium which has already had a primary extraction with TBP, followed by a reducing reextraction.

The method consists of three parts: (1) a new solvent extraction of plutonium followed by a reducing reextraction, which improves the decontamination of fission products, (2) passage of the solution obtained on ion exchange resins, which gives a concentrated solution of very pure plutonium, and (3) precipitation of plutonium trifluoride which is used for the production of the metal. The theoretical study which led to the establishment of the procedure is discussed. A detailed description is given of the process along with some of the practical results obtained. (J.S.R.)

8731 ✓
PLUTONIUM RECOVERY BY A REFLUX SOLVENT-EXTRACTION SYSTEM. Burton F. Judson (Hanford Atomic Products Operation, Richland, Wash.). pp. 302-6 in "Progress in Nuclear Energy. Series III. Process Chemistry. Volume 2."

Plutonium may be purified and concentrated by a product-reflux solvent-extraction system. A process involving tributyl phosphate in carbon tetrachloride has been developed and tested on the semi-works scale. The advantages and disadvantages of employing product-refluxing are discussed with respect to attainable concentration, product purity, inventory and ease of plant design and operations. (auth)

~~6503~~ (A/CONF.15/P/137)
THE EXTRACTION OF PLUTONIUM AT VARIOUS OXIDATION POTENTIALS. Göran Carleson (~~AB Atomenergi~~, Stockholm). 16p.

The extraction of plutonium in tracer amounts by 40% tributyl phosphate (TBP), diluted in kerosene, from nitric acid solutions of varying concentration, either pure or containing small amounts of some oxidizing or reducing agents was investigated. Distribution coefficients were determined for Pu(III), Pu(IV), and Pu(VI), and their partition curves were constructed. For acidities <2 M, higher valency states of Pu are rapidly and quantitatively reduced to Pu(III) by very dilute solutions of ferrosulfamate and hydroxylamine, while only a partial reduction is obtained with hydrazine. In higher HNO_3 concentrations, however, the process is much slower. The rate as well as the upper limit of acidity for a complete reduction was found to be a function of reducing conditions and shaking time. A slight increase of the upper limit can be obtained in certain mixtures of the reducing agents mentioned above. The stabilities of TBP are also given. The mechanism of the extraction of Pu in different valency states is discussed, and it is shown that at equilibrium with a water phase of acidity <7 M, Pu is extracted to the organic phase as the neutral complexes $Pu(NO_3)_3(TBP)_3$, $Pu(NO_3)_4(TBP)_2$, and $PuO_2(NO_3)_2(TBP)_2$ in competition with the less strongly coördinated complexes $(HNO_3)(TBP)$ and $(H_2O)(TBP)$.

OTHER THAN TBP 3.03.14.02

20009
SEPARATION OF PLUTONIUM. (to UK Atomic Energy Authority (Canada)). British Patent 805,302. Nuclear Eng. 4, 324(1959) July-Aug.-Sept.

The use of triglycoldichloride has been proposed as the non-aqueous solvent for a fractioning method in which the plutonium is extracted from aqueous nitrate solutions into a non-aqueous phase to uranium and/or fission products, which remain in the aqueous phase. It has been found that triglycoldichloride is much more effective when it has added to it a saturated aliphatic-aldehyde containing at least two carbon atoms per molecule, benzaldehyde, or a polyhydric phenol.

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OTHER THAN TBP 3.03.14.02

ethylether to selectively extract the uranium, making the residual aqueous solution acid with nitric acid, adding an oxidizing agent such as ammonium bromate to oxidize the plutonium to the hexavalent state, and selectively extracting the plutonium by means of an immiscible solvent, such as dibutoxy diethylether.

SEPARATION OF PLUTONIUM FROM ELEMENTS HAVING AN ATOMIC NUMBER NOT LESS THAN 92. F. T. Fitch and D. S. Russell (to U. S. Atomic Energy Commission). U. S. Patent 2,852,338. Sept. 16, 1958.

A method is described for separating plutonium from other elements having atomic numbers not less than 92. It has been proposed in the past to so separate plutonium by solvent extraction into an organic solvent using triglycoldichloride as the organic solvent. The improvement lies in the discovery that triglycoldichloride performs far more efficiently as an extractant, when certain second organic compounds are added to it. Mentioned as satisfactory additive compounds are benzaldehyde, saturated aliphatic aldehydes containing at least two carbon atoms, and certain polyhydric phenols.

14693 A/CONF.15/P/544
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
TERTIARY AMINE EXTRACTION OF PLUTONIUM FROM NITRIC ACID SOLUTIONS. A. S. Wilson. 7p. $0.50(OTS).

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

Data are presented to show the effect of pertinent variables on the extraction of plutonium. The extraction of plutonium(III) and (VI) is shown to be small. The data on the extraction of plutonium(IV) by several amines are presented and discussed. Some experiments on the effect of the organic diluent on the solubility of the plutonium(IV)-amine complex are presented. Temperature effects were also investigated and these results are summarized. Some discussion on the probable mechanism responsible for the extraction of plutonium(IV) by tertiary amines is presented. (auth)

2281
SEPARATION AND DETERMINATION OF PLUTONIUM BY LIQUID-LIQUID EXTRACTION. F. L. Moore and J. E. Hudgens, Jr. (Oak Ridge National Lab., Tenn.). Anal. Chem. 29, 1767-70(1957) Dec.

A rapid and quantitative radioachemical method for separation and determination of plutonium is based on liquid-liquid extraction of plutonium(IV) with 0.5M 2-thenoyltrifluoroacetone in xylene. Plutonium tracer is stripped from organic phase with 10M nitric acid and is recovered free of interferences, both radioactive and nonradioactive. Plutonium recovery is 99 ± 3%. (auth)

LIQUID-LIQUID EXTRACTION OF URANIUM AND PLUTONIUM FROM HYDROCHLORIC ACID SOLUTION WITH TRI(ISO-OCTYL) AMINE; SEPARATION OF URANIUM AND PLUTONIUM FROM THORIUM AND FISSION PRODUCTS. Fletcher L. Moore. June 18, 1957. 21p. (CF-57-6-61)

A new and rapid method for the liquid-liquid extraction of U and Pu from HCl solution is based on the use of tri(iso-octyl)amine dissolved in xylene or methylisobutylketone. U and/or Pu are separated from Th, alkalis, alkaline earths, rare earths, Zr, Nb, Ru, and other elements which do not form anionic species under the conditions described. The technique may be used for either tracer or macro quantities of U. Several practical applications of the method for the separations chemist are proposed.

11143
THE SEPARATION OF PLUTONIUM BY EXTRACTION WITH THENOLYTRIFLUORACETONE(TTA). J. G. Cuninghame and G. L. Miles (Atomic Energy Research Establishment, Harwell, Berks, England). J. Inorg and Nuclear Chem. 3, 54-63(1956) Aug.

The separation of Pu by solvent extraction with TTA solutions is discussed. Distribution ratio data are presented together with an experimental survey of the factors affecting them. Values have been calculated for the equilibrium constant of the solvent extraction reaction for Pu^{4+}. (auth)

7481
EXTRACTION OF PLUTONIUM VALUES FROM ORGANIC SOLUTIONS. G. T. Seaborg (to U. S. Atomic Energy Commission). U. S. Patent 2,918,349. Dec. 22, 1959.

A process is described for back-extracting plutonium, present in an organic solvent in at least the tetravalent state, with nitric acid.

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SOLVENT EXTRACTION 3.03.14

OTHER THAN TBP 3.03.14.02

10097
PRIMARY SEPARATION PLANT AT THE WINDSCALE WORKS. H..G. Davey. Nuclear Power 1, 53-61(1956) June.

Plutonium is manufactured in Britain in the two air-cooled, graphite-moderated piles of the Windscale works of the United Kingdom Atomic Energy Authority. After removal from the piles the irradiated fuel rods are first stored under water for several weeks. They are then dissolved in nitric acid and the solution passed through three towers from which streams of fission products, plutonium and uranium are obtained by solvent extraction with dibutyl carbitol. The plutonium and uranium streams undergo further purification while the fission products are concentrated and stored. The plant has a highly active section and a moderately active one and although it was at first doubted whether access even to the medium active cell would be possible, methods have since been developed for achieving this. Full details of the process are given, and some of the operational problems discussed. (auth)

PROPERTIES OF THE SYSTEM-URANYL NITRATE-ALUMINUM NITRATE-NITRIC ACID-WATER-HEXONE. PART VII. DISTRIBUTION OF URANYL NITRATE, NITRIC ACID, PLUTONIUM(IV) AND PLUTONIUM(VI) FROM 0 TO 60°C. L. L. Burger, I. M. Rehn, and C. M. Slansky. Feb. 1, 1952. Decl. Apr. 3, 1957. 24p. (HW-19949)

6539 A/CONF.15/P/2216
OTDELENIE URANA I PLUTONIYA OT PRODUKTOV DELENIYA EKSTRAKTSIEI SMES'YU DIBUTILOVOGO EFIRA I CHETYREKHKHLORISTOGO UGLERODA. (Separation of Uranium and Plutonium from Fission Products by a Mixture of Dibutyl Ether and Carbon Tetrachloride.) V. M. Vdovenko and M. P. Koval'skaya (U.S.S.R.). 11p.

A description is given of an extraction technique for processing irradiated uranium. The solvent is an explosion-proof mixture of dibutyl ether and carbon tetrachloride. After coextraction of hexavalent uranium and plutonium nitrates so that the bulk of fission elements remains in the aqueous waste solution, plutonium is separated from uranium by reductive re-extraction. Further purification of plutonium is carried out in a 2-cycle extraction-re-extraction process. The separation of uranium from fission products is carried out in a 1-cycle operation. Uranium recovery yield is 99.9%; plutonium yield is 98%. (auth)

6506 A/CONF.15/P/307
THE CHEMICAL PROCESSING OF IRRADIATED FUELS FROM THERMAL REACTORS. G. R. Howells, T. G. Hughes, D. R. Mackey, and K. Saddington (United Kingdom Atomic Energy Authority, Sellafield, Cumb., Eng.). 49p.

The chemical plants located at the Windscale Works of the U.K. Atomic Energy Authority are designed to process natural or near-natural uranium fuels discharged from thermal reactors. The purpose is to separate the uranium and plutonium in pure forms from the associated fission products and from one another. This is accomplished by the use of solvent extraction methods after dissolution of the fuel rods in nitric acid. The organic solvents used at different stages of the process are dibutoxytetraethylene glycol ("Butex") and tributyl phosphate diluted with kerosene. The pure uranyl nitrate solution is returned to the Springfields Works for further treatment prior to re-enrichment and re-fabrication. The pure plutonium nitrate solution is converted to the metal billet form. The chemistry, chemical engineering, and engineering aspects of the existing plants and processes are described, and the experience gained as a result of some six years of operation is presented. (auth)

7465
SEPARATION OF URANIUM, PLUTONIUM AND FISSION PRODUCTS. C. M. Nicholls, I. Wells, and R. Spence (to U. S. Atomic Energy Commission). U. S. Patent 2,908,547. Oct. 13, 1959.

The separation of uranium and plutonium from neutron-irradiated uranium is described. The neutron-irradiated uranium is dissolved in nitric acid to provide an aqueous solution 3N in nitric acid. The fission products of the solution are extruded by treating the solution with dibutyl carbitol substantially 1.8N in nitric acid. The organic solvent phase is separated and neutralized with ammonium hydroxide and the plutonium reduced with hydroxylamine base to the trivalent state. Treatment of the mixture with saturated ammonium nitrate extracts the reduced plutonium and leaves the uranium in the organic solvent.

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2466 ✓ ORNL-2830
Oak Ridge National Lab., Tenn.
RECOVERY OF URANIUM AND PLUTONIUM FROM SULFURIC ACID DECLADDING SOLUTIONS. D. E. Horner and C. F. Coleman. Nov. 25, 1959. 46p. Contract W-7405-eng-26. OTS.

Uranium and plutonium were recovered by liquid-liquid extraction from simulated sulfuric acid stainless steel decladding solution with several extractants. Consecutive extraction of U(IV) and Pu(III) or (IV) by 0.1 to 0 3 M primary amine in hydrocarbon–alcohol diluent appeared promising, and chemical flowsheets were demonstrated in laboratory-scale continuous countercurrent extraction. Extraction of U(VI) with a dialkylphosphoric acid appeared promising when plutonium recovery is not needed. Recovery is also chemically feasible by extraction of U(VI) and Pu(IV) with an N-benzyl secondary alkyl amine or a trialkylphosphine oxide. The amine extracts are stripped with nitric acid, giving a sulfate-nitrate product solution. The organophosphorus extractants permit elimination of the sulfate but require sodium carbonate for stripping. (auth)

DISPROPORTIONATION OF Pu(IV) AND Pu(V) IN NITRIC ACID SOLUTIONS. P. I. Artyukhin, V. I. Medvedovskii, and A. D. Gel'man (Inst. of Physical Chemistry, Academy of Sciences, U.S.S.R.). Zhur. Neorg. Khim. 4, 1324-31(1959) June. (In Russian)

The analysis of disproportionation of Pu(IV) and Pu(V) in nitric acid solutions at different concentrations showed that disproportionation of Pu(IV) follows an identical mechanism to that in perchloric acid. Because of complex formation, the equilibrium constant for the reaction $3Pu(IV) \rightleftharpoons 2Pu(III) + Pu(VI)$ is an inverse ratio to nitric acid concentration at 5.3°C. The rate of disproportionation of Pu(V) in nitric acid solutions increases with hydrogen ion concentration. It is also shown that at the beginning the disproportionation of Pu(V) is determined by the reaction $Pu(V) + Pu(V) \rightleftharpoons Pu(IV) + (Pu(VI)$, but with the accumulation of Pu(III), the reaction $Pu(V) + Pu(III) \rightleftharpoons 2\ Pu(IV)$ begins to prevail, leading to the autocatalytic acceleration of the process. (R.V.J.)

THE IMPORTANCE OF SOLVENT EXTRACTION IN ATOMIC ENERGY. E. Glueckauf (Atomic Energy Research Establishment, Harwell, Berks, Eng.). Ind. chim. belge 23, 227-32(1958) Mar.

The large-scale utilization of solvent extraction for the purification and isolation of inorganic materials in atomic industry is surveyed. The separation of U and Pu and their fission products, of Th and U and their fission products, of Hf and Zr, of Nb and Ta, and of the rare earths and protactinium are discussed. (J.S.R.)

Chemical Processing of Nuclear Fuels, C.M. Nicholls, R. Spence. Nuclear Power 2, 12, Apr. 1957, p 152-4.

How early processes for separation of plutonium are now superseded by solvent extraction processes; for production of power rather than weapon material, moderate decontamination only is required; separation by phase contacting methods; avoidance of criticality; factors relating to processing cost.

316
PERFORMANCE OF A PLUTONIUM REFLUX SOLVENT EXTRACTION SYSTEM. B. F. Judson. May 1, 1957. p.296-302 of SYMPOSIUM ON THE REPROCESSING OF IRRADIATED FUELS HELD AT BRUSSELS, BELGIUM, MAY 20-5, 1957. 1165p. (TID-7534(Bks. 1-3); HW-49441A)

The operability of a product-refluxing flowsheet in a Pu solvent extraction system was demonstrated during a six-month semi-works program to develop a Pu purification process. The semi-works performance of the reflux flowsheet is described briefly and the advantages and disadvantages of employing product refluxing as a solvent extraction technique are summarized.

1323 HW-44744
General Electric Co. Hanford Atomic Products Operation, Richland, Wash.
ABSORPTION SPECTRA OF PLUTONIUM AND IMPURITY IONS IN NITRIC ACID SOLUTION. M. N. Myers. July 31, 1956. Decl. Sept. 16, 1957. 22p. Contract W-31-109-Eng-52. $0.50(OTS).

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GENERAL 3.03.14.03

6384 ✓ A/CONF.15/P/1587
THE DISSOCIATION OF ORGANIC COMPLEXES OF PLUTONIUM IN ORGANIC MEDIA. M. Taube (Inst. of Nuclear Research, Warsaw). 5p.

Possible mechanisms of dissociation of organic complexes of plutonium in organic media are considered, particularly in connection with the possible role of such dissociation in mechanisms of extraction from water to organic solvents. Studies of migration in electrical fields were used to investigate the dissociation of such organic complexes as those with iso-butyl-methyl-ketone, tributyl phosphate, tetrabutylammonium nitrate, furfural, cyclohexanol, and acetone, in such media as the above solvents themselves, together with chloroform, carbon tetrachloride, and benzene. Apart from free migration studies, particular attention was paid to the use of paper electrophoresis for studying dissociation of complexes. Suitable apparatus for these studies

VOLATILITY METHODS 3.03.15

See also, this section,

FUSED SALTS AND LIQUID METALS (3.02.11),

and,

under FISSION PRODUCT, POISON, AND RADIOISOTOPE REMOVAL,

FUSED SALTS AND LIQUID METALS (2.20),

and,

under PROCESS CHEMISTRY AND ENGINEERING,

SPECIAL PROCESSES--Zircex (6.17.24).

20007
METHOD OF SEPARATING PLUTONIUM. (to UK Atomic Energy Authority (U.S.A.)). British Patent 804,999. Nuclear Eng. 4, 324(1959) July-Aug.-Sept.

A vaporous mixture of the hexafluorides of uranium and plutonium is passed over a heated copper surface (100 to 455°C). Plutonium is removed through deposition on the copper.

22161
VOLATILE FLUORIDE PROCESS FOR SEPARATING PLUTONIUM FROM OTHER MATERIALS. F. H. Spedding and A. S. Newton (to U. S. Atomic Energy Commission). U. S. Patent 2,882,125. Apr. 14, 1959.

The separation of plutonium from uranium and/or fission products by formation of the higher fluorides of uranium and/or plutonium is described. Neutron-irradiated uranium metal is first converted to the hydride. This hydrided product is then treated with fluorine at about 315°C to form and volatilize UF_6 leaving plutonium behind. The plutonium may then be separated by reacting the residue with fluorine at about 500°C and collecting the volatile plutonium fluoride thus formed.

METHOD OF SEPARATING URANIUM, PLUTONIUM AND FISSION PRODUCTS BY BROMINATION AND DISTILLATION. A. H. Jaffey and G. T. Seaborg (to U. S. Atomic Energy Commission). U. S. Patent 2,865,704. Dec. 23, 1958.

The method for separation of plutonium from uranium and radioactive fission products obtained by neutron irradiation of uranium consists of reacting the irradiated material with either bromine, hydrogen bromide, aluminum bromide, or sulfur and bromine at an elevated temperature to form the bromides of all the elements, then recovering substantially pure plutonium bromide by distillation in combination with selective condensation at prescribed temperature and pressure.

14399
FLUORINE PROCESS FOR SEPARATION OF MATERIALS. G. T. Seaborg and H. S. Brown (to U. S. Atomic Energy Commission). U. S. Patent 2,833,617. May 6, 1958.

A process is described for separating plutonium from neutron-irradiated uranium, which consists of reacting the irradiated uranium mass with HF to form the tetrafluorides of U, Pu, and Np, and then reacting this mixture of tetrafluorides with fluorine at temperature between 140 and 315°C. This causes volatile hexafluorides of U and Np to form while at the temperature employed the Pu tetrafluoride is unaffected and remains as a residue.

RECOVERY OF PLUTONIUM 3.03

VOLATILITY METHODS 3.03.15

THE PYROCHEMICAL RECYCLING OF PLUTONIUM FROM SPENT TO FRESH URANIUM FUEL USING MOLTEN UF_4 AS TRANSFER MEDIUM. Archie G. Buyers. Oct. 1, 1955. Decl. Feb. 27, 1957. 31p. (NAA-SR-1435)

The pyrochemical transfer of Pu from spent to fresh U using molten UF_4 as transfer agent is demonstrated. The resultant U–Pu alloy contained fission products in small fractions of the amounts originally present in the spent fuel. Distribution of Pu and fission products throughout recycling is correlated with thermodynamic data. Application of this method for control of Pu concentration in a Na graphite reactor fuel cycle and fast breeder reactors is suggested.

7172 KAPL-M-PF6-1
Knolls Atomic Power Lab., Schenectady, N. Y.
PURIFICATION OF PLUTONIUM HEXAFLUORIDE. J. W. Codding, N. J. Hawkins, and W. W. Sabol. July 8, 1955. Decl. Feb. 7, 1956. 12p. Contract W-31-109-Eng-52. $3.30(ph OTS); $2.40(mf OTS).

The three-part distillation described represents a quick and convenient method for removing the commonly occurring gaseous impurities from PuF_6. In actual practice, the first distillation (from liquid nitrogen) can take place directly after the preparation, while the PuF_6 is still condensed in the coils of the reactor, since liquid nitrogen is used as the coil coolant. The distillation method as described provides for the rapid and essentially complete removal of HF, with a PuF_6 loss as low as desired. (auth)

11690 √ NAA-SR-Memo-729
North American Aviation, Inc., [Downey, Calif.].
THE HIGH TEMPERATURE SEPARATION OF PLUTONIUM AND FISSION PRODUCTS FROM IRRADIATED URANIUM CARBIDE. Charles R. F. Smith. July 10, 1953. Decl. Nov. 6, 1958. 18p. $3.30(ph), $2.40(mf) OTS.

A brief study was made on the high-temperature diffusion of tracer plutonium and fission products from uranium carbide. Uranium carbide powder was pressed into small pellets and sintered in vacuo at about 1900°C. The pellets were neutron irradiated in the Los Alamos Water Boiler and were placed inside graphite susceptors. They were then heated by induction in vacuo and in argon for periods of 40 to 200 minutes at average temperatures ranging from 2300 to 2625°C. Analyses of the material remaining after heating showed that up to 90% of the plutonium, 85% of the gross beta activity, and 28% of the gross gamma activity were removed. Up to 8% loss of uranium was also observed but this could be minimized by carefully controlling maximum firing temperature.