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ANALYTICAL CHEMISTRY DIVISION
ANNUAL PROGRESS REPORT
For Period Ending November 15, 1965

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ORNL-3889

Contract No. W-7405-eng-26

ANALYTICAL CHEMISTRY DIVISION
ANNUAL PROGRESS REPORT
For Period Ending November 15, 1965

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JANUARY 1966

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ORNL-3243	Period Ending December 31, 1961
ORNL-3397	Period Ending December 31, 1962
ORNL-3537	Period Ending November 15, 1963
ORNL-3750	Period Ending November 15, 1964

Preface

Analytical chemistry at ORNL is centralized within the Analytical Chemistry Division. As the content of this report attests, the activities of the Division are multifarious. This diversity is necessary to meet the general mission of the Division – to provide the analytical know-how required to solve the problems generated by the many and varied research programs at ORNL. Thus, the Division program encompasses (1) research in analytical chemistry, (2) development of new and improved methods of analysis, and (3) service analyses.

Research and development efforts within the Division characteristically are of an applied nature and in some cases are closely allied to specific ORNL research programs such as the MSRE and TRU projects. Analytical research on molten-salt media, particularly molten fluoride salts that are of primary concern in the reactor program at ORNL, is centered on electroanalytical technology, an area of competence within the Division, and on absorption spectrum measurements of the molten state. The goal of these studies is to provide automated continuous analyses for molten-salt reactors. The objectives of the effort in heavy-element analytical chemistry are first to provide required methods of analysis for the processing program and simultaneously to develop in a systematic manner the analytical chemistry of the heavy elements.

Fundamental investigations are also under way to accumulate the background information necessary for establishing the validity of new analytical principles and techniques. Typical examples are studied of (1) solid electrodes; (2) refined electroanalytical instrumentation; (3) applications of vapor and liquid chromatography, of mass spectrometry (in particular of the spark-source mass spectrograph), of x-ray and electron microscopy, and of NMR and infrared spectrometry; and (4) activation analysis with fast neutrons and ^{3}He ions. This year, a highlight of this type of effort has been the development of the new concept of the use of a radioisotopic light source in precision photometric analysis. The excellent precision inherent in this technique makes absorption photometry competitive with conventional techniques for the determination of macro amounts of materials.

A relatively new and expanding program within the Division is the bioanalytical effort. In cooperation with the Biology and Chemical Technology Divisions, the Analytical Chemistry Division is participating in the program to separate macromolecules, in particular the tRNA molecules. Of primary concern is the automation of the numerous and tedious analyses required to interpret those separations and to provide analyses of purity for the separated products. A complementary effort is in progress on the analysis of urine by use of the nucleotide analyzer developed by N. G. Anderson of the Biology Division.

These programs involve about one-third of the Division members; the other members are concerned with the analysis of samples submitted by ORNL research divisions. The diversity of the analyses requested continued to increase during this past year. It is this ever-increasing complexity of research at ORNL that requires the large centralized effort of the Analytical Chemistry Division.

Contents

PREFACE iii
SUMMARY xi

PART A. ANALYTICAL RESEARCH

1. ANALYTICAL INSTRUMENTATION 1
   1.1 Chronopotentiometer with Compensation for Extraneous Currents 1
   1.2 Evaluation of Technique of Chronopotentiometry with Compensation for Extraneous Currents 2
   1.3 Comparative Amperometric DC Polarography: A New High-Precision and High-Accuracy Method 3
   1.4 Controlled-Potential DC Polarograph-Voltammeter, ORNL Model Q-2792 4
   1.5 High-Sensitivity Coulometric Titrator, ORNL Model Q-2564 6
   1.6 Controlled-Potential Derivative DC Polarography: Application to Determination of Microgram Quantities of Uranium in a Tri-*n*-octylphosphine Oxide (TOPO) Extract 6
   1.7 Controlled-Potential Derivative DC Polarography and Controlled-Potential Coulometry as Microanalytical Techniques 7
   1.8 Utility of *i* vs *Q* Plots in Controlled-Potential Coulometry 8
   1.9 Derivative Chronopotentiometry 8
   1.10 Preparation of Chapter for Welcher's *Standard Methods of Chemical Analysis*, Vol III 9
   1.11 Differentiating Circuit for the Controlled-Potential Voltammeter 9
   1.12 Sample-Irradiation Assembly for ^{60}Co Source 9
   1.13 Computer Programs for Analysis of Polarograms and Chronopotentiograms 10
   1.14 A Square-Wave Polarograph 10
   1.15 Controlled-Potential Differential DC Polarography 10

2. ANALYTICAL STUDIES OF MOLTEN-SALT SYSTEMS 12
   2.1 Absorption Spectrophotometric Studies Related to Molten-Salt Systems 12
      2.1.a Absorption and Reflectance Spectra of Irradiated $LiF-BeF_2-ZrF_4-ThF_4-UF_4$ 12
      2.1.b Absorption Spectrum of Co^{2+} in LiF-NaF-KF 12
      2.1.c Absorption and Diffuse-Reflectance Spectra of Solid Materials 13

2.2 Voltammetry of Molten-Fluoride Salt Systems 14
2.2.a Reversible Deposition of Metals on Solid Electrodes by Voltammetry with Linearly Varying Potential 14

3. EFFECTS OF RADIATION ON ANALYTICAL METHODS 14

3.1 Gamma Radiolysis of Acid Chloride Solutions 14

3.2 Gas Chromatographic Analysis of Gamma-Irradiated Mixtures of Methanol and Hydrochloric Acid 15

3.3 High-Temperature Chemical Dosimetry 15

3.4 Modifications to the ^{60}Co Source Assembly 16

4. ANALYTICAL CHEMISTRY FOR REACTOR PROJECTS 17

4.1 Molten-Salt Reactor Experiment 17
4.1.a MSRE Sample Preparation Apparatus 17
4.1.b Remotely Servo-Controlled Corrosion-Resistant Pipetter, ORNL Model Q-1348B 18
4.1.c Remotely Operated Filter Photometer 18
4.1.d Controlled-Potential Coulometric Titration of Uranium(VI) in a Synthetic MSRE Fuel 18
4.1.e Polarographic Determination of Nickel in MSRE Fuel 18

4.2 Transuranium Element Processing Program 19

4.3 Medium-Power Reactor Experiment 19
4.3.a Analysis of the Alkali Metals 19

4.4 Nuclear Safety 22
4.4.a Nuclear Safety Program 22
4.4.b Spectrophotometric Study of the Stability of Iodine in Cyclohexane and in Benzene 22
4.4.c Gas Chromatographic Analysis of the Products of the Reaction of Iodine with Cyclohexane and with Benzene at 300°C and 20 atm 23

5. SPECIAL RESEARCH AND DEVELOPMENT PROBLEMS 25

5.1 Gas Chromatography 25
5.1.a Radio-Gas Chromatography of the Tritium-Water-Methane System 25
5.1.b Pyrolysis and Gas Chromatographic Determination of Styrofoam Dielectric Film 26

5.2 Studies with Solid Indicator Electrodes 27
5.2.a Glassy-Carbon Indicator Electrode (G.C.E.) 27
5.2.b Oxide Film Formation on the Pyrolytic-Graphite Electrode (P.G.E.) 27
5.2.c Differential Titrimetry with the Pyrolytic-Graphite Electrode (P.G.E.) and the Glassy-Carbon Electrode (G.C.E.) 27
5.2.d Voltammetry of the Iodine System 27
5.2.e Chronopotentiometry of the Iodine System 27

5.3 Studies with the Teflon Dropping-Mercury Electrode (D.M.E.) 28
5.3.a Polarography in Hydrofluoric Acid and Other Glass-Corroding Media 28
5.3.b Signal-to-Noise Ratios (S/N) of Glass and Teflon D.M.E. Capillaries 28

5.4 New and Modified Methods 29
5.4.a Determination of Titanium in Seawater 29
5.4.b Determination of Trace Metals in Niobium-Iron Alloy 29
5.4.c Spectrophotometric Determination of Uranium with Arsenazo III 29

5.4.d Determination of Nitrite in a Neutral Nitrate-Containing Solution of Potassium Iodide 29
5.4.e Spectrophotometric Determination of Nitrated 4-sec-Butyl-2-(α-methylbenzyl)phenol (BAMBP) 30
5.4.f Determination of Iodine and Fluorine in KIO_2F_2 30
5.4.g Determination of Uranium(VI) in Uranium Oxide–Thorium Oxide Fuels 30
5.4.h Spectrophotometric Thiocyanate Determination of Niobium in the Presence of Fluoride 30
5.4.i Spectrophotometric Heteropoly-Blue Determination of Silicon 31
5.4.j Determination of Nitrate by Controlled-Potential Coulometric Titration 31
5.4.k Analysis of Alkylbenzenesulfonate Detergents 31
5.4.l Determination of Oxidizing Power of Solutions of Hydrolyzed Xenon Hexafluoride 32
5.4.m Absorption Spectral Measurements 32
5.4.n Technetium Studies 32

6. ANALYTICAL BIOCHEMISTRY 32
6.1 Transfer Ribonucleic Acid (tRNA) Analytical Studies 32
6.2 Studies on the Phenylalanyl Transfer Ribonucleic Acid (tRNA) System 33
6.3 Gas Chromatography of Pyrolytic Products of Purines and Pyrimidines 34
6.4 Dissolution or Decomposition of Various Organic Materials 35

7. X-RAY AND SPECTROCHEMICAL ANALYSES 36
7.1 X-Ray and Spectrochemical Analyses (X-10) 36
7.1.a X-Ray Analysis 36
7.1.b Atomic Absorption 36
7.1.c Spectrographic Analysis of Radioactive Materials 37
7.1.d Flame Photometry 38
7.1.e Tissue Analysis 38
7.1.f Semiquantitative Spectrographic Analysis (Visual Interpolation) 38
7.2 X-Ray and Spectrochemical Analyses (Y-12) 39
7.2.a Determination of Lead, Bismuth, Cadmium, Tin, and Zinc in Airborne Dust Samples 39

8. MASS SPECTROMETRY 39
8.1 Spark-Source Mass Spectrometry 39
8.2 Transuranium Element (TRU) Mass Spectrometry 42
8.3 Surface Ionization Studies 42
8.4 Isotopic Comparison of Lead from Tektites and Contemporaneous Impact Craters 43

9. INFRARED AND NUCLEAR MAGNETIC RESONANCE SPECTROMETRY 44
9.1 Infrared Spectrometry 44
9.2 Nuclear Magnetic Resonance Spectrometry 44

10. OPTICAL AND ELECTRON MICROSCOPY 46
10.1 Electron Microscopy of Radioactive Materials 46
10.1a Electron Microscope Facility for Studying Radioactive Materials 46

10.2 Nuclear Safety Program 47

10.3 Methods Development for Electron Microscopy 47

10.4 Electron and Optical Microscopy and Electron Diffraction Research Assistance to Other Divisions 47

10.5 Personnel Education 48

11. NUCLEAR AND RADIOCHEMICAL ANALYSES 48

11.1 Nuclear Properties of Radionuclides 48
- 11.1.a ^{123}I 48
- 11.1.b ^{197}Hg 49
- 11.1.c Gamma-Ray Branchings in ^{237}U 50
- 11.1.d Half-Lives of Long-Lived Beta Emitters 50
- 11.1.e Nuclear Spectroscopy of Neutron-Deficient Hafnium and Rare-Earth Radionuclides 50
- 11.1.f Studies of Rare-Earth Alpha-Emitting Radionuclides 51

11.2 Cooperative Isotopes Program 51
- 11.2.a Reviews and Technical Assistance 51
- 11.2.b ^{131}I Products 52
- 11.2.c ^{14}C Products 52
- 11.2.d ^{144}Ce Product 52
- 11.2.e Trace High-Energy Gamma Radioactivity in Isotope Products 53
- 11.2.f ^{85}Rb(n,2n) Cross Section 53
- 11.2.g Release of Tritium by Tritiated Paint 53
- 11.2.h ^{210}Pb in Auto Exhaust Fumes and in Natural Materials 53

11.3 Measurement of Radioactivity 54
- 11.3.a Preparation by Electrodeposition of Mounts for 4π Beta Counting 54
- 11.3.b Detection of Natural Radionuclides by Well-Type Scintillation Counters 55
- 11.3.c ^{33}S(n,p) Cross Section 57
- 11.3.d Half-Life Measurements 57
- 11.3.e Experimental Study of Artifacts and Errors Encountered in Gamma-Ray Spectrometry for Radiochemistry and Activation Analysis 58
- 11.3.f Artifacts in Gamma-Ray Spectrometry 58

11.4 Radiochemical Studies 58
- 11.4.a N,N′,N″-Trihexylphosphoric Triamide (THPA) as an Extractant for Metal Ions 58
- 11.4.b Review of Organic Compounds that Contain Phosphorothioate, P=S, and Phosphorodithioate, P(S)SH, Groups as Separation and Analytical Reagents 59
- 11.4.c Rapid Separation of Technetium from Fission Products 59
- 11.4.d High-Voltage Paper Electrophoresis 59
- 11.4.e Separation of Trace Metals from Uranium by Chromatography on a Cellulose Column 59

11.5 Liquid Scintillators 60
- 11.5.a N,N′,N″-Trimethylborazine as a Liquid-Scintillator Solvent for Thermal-Neutron Detection 60
- 11.5.b Color-Quench Correction in Liquid-Scintillator Systems by Use of an Isolated Internal Standard 60

11.6 Application of Computers 60
11.6.a Electronic Gamma-Ray Spectral Resolution 60
11.6.b Evaluation of Computer Programs for Gamma-Ray Spectrometry in Activation Analysis 60
11.6.c Computer Program for Calculating Correlation Coefficients 61
11.6.d Use of Computers in Applied Activation Analysis 61
11.6.e Program NETAREA 62
11.6.f Computer Program for Calculating Neutron Flux 62

11.7 Application of Radioisotopes to Analytical Chemistry 63
11.7.a A New Concept in Precision Photometric Analysis: Use of a Radioisotopic Light Source 63
11.7.b Radio-Release Methods for the Evaluation of Atmospheric Pollution: Sulfur Dioxide 63
11.7.c Measurement of Wear Rates in Automotive Engines by Liquid-Scintillation Counting of ^{55}Fe 63
11.7.d Radiotracer Method for the Determination of Lead 64

11.8 Activation Analysis 64
11.8.a Studies of ^{3}He Nuclear Reactions 64
11.8.b Study of Precision and Accuracy in Neutron Activation Analysis 65
11.8.c Determination of ^{99}Tc by Neutron Activation Analysis 65
11.8.d Neutron Activation Analysis of Human Hair: Forensic Applications 67
11.8.e Applied Neutron Activation Analysis 67
11.8.f Gold Atoms Sputtered from Neutron-Irradiated Gold Foils 68
11.8.g Determination of Uranium in Seawater by Neutron Activation Analysis 68
11.8.h Neutron Activation Analysis of River Water 68
11.8.i Wool Analysis 68

11.9 14-Mev Neutron Generator 69
11.9.a Catalog of 14-Mev Neutron Reactions 69
11.9.b Studies of Neutron-Producing Targets 69
11.9.c Standardization of Neutron-Flux Measurements 69
11.9.d Activation of ^{64}Ni with 14-Mev Neutrons 69
11.9.e Activation Cross Sections for the Reactions of 14-Mev Neutrons with ^{99}Tc 70
11.9.f Monitoring of Leached Fuel Elements with a Neutron Generator 70

12. INORGANIC PREPARATIONS 71

13. ORGANIC PREPARATIONS 73

13.1 Compounds Prepared 73
13.1.a Quaternary Phosphonium Halides 73
13.1.b 2,6-Dioximinocyclohexanone 73
13.1.c ^{10}B Diborane 74
13.1.d Methyl Methanesulfonate 74
13.1.e Heptadecylamine 74
13.1.f Azo Dyes 74

PART B. SERVICE ANALYSES

14. QUALITY CONTROL 78

15. MASS SPECTROMETRIC ANALYSIS 80

16. SPECTROCHEMICAL ANALYSIS LABORATORY 80

17. PROCESS ANALYSES 81

17.1 High-Level Alpha Radiation Laboratory 81

17.2 General Analyses Laboratory 81

17.3 High-Radiation-Level Analytical Laboratory (HRLAL) 82

17.4 Radioisotopes-Radiochemistry Laboratory 84

17.5 High-Radiation-Level Analytical Facility (HRLAF) 85

PART C. *ORNL MASTER ANALYTICAL MANUAL*

18. *ORNL MASTER ANALYTICAL MANUAL* 87

18.1 Cumulative Indexes to the *ORNL Master Analytical Manual* 87

18.2 Maintenance of the *ORNL Master Analytical Manual* 87

PART D. ACTIVITIES RELATED TO EDUCATIONAL INSTITUTIONS

19. ACTIVITIES RELATED TO EDUCATIONAL INSTITUTIONS 89

19.1 Consultants 89

19.2 Research Participants 90

19.3 AEC Postdoctoral Program 90

19.4 Graduate Thesis Research Programs 90

19.4.a Educational Assistance – Ph.D. Program 90

19.4.b Oak Ridge Resident Graduate Program of the University of Tennessee 91

19.4.c Oak Ridge Graduate Fellowship Programs 91

19.5 Loanees 91

19.6 Summer Student Trainee Program 92

19.7 Alien Guests in Residency 92

PRESENTATION OF RESEARCH RESULTS 94

TRANSLATIONS 112

INDEXES 114

Key Word Index 117

Author Index 143

ORGANIZATION CHART 153

Summary

PART A. ANALYTICAL RESEARCH

1. Analytical Instrumentation

Two electroanalytical methods have been originated: chronopotentiometry compensated for extraneous currents and one-cell, comparative, amperometric dc polarography. The utility of chronopotentiometry for measuring concentrations below millimolar has been limited, because extraneous processes (double-layer charging and faradaic processes other than that of interest) prevent the electrode reaction being studied from advancing with 100% current efficiency relative to the selected input current. A *chronopotentiometer with compensation for extraneous currents* was designed to minimize this limitation. The potential of the working electrode in a second cell that does not contain the species of interest is potentiostatically forced to equal that of the working electrode in the first cell. The current in the second cell is thus a measure of the current due to extraneous processes; it is scaled (in the simplest case, to allow for unequal working electrode areas) and is added to the selected input current in the first cell so that the reaction of interest should proceed in the first cell at 100% current efficiency relative to the selected input current. An efficiency of 100% is realized only if the scaled current in the second cell is indeed equal to the sum of the extraneous currents in the first cell. Whether these currents are equal is not readily predictable for various kinds of extraneous processes. Also, the best method for obtaining the scale factor must be established experimentally. For evaluation of the validity of this compensation technique, four redox reaction–platinum electrode systems have been investigated. The reduction of $K_3Fe(CN)_6$ in 1 *F* KCl at platinum was the system chosen as representative of a reversible process in which the only important extraneous process is double-layer charging. Good compensation (excellent agreement with theory) was achieved down to 10^{-4} *M*. Further evaluations of compensation are planned for systems in which substantial extraneous faradaic processes and electrode-surface changes occur concurrently with the process of interest and also for redox–mercury electrode systems. *Comparative amperometric dc polarography* is a new polarographic method that has high precision and accuracy. The use of two cells in a differential mode is the reason given for the achievement of $S = 0.1\%$ and high accuracy in the Davis and Seaborn method of comparative polarography. The question was asked here as to whether the achievement of $S = 0.1\%$ by comparative polarography is due primarily to the close control of experimental conditions, which results in very reproducible cell currents, and to the use of scale expansion rather than that the currents in the two cells vary in the same compensating manner so that scale expansion (comparative measurement) can be used. If this hypothesis were correct, and if the average values of the D.M.E. current were measured at a fixed potential on the diffusion-controlled plateau of the wave, it should be possible to obtain high precision and accuracy by replacing the second, standard cell by its electrical analog. It has been shown that high precision and low error (0.1%) can indeed be achieved by this method. The substantial advantages are that only one D.M.E. having highly reproducible constant characteristics has to be operated for the duration of the measurements rather than two D.M.E.'s and that only one cell well isolated from the atmosphere is required.

Five functional improvements were made in the ORNL model Q-2792 controlled-potential dc polarograph-voltammeter, which is built with solid-state operational amplifiers and power supplies. Also, an apparatus that provides mechanically controlled

drop time (D.M.E. hammer) is being designed; there are four significant advantages in the use of hammering to obtain a short drop time for rapid regular and derivative dc polarography. The performance of this hammer is to be evaluated. The Q-2792 instrument with stationary electrodes performed satisfactorily. Three instruments were built, checked out, and delivered for use; a check-out and test procedure and operating instructions were prepared. Continued research in polarography and voltammetry with this instrument is planned.

Two more ORNL model Q-2564 high-sensitivity coulometric titrators were fabricated and checked out; a total of five are now in use. A method on this instrument was prepared for issue in the *ORNL Master Analytical Manual;* also, a paper that describes it was published.

A controlled-potential derivative dc polarographic method was developed for determining microgram quantities of uranium directly in the organic phase from a tri-*n*-octylphosphine oxide (TOPO) extraction; two papers on this work are in press. The highly efficient and selective TOPO solvent-extraction step prior to the polarographic finish separates the uranium from interferences and concentrates it, thus giving an overall increase in sensitivity. Controlled-potential electrolysis makes practical polarographic measurements in high-specific-resistance organic extracts and also greatly simplifies the design of micro cells. First-derivative dc polarograms were very reproducible. With a 0.5-ml cell, a 250-fold concentration advantage was demonstrated. Uranium(VI) in aqueous samples in concentrations as low as 5×10^{-9} *M* was measured satisfactorily.

Also, controlled-potential coulometric titration was investigated as a microanalytical technique. By applying suitable blank corrections and refining the procedure, reducible and oxidizable constituents at a level of 0.02 microequivalent were determined with the model Q-2564 titrator; $S = 1\%$. The methods used and results obtained in the determination of iron, uranium, and plutonium in the range from 0.02 to 7 microequivalents are described in a paper that is in press. The utility of plots of i vs Q in controlled-potential coulometric titration was explored with the model Q-2564 titrator; microgram quantities of plutonium and uranium were determined. The results are comparable in precision and accuracy with those obtained by the Q vs time technique. The i vs Q method has the advantages of timesaving and of warning of the occurrence of extraneous processes.

A communication is being prepared on the theory of time-derivative chronopotentiometry. Theoretically, with certain current-time programs, concentration or kinetic measurements can be obtained prior to the transition time. This possibility avoids measurements in the transition-time region, where considerable distortion can be caused by double-layer charging, and minimizes convective disturbances arising from electrolysis-induced density gradients. Also, according to theory, resolution of overlapped waves may be enhanced.

A chapter was written for the forthcoming edition of Welcher's *Standard Methods of Chemical Analysis* that presents a concise theoretical and practical treatment of the techniques that comprise coulometric methods.

An operational amplifier system was added to our controlled-potential voltammeter. It is now possible to record either regular or first time-derivative controlled-potential voltammograms with this instrument.

A machine was designed and constructed for inserting a sample into the shielding cask of a ^{60}Co source, exposing it for a reproducible time at a reproducible position, and then withdrawing it.

Computer programs (FORTRAN 63) were written to facilitate analysis of chronopotentiograms or dc polarograms made with simple, reversible, diffusion-controlled electrode processes. Programs PØLARGRM and CHRNØGRM are based on the Heyrovský-Ilkovič and the Karaoglanoff equations respectively.

A compact square-wave polarograph that incorporates solid-state integrated circuits, modular operational amplifiers, and X-Y recording was designed and is under construction at AERE, Harwell. The performance characteristics of this new polarograph are expected to exceed those of the older vacuum-tube model in most respects. The instrument will be tested by use of conventional and modified glass D.M.E.'s and Teflon D.M.E.'s.

A controlled-potential differential dc polarograph was designed and constructed. The seven principal design features are described. This instrument is being used in an extensive investigation (restricted to work with D.M.E.'s of natural drop times) of several differential polarographic techniques, each of which is defined: ΔE-differential, single- and dual-cell comparative, and subtractive. Equations for the ΔE-differential technique were derived and verified; results obtained were compared with

those obtained by derivative dc polarography. By both single- and dual-cell comparative dc polarography, results having both S and an error of 0.1% were obtained. Subtractive polarography was shown to provide increased sensitivity or selectivity or both; for example, it is possible to detect Cd^{2+} at 10^{-7} M concentration and to determine 10^{-4} M or more Cd^{2+} in oxygen-saturated solutions with the same accuracy and precision as are obtained for oxygen-free solutions.

The development of instruments necessary to the MSRE and TRU programs was continued (Chap. 4).

For the MSRE program, a considerable amount of instrument development was done. A paper that describes our apparatus for MSRE sample preparation was written. Final engineering drawings for the fabrication of our model Q-1348B remotely servo-controlled corrosion-resistant pipetter were completed; a method on the pipetter was issued to the *ORNL Master Analytical Manual.* A paper was written that describes our remotely operated filter photometer which was designed and built for MSRE analyses. Assistance was given in the design of an integrating readout circuit for a Keidel moisture cell for the indirect determination of oxygen in MSRE salts.

For the TRU program, the following instrument development work was done or is in progress. Design, fabrication, and testing of our model Q-1348C remotely servo-controlled corrosion-resistant pipetter for the TRU facility are complete. The S of successive deliveries of 0.1-ml volumes is ~0.2%; as much as 1 ml can be delivered per filling. Fabrication drawings and a paper were completed, and a method on the pipetter was issued to the *ORNL Master Analytical Manual.* Design, fabrication, and testing of our model 65-2590 remotely operated bottle decapper were completed. The decapper can be dismantled and removed from the TRU cell through the conveyor. Our model 64-3171 remotely operated filter photometer was designed and fabricated for use in the TRU facility; a paper that describes this instrument was written. Remotely operated solvent-extraction apparatus was designed for use in analyses done in the TRU facility. A predetermined volume of the lower phase is left in the expendable sample vial. Only one hand of the master-slave manipulator is required to perform any of the in-cell operations; both controlled agitation during the extraction and transfer of the upper phase are provided. A timed controller for regulating the speed of small motors was designed; six were fabricated for use with centrifuges. Two of our model Q-1728A velocity-servo potentiometric titrators were fabricated and checked out for use in analyses required for the Curium Program and in the TRU facility. Delivery units were fabricated that were designed for remote use in the HRLAF and in the HRLAL; a new delivery unit is being designed specifically for use in the TRU analytical cells. A remotely operated analytical balance for weighing TRU samples prior to their analysis is being designed. A flame spectrophotometer is being designed for use in the TRU facility.

2. Analytical Studies of Molten-Salt Systems

Spectral and electrochemical studies were continued in the Division's program on analytical studies of molten-salt systems. Reflectance spectra of the solid state and absorption spectra of the molten state were obtained on samples of irradiated $LiF\text{-}BeF_2\text{-}ZrF_4\text{-}ThF_4\text{-}UF_4$ in an attempt to determine the oxidation state of the uranium in the salt. Little evidence of the presence of trivalent uranium was observed.

In addition to the spectral studies on the molten state, considerable effort was expended on spectral investigations of the solid state. Transmission spectra of small crystals (0.1-mm diam) and diffuse-reflectance spectra were obtained to aid in elucidating the symmetry and coordination of light-absorbing species. Solidified UF_3 and UF_4 were examined over the wavelength range from 250 to 1200 mμ.

A paper was published on the reversible deposition of iron and nickel on silver and pyrolytic-graphite electrodes from molten-fluoride-salt media. The experimental data agreed satisfactorily with the published theory of Berzins and Delahay.

3. Effects of Radiation on Analytical Methods

The effects of gamma radiolysis of acid chloride solutions were investigated using the spectrophotometric determination of zirconium with arsenazo III as the test medium. At dosages below 1000 rads, no deleterious effect is observed. The role of methanol as an inhibitor of the formation of free chlorine during irradiation was confirmed; however, depletion of free acid was not arrested,

and the rate of acid loss was unaffected by the presence of methanol. Gas chromatographic analyses of the gaseous products of irradiation showed the presence of much methyl chloride and traces of methane and ethane.

Studies on a high-temperature chemical dosimeter using deaerated ferrous sulfate at 150, 200, and 250°C were made that showed G values slightly lower, about 10%, than the accepted value of 8.2 at 25°C.

Modifications were made to the ^{60}Co source used in the study of effects of radiation on analytical methods.

4. Analytical Chemistry for Reactor Projects

Analytical research and development is being done for the Molten-Salt Reactor Experiment, Transuranium Element Processing Program, and Medium-Power Reactor Experiment. Only brief summaries are presented of these investigations since more detailed and frequent reports are made in the appropriate progress reports from the programs. Included in this chapter are discussions of several projects that were done as part of the Nuclear Safety Program. Among these are investigations of the presence of nonelemental iodine compounds originating from fission-product-produced iodine. Gas chromatography and spectrophotometry are the methods primarily used in this work.

5. Special Research and Development Problems

A two-column gas chromatographic apparatus with a scintillation detector was assembled for the study of the exchange reaction between tritiated water and methane. Some problems with a commercial converter, which generates tritium gas from effluent tritiated compounds, are expected to be resolved by replacing the iron catalyst with a synthetic ammonia catalyst.

Relative concentrations of the pyrolysis products (methane, ethylene, benzene, toluene, ethylbenzene, and styrene) of the dielectric films from acceptable and defective dosimeters were determined by gas chromatography.

Electrochemical studies with solid electrodes of pyrolytic graphite and glassy carbon have shown that these materials are useful in voltammetry, differential titrimetry, and chronopotentiometry.

The fundamental polarographic studies of the use of Teflon D.M.E. capillaries have resulted in several publications, which are abstracted in this report. A study was begun of signal-to-noise ratios of Teflon D.M.E. capillaries relative to that of a typical reference glass D.M.E. capillary.

Titanium in seawater has been determined by a concentration step followed by a polarographic estimation; the limit of detection is 0.5 ppb.

A niobium-iron alloy was analyzed polarographically for trace quantities of Cu, Bi, Pb, and Zn. The selectivity of arsenazo III for the spectrophotometric determination of uranium was improved by use of cyclodiaminetetraacetic acid as masking agent and maleic acid as a buffer.

Dissolutions or decompositions of organic materials have been accomplished by means of various acid mixtures.

The oxidizing ability of hydrolyzed xenon hexafluoride has been studied. The absorption spectra of several solar-cell windows that were irradiated in the 86-in. cyclotron by NASA personnel were examined by means of a Cary model 14 spectrophotometer. Also, ribonuclease and Alipal-LO-436 were measured from their absorption spectra.

Miscellaneous special determinations included: nitrite in a neutral solution of potassium iodide and potassium nitrate, degree of nitration of BAMBP, iodine and fluorine in KIO_2F_2, and U^{6+} in UO_2-ThO_2 fuels. The interference of fluoride in the spectrophotometric determination of niobium has been obviated by use of aluminum. The problem of color instability in the spectrophotometric heteropoly-blue method for silicon was corrected. Nitrate has been determined by controlled-potential coulometric titration; alkybenzenesulfonate detergents were analyzed for Na^+, Cl^-, SO_4^{2-}, HCO_3^-, and water.

6. Analytical Biochemistry

Analytical assistance to the Biology and Chemical Technology Divisions was continued on the cooperative program to separate and purify transfer ribonucleic acids (tRNA) from *Escherichia coli* bacteria. Special attention was given to establishing the optimum conditions for the phenylalanine-phenylalanyl tRNA charging reaction in order to achieve quantitative determinations of the nucleic acid.

Pyrolytic products of purines and pyrimidines at 880°C were analyzed by gas chromatography to establish whether characteristic pyrochromatograms could be obtained for the different bases. Definite differences were observed between purines and pyrimidines by this analysis. Methyl cyanide was identified in significant concentrations from uracil, in addition to traces of ethyl cyanide. Cytosine also produced significant amounts of methyl cyanide; the other nitrogenous bases produced comparatively small amounts of methyl cyanide.

7. X-Ray and Spectrochemical Analyses

A program was written to perform the calculations for analyzing eight-component alloys by x-ray fluorescence. Analyses were very accurate when polished specimens were used. The lattice spacing of the lead stearate film on the mica crystal in the Philips electron probe analyzer was measured, thus making possible the use of wavelength tables to identify low-Z elements. The areal density of gold coatings on silicon radiation detectors was measured by x-ray fluorescence and by atomic absorption; results by the two methods agreed to within experimental errors. The limits of detection (μg/ml) for the atomic absorption method were: Au, 0.03; Cu, 0.01; Ag, 0.01. A method was developed for isolating traces of chromium from MSRE salt and determining the chromium spectrographically. Di-(2-ethylhexyl)phosphoric acid was studied as a reagent for separating curium from contaminant metals. It was found that when $MgNH_4PO_4$ is formed in tap water, it coprecipitates barium almost completely, especially if calcium is present; this coprecipitation is the basis of a technique for collecting barium from tap water for flame photometric determination. In the tissue-analysis program, a technique was worked out for homogenizing large samples of muscle, fat, or bone; contamination during the homogenization was negligible. A survey of universally applicable methods of emission spectrographic analysis was begun. For visual estimation of impurity concentrations, the Kroonen-Vader (Li_2CO_3-base) method appears to be more accurate than graphite-base methods. A solution technique was established for determining five elements on filter papers from air samples; the Paschen Direct Reader is used.

8. Mass Spectrometry

An MS-7 spark-source mass spectrograph has been used for survey and semiquantitative determinations of trace elements in a variety of materials. Nonconductors are mixed with a powdered metal matrix and are isostatically pressed in a mold of naphthalene, which is later removed with hot acetone. The laboratory cooperated in the analysis of a standard copper sample.

The TRU mass spectrometry laboratory has now made isotopic determinations on the transplutonium elements up to and including californium. In collaboration with A. Chetham-Strode of the Chemistry Division, the half-lives of the californium isotopes were checked by mass spectrometric analyses. A total of 482 "hot" samples were run during the year.

Investigation of a real, but relatively small, isotope discrimination effect observed in uranium samples in the nanogram range has shown that the effect seems not to be temperature dependent. At temperatures above ~2200°K the production of uranium ions from a molecular beam of UF_4 impinging on a filament follows the Saha-Langmuir equation. Below that temperature there are marked deviations. A study of the lower-temperature region is contemplated with control of the amounts of "impurities" admitted to the ionization region.

The isotopic composition of lead from tektites and in material from impact craters that appear to be contemporaneous has been studied. The data have not been evaluated thoroughly, but the similarity between lead from Ivory Coast tektites and from Lake Bosumtwi phyllite suggests strongly that the tektites were formed from terrestrial material during the formation of the crater.

9. Infrared and Nuclear Magnetic Resonance Spectrometry

Nuclear magnetic resonance (NMR) spectrometry was made available by the Division as a service function. The NMR spectra of a wide variety of organic compounds were obtained and were recorded for reference. A considerable and diversified number of organic and inorganic compounds were also examined by infrared spectrometry. These techniques are used primarily to identify organic compounds and to ascertain relative degrees of

purity. Quantitative analyses are done when feasible, particularly for functional groups, for oil in waters from various sources, and for water in engine and lubrication oils.

10. Optical and Electron Microscopy

Examination of radioactive materials by electron microscopy has been in progress since December 1964 at the new microscopy facility in Building 3019. Assistance to research groups concerned with the Nuclear Safety Program continued. A variety of methods were developed for replicating powders, extracting inclusions from metals, and observing particulates collected on cellulose triacetate filters. Optical and electron microscopy and electron diffraction services were provided to other divisions when knowledge of the internal and external ultrastructure or microstructure of a wide variety of materials was important. Members of the group attended special classes in ultramicrotomy and ultrafiltration methods.

11. Nuclear and Radiochemical Analyses

Nuclear parameters associated with ^{123}I and ^{197}Hg, two radionuclides of medical interest, were measured, as well as gamma branchings in ^{237}U; half-lives of ^{36}Cl and ^{10}Be were redetermined. Nuclear spectroscopy of neutron-deficient rare earths was continued, as was the search for rare-earth alpha emitters.

A formalized cooperative program between the Isotopes and Analytical Chemistry Divisions has resulted in improved nuclear data and analytical procedures. Among the analytical methods studied were those for ^{131}I, ^{14}C, and ^{144}Ce; additional products were examined for high-energy gamma rays. Also, ^{84}Rb was identified in ^{86}Rb products. At elevated temperatures, tritium was shown to be released from tritiated paints chiefly as tritiated water. The problem of ^{210}Pb in automobile exhaust fumes and natural materials was also investigated.

Attempts to prepare 4π beta mounts of ^{99}Tc by electrodeposition were not successful quantitatively. Prediction and experiment were compared for detection of natural radionuclides in a standard well-type scintillation counter. The $^{33}S(n,p)^{33}P$ cross section was found to be 2.3 mb in a region of well-thermalized neutrons. Half-life values for ^{137}Cs, ^{153}Sm, ^{77}As, ^{198}Au, ^{133}I, and ^{33}P were determined. Artifacts and errors in gamma-ray spectrometry were studied, but a search for an annihilation artifact in ^{86}Rb gave negative results.

The properties of N,N′,N″-trihexylphosphoric triamide as an extractant for metal ions were studied, and a review was prepared which describes the analytical potential of organic compounds that contain P=S and P(S)SH groups. A method for rapid separation of technetium from fission products was devised.

High-voltage paper electrophoresis was investigated for use in fast chemical separations; a paper on the separation of trace metals from uranium by cellulose chromatography is to be published. N,N′,N″-Trimethylborazine was used as a liquid-scintillator solvent for thermal-neutron detection. A new technique, the isolated internal standard, was developed for use in color-quench correction in liquid-scintillation counting.

A definitive study of electronic gamma-ray resolution was completed, and the standard tape library was shown to function satisfactorily. In another study, three computer programs for resolving gamma-ray spectra were evaluated. A computer program was written to calculate correlation coefficients for all pairs of elements in a set of measurements. Computer techniques were applied to activation analysis, and a program LIMIT was written to calculate detection limits. Another program ABSNAA was written for use in absolute activation analysis. A program NETAREA calculates photopeak areas of gamma-ray spectra, and a new program to calculate neutron flux from flux-monitor data was written.

Extremely accurate and precise measurements are possible by use of a unique photometric method of analysis that employs a radioisotopic light source; the method provides a new physical basis of measurement for absorption studies. A radio-release method for determination of sulfur dioxide in the atmosphere was proved, and the wear rates in automobile engines were measured by use of radioactive isotopes. A method for determining ^{32}P in water was devised, and a radiotracer method for determination of lead was developed.

Research into the use of ^{3}He ions for activation analysis was continued, and excitation functions were obtained for a number of observed reactions. A study of precision and accuracy in neutron activation analysis was made. Contamination in

sample-mount material has been the limiting factor in achieving high sensitivity for ^{99}Tc by neutron activation. The forensic application of neutron activation analysis of human hair was carefully evaluated; an attempt to use the technique in the investigation of an actual crime gave nondefinitive results. Neutron activation analysis was applied to the determination of trace elements in biological, metallurgical, radiochemical, and other samples; for example, gold atoms sputtered from neutron-irradiated gold foils were determined by activation analysis. Methods for determining uranium in seawater are being studied; also, copper, zinc, nickel, and chromium were determined in river water at concentrations below 50 ppb. Use of neutron activation analysis for wool was studied.

Work continued on the cataloging of 14-Mev neutron reactions; the evaluation of neutron-producing targets was completed. A method for standardization of neutron-generator flux measurements is under development. Study of 14-Mev neutron reactions on ^{64}Ni has given information on previously unpublished nuclear species and will result in improvement of other nuclear data. Activation cross sections for 14-Mev neutron reactions on ^{99}Tc have been determined. A useful application of the neutron generator is the monitoring of leached fuel elements for uranium; the number of delayed neutrons from ^{235}U fission is correlated with the amount of uranium present. Another important application of the generator has been the determination of oxygen in alkali metals.

12. Inorganic Preparations

Programs that continued from preceding years were the preparation of fused salts for the Metals and Ceramics Division and for the Chemistry Division and the preparation of high-purity KCl for the Solid State Division. A number of alkali-metal oxides were prepared: KO_2 and RbO_2 for the Solid State Division and Na_2O and K_2O for the Chemistry Division. For the latter division, $LiOBr \cdot 2H_2O$ and K_2ReCl_6 were also prepared. A number of rare-earth-metal nitrides, phosphides, and sulfides were made for the Solid State Division; three different forms of Ho_2S_3 were obtained. For the Isotopes Division some uranium trihalides were made; one of these, $^{235}UCl_3$, was incorporated as a solid solution in $LaCl_3$. Some further work was done for the Physics Division to substitute ^{57}Fe for Al in alums.

13. Organic Preparations

Some special organic compounds, most of which are not available commercially, were synthesized on request from other research divisions. These compounds were needed by staff members as reagents accessory to fundamental and applied research; they were: benzyltributylphosphonium bromide, benzyltributylphosphonium chloride, 2,6-dioximinocyclohexanone, ^{10}B diborane, methyl methanesulfonate, heptadecylamine, Calcichrome, and "chlorophosphonazo III."

PART B. SERVICE ANALYSES

14. Quality Control

The Statistical Quality Control Program decreased during the year; some 4000 control tests were made. The quality of the work remained about the same. The quarterly quality-control calculations were programmed for the computer.

15. Mass Spectrometric Analysis

The X-10 and Y-12 stores departments now stock mass spectrometrically analyzed cylinders of helium, nitrogen, and argon. This arrangement evens out the sample load on the instruments used for analyzing gases. Thirty-five samples of enriched isotopes were analyzed for the United Kingdom AERE installation at Harwell. Strontium samples were analyzed that had been collected by placing the standard-sample filaments in calutron receivers along the *Z* direction of the ion beam. New mass spectrometric data indicate that the neutron cross sections of ^{36}Cl and ^{40}K may be much lower than the accepted values.

16. Spectrochemical Analysis Laboratory

The sample load on the Spectrochemical Analysis Laboratory increased 50% over the previous year. The Paschen Direct Reader was applied to several new sample types, particularly new types of alloys.

17. Process Analyses

The Process Analyses Laboratories performed 156,175 analyses, an 8% increase over the past year. The number of persons associated with the laboratories decreased 3%. The Materials Testing Laboratory was consolidated with the General Analyses Laboratory. The new High-Level Analytical Laboratory was put into service, and the Radioisotopes-Radiochemistry Laboratory was moved to Building 3019. Brief statements of the new developments in each laboratory follow.

The High-Level Alpha Radiation Laboratory analyzed samples from the sol-gel process; O/U ratio, nitrate, and carbon were determined. Plutonium was determined in a variety of samples from studies of the volatility of plutonium fluoride. Americium alpha radioactivity was determined in samples that contained high levels of curium radioactivity.

The General Analyses Laboratory continued to work on an improved vacuum-fusion analyzer. A Leco inert-gas-fusion apparatus was used to determine O_2 in U_3O_8, Al_2O_3, ThO_2, and Nb_2O_5. Pyrolytic tungsten was analyzed for O_2, C, and F at <10-ppm levels. Several new computer programs were completed.

The High-Radiation-Level Analytical Laboratory (HRLAL) was placed in service in December 1964; the first sample analyzed was a 50-curie ^{147}Pm source. Preparations for analyzing samples of MSRE fuel salt were completed, and samples were received and analyzed during the precritical and zero-power experiments. Dissolution of unusual materials and the analysis of samples by gas chromatography were continued in the HRLAL. The analysis of samples from the Curium Program was continued in Building 3019.

In the Radioisotopes-Radiochemistry Laboratory, the number of analyses performed during the year increased 35% without increase in the number of personnel. The increase was achieved by assaying a large number of samples by gamma-ray spectrometry and computer codes. Reproducible decontamination methods were devised for evaluating various structural and coating materials.

The High-Radiation-Level Analytical Facility (HRLAF) continued to analyze materials having high levels of alpha radioactivity. This work necessitated improved cell-containment and waste-handling techniques and stringent cell-entry requirements. Radiation damage to zinc bromide viewing windows in the HRLAF work cells has increased.

The laboratories in the Transuranium Facility were completed. Equipment is being moved in and checked. These laboratories will be ready to receive radioactive samples by December 1965. The cell block in this facility will be ready for operating in March 1966.

The conceptual design of a new alpha laboratory has been started. This laboratory will be known as the High-Level Alpha Laboratory.

PART C. ORNL MASTER ANALYTICAL MANUAL

18. ORNL Master Analytical Manual

The cumulative indexes to the *ORNL Master Analytical Manual* were updated to make the indexes cumulative for the years 1953 through 1964. The updated indexes are available from the Clearinghouse for Federal Scientific and Technical Information; they are designated TID-7015 (Indexes), Revision 2.

The seventh supplement to the reprinted form of the *Manual* (TID-7015, Suppl. 7) was issued; it includes 15 new methods and revisions to 19 methods. Six additional methods, not included in Suppl. 7, were written for record only; seven obsolete methods were discontinued. The Table of Contents to the *Manual* was revised.

The need for new methods for the Division was determined, and the writing of methods was planned accordingly.

Part A. Analytical Research

Research in a variety of fields of analytical chemistry was conducted during the past year for the Reactor, Physical Research, and Isotopes Development Divisions of the Atomic Energy Commission. Research was also conducted for the various research divisions of the Oak Ridge National Laboratory on specific problems associated with the programs of the Laboratory.

The progress in these investigations is presented in the following sections.

1. Analytical Instrumentation

M. T. Kelley
D. J. Fisher

1.1 CHRONOPOTENTIOMETER WITH COMPENSATION FOR EXTRANEOUS CURRENTS

W. D. Shults
F. E. Haga[1]
T. R. Mueller
H. C. Jones

The analytical utility of chronopotentiometry has always been limited in practice by extraneous processes (e.g., double-layer charging) that prevent the electrode reaction of interest from advancing with 100% current efficiency. A chronopotentiometer was built that is intended to minimize this limitation. The instrument requires the use of two cells: one contains the electroactive species of interest in an appropriate supporting-electrolyte solution; the second contains a solution identical with the other except that it is free of the electroactive species of interest. A selected input current is established in the first cell, and the potential of the working vs the reference electrode in this cell is monitored. This potential also is used as the command signal for cell 2, which is operated in a controlled-potential configuration. Thus, the potential of the controlled electrode (vs the reference) in cell 2 is always equal to the potential of the working electrode (vs the reference) in cell 1. Hence, the current in cell 2 (which contains no electroactive species of interest) is an instantaneous measure of the current due to extraneous processes that occur during the chronopotentiometric experiment. This cell-2 current is scaled and is added to the selected input current in cell 1 to accomplish compensation. The current in cell 1 is then the sum of two currents – the selected input current plus the scaled compensation current – but the current available for advancing the reaction of interest is equal to the selected input current. Therefore, effectively, this reaction proceeds with 100% current efficiency relative to the selected input current. A Scientific Communication that describes the conceptual design of this instrument and presents some preliminary performance data was published.[2] The technique of extraneous-current compensation is being evaluated

[1]Present address: Southern Railway Co., Atlanta, Ga.

[2]W. D. Shults, F. E. Haga, T. R. Mueller, and H. C. Jones, "Chronopotentiometer with Compensation for Extraneous Currents," *Anal. Chem.* 37, 1415 (1965).

further (Sect. 1.2). The addition of other circuits is planned to provide programmed selected input currents and to compute the first and second time derivatives of the compensated chronopotentiograms (Sect. 1.9).

1.2 EVALUATION OF TECHNIQUE OF CHRONOPOTENTIOMETRY WITH COMPENSATION FOR EXTRANEOUS CURRENTS

D. G. Peters[3] T. R. Mueller
W. D. Shults

Extraneous electrode processes that occur during chronopotentiometry of an electroactive species are the charging of the double layer and the faradaic reactions not associated with the process of interest. The extent to which each type of interference can be eliminated by the extraneous-currents-compensation technique described previously[2] (Sect. 1.1) is not readily predictable. Consequently, a number of systems must be studied to assess adequately the validity of the compensation technique.

The four systems that have been studied are

$$K_3Fe(CN)_6 \text{ in } 1\ F\ KCl\ ,$$

$$Na_2C_2O_4 \text{ in } 1\ F\ H_2SO_4\ ,$$

$$Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O \text{ in } 1\ F\ H_2SO_4\ ,$$

$$Fe(ClO_4)_3 \text{ in } 1\ F\ HClO_4\ .$$

These systems were chosen for studies at platinum electrodes, because the behavior of each illustrates a different requirement for compensation. The reduction of ferricyanide represents a reversible process in which the only important extraneous process is double-layer charging. The highly irreversible oxidation of oxalic acid is a severe test of the practicality of the technique in that the electrodes are alternately oxidized and reduced electrochemically with the attendant effect of a continuously changing double-layer capacity in addition to the extraneous faradaic processes of electrode oxidation and reduction. Ferrous iron reduces platinum oxide, and ferric iron is believed to oxidize a platinum electrode. The oxide layer or reduced-metal surface produced by this chemical action on the electrode in the cell that contains iron may thus differ from the surface of the electrode in the cell that contains the solution of the supporting electrolyte only.

Much of the investigation to date has been concerned with determining the best method for obtaining and maintaining the balance (i.e., the scale factor required to feed back the proper compensation current) between the two cells. It was found – not unexpectedly – that a balance setting which compensates for differences in *geometrical areas* of the electrodes in the two cells is adequate when the concentration of electroactive species is high, since under these conditions double-layer charging is accomplished with a relatively insignificant fraction of the total cell current. At very low concentrations, however, the wave shape is determined primarily by double-layer charging, and the balance must be achieved on the basis of the *micro areas* of the two electrodes.

In this study, concentrations of the electroactive species of interest ranged from 2×10^{-5} to 1×10^{-2} M. For a species having a diffusion coefficient of 0.7×10^{-5} cm^2/sec and undergoing a one-electron process, 10^{-4} M appears to be the lowest concentration practical for analytical purposes when a platinum electrode is used.

Results of this investigation show that excellent agreement between experimental data and theory can be obtained by this extraneous-currents-compensation technique when the important extraneous process is double-layer charging and when the electrode is not subjected to uncontrolled conditions that alter its surface characteristics. The study of systems in which electrode-surface changes occur was not exhaustive, but the preliminary results are sufficiently encouraging to warrant further investigation.

In addition to studies with platinum electrodes, it is planned to continue the evaluation of this technique with other electrodes (principally mercury) and with other systems.

[3]Research Participant, summer 1965; Assistant Professor of Chemistry, Department of Chemistry, Indiana University, Bloomington.

1.3 COMPARATIVE AMPEROMETRIC DC POLAROGRAPHY: A NEW HIGH-PRECISION AND HIGH-ACCURACY METHOD

D. J. Fisher W. D. Shults
Dietrich Thiele[4] W. L. Belew

The precision ($S = 0.1\%$) and accuracy of the Davis and Seaborn method of comparative polarography are high. The reason given[5,6] is the use of two polarographic cells in a differential mode. It is important to realize that it is necessary[5] to control very closely and to match experimental conditions, such as the internal diameters of the cells, D.M.E. potentials, and temperature, and to maintain the dissolved-oxygen concentrations equal and as low as possible. Matched, balanced, and mechanically synchronized D.M.E.'s are used; thus, the areas of the electrodes are equal.[5] However, D.M.E.'s having unequal flow rates and unsynchronized drop times can be used for differential dc polarography if current-averaging and electrical-gain adjustment are provided.[7] Residual currents in the two cells, which are small relative to the i_d's for millimolar concentrations of the species of interest, are supposed to be equal and therefore cancel. This method of comparative polarography is a scale-expansion technique and is analogous in principle to other methods, including differential spectrophotometry and high-precision weighing-titration. A closely related method is null-point differential polarographic titration.[8]

In the method of absolute polarography[9] the cell conditions and D.M.E. characteristics are held constant. However, even when the experimental conditions are well controlled, high precision and accuracy are not achieved, because the total cell current is measured per se. The method of comparative polarography also requires that the D.M.E. characteristics be constant for the duration of the series of measurements. Therefore, it must be that the i_d at the D.M.E. in the standard cell is, in fact, highly reproducible for each standard concentration. The question was asked here as to whether the achievement of $S = 0.1\%$ by comparative polarography is due primarily to the close control of experimental conditions, which results in a very reproducible current for the standard cell, and to the use of scale expansion.

It seemed more gravid to consider that the experimental conditions required for comparative polarography are such that the average current of neither cell (at each concentration and potential) varies significantly, rather than that the average currents of both cells vary in the same compensating manner. The hypothesis is that maintenance of highly reproducible currents at both D.M.E.'s rather than compensation by means of the second cell is the most significant reason for the high precision and accuracy of two-cell comparative polarography. If this hypothesis were correct, and if the average values of the current at the D.M.E. were measured at a fixed potential on the diffusion-controlled plateau, it should be possible to replace the standard cell by its electrical analog (a bucking current corresponding to $\bar{i}_d + \bar{i}_r$ for the standard cell). Also the usual mechanical synchronization of the D.M.E.'s should not be required if average values of D.M.E. currents are measured[7] with a highly stable polarograph. For comparative polarography, mechanical synchronization is also required, because it results in a constant drop time that is independent of potential and thus avoids the small error due to unequal variations of drop time with potential for two otherwise-matched D.M.E.'s. If measurements are made at a fixed potential, this result of mechanical synchronization is irrelevant. The average current at the D.M.E. is measured by the use of an ORNL parallel-T, low-pass filter assembly.[10] It is apparent that if high accuracy and precision could be achieved by replacing the standard cell by its analog and by measuring net average current at a fixed potential, substantial advantages would result. Only one D.M.E., rather than two, having highly reproducible constant characteristics would have to be operated for the duration of the measurements.

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[4] Alien Guest from Kernforschungszentrum Karlsruhe, Institut für Heisse Chemie, Karlsruhe, Germany.

[5] H. I. Shalgosky and J. Watling, "High Precision Comparative Polarography," *Anal. Chim. Acta* **26**, 66 (1962).

[6] H. M. Davis, "Komparative Polarographie hoher Präzision," *Chem. Ingr.-Tech.* **37**, 715 (1965).

[7] M. T. Kelley and D. J. Fisher, "Instrumental Methods of Derivative Polarography," *Anal. Chem.* **30**, 929 (1958).

[8] E. T. Verdier, "A New Chemical Titration," *S. African J. Sci.* **52**, 203 (1956).

[9] I. M. Kolthoff and J. J. Lingane, *Polarography*, 2d ed., pp. 381–85, Interscience, New York, 1952.

[10] M. T. Kelley and D. J. Fisher, "Unique Polarographic Damping Circuit for Selective Elimination of Current Fluctuations Due to Dropping Mercury Electrode," *Anal. Chem.* **28**, 1130 (1956).

Also, only one cell isolated well from the atmosphere would be required.

A method – comparative amperometric dc polarography – based on the above hypothesis was developed; it is as follows. Only one polarographic cell is used; it is thermostated at 25 ± 0.05°C and is provided with good sparging and with a tight seal to the atmosphere (the sparge gas is vented through a water seal). Sporadic mechanical shocks and vibration are avoided. An accurately standardized solution, about millimolar (C_1) in the species of interest, is introduced into the cell and is sparged. The cell voltage is set at a constant value on the diffusion-controlled plateau of the wave. With the current range required for recording ($\bar{i}_d + \bar{i}_r$) and with the parallel-T, low-pass filter switched in, a dc current (initial-current compensation) is applied to bring the net filtered output of the current amplifier to about zero. Then, a more sensitive (about tenfold) current range is switched in, and the net filtered current is adjusted to be zero (the recorder-pen position is brought to the point corresponding to zero cell current). The amount of compensating current required need not be measured; it is maintained constant at this nulling value throughout the following steps. The first standard solution is replaced by a second accurately standardized solution whose concentration (C_2) is greater than C_1 by an accurate increment of ~0.00002 to 0.0001 *M*. After sparging, the average value of the corresponding change in current, $\Delta\bar{i}_{ds}$, is measured on the more sensitive current range. The second standard solution is then replaced by the sample solution (same supporting electrolyte) whose concentration (C_x) is to be measured; as in comparative polarography, C_x differs from C_1 and from C_2 by an amount similar to ($C_2 - C_1$) and is usually greater than C_1. After sparging, the value for $\Delta\bar{i}_{dx}$, that is, ($\bar{i}_d$ for C_x) – ($\bar{i}_d$ for C_1), is measured on the more sensitive current range. Then,

$$C_x = C_1 + \left(\frac{\Delta\bar{i}_{dx}}{\Delta\bar{i}_{ds}}\right)(C_2 - C_1) .$$

The precision achievable by this method was estimated. The ORNL model Q-1988-FES dc controlled-potential polarograph[11] and a hammered D.M.E. were used. The cell contained 10 ml of test solution. A significant concentration change resulted from evaporation during sparging, even though the sparge gas was presaturated with water. This concentration change is significant relative to $C_2 - C_1$ but is negligible in terms of the increase in the total wave height, which would be nonobservable if measured on a current range that would be needed for that purpose. This evaporation error was reduced by controlling the rate and duration of the sparge; a more satisfactory solution to this problem is to also use a larger (about threefold) volume of solution. The loudspeaker type D.M.E.-hammering device used to obtain a 0.5-sec drop time with a low mercury flow rate (~3 mg/sec) was not entirely satisfactory, because the cell current was not independent of the hammering intensity. A small amount of convection must have been introduced due to the inadequate design of this hammering device. In spite of less than adequate experimental conditions, the relative standard deviation of the determination of 2×10^{-3} and of 4×10^{-3} *M* Cd^{2+} in 1 *M* KCl–0.001% thymol was 0.2%. The results indicate that, with better control of experimental conditions, it might be possible to attain $S = 0.1\%$.

The precision and accuracy were studied further (Sect. 1.15). A larger volume (~30 ml) of test solution and a 1-sec, vertical-orifice, Smoler D.M.E. having a natural drop time were used. The results show that high precision and accuracy can indeed be achieved by one-cell, comparative, amperometric dc polarography; both the relative error and *S* (~0.1%) were the same as those obtained concurrently by two-cell comparative dc polarography.

1.4 CONTROLLED-POTENTIAL DC POLAROGRAPH-VOLTAMMETER, ORNL MODEL Q-2792

W. L. Belew T. R. Mueller
H. C. Jones

The following functional changes were made in the ORNL model Q-2792 controlled-potential dc polarograph-voltammeter:[12]

[11] D. J. Fisher, W. L. Belew, and M. T. Kelley, "A Controlled-Potential and Derivative DC Polarograph for Rapid Regular and First and Second Derivative DC Polarography: Circuits, Theory and Performance Characteristics," to be submitted for publication.

[12] D. J. Fisher, M. T. Kelley, H. C. Jones, R. W. Stelzner, and W. L. Belew, "Controlled-Potential DC Polarograph-Voltammeter Design and Evaluation," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1964,* ORNL-3750, p. 3.

1. A provision for using the potential-scan generator to produce a linear time base. With this provision, current-time curves at constant controlled electrode potential can be plotted on the X-Y recorder.
2. An automatic cutoff circuit for linear-potential-sweep applications that terminates the sweep at a preselected potential. This circuit has two related advantages; its automatic feature frees the operator for other work during slow scans, and, during both slow and fast scans, the cutoff prevents excursions of potential that could damage the working electrode.
3. The addition of a potential scan that cycles between selectable initial and switching potentials from +3 to −3v. The recorder pen is coupled to this circuit in such a way that it writes either continuously or only during one half cycle.
4. A simple switching arrangement that permits all signals, required for effecting the check-out and testing procedures, to be monitored with the X-Y recorder.
5. A maximum scan rate of 10 v/min. This scan rate was found to be the maximum compatible with the recorder used with the instrument. Accordingly, the 20-v/min scan rate originally contemplated[12] was not included in the instrument.

When the rapid scan rates and fast derivative circuits available on the model Q-2792 polarograph-voltammeter are used in conjunction with a D.M.E., a drop time of 1 sec or less is advantageous. Rapid drop times (1 sec or less) can be obtained in either of two ways: by use of a Smoler vertical-orifice D.M.E. or by detaching the mercury drops (without introducing deleterious convection) before they would normally fall from the capillary. The Smoler vertical-orifice D.M.E. is satisfactory for obtaining drop times of ~1 sec. Capillaries of this type can be fabricated that give satisfactory results with drop times as short as 0.5 sec. However, it may be necessary to select carefully from several capillaries in order to find one having a 0.5-sec drop time and a flow rate of mercury not greater than ~4 mg/sec. When the 0.5-sec drop time is obtained mechanically by detaching the drop from the capillary at controlled intervals, the problem of capillary selection is circumvented. When a controlled drop time is used, the following advantages are gained: constant drop time vs potential, possibility of varying the flow rate of mercury independently of the drop time (attainment of a low mercury flow rate at short drop times is thus greatly simplified), more precise tuning of the parallel-T average-current filters to remove drop oscillations, and greater tolerance to sporadic mechanical shocks and vibration. Thus, the most practical way to obtain a short drop time for rapid-scan dc polarography is with an apparatus that mechanically controls the drop time, that is, a "drop hammer."

Several types of drop hammers were built and evaluated. Also, one commercial apparatus having this means for obtaining rapid drop times was purchased, evaluated, and found to be unsatisfactory. As a result of these evaluations, another drop hammer is being designed and fabricated. It will be evaluated for use with the Q-2792 polarograph-voltammeter in the following way. The achievable sensitivity will be determined from the signal-to-noise ratio[13] for polarograms of micromolar solutions. This sensitivity will be compared with that obtained previously for D.M.E.'s having natural drop times; these will include both Smoler vertical-orifice capillaries and 2-to-5-sec Sargent horizontal-orifice capillaries. To determine the shortest drop time usable with the drop hammer without disturbance of the diffusion layer at the mercury-drop surface by introducing stirring, polarographic diffusion coefficients will be measured for several systems over a wide range of drop times.

The Q-2792 polarograph-voltammeter was checked with stationary electrodes at potential-sweep rates up to 10 v/min. Hanging-mercury-drop and smooth platinized platinum electrodes were used. Areas of the mercury electrodes varied from ~0.05 to 0.07 cm^2; those of the platinum electrodes from ~0.1 to 3 cm^2. A 3-cm^2 heavily platinized electrode (large double-layer capacity) was also used successfully. Results consistent with theory were obtained for all modes of instrument operation except for the most rapid sweep rate, at which nonlinear recorder damping distorted the second-derivative recordings.

Three of the Q-2792 polarograph-voltammeters were fabricated by the Instrument Department of the Instrumentation and Controls Division. They

[13] D. J. Fisher, W. L. Belew, and M. T. Kelley, "Recent Developments in DC Polarography," pp. 89–134 in *Polarography 1964* (ed. by G. J. Hills), Macmillan, London (in press).

were checked out and delivered to the General Analyses Laboratory, General Hot Analyses Laboratory, and Methods Development Group. Since the development of the drop hammer is incomplete, the instruments were provided with plug-in filters for use with naturally detached 1-sec drops. These filters will be changed as required on completion of the design and fabrication of hammering devices to give a controlled drop time. A check-out and test procedure[14] was prepared for use in the check-out of new instruments and in the maintenance of instruments in service. Operating instructions for the instrument were prepared[15] that contain procedures for placing the instrument in operation and a description of the function of each front-panel control.

Work in progress with this instrument[12] will be continued. These studies include investigation of compensation for resistance in the polarographic cell, evaluation of the analytical utility of third-derivative dc polarography, and appraisal of a material for fabricating stationary electrodes for voltammetry.

1.5 HIGH-SENSITIVITY COULOMETRIC TITRATOR, ORNL MODEL Q-2564

H. C. Jones W. D. Shults

Two additional ORNL model Q-2564 high-sensitivity coulometric titrators[16–18] were fabricated, checked out, and delivered. Five of these instruments (including the prototype) are now in use at ORNL, two each in the General Hot Analyses and High-Level Alpha Radiation Laboratories and one in the Methods Development Group.

A method that describes the Q-2564 titrator was prepared[19] for inclusion in the *ORNL Master Analytical Manual.* This method contains check-out, test, and troubleshooting information. A paper was published[20] on the design, performance, and typical applications of the instrument; discussion of blank- and background-correction procedures for controlled-potential coulometric titrations is included. An information package[21] on the Q-2564 titrator is available; it contains all construction drawings and other information. This package is being updated.

1.6 CONTROLLED-POTENTIAL DERIVATIVE DC POLAROGRAPHY: APPLICATION TO DETERMINATION OF MICROGRAM QUANTITIES OF URANIUM IN A TRI-*n*-OCTYLPHOSPHINE OXIDE (TOPO) EXTRACT

W. L. Belew M. T. Kelley
D. J. Fisher J. A. Dean[22]

A study was completed of the determination of microgram quantities of uranium by extraction with tri-*n*-octylphosphine oxide (TOPO) followed by derivative dc polarographic measurement directly in the extract.[23] The ORNL models Q-1988-FES (ref. 11) and Q-2792 (ref. 24) controlled-potential, derivative dc polarographs were used.

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[14]H. C. Jones, *Check Out and Test Procedure for Q-2792 Controlled-Potential DC Polarograph-Voltammeter,* ST-293 (Apr. 9, 1965).

[15]H. C. Jones and W. L. Belew, "Operating Instructions for Controlled-Potential DC Polarograph-Voltammeter ORNL Model Q-2792" (Sept. 9, 1965); unpublished instructions to be included in a method for the *ORNL Master Analytical Manual.*

[16]H. C. Jones, D. J. Fisher, and W. D. Shults, "High-Sensitivity Coulometric Titrator," *Anal. Chem. Div. Ann. Progr. Rept. Dec. 31, 1962,* ORNL-3397, pp. 3–4.

[17]H. C. Jones, "ORNL Model Q-2564 High-Sensitivity Coulometric Titrator," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1963,* ORNL-3537, p. 3.

[18]H. C. Jones, "ORNL Model Q-2564 High-Sensitivity Coulometric Titrator," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1964,* ORNL-3750, p. 4.

[19]H. C. Jones, "Coulometric Titrator, Controlled-Potential, High-Sensitivity, ORNL Model Q-2564," Method Nos. 1 003025 and 9 003025 (8-20-65), *ORNL Master Analytical Manual;* TID-7015, suppl. 8 (in press).

[20]H. C. Jones, W. D. Shults, and J. M. Dale, "High-Sensitivity Controlled-Potential Coulometric Titrator. Controlled-Potential Coulometric Determination of Milli- and Microgram Quantities of Uranium and Iron," *Anal. Chem.* 37, 680 (1965).

[21]Anon., "High-Sensitivity Coulometric Titrator /Q-2564/ in *Engineering Materials List,*" TID-4100, suppl. 28, CAPE-1196.

[22]Consultant; Professor of Chemistry, University of Tennessee, Knoxville.

[23]W. L. Belew, D. J. Fisher, J. A. Dean, and M. T. Kelley, "Controlled-Potential and Derivative Polarography: Application to the Determination of Metal Complexes in Organic Extracts," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1963,* ORNL-3537, pp. 12–14.

[24]D. J. Fisher, M. T. Kelley, H. C. Jones, R. W. Stelzner, and W. L. Belew, "Controlled-Potential DC Polarograph-Voltammeter. Design and Evaluation," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1964,* ORNL-3750, p. 3.

Controlled-potential polarography permits the use of test solutions of relatively high specific resistance, which makes practical the combination of the techniques of solvent extraction and polarography. The effect of *iR* drop in a high-specific-resistance solution can be minimized when a controlled-potential polarograph is used properly.[13,25] The controlled-potential technique in polarography also allows the use of a very small reference electrode, such as a Q.R.E.,[26] which facilitates the design of micro polarographic cells.

The determination of uranium at trace levels, for example, in seawater (~3 μg/liter) and in waste streams, is of practical interest. By the highly efficient and selective TOPO solvent extraction[27] done prior to the polarographic determination, uranium is separated from interfering ions and is concentrated; the overall sensitivity of the method is thereby increased. The results obtained are described in two articles accepted for publication.[28,29]

In the method developed, uranium is extracted into a cyclohexane solution of TOPO from a 0.5 *M* HNO_3 solution; the 0.5 *M* HNO_3 is purified previously by reaction with sulfamic acid to minimize the blank. A portion of the extract is diluted with an equal volume of ethyl alcohol that is 0.2 *M* in $LiClO_4$; the resulting solution has a specific resistance of ~1400 ohm-cm. Very reproducible first-derivative polarograms are obtained. The uranium concentration vs i_d and vs first-derivative peak height relationships are linear over the range from 1×10^{-6} to 1×10^{-4} *M*. The limit of detection of uranium in the solution polarographed, defined as three times the noise level, is $\sim 1 \times 10^{-6}$ *M* (0.24 μg/ml).

The maximum aqueous-to-organic phase ratio that permits complete extraction of the uranium in one equilibration is 50:1.[28] Thus, with a 250-ml sample and a 5-ml polarographic cell, a net 25-fold concentration advantage is achieved. For example, a 250-ml aqueous sample that contains as little uranium as 0.01 μg/ml (5×10^{-8} *M*) can be analyzed.

To further increase the sensitivity of the method, a 0.5-ml polarographic cell was fabricated.[29] For a given uranium concentration, the signal-to-noise ratio for the 0.5-ml cell was found to be equal to that of the 5-ml cell. Thus, use of the 0.5-ml cell has the advantage that a smaller sample can be analyzed, or with a large sample, either a higher concentration can be achieved in the solution polarographed to improve the precision or the uranium concentration can be increased relative to that of the aqueous sample to increase the sensitivity of the method. To achieve a large concentration increase, the volume of the extract is reduced tenfold by evaporation. The net concentration of the uranium in the cell can thus be increased 250-fold relative to the aqueous sample; an aqueous sample having uranium concentration as low as 5×10^{-9} *M* can be analyzed.

With 0.6 μg of uranium in the 0.5-ml cell, the first-derivative peak height can be determined with satisfactory precision ($S \cong 4\%$). A cursory analysis indicated that this method is applicable to the determination of uranium in seawater.

[25] W. L. Belew, D. J. Fisher, M. T. Kelley, and J. A. Dean, "Compensation of Cell Resistance in Controlled-Potential Polarography," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1963*, ORNL-3537, pp. 14–16.

[26] D. J. Fisher, W. L. Belew, and M. T. Kelley, "A Simple Quasi Reference Electrode. . . .Applications in Controlled-Potential Polarography and Voltammetry and in Chronopotentiometry," pp. 1043–1059 in *Polarography 1964* (ed. by G. J. Hills), Macmillan, London (in press).

[27] J. C. White, *The Use of Trialkyl Phosphine Oxides as Extractants in the Fluorometric Determination of Uranium*, ORNL-2161 (Nov. 1, 1956).

[28] W. L. Belew, D. J. Fisher, M. T. Kelley, and J. A. Dean, "Determination of Trace Quantities of Uranium by Controlled-Potential DC Polarography in Tri-*n*-Octylphosphine Oxide Extract," *Microchem. J.* 10, in press (1965).

[29] M. T. Kelley, W. L. Belew, G. V. Pierce, W. D. Shults, H. C. Jones, and D. J. Fisher, "Controlled-Potential Polarography and Coulometry as Microanalytical Techniques," *Microchem. J.* 10, in press (1965).

1.7 CONTROLLED-POTENTIAL DERIVATIVE DC POLAROGRAPHY AND CONTROLLED-POTENTIAL COULOMETRY AS MICROANALYTICAL TECHNIQUES

M. T. Kelley
W. L. Belew
G. V. Pierce[30]
W. D. Shults
H. C. Jones
D. J. Fisher

Controlled-potential derivative dc polarography has been described as a microanalytical technique for determining uranium extracted into tri-*n*-octylphosphine oxide (TOPO)[28,29] (Sect. 1.6).

[30] High-Level Alpha Radiation Laboratory.

At this laboratory, small samples (~0.04 meq) are normally used, and high precision ($S = 0.1\%$) is obtained. Work was done to extend controlled-potential coulometry to the micro level. It was possible to determine reducible and oxidizable constituents at a level of 0.02 microequivalent with high precision ($S = 1\%$)[29] by: reducing the size of the cell and the volume of the test solution, giving careful attention to such details as purity of reagents and exclusion of oxygen, use of the ORNL model Q-2564 high-sensitivity coulometric titrator,[20] and application of one of several suitable blank corrections. One of the blank-correction techniques, that is, the compensated-i vs Q technique, is of considerable utility (Sect. 1.8). The applicability of controlled-potential coulometry as a microanalytical technique was demonstrated by determining 0.02 to 7 microequivalents of iron, uranium, and plutonium.[29]

1.8 UTILITY OF i VS Q PLOTS IN CONTROLLED-POTENTIAL COULOMETRY

W. D. Shults, D. J. Fisher, H. C. Jones, M. T. Kelley, G. V. Pierce[30]

Hanamura has demonstrated that plots of electrolysis current (i) as ordinate vs integrated current (Q) as abscissa can be used to advantage in potentiostatic coulometry.[31,32] He developed an i-Q recorder[33] specifically for this application. The utility of this technique was explored as part of a study of the potentiostatic coulometric determination of microgram quantities of plutonium (Sect. 1.7); the results are described in some detail.[29] It is pertinent to summarize the conclusions here.

1. The ORNL model Q-2564 coulometric titrator is well suited for this application when used in conjunction with an X-Y recorder. It is necessary to RC-damp the current range (Y) so as to rid the plot of minor fluctuations due to momentary variations in mass transfer within the cell. It is sometimes necessary to nullify the continuous faradaic current by inserting a constant current of suitable magnitude and polarity into the current-control-amplifier input of the Q-2564 titrator. (Hanamura's instrument[33] also incorporates compensation for the continuous faradaic current.)
2. The resulting plot is linear and can be easily extrapolated to zero current to provide an estimate of Q, and thence the analytical result. Extrapolation can be made before the electrolysis is complete; therefore, the i vs Q technique is considerably faster than graphical techniques based on plots of Q vs time (see Fig. 6 of ref. 29).
3. The results obtained are comparable in precision and accuracy with those obtained by the Q vs time technique. This conclusion is based on the determination of ~10-μg quantities of plutonium and of ~550-μg quantities of uranium.
4. The i vs Q technique has the additional advantage that nonlinearity of the plot (or perhaps a change in slope) signals the occurrence of an extraneous process.

An i vs Q method was also applied to the controlled-potential coulometric titration of uranium in simulated MSRE samples (Sect. 4.1.d).

1.9 DERIVATIVE CHRONOPOTENTIOMETRY

T. R. Mueller, W. D. Shults

The first time derivative of the potential during electrolysis with controlled current has a concentration-dependent minimum. For certain current-time programs, this point occurs well before the normal transition time. Thus, the derivative technique permits obtaining pertinent information about the concentration of a species or the kinetics of a reaction without the necessity to prolong the electrolysis until the normal transition time is reached. This technique has several advantages. First, the distortion of the chronopotentiometric wave in the transition-time region by double-layer

[31] S. Hanamura, "Rapid Method for Coulometric Analysis Using an I-Q Recorder," *Talanta* 2, 278 (1959).

[32] S. Hanamura, "A Direct-Reading Current Integrator for Coulometric Analysis," *Talanta* 3, 14 (1959).

[33] S. Hanamura, "An I-Q Recorder and Its Application to Rapid Coulometric Analysis and Micro-Coulometry," *Talanta* 9, 901 (1962).

charging and other extraneous processes is avoided. Second, convective disturbances that arise from electrolysis-induced density gradients (and perhaps thermal gradients as well) are minimized. Finally, resolution of overlapping waves may be enhanced.

Murray and Reilley[34] have derived expressions for the potential-time behavior of reversible and totally irreversible electrode reactions. Their equations are derived from the general current function

$$i = \beta t^r ,$$

where i is the current density, β is a constant, t is time, and r is any number greater than -1. We have used their equations to obtain expressions for $(dE/dt)_{min}$, t_{min}, and E_{min} as functions of r. Their equations are based on the assumption that the electrode reaction of interest proceeds with 100% current efficiency. This condition can be approached closely with the chronopotentiometer that makes possible compensation for extraneous processes[2] (Sects. 1.1 and 1.2). The instrument was designed and constructed in such a way that current-programming and derivative-taking circuitry can be added. A communication is being prepared for publication that presents the generalized theoretical basis for derivative chronopotentiometry.

1.10 PREPARATION OF CHAPTER FOR WELCHER'S *STANDARD METHODS OF CHEMICAL ANALYSIS*, VOL III

W. D. Shults

The forthcoming edition of Welcher's *Standard Methods of Chemical Analysis* is to contain a third volume that deals exclusively with the various instrumental techniques of analysis. A chapter[35] was written for that volume which presents a concise theoretical and practical treatment of coulometric methods.

[34]R. C. Murray and C. N. Reilley, "Chronopotentiometry with Current Programmed as a Function of Time. I. Constant Power Functions of Time," *J. Electroanal. Chem.* 3, 64 (1962).

[35]W. D. Shults, "Coulometric Methods," chap. 23 in vol III of *Standard Methods of Chemical Analysis* (ed. by F. J. Welcher), Van Nostrand, Princeton, N.J. (in press).

1.11 DIFFERENTIATING CIRCUIT FOR THE CONTROLLED-POTENTIAL VOLTAMMETER

W. L. Maddox D. J. Fisher

The prototype controlled-potential voltammeter[36] was originally used by the Reactor Chemistry Division for electrochemical studies in molten salts and is now in use in the Analytical Chemistry Division. Its capabilities have been increased by the addition of an operational-amplifier differentiating system that has a 2-sec time-constant network.[37] It is now possible to record either regular or first-derivative controlled-potential voltammograms with this instrument.[38]

1.12 SAMPLE-IRRADIATION ASSEMBLY FOR ^{60}Co SOURCE

W. L. Maddox

A safe and reliable means of inserting a sample into the shielding cask of a ^{60}Co source, exposing it for a reproducible time at a reproducible position, and then withdrawing it was requested by H. E. Zittel[39] and H. Kubota for use in the study of effects of radiation on analytical methods (Sect. 3.4). A machine for doing these operations by remote control was designed and constructed. This machine comprises chain-operated, electrically driven, sample-elevating and shield-shifting sections which are interconnected in such a way that a shield is drawn back from the opening of the source tube just before the sample elevator begins to move down. The sample carrier deposits a plug in the top of the tube as it descends and picks up the plug as it rises; the opening is unshielded only briefly while the sample is being moved. When the elevator reaches the top of its travel, a contact is broken, and the shield is thus allowed

[36]D. J. Fisher, M. T. Kelley, W. L. Maddox, and R. W. Stelzner, "Controlled-Potential Voltammeter," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1963*, ORNL-3537, pp. 16–17.

[37]G. A. Philbrick Researches, Inc., Boston 16, Mass., "IV. Some Applications," in *Utility Packaged Amplifier, Technical Data, Model UPA-2*, pp. 7–8, UPA-2/and/4M/663.

[38]G. Mamantov, "Electrochemistry of UF_4 in Molten Fluorides," *Reactor Chem. Div. Ann. Progr. Rept. Jan. 31, 1965*, ORNL-3789, p. 80.

[39]Research Group.

to roll back into place over the opening. The aluminum carrier is of seamless construction to prevent damage to the source if a sample container should break.

Preliminary tests have shown that the mechanism is satisfactory but that some additional fixed shielding may be required; final testing will be done when the laboratory for the radiation-effects program is completed.

1.13 COMPUTER PROGRAMS FOR ANALYSIS OF POLAROGRAMS AND CHRONOPOTENTIOGRAMS

Serena Savage[40] W. D. Shults

Computer programs (FORTRAN 63) were written for the ORNL CDC 1604-A computer to facilitate analysis of dc polarograms and chronopotentiograms that derive from the study of simple, reversible, diffusion-controlled electrode processes. Experimental current-potential dc polarographic data are used to compute a "linear least-squares" fitted line that corresponds to the conventional semilogarithmic plot based on the Heyrovský-Ilkovič equation.[41] The reciprocal slope ($2.303\ RT/nF$) and intercept ($E_{1/2}$) and their standard deviations are printed out. The input data and values for the logarithmic term in the Heyrovský-Ilkovič equation are also printed out to facilitate construction of the conventional plot when this is desirable. This program is termed PØLARGRM. Chronopotentiometric time-potential data are treated similarly except that the analysis is based on the Karaoglanoff equation;[42] this program is termed CHRNØGRM. Reports[43,44] were issued that present these programs and explain how they can be used; the programs were added to the Computer Program Library at ORNL.

[40]Summer Student Trainee from Rice University, Houston, Tex.

[41]J. Heyrovský and D. Ilkovič, "Polarographic Studies with the Dropping Mercury Electrode. – Part II. – The Absolute Determination of Reduction and Depolarization Potentials," *Collection Czech. Chem. Communs.* 7, 198 (1935).

[42]Z. Karaoglanoff, "Uber Oxydations- und Reduktionsvorgänge bei der Elektrolyse von Eisensalzlösungen," *Z. Elektrochem.* 12, 5 (1906).

[43]Serena Savage and W. D. Shults, *PØLARGRM: A Computer Program for Analysis of Polarograms*, ORNL-TM-1253 (Aug. 31, 1965).

[44]Serena Savage and W. D. Shults, *CHRNØGRM: A Computer Program for Analysis of Chronopotentiograms*, ORNL-TM-1254 (Aug. 31, 1965).

1.14 A SQUARE-WAVE POLAROGRAPH

G. C. Barker[45] R. W. Stelzner

A compact square-wave polarograph that incorporates solid-state integrated circuits, modular operational amplifiers, and X-Y recording was designed and is under construction at AERE, Harwell, in the Analytical Chemistry Branch. The performance characteristics of this new polarograph are expected to exceed those of the older vacuum-tube model[46] in most respects, except perhaps sensitivity.

A 225-cps square wave of 7- or 35-mv amplitude is superimposed on a linearly increasing dc voltage and is applied subsequently to a low-resistance cell (~50 ohms). The electrochemical cell consists of a dropping-mercury electrode, a saturated calomel reference electrode, and a counter electrode. Current is measured during a 25-msec interval near the end of drop life by a sample-and-hold circuit. A Moseley type 2D-4 X-Y recorder plots the current as a function of the applied dc voltage.

Linear sweeps of 20, 50, and 100 mv/min and synchronized linear sweeps of 0.6, 3.0, and 6.0 v/min are produced by an analog integrator circuit that is actuated by a selected constant-current input. Double-layer capacitance measurement is achieved by switching a 1-μf capacitor into the feedback loop of the low-level current amplifier.

The polarograph will be tested with conventional and modified glass D.M.E. capillaries and with ORNL-fabricated Teflon D.M.E. capillaries.[47]

1.15 CONTROLLED-POTENTIAL DIFFERENTIAL DC POLAROGRAPHY

W. D. Shults W. B. Schaap[48]
D. J. Fisher H. C. Jones

A controlled-potential differential dc polarograph was designed and constructed. Basically, it

[45]Chemistry Division, A.E.R.E., Harwell, Didcot, Berkshire, England.

[46]A.E.R.E. Instruction Manual for Square Wave Polarograph Equipment Type 1336A.

[47]Helen P. Raaen, R. J. Fox, and V. E. Walker, *Fabrication and Assembly of a Teflon Dropping-Mercury Electrode*, ORNL-3344 (Nov. 30, 1962).

[48]Consultant; Professor of Chemistry, Indiana University, Bloomington.

consists of two separate channels, each of which contains potential-controlling and current-measuring circuitry similar in principle to that utilized in the ORNL model Q-1988A polarograph. Two independent polarographic cells are operated by these two channels. The signal from channel 1 (i_1) or from channel 2 (i_2) or the differential signal ($i_1 - i_2$) can be recorded as a function of the controlled-electrode vs reference-electrode potential in channel 1. The instrument incorporates:

1. X-Y recorder readout. This readout facilitates the use of various scan rates and provides for amplification of the polarographic signal through use of the several ranges of the recorder.
2. Filter circuitry that removes current fluctuations (caused by growth and fall of mercury drops) from the polarographic signal. This feature eliminates the need for drop-time synchronization in many applications of differential polarography.
3. Variable gain in channel 2 relative to channel 1. This feature obviates the requirement that capillaries with identical characteristics be used in differential polarography – small differences in characteristics can be nullified effectively by proper adjustment of the channel-2 gain.
4. A circuit that allows the potential in channel 2 to be offset with respect to that in channel 1 by a constant amount.
5. Initial-current-compensation circuitry in both channels.
6. A wide selection of scan rates.
7. A circuit that computes the derivative of the polarographic signal from either channel or from the difference between the two channels.

This instrument is being used in a general investigation of several differential-polarographic techniques. The investigation has been restricted to work with dropping-mercury electrodes that have natural drop times.

A brief theoretical and experimental study of ΔE-*differential* polarography was conducted. This is the technique in which a constant difference in applied potential is maintained between the two channels throughout the potential scan in order to obtain derivative-like curves. Equations pertinent to the ΔE-differential technique were derived and were verified; the curves obtained were compared with analogically computed derivative curves.

Comparative polarography is being evaluated, and both dual- and single-cell techniques are being used. In the dual-cell technique, a solution of unknown electroactive-species concentration is placed in one cell, and a similar solution of accurately known electroactive-species concentration is placed in the second cell. The differential diffusion current can then be measured and used to determine the unknown concentration. In the single-cell technique, the diffusion current of the reference solution is first nullified electronically; then the reference solution is replaced with the unknown solution and the differential diffusion current is measured. Each of these two techniques permits highly precise and highly accurate polarographic analyses to be made. Table 1.1 presents a summary of the results obtained when cadmium was determined in 1.0 *F* KCl–0.001 *F* HCl–0.001% Triton X-100 medium; ten or more replicate analyses were made at each concentration.

Subtractive polarography is also being evaluated. This is the technique whereby unwanted signals are removed from the total polarographic signal

Table 1.1. Results Obtained by Comparative Polarographic Determination of Cadmium

Supporting medium, 1.0 *F* KCl–0.001 *F* HCl–0.001% Triton X-100

Cd^{2+} Concentration (*M*)	Single-Cell Technique		Dual-Cell Technique	
	Relative Standard Deviation (%)	Relative Error (%)	Relative Standard Deviation (%)	Relative Error (%)
10^{-4}	0.2	+0.03	0.4	+0.04
10^{-3}	0.1	0.00	0.1	+0.01
10^{-2}	0.1	-0.24	0.3	-0.13

by operating in the dual-cell differential mode. Thus, a solution that contains the electroactive species of interest plus the supporting electrolyte (and possibly other reactants) is placed in cell 1, and a similar solution free of the electroactive species is placed in cell 2; the differential signal is then due only to the electroactive species of interest. This technique can provide increased sensitivity or selectivity or both. By subtractive polarography, for example, Cd^{2+} can be detected at 10^{-7} *M* concentration in air-free solution and determined at 10^{-4} *M* concentration (or more) in an oxygen-saturated solution with about the same accuracy and precision as are achieved in oxygen-free solution.

This work is to be published in thesis form and is to be submitted in part for publication in the open literature.

2. Analytical Studies of Molten-Salt Systems

J. C. White

2.1 ABSORPTION SPECTROPHOTOMETRIC STUDIES RELATED TO MOLTEN-SALT SYSTEMS

J. P. Young

Absorption spectrophotometric studies related to various molten-salt systems were continued. One of the objectives is to evaluate the analytical usefulness of such spectra. Presently, the possibility of continuously determining U^{3+} in molten-fluoride-salt reactor fuels by in-line spectrophotometry is being investigated. The progress of this work is reported elsewhere.[1]

2.1.a Absorption and Reflectance Spectra of Irradiated $LiF-BeF_2-ZrF_4-ThF_4-UF_4$

To evaluate the radiation stability of a solidified sample of $LiF-BeF_2-ZrF_4-ThF_4-UF_4$, persons of the Reactor Chemistry Division subjected the sample to gamma radiation. Fluorine gas was evolved during the experiment. In an effort to determine the oxidation state of uranium in the sample, reflectance spectra of the solid sample and absorption spectra of the molten salt, after it was melted in the high-temperature cell assembly, were obtained. Before irradiation, the solid was green; after irradiation, it was a charcoal color. Both the color and the absorption spectrum of the melted irradiated sample indicated that essentially all the uranium was present as U^{3+}. This evidence did not mean, however, that U^{3+} was present in the solid; any reduced species in the solid probably could reduce U^{4+} to U^{3+}. Reflectance spectra of the solid sample were also obtained. Techniques have been developed by which U^{3+} in concentrations $\sim$0.5 w/w % or greater in solids can be determined. In the solid irradiated sample the U^{3+} concentration was below this detection limit. Work on this problem is continuing.

2.1.b Absorption Spectrum of Co^{2+} in LiF-NaF-KF

Further spectral work was done pertaining to the quantitative nature of the spectrum of Co^{2+} in LiF-NaF-KF. Previously,[2] the spectrum was described only qualitatively. The position of the double absorption peak is essentially as reported.

[1] J. P. Young, "Spectrophotometric Studies of Molten-Salt Reactor Fuels," *MSR Program Semiann. Progr. Rept. Aug. 31, 1965*, ORNL-3872, p. 145.

[2] J. P. Young and J. C. White, "Absorption Spectra of Molten Fluoride Salts. Solutions of Several Metal Ions in Molten Lithium Fluoride—Sodium Fluoride—Potassium Fluoride," *Anal. Chem.* **32**, 799 (1960).

The maximum molar absorptivity at ~580 mμ is 14. As was pointed out[3] from the earlier qualitative data,[2] the position and intensity of the Co^{2+} spectrum in LiF-NaF-KF suggest that Co^{2+} is octahedrally coordinated in this melt.

2.1.c Absorption and Diffuse-Reflectance Spectra of Solid Materials

To understand the nature of the spectra of solutes dissolved in molten-salt solvents, it is advantageous to know the spectral characteristics of the solid-solute species. Techniques developed for this purpose are of value, then, to other investigators as well, since spectral measurements can indicate the purity of solids and aid in determining the symmetry and coordination of the absorbing species. Two techniques have been used to obtain spectra of solids, namely, transmission and diffuse-reflectance measurements.

The use of diffuse reflectance to detect and estimate U^{3+} in solids was discussed in Sect. 2.1.a. For these studies, it was necessary to design and fabricate a vacuum-tight reflectance cell, since the samples were unstable to moisture and were toxic. The spectra were obtained with a Cary model 14 M spectrophotometer provided with the Cary model 1411 type 1 reflectance attachment. The cell consists of a black anodized aluminum cup and cover plate. A rubber O-ring is placed in a groove on the rim of the cup, and a quartz plate covers the cup and O-ring. The assembly is held together and sealed by six screws that pass through the cover plate and into the base of the cup. A slight decrease in absorbance (~2%) results from the fact that the thickness of the quartz plate causes the surface of the powdered sample in the sealed cell to be below the focal plane of the sample beam. No detectable decrease in absorbance is caused by surface reflection from the quartz plate. Reflectance spectra of a number of pure solids were measured,[1] as were those of solidified melts of U^{3+} or U^{4+} in alkali-metal fluorides and of mixtures of U^{3+} and U^{4+} fluorides in the wavelength range from 250 to 800 mμ. Both U^{3+} and U^{4+} exhibit many absorption peaks in this region. Nevertheless, U^{3+} can be detected in mixtures with U^{4+} by an absorption peak at 710 mμ; the limit of detection is less than 0.5 w/w %. The presence of U^{4+} can be detected by an absorption peak at 660 mμ.

The apparatus for obtaining the transmission spectra of small crystals was described previously.[4] In cooperation with G. W. Clark,[5] we are obtaining absorption spectra of a number of rare-earth germanomolybdates and rare-earth-doped oxides. The data should be useful in delineating the nature of the rare-earth ions in these crystals and possibly also in estimating the energy of ground-state transitions caused by thermal processes. This work is continuing.

The transmission spectra of small crystals of UF_4 were obtained in the wavelength range from 250 to 1200 mμ. The transmission and diffuse-reflectance spectra of UF_4 are essentially the same with respect to positions of absorption peaks. The only differences seem to be related to the fact that transmission spectra are obtained from crystals oriented in a particular direction and reflectance spectra arise from randomly oriented powdered crystals. Only from transmission spectra, however, can molar absorptivities be calculated. Such data were obtained and will be published. A general report[6] describes much of this work.

The absorption spectrum of a very small piece of translucent boron nitride, BN, was obtained. This type of BN sometimes results from studies of the synthesis of pyrolytic BN that are being carried out in the Metals and Ceramics Division. A transparent piece of BN might prove useful as an inert window for visual and spectrophotometric studies of molten-fluoride salts. The sample was essentially transparent from ~450 to 1000 mμ; no data were obtained at wavelengths greater than 1000 mμ. The spectrum of the sample exhibited an optical cutoff at 300 mμ and a shoulder at 370 mμ; it is not known whether this absorption is due to BN or to impurities.

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[3] G. P. Smith, "Electronic Absorption Spectra of Molten Salts," pp. 428–505 in *Molten Salt Chemistry*, ed. by M. Blander, Interscience, New York, 1964; esp. p. 453.

[4] J. P. Young, "Absorption Spectrophotometric Studies Related to Molten-Salt Systems," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1964*, ORNL-3750, pp. 5–8.

[5] Metals and Ceramics Division.

[6] J. P. Young and G. W. Clark, "Absorbance Spectra of Single Crystals," presented at the Ninth Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Oct. 12–14, 1965.

2.2 VOLTAMMETRY OF MOLTEN-FLUORIDE SALT SYSTEMS

Gleb Mamantov[7] D. L. Manning
J. M. Dale

2.2.a Reversible Deposition of Metals on Solid Electrodes by Voltammetry with Linearly Varying Potential

It has been shown by extending the treatment of Berzins and Delahay[8] that, for the reversible deposition of metals on solid electrodes by voltammetry with linearly varying potential, a plot of log $(i_p - i)$ vs E approaches linearity over the current range from about 0.5 to 0.9 i_p, the slope of the plot being $2.2(nF/RT)$. The experimental data for the reduction of Ag^+ in 1 *M* KNO_3 at silver and pyrolytic-graphite electrodes agree satisfactorily with the theory. This agreement likewise exists for the reduction at 500°C of Fe^{2+} in LiF-BeF_2 (66-34 mole %) and in LiF-NaF-KF (46.5-11.5-42 mole %) and of Ni^{2+} in LiF-NaF-KF at pyrolytic-graphite electrodes. An article on this work has been published.[9]

[7]Consultant; Assistant Professor of Chemistry, University of Tennessee, Knoxville.

[8]T. Berzins and P. Delahay, "Oscillographic Polarographic Waves for the Reversible Deposition of Metals on Solid Electrodes," *J. Am. Chem. Soc.* **75**, 555 (1953).

[9]G. Mamantov, D. L. Manning, and J. M. Dale, "Reversible Deposition of Metals on Solid Electrodes by Voltammetry with Linearly Varying Potential," *J. Electroanal. Chem.* **9**, 253 (1965).

3. Effects of Radiation on Analytical Methods

J. C. White

3.1 GAMMA RADIOLYSIS OF ACID CHLORIDE SOLUTIONS

Hisashi Kubota

The effect of the chlorine produced by the gamma-induced radiolysis of acid chloride solutions on actual analytical procedures was investigated by taking as an example the determination of zirconium with arsenazo III. Arsenazo III forms colored complexes with zirconium in acid solution. The reaction is most sensitive in HCl solution, the molar absorptivity increasing with increasing acid molarity up to 9 *M*. Arsenazo III is readily destroyed by strong oxidants; therefore, it is not surprising that both the reagent itself and the zirconium complex degrade when irradiated with ^{60}Co gamma radiation. The rate of decomposition is proportional to acid molarity. This effect is the same as that from the radiolysis of acid chloride systems. A 30,000-rad dose causes the absorbance of the complex in 3 *M* HCl to diminish by 20%; in 9 *M* HCl, 50%. The sensitivity of the reaction in 9 *M* HCl is about four times that in 3 *M* HCl. If the dose does not exceed 1000 rads, zirconium can be determined with arsenazo III with a maximum radiolytic effect of 3%.

Methanol has been used to prevent the formation of free chlorine, as well as the depletion of acid, in concentrated lithium chloride solutions that contain high alpha radioactivity.[1] The present work has confirmed that methanol inhibits chlorine production in gamma-irradiated systems; however, methanol does not arrest the depletion of acid, the rate of acid loss being unaffected. Furthermore, methanol does not negate radiolytic effects on arsenazo III solutions; they decompose as readily in the presence as in the absence of methanol. It was suspected that the role of methanol is to react with the chlorine produced by the radiolysis.

[1]R. D. Baybarz, "Alpha Radiation Effects on Concentrated LiCl Solutions Containing HCl, and the Use of Methanol as an Inhibitor of Acid Radiolysis," *J. Inorg. Nucl. Chem.* **27**, 725 (1965).

Gas chromatographic analysis of the gaseous products of the radiolysis in the presence of methanol showed that methyl chloride is produced in large amounts together with traces of methane, ethane, and an unidentified component (Sect. 3.2).

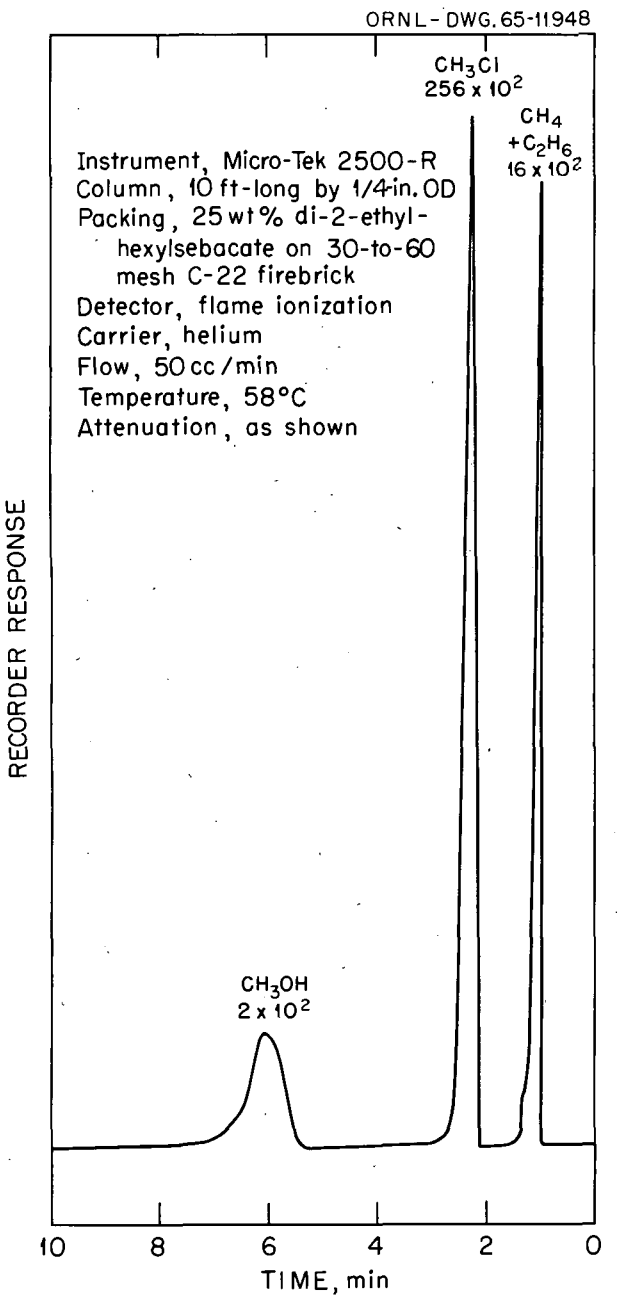

Fig. 3.1. Typical Gas Chromatogram of the Reaction Products of Gamma-Irradiated Mixtures of Methanol and Hydrochloric Acid. Radiation time, 30 hr; radiation intensity, $\sim 10^7$ rads/hr; sample size, unknown.

It is believed that the role of methanol is to scavenge the chlorine rather than its precursor hydroxyl radical. The following reactions are thought to occur in gamma-irradiated acid chloride systems in the presence of methanol:

$$Cl^- + OH\cdot + H^+ \longrightarrow Cl^0 + H_2O \,,$$

$$CH_3OH + Cl^0 \longrightarrow CH_3Cl + OH\cdot \,.$$

3.2 GAS CHROMATOGRAPHIC ANALYSIS OF GAMMA-IRRADIATED MIXTURES OF METHANOL AND HYDROCHLORIC ACID

A. D. Horton

Several sealed glass bulbs that contained mixtures of methanol and hydrochloric acid were irradiated in the ^{60}Co source and were submitted for analysis of the vapor (Sect. 3.1). For each bulb, the concentrations of methanol and hydrochloric acid in the solution and the duration and intensity of the irradiation were different.

The gaseous contents of the bulbs were analyzed by gas chromatography; Fig. 3.1 gives typical conditions and results. The compounds identified from the chromatograms were methane, ethane, methanol, and methyl chloride. No quantitative data were taken, but the following general correlations were obtained. The amount of reaction products increased with increase in duration and in intensity of the irradiation. At a constant radiation intensity, the reaction products increased with increase in the concentration of hydrochloric acid and in the duration of irradiation. Ethane did not appear in samples that were irradiated for only a short time.

3.3 HIGH-TEMPERATURE CHEMICAL DOSIMETRY

Hisashi Kubota

Chemical dosimeters are a convenient and reliable means of monitoring gamma-radiation dose in the range from 10^3 to 10^5 rads. The response is nearly constant over the range of beam energy

between 0.1 and 3 Mev.[2] Most applications are made at ambient temperature; however, the $FeSO_4$ dosimeter has been studied up to 65°C[3] and the $Ce(SO_4)_2$ to 90°C.[4] For some work in high-level waste disposal it is desirable to have a chemical dosimeter that can be used at temperatures as high as 250°C. Preliminary investigation indicated that dosimetric solutions sealed into glass tubes might be usable. It became apparent very quickly that both the oxalic acid and $Ce(SO_4)_2$ dosimeters are unsuitable. The former decomposes above 100°C, and the latter is reduced and hydrolyzed above 150°C. The air-saturated $FeSO_4$ solution undergoes slight oxidation; however, the oxygen-free solution is stable at least to 250°C.

Ferrous sulfate solution was deaerated with argon and sealed into glass tubes. These tubes were irradiated at ambient temperature, 150, 200, and 250°C. The *G* values obtained were 7.6, 7.6, 7.5, and 7.3, respectively, with reference to a *G* value of 15.6 for an air-saturated dosimeter at 25°C. The reproducibility is within 8%, and the response is linear with dose.

The *G* value for the deaerated solution at room temperature should be 8.2. It is not yet understood why the lower values are being obtained; study is still under way to clarify this point. Nevertheless, the results to date indicate that the basic radiolytic reactions are essentially temperature-independent.

[2]A. O. Allen, *The Radiation Chemistry of Water and Aqueous Solutions*, p. 22, Van Nostrand, Princeton, N.J., 1961.

[3]C. J. Hochanadel and J. A. Ghormley, "Effect of Temperature on the Decomposition of Water by Gamma Rays," *Radiation Res.* **16**, 653 (1962).

[4]H. Hotta and K. Shimada, "Temperature Dependence of the Ceric-Cerous Gamma-Ray Dosimeter," *Bull. Chem. Soc. Japan* **33**, 114 (1960).

3.4 MODIFICATIONS TO THE ^{60}Co SOURCE ASSEMBLY

H. E. Zittel Hisashi Kubota

To conform to health-physics requirements, certain modifications were made to the sample-irradiation assembly for the ^{60}Co source obtained from surplus for use in the study of effects of radiation on analytical methods. W. L. Maddox[5] designed and fabricated the assembly (Sect. 1.12), which mechanically lowers a sample tube into and raises it out of the source compartment. When the prototype assembly has been fully tested, another similar assembly will be fabricated for the second tube source, which was also obtained from surplus. The intensities of the two sources differ by a factor of ~10; therefore, use of the two sources together should make possible the study of the effects of gamma radiation on analytical methods over a wide range of dosage levels. It is contemplated that the weaker source will be used for in-situ studies of electrochemical procedures; the more intense source will be used in those studies in which the radiolysis products are to be determined external to the source.

[5]Analytical Instrumentation Group.

4. Analytical Chemistry for Reactor Projects

M. T. Kelley
J. C. White L. T. Corbin

The analytical Chemistry Division continued its program on analytical chemistry research and development for the Molten-Salt Reactor Experiment (MSRE) and the Transuranium Element Processing (TRU) Program; details of these investigations are reported regularly in the following progress reports:

MSRE semiannual progress reports:
ORNL-3812 for Period Ending February 28, 1965
ORNL-3872 for Period Ending August 31, 1965
TRU quarterly progress reports:
ORNL-3847 for Period Ending November 30, 1964
ORNL-3880 for Period Ending February 28, 1965

Also, the Division began work for the Medium-Power Reactor Experiment (MPRE).

Brief summaries of the analytical programs for these reactor projects are given in the following sections. For details of the work, the reader is referred to the appropriate reactor-project progress report.

4.1 MOLTEN-SALT REACTOR EXPERIMENT

R. F. Apple
J. M. Dale
D. J. Fisher
F. E. Haga[1]
F. K. Heacker
M. T. Kelley
Urpo Koskela
C. E. Lamb
J. R. Lund
W. L. Maddox
D. L. Manning
A. S. Meyer
T. R. Mueller
C. K. Talbott
P. H. Thomason

The program in analytical chemistry for the MSRE was directed primarily to development and refinement of methods and techniques for the analysis of MSRE samples. The reactor went critical in May 1965; it was then shut down after zero-power experiments and is scheduled for operation at 10 Mw late in 1965. Some experience has thus been gained in the analysis of the samples in the High-Radiation-Level Analytical Facility (HRLAF) hot cells.

A prototype apparatus was constructed and tested for determining oxides in the MSRE fuel by use of the hydrofluorination principle. The water produced from the reaction of HF gas with oxides is measured automatically by means of an electrolytic moisture monitor. The entire apparatus is being assembled for insertion into a hot cell in order to analyze the fuel after the reactor has gone to power.

Studies were continued on adapting electrochemical methods to in-line determinations of impurities in the fuel. When the simulated fuel is subjected to controlled-potential electrolysis, evolution of gases (primarily CO, CO_2, and O_2) occurs at the indicating electrode. This gas evolution is evidence of removal of oxide from the melt by electroreduction. This discovery holds promise for a possible in-line determination of oxide. Absorption spectrophotometric studies are also under way to determine trivalent uranium and tetravalent uranium in the fuel by their characteristic absorption peaks.

A process gas chromatograph equipped with a helium breakdown-voltage detector is under construction for the continuous analysis of the helium cover gas in the MSR. A special metal-diaphragm sampling valve was designed to withstand the temperature and radiation effects that nullify the use of conventional sampling valves.

Samples from the MSRE precritical and zero-power experiments were analyzed in the High-Radiation-Level Analytical Laboratory (HRLAL) hot cells. The results, obtained with the specially developed equipment and analytical methods, were satisfactory. Statistical evaluation of the control data indicated a negative bias of ~0.8% for uranium and none for beryllium.

4.1.a MSRE Sample Preparation Apparatus

The completion of the design and testing of our apparatus for MSRE sample preparation was reported.[2,3] This apparatus, which is now in use

[1]Present address: Southern Railway Co., Atlanta, Ga.

[2]"Analytical Studies of Molten-Salt Systems," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1964*, ORNL-3750, pp. iv–v.

[3]A. S. Meyer, R. F. Apple, D. J. Fisher, M. J. Gaitanis, F. E. Haga, F. K. Heacker, J. M. Dale, W. L. Maddox, and R. W. Stelzner, "Molten-Salt Reactor Experiment," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1964*, ORNL-3750, pp. 14–15.

for analyses of MSRE samples, includes a transport container, a ladle cutter, a copper pulverizer/mixer capsule, a modified mixer/mill, and a powder-transfer shaker. A paper that describes this apparatus has been written.[4]

4.1.b Remotely Servo-Controlled Corrosion-Resistant Pipetter, ORNL Model Q-1348B

The design and testing of our model Q-1348B, remotely servo-controlled corrosion-resistant pipetter are complete. Several pipetters of this model are in use and are performing satisfactorily for analyses needed in the MSRE program.

Engineering drawings for the fabrication of this pipetter were completed. Copies of these drawings have been sent to interested installations in the United States and abroad and to the AEC's Division of Technical Information Extension. Copies of the drawings are being offered for sale to the public.[5] Also, a method was written that describes procedures for the use of this pipetter; information for its maintenance is included.[6]

4.1.c Remotely Operated Filter Photometer

Our remotely operated filter photometer is being used in the HRLAL for analyses required for the MSRE program. A paper has been written that describes this instrument.[7]

4.1.d Controlled-Potential Coulometric Titration of Uranium(VI) in a Synthetic MSRE Fuel

Titration of ~500 μg of U^{6+} in a sample of radioactive MSRE fuel is normally done with the ORNL model Q-2564 high-sensitivity coulometric titrator. The titration is usually terminated when a cutoff current of 5 μa is reached, which normally takes ~15 min. The correction for faradaic current for this period of time is appreciable and is difficult to determine, because the true background current varies. A background current as low as 1 μa can be achieved if the titration is permitted to continue for 30 min.

Since the precise and accurate coulometric titration of 5-mg quantities of U^{6+} is arbitrarily terminated at 50 μa, a similar titration was studied for determining submilligram quantities of U^{6+}; a synthetic MSRE fuel was used. An X-Y recorder was connected to the titrator so that current (i) vs Q (coulombs) was plotted as a function of time. It was possible to obtain a cutoff current of 50 μa in ~5 min when ~500 μg of U^{6+} was titrated. When Q (coulombs on the Y axis) was extrapolated to zero current (X axis), a correction to the readout voltage (measure of coulombs) of +0.8% was sufficient to give the correct amount of U^{6+} present. For the titration of 29 aliquots of a synthetic MSRE fuel, S was 0.40%.

Advantages of this empirical titration procedure over the regular method are: speed (5 vs 15 min); pictorial presentation of the titration, which can reveal interferences by showing changes in the slope of the i vs Q recording; improved precision; and no need to correct for blank or for faradaic current. The procedure is now being evaluated in the HRLAF.

4.1.e Polarographic Determination of Nickel in MSRE Fuel

As an auxilary way to determine nickel in MSRE fuel ($LiF-BeF_2-UF_4-ZrF_4$), a polarographic method was adapted; the ORNL model Q-1338 polarograph was used. Test portions of the fuel were treated with a mixture of boric, nitric, and sulfuric acids and then were heated to fumes of sulfur trioxide. The cooled solution was diluted to volume with water; it was free of borate, nitrate, and fluoride and was ~1 M in H_2SO_4. Satisfactory polarographic waves for nickel were obtained in the supporting medium 0.25 M HCl–0.5 M H_2SO_4–1.4 M NH_4OH–25 v/v % pyridine–0.05 w/v % gelatin. The results were in the range from 0.002 to 0.02 % nickel with S = 10%.

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[4]W. L. Maddox and F. E. Haga, "Analytical Laboratory Hot-Cell Equipment: I. Sample Preparation Apparatus for the Molten Salt Reactor Experiment," pp. 179–81 in *Proceedings of the 13th Conference on Remote Systems Technology*, American Nuclear Society, Hinsdale, Ill., 1965.

[5]Anon., "Pipetters" in *Engineering Materials List*, TID-4100, suppl. 28, CAPE-1220.

[6]F. E. Haga and D. J. Fisher, "Pipetters, Remotely Servo-Controlled, Corrosion-Resistant, ORNL Models Q-1348A, Q-1348B, and Q-1348C," Method Nos. 1 003102 and 9 003102 (3-26-65), *ORNL Master Analytical Manual*; TID-7015, suppl. 8.

[7]W. L. Maddox, "Analytical Laboratory Hot-Cell Equipment: III. A Remotely Operated Filter Photometer," pp. 184–85 in *Proceedings of the 13th Conference on Remote Systems Technology*, American Nuclear Society, Hinsdale, Ill., 1965.

4.2 TRANSURANIUM ELEMENT PROCESSING PROGRAM

J. H. Cooper
D. A. Costanzo
D. J. Fisher
F. E. Haga[1]
W. L. Maddox
H. C. Meyer
F. L. Moore
T. R. Mueller
R. W. Stelzner
P. F. Thomason

The program by the Analytical Chemistry Division for the TRU Program has continued to center on the development of special equipment and apparatus and of methods of analysis for control of the TRU process.

Studies either completed or under way include: liquid-liquid extraction of metal ions from aqueous solutions of organic acids with high-molecular-weight amines, isolation of trivalent actinide-lanthanide elements from nitrate solutions, determination of free acid, spectrophotometry of californium, determination of nitrate, and effects of alpha radiation on solutions of LiCl. Special equipment and instrumentation include: remotely servo-controlled corrosion-resistant pipetter, remotely operated decapper, filter photometer, remotely operated solvent-extraction apparatus, timed centrifuge-speed controller, velocity servo potentiometric titrator, and remotely operated analytical balance.

The organization of methods for the TRU section of the *ORNL Master Analytical Manual* is practically complete. Equipment for the analytical cells is about 65% complete; for the counting room 90% complete.

4.3 MEDIUM-POWER REACTOR EXPERIMENT

Studies were initiated on the determination of interstitials in alkali metals in connection with the MPRE. None of the following material has appeared in the MPRE progress reports; hence, a more detailed treatment of the analytical investigations is given in this section.

4.3.a Analysis of the Alkali Metals

Gerald Goldberg

Interest in determining impurities (~10 ppm or less) in the alkali metals is increasing. This interest has raised the question of what influence atmospheric impurities, within the dry box in which the samples are handled, have on the reliability of the analytical methods for determining these impurities. The atmospheric impurities most likely to be present and to affect the analytical results are H_2O, O_2, H_2, N_2, and CO_2. We were requested by the AEC to establish maximum permissible concentration levels for these impurities in the atmosphere of the dry box in order to increase the reliability of the analytical methods.

Table 4.1. Recommended Maximum Concentrations of Impurities in the Atmosphere of the Dry Box Used in the Handling of Alkali Metals

Impurity	Maximum Concentration (ppm)
H_2O	5
O_2	10
H_2	5
N_2	10[a]
CO_2	10

[a]Applies only to the handling of lithium.

Several factors were considered: conditions that control the rates of reactions between the alkali metals and the impurities, types of environmental systems now in use for handling the metals, analytical techniques used to determine these impurities, and ability to detect and reduce the concentrations of the impurities. A comprehensive study of the literature was made; also, individuals actively engaged in work with the alkali metals were contacted directly. Table 4.1 lists our recommendations as they appear in a published paper.[8]

Analysis for Carbon. – At present we have two methods for determining carbon in the alkali metals. High-temperature oxidation[9] is used to determine total carbon. Carbon species such as carbonate, cyanide, and acetylide are determined

[8]G. Goldberg, "The Effects of Trace Impurities of Moisture, Oxygen, Hydrogen, and Carbon Dioxide in Glove Box Atmospheres on the Analysis of the Alkali Metals," presented at the Joint AEC-NASA Meeting on the "Second Round-Robin Analysis of Oxygen in Sodium and Potassium," Lewis Research Center, NASA, Cleveland, Sept. 22–23, 1965.

[9]G. Goldberg, "Determination of Carbon in Alkali Metals by High-Temperature Oxidation," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1964*, ORNL-3750, p. 26.

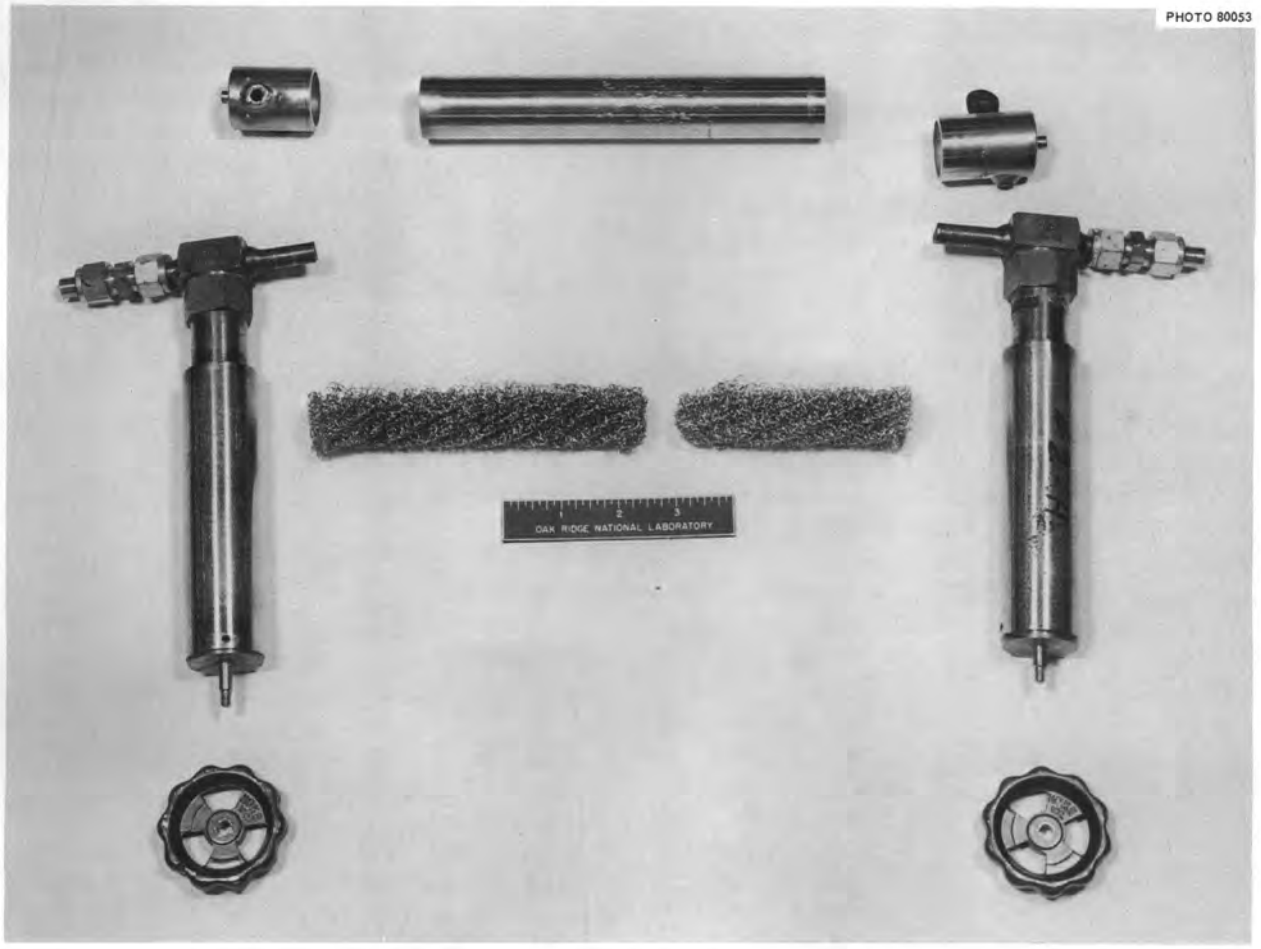

Fig. 4.1. Exploded View of NaK-Loop Mesh-Packed Cold Trap.

by the addition of either deaerated water or dilute acid, under vacuum, to the residue that remains after amalgamation. The resulting gases are transferred to an evacuated sample bulb by means of a Toepler pump. The species present are then determined by either gas chromatography or mass spectrometry. These methods are lacking in that they require separate samples of the metal to be analyzed.

An apparatus is being designed that will allow us to determine total carbon in a single sample as a function of the species present as well as of the elemental carbon. Basically, the sample will be dissolved in water; dilute acid will then be added. The resulting gases, with the exception of hydrogen, will be collected on a silica-gel column at liquid-nitrogen temperature for analysis by gas chromatography. The remaining elemental carbon will be oxidized by suitable wet-chemical means, and the gas evolved will also be collected and analyzed. A gas chromatograph is being requisitioned for this analysis.

Analysis for Hydrogen. – The amalgamation–vacuum extraction procedure[10] is applied routinely to the determination of hydrogen in sodium, potassium, and NaK. Of particular interest was a small mesh-packed cold trap from a SNAP-8 NaK loop submitted for hydrogen determination by this method. Deuterium had been injected into the loop to check on the efficiency of the cold trap, among other tests. The trap created something of an analytical problem, since it was filled

[10]G. Goldberg, "Determination of Hydrogen in Alkali Metals by Amalgamation and Vacuum Extraction," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1964,* ORNL-3750, p. 26.

with more than 100 g of NaK, and both the NaK and all sections of the trap were to be analyzed for hydrogen.

Figure 4.1 shows the trap divided into the sections that were analyzed by use of enlarged amalgamation reaction vessels, such as the one pictured in Fig. 4.2. Figure 4.2 also shows the stainless steel mesh coated with both the NaK and the hydrides prior to amalgamation, as well as a section of the mesh (outside the container) after amalgamation. This is one of the few photographs ever taken of the mesh packing from an NaK-system trap prior to analysis or atmospheric contamination. Essentially all the deuterium injected into the system was recovered from the trap.

Analysis for Oxygen. – A second round robin[11] to determine oxygen in sodium and in potassium was conducted in conjunction with an AEC-NASA endeavor. The samples were handled routinely in that each tube of sample was cut into sections within the dry box, and each section was placed in a separate amalgamation reaction vessel. Table 4.2 gives the results of the analyses, which were also reported at the AEC-NASA meeting.[8] The consensus from the meeting is that the amalgamation procedure[12] is applicable to the routine determination of oxygen in sodium and in potassium at concentrations <50 ppm.

Determination of Oxygen in Alkali Metals by 14-Mev Neutron Activation Analysis (J. E. Strain, W. J. Ross). – Preliminary experiments have shown that it is practical to determine oxygen in concentrations of ~10 ppm in the alkali metals with $S = 5$ to 10% by 14-Mev neutron activation analysis.[13] As a result, the Analytical Chemistry Division in cooperation with the Metals and Ceramics Division is designing a neutron-generator facility specifically to determine oxygen in alkali metals. The sample will be totally enclosed in an inert atmosphere during sample preparation, irradiation, and the counting sequence. It is anticipated that a single determination can be made in about 2 min. The design of the transport system and the engineering drawing of the building modification have been completed.

Fig. 4.2. Enlarged Amalgamation Reaction Vessel That Contains NaK-Coated Stainless Steel Mesh.

Table 4.2. Results of Analyses of AEC-NASA Round-Robin Samples of Sodium and Potassium

Sample-Tube Identity	Sample Weight (g)	Oxygen Content (ppm)
Potassium P-5	2.85	12.1
	3.40	13.5
	4.42	12.1
Sodium O-37	2.76	8.3
	3.18	9.1
Sodium Q-38	2.44	14.2
	3.17	14.0
	4.55	13.5

[11] G. Goldberg, "Determination of Oxygen in Alkali Metals," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1963*, ORNL-3537, pp. 55–57.

[12] J. C. White, A. S. Meyer, and G. Goldberg, "Determination of Oxygen in Higher Alkali Metals," *Anal. Chem. Div. Ann. Progr. Rept. Dec. 31, 1961*, ORNL-3243, p. 41.

[13] J. E. Strain and A. P. Litman, *Trip Report to General Atomic, Sept. 28, 1964*, Oct. 30, 1964 (unpublished report).

4.4 NUCLEAR SAFETY

4.4.a Nuclear Safety Program

J. E. Attrill

Several reports[14–16] indicate that nonelemental iodine is produced from, or along with, radioactive I_2 when uranium metal is exposed to ionizing radiation. The iodides, being less reactive than the parent I_2, are difficult to remove either by chemical or by physical methods. Therefore, a study to identify and to characterize them was of prime importance. Gas chromatography was chosen as the most suitable analytical tool because of its sensitivity.

It has been shown[16] that nonradioactive I_2, prepared either by the palladium iodide distillation method or by heating a mixture of potassium dichromate and potassium iodide, also contains these same contaminants. Therefore, for obvious reasons, these nonradioactive methods of preparing I_2 sources were chosen for initial study. The iodides pass through a cold trap at –70°C, which is used to remove the I_2, but they are retained in a cold trap at the temperature of liquid nitrogen (–190°C). This difference offered a way to purify and concentrate these materials for analysis. Samples of these highly volatile compounds were thus collected and were analyzed by use of a Barber-Colman model 20 gas chromatograph with an argon ionization detector. With an 8-ft-long by 1/4-in.-OD stainless steel column packed with a silicone-rubber-coated support, the resolution was good; two to six peaks were obtained from samples so analyzed. These peaks were identified as air, water, methyl iodide, ethyl iodide, propyl iodide, and hydrogen iodide. Methyl iodide was always present in the greatest concentration by several orders of magnitude. The total amounts of these compounds varied and appeared to depend on the amount of moisture, acids, or metallic compounds present during the preparation of the I_2 source. Also, their concentrations were considerably higher in aged iodines.

Because methyl iodide was the main constituent of these nonelemental iodine compounds, further studies were confined to its removal from an atmosphere that simulated that which might result from a reactor accident. The air-exhaust systems of most reactors include a charcoal filter bed as a safety provision in case of such an accident. Methyl iodide penetrates further through this bed at lower concentrations than do the other iodide contaminants and therefore presents a potential hazard. This is especially true at high humidity. Thus, it is desirable to improve the trapping characteristics of the charcoal. Various types of charcoal were coated with chemical compounds (e.g., oxidizing agents and amines) in an attempt to induce a rapid chemical reaction. Oxidizing compounds, such as potassium bromate and silver bromate, showed some tendency to aid the retention of methyl iodide, but they behaved erratically. Some 30 amines were also tested under various conditions of temperature and humidity; they showed considerable promise. Heterocyclic amines (pyridines, morpholines, piperidines, and piperazines) tremendously increased the adsorptive capacity of these traps, even at high humidity. The high volatility of the amines and their subsequent loss from charcoal at higher temperatures is a drawback. Substituted amines of this type are being tested for improved performance.

4.4.b Spectrophotometric Study of the Stability of Iodine in Cyclohexane and in Benzene

J. P. Young

As an aid in the gas chromatographic study of the stability of solutions of I_2 in either cyclohexane or benzene as a function of time and temperature, a number of spectrophotometric studies were carried out. By spectrophotometric measurements, I_2 vapor could be determined directly in sealed cylindrical quartz ampuls. Each ampul contained a different amount of I_2 and of solvent. The results indicate that at 150°C, only a negligible percent of the I_2 (<10% at best) reacts with either cyclohexane or benzene. At 300°C the benzene-I_2 mixtures are still relatively stable; I_2 absorbance decreases ~6% per hour, whereas

[14] D. H. F. Atkins and A. E. J. Eggleton, *Iodine Compounds Formed on Release of Carrier-Free Iodine-131*, AERE-M-1211 (May 1963).

[15] J. B. Morris and C. H. Rumary, *Tests on Absorbers for Iodine at Low Concentrations*, AERE-R-4219 (February 1963).

[16] W. E. Browning, Jr., R. D. Ackley, and R. E. Adams, "Investigation of the Occurrence and Behavior of Different Forms of Radioiodine," *Nucl. Safety Program Semiann. Progr. Rept. Dec. 31, 1963*, ORNL-3547, pp. 60–64.

cyclohexane-I_2 mixtures react rapidly and completely. No spectrophotometric identification of the products was possible.

4.4.c Gas Chromatographic Analysis of the Products of the Reaction of Iodine with Cyclohexane and with Benzene at 300°C and 20 atm

A. D. Horton A. S. Meyer

This research was undertaken as a part of the study of the mobility of radioiodine in a stainless steel reactor loop at 300°C and 20 atm (abs) of helium. A solution of I_2 in cyclohexane or in benzene was used as a convenient means of injecting I_2 into the loop at maximum pressure. The reaction of I_2 with the organic solvent would invalidate the results of the experiment.

In a three-part experiment, an 0.84 mole % solution of I_2 in cyclohexane was used to determine the reactivity of I_2 with cyclohexane under various conditions. First, 0.5 μl of the solution was injected into the column, and the chromatograph was programmed as shown in Fig. 4.3; second, the helium flow through the injector block was interrupted, and another 0.5 μl of the solution was injected into the block and held for 4 min before the helium flow was restarted. The program was then completed. For the third part, an amount of the I_2-cyclohexane solution

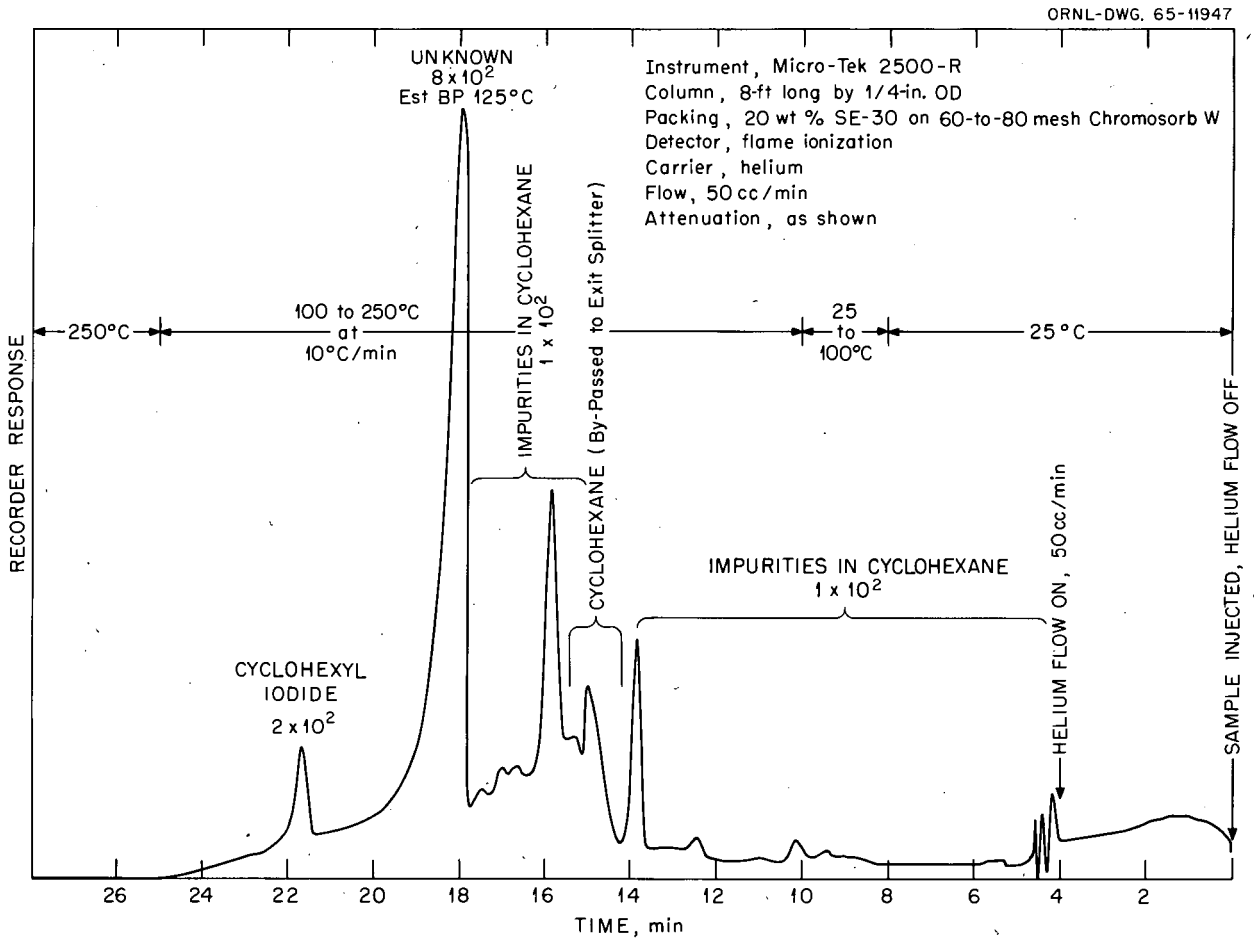

Fig. 4.3. Gas Chromatogram of the Products of the Reaction of Iodine with Cyclohexane. Sample size, 4 μl; sample age, 72 hr; temperature of injector block, 300°C.

that would produce a pressure of 20 atm (abs) at 300°C was sealed into a 2.5-ml quartz ampul. When the ampul was heated to 300°C, the solution was completely decolorized, and a small amount of polymeric solid was found in the ampul. The colorless solution was injected onto the column as in part 1. Figure 4.3 gives the conditions and a chromatogram of the results of this experiment; the data in Table 4.3 show the extent of the reaction of I_2 with cyclohexane.

In a similar experiment, an 0.84 mole % solution of I_2 in benzene was injected into the chromatograph as in parts 1 and 2 above. There was no evidence of products of a reaction of I_2 with benzene.

The results indicate that the I_2-cyclohexane solution is unsatisfactory as a means of injecting I_2 into the reactor loop. The I_2-benzene solution is satisfactory within the stated limits of these tests.

Table 4.3. Results of Gas Chromatographic Determination of Products of the Reaction of Cyclohexane with I_2

Concentration of I_2, 0.84 mole %

Sample Age (hr)	Mole Percent					
	125°C-bp Component		Cyclohexyl Iodide		210°C-bp Component	
	Direct Injection	4 min at 300°C and 3 atm	Direct Injection	4 min at 300°C and 3 atm	Direct Injection	4 min at 300°C and 3 atm
Fresh	0.72	0.14	0.006	0.02	Not found	Not found
24	1.72	0.93	0.001	0.02	Not found	Not found
48	3.52	0.63	0.005	0.04	Not found	Not found
72	4.48	0.74	0.003	0.01	Not found	Not found
96	4.60	0.86	0.001	0.01	Not found	Not found
Heated in sealed quartz cell to 300°C and 20 atm (abs)	Not found		0.10		0.70	Not found

5. Special Research and Development Problems

J. C. White

5.1 GAS CHROMATOGRAPHY

A. S. Meyer

5.1.a Radio-Gas Chromatography of the Tritium-Water-Methane System

A. D. Horton J. M. Schmitt[1]

The tritium-water-methane system is being studied by the Chemical Technology Division to determine the effects of tritium, produced by the Plowshare Experiment, on ground water and natural gas.

A sampling manifold and gas chromatographic analytical train (Fig. 5.1) were designed to permit examination of the products of the reaction of tritium with methane and water. At the start of the experiment, an evacuated 1-liter flask is saturated with water vapor from a mixture of H_2O and ^3H_2O at 25°C. The flask is then filled with methane to 1 atm (abs) pressure. The flask remains at room temperature. The calculated water concentration is 20 μl of liquid water per liter, and the tritium activity is 74×10^5 dis/sec. The sample is transferred from the manifold to the ice-cooled Haloport-Carbowax 20M column in which ^3H_2O and H_2O are trapped. Hydrocarbon gases, including tritiated gases, plus $^3H_2$ and H_2 pass through the water trap and into the silica gel–squalane or the molecular-sieve 13X column. The silica gel–squalane column separates the hydrocarbon gases, and the molecular-sieve column separates (H_2 + $^3H_2$) and methane. Water ($^3H_2O + H_2O$) is eluted by warming the water trap to 60°C. The eluate from the columns passes through the converter furnace, where tritiated compounds are reduced to CO_2 plus $^3H_2$; the activity of the eluate corresponding to each component is determined by scintillation spectrometry. A modified Aerograph Autoprep chromatograph with

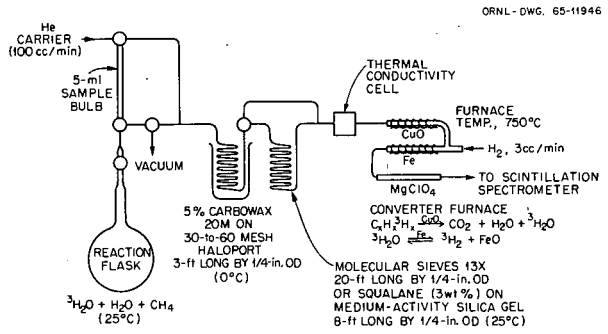

Fig. 5.1. Schematic Diagram of Sampling and Analytical System for the Determination of the Reaction Products of ^3H_2O with Methane.

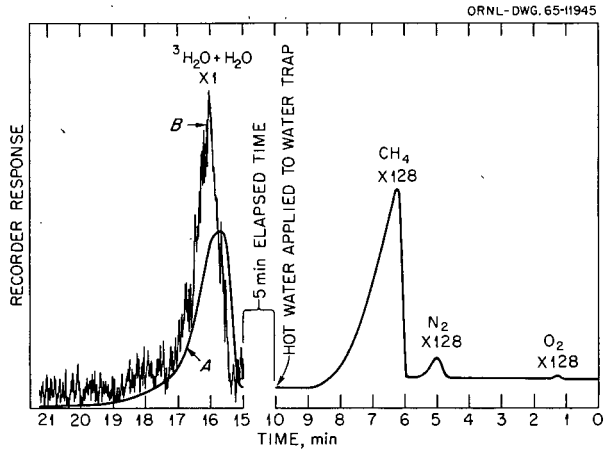

Fig. 5.2. Gas Chromatogram (*A*) and Scintillation Spectrogram (*B*) of the Reaction Product of ^3H_2O with Methane.

a thermal-conductivity detector, a Packard model 325 Tri-Carb combustion furnace, and a Packard Tri-Carb scintillation spectrometer are used to monitor the eluates. The converter furnace has given some difficulty because of the inefficiency of the column of steel wool used to convert ^3H_2O to $^3H_2$. A supply of synthetic ammonia catalyst

[1]Chemical Technology Division.

(iron promoted with silica and zirconia) was obtained from P. H. Emmett;[2] the catalyst is expected to improve the efficiency of the conversion (now ~5%) by severalfold.

The content of the reaction flask has been sampled frequently during a period of several weeks. There has been some evidence of tritium exchange between ^3H_2O and CH_4; however, the behavior of the converter furnace has caused erratic results. Figure 5.2 shows a chromatogram of a 5-ml aliquot of the content of the reaction flask. In this case, there was no tritium activity peak from the methane fraction. The overall efficiency of the conversion of ^3H_2O to $^3H_2$ is about 0.3%. The stated efficiency of the spectrometer for $^3H_2$ is about 10%; therefore, the efficiency of the converter is 5% or less.

5.1.b Pyrolysis and Gas Chromatographic Determination of Styrofoam Dielectric Film

A. D. Horton

A popular gamma dosimeter for personnel protection is in the form of a plastic tube the size of a fountain pen and is worn in the coat pocket. The tube contains a charged Styrofoam dielectric film that discharges in proportion to the intensity of the radiation field and causes an indicating electrometer fiber to move along a scale graduated from 0 to 100 mr. The scale is observed through a magnifying lens in the end of the tube. Some of these dosimeters contained defective dielectric film that would not hold an electrical charge.

Samples of both the defective dielectric film and the good film were pyrolyzed at 900°C. The pyrolyzates were determined by gas chromatography; Fig. 5.3 shows the conditions and the chromatograms of the two films. Methane and ethylene were determined by pyrolyzing separate samples and obtaining chromatograms of the pyrolyzates by use of an 8-ft-long by ¼-in.-OD column that contained 3 wt % squalane on 30- to 60-mesh medium-activity silica gel. Table 5.1 lists the major pyrolyzates of the Styrofoam polymer. No attempt was made to interpret chemically the differences in the compositions of the pyrolyzates of the two samples.

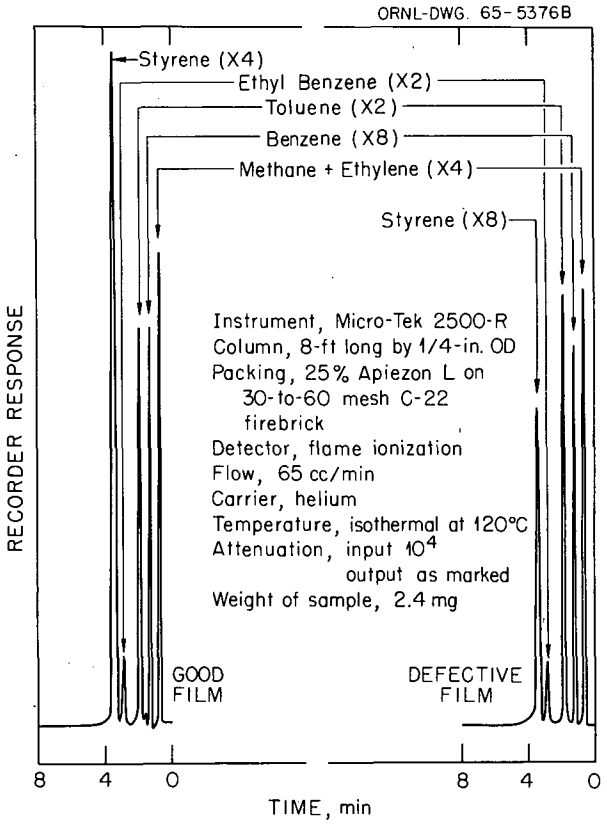

Fig. 5.3. Gas Chromatogram of the Pyrolysis Products of Polystyrene Dielectric Film.

Table 5.1. Pyrolyzates from Good and Defective Styrofoam Dielectric Film

Identity	Amount (mole %)	
	Good Film	Defective Film
Methane	12.7	11.2
Ethylene	9.0	9.7
Benzene	36.5	36.7
Toluene	9.1	10.3
Ethylbenzene	1.8	1.6
Styrene	30.9	30.5

[2]Consultant to the Director's Division; Professor of Chemistry, The Johns Hopkins University, Baltimore, Md.

5.2 STUDIES WITH SOLID INDICATOR ELECTRODES

P. F. Thomason
F. J. Miller H. E. Zittel

5.2.a Glassy-Carbon Indicator Electrode (G.C.E.)

An article was published[3] that describes the completed study of the usefulness of the glassy-carbon electrode (G.C.E.) as an indicator electrode for voltammetry.

5.2.b Oxide Film Formation on the Pyrolytic-Graphite Electrode (P.G.E.)

The investigation of oxide film formation on the pyrolytic-graphite electrode (P.G.E.), done together with D. B. Freeman[4] and G. Mamantov,[5] was completed. The results were published both as a journal article[6] and in more detail by D. B. Freeman as a thesis for the Master of Science degree.[7]

5.2.c Differential Titrimetry with the Pyrolytic-Graphite Electrode (P.G.E.) and the Glassy-Carbon Electrode (G.C.E.)

Study was completed of the use of the P.G.E. and G.C.E. as a coupled electrode system for differential titrimetry. An article that describes the work was submitted for publication.[8]

[3]H. E. Zittel and J. F. Miller, "A Glassy Carbon Electrode for Voltammetry," *Anal. Chem.* **37**, 200 (1965).

[4]Student; Graduate School of Chemistry, University of Tennessee, Knoxville.

[5]Consultant; Assistant Professor of Chemistry, University of Tennessee, Knoxville.

[6]G. Mamantov, D. B. Freeman, F. J. Miller, and H. E. Zittel, "Film Formation on Pyrolytic Graphite Electrodes," *J. Electroanal. Chem.* **9**, 305 (1965).

[7]D. B. Freeman, "Electroanalytical Studies with the Pyrolytic Graphite Electrode," Master of Science thesis, University of Tennessee, August 1965.

[8]F. J. Miller and H. E. Zittel, "Use of the Glassy Carbon–Pyrolytic Graphite Electrode System for Potentiometric Titrations," *Anal. Chem.* (in press).

5.2.d Voltammetry of the Iodine System

The voltammetry of the iodine system in aqueous medium with the P.G.E. was investigated. The results of the investigation are presented in an article to be published.[9] At the P.G.E. a voltage scan of I^- in acid or neutral medium shows three voltammetric waves that correspond to three successive oxidations. The peak potentials for these waves occur at +0.5, +0.9, and +1.2 v vs S.C.E. A solution of I_2 in the same medium exhibits two anodic waves that correspond to the latter two seen when I^- is the electroactive species. The data indicate that the observed anodic waves result from the stepwise reaction

$$I^- \xrightarrow{+0.5\ v} I_2 \xrightarrow{+0.9\ v} IO^- \xrightarrow{+1.2\ v} IO_3^- .$$

5.2.e Chronopotentiometry of the Iodine System

Both the P.G.E. and the G.C.E. were used for a comprehensive study of the chronopotentiometry of the iodine system. The results corroborate and clarify the phenomena observed in an earlier study.[9] The first anodic reaction, $I^- \rightarrow \frac{1}{2} I_2$, is fairly reversible and fits nicely the theoretical treatment of such a reaction. The second anodic reaction, $\frac{1}{2} I_2 \rightarrow IO^-$, is not amenable to the treatment for either a reversible or nonreversible reaction. The τ values obtained and various plots of potential vs τ indicate that the reaction may in itself be reversible, but that it is followed immediately by a chemical reaction. The third wave, $IO^- \rightarrow IO_3^-$, is highly irreversible. After correction, the τ values for the three waves are each proportional to the electroactive species present in the bulk of the solution, be it I^- or I_2. The cathodic reaction, $I_2 \rightarrow 2I^-$, is also highly reversible. In the case of each of the electroactive species the precision and accuracy of measurement are sufficient to warrant use of the method for analytical purposes.

The P.G.E. is more suitable than the G.C.E. for routine use in chronopotentiometry, but the G.C.E. is more durable at high current density and under extreme oxidizing conditions.

The results of this study are being prepared for publication.

[9]F. J. Miller and H. E. Zittel, "Voltammetry of the Iodine System in Aqueous Medium at the Pyrolytic Graphite Electrode," *J. Electroanal. Chem.* (in press).

5.3 STUDIES WITH THE TEFLON DROPPING-MERCURY ELECTRODE (D.M.E.)

P. F. Thomason

5.3.a Polarography in Hydrofluoric Acid and Other Glass-Corroding Media

Helen P. Raaen

Polarography with the Teflon dropping-mercury electrode (D.M.E.)[10] was continued. Four articles on this work were published;[11–14] these are summarized below.

Evaluation of the Horizontal-Orifice Teflon D.M.E. for Obtaining Fundamental Polarographic Data. – The evaluation of the horizontal-orifice Teflon D.M.E. for obtaining fundamental polarographic data was completed. Two reference redox reactions in noncorroding media were used.

The $Tl^+ \rightleftharpoons Tl^0$ Reaction. – Evaluation of the Teflon D.M.E. by means of the $Tl^+ \rightleftharpoons Tl^0$ reaction in 0.1 *M* KCl–1 m*M* HCl is described in a published article.[11]

The $Pb^{2+} \rightleftharpoons Pb^0$ Reaction. – A paper on the use of the $Pb^{2+} \rightleftharpoons Pb^0$ reaction in 1 *M* HCl to evaluate the Teflon D.M.E. was published.[12]

Polarography of the $Pb^{2+} \rightleftharpoons Pb^0$ Reaction in Aqueous Hydrofluoric Acid with the Horizontal-Orifice Teflon D.M.E. – The study of the polarography of the $Pb^{2+} \rightleftharpoons Pb^0$ reaction in aqueous hydrofluoric acid (1 to 12 *M*) with the horizontal-orifice Teflon D.M.E. was completed. An article on this work was published.[13]

Significance of Junction Potentials in the Measurement of $E_{1/2}$ Values. – A Short Communication was published[14] on the significance of junction potentials in the measurement of $E_{1/2}$'s as observed during evaluation of the Teflon D.M.E. This work was done with W. L. Belew.[15]

5.3.b Signal-to-Noise Ratios (S/N) of Glass and Teflon D.M.E. Capillaries

D. J. Fisher[15] W. L. Belew[15]
Helen R. Raaen Bonnie C. Johnson[16]

A study of the signal-to-noise ratios (S/N) of Teflon D.M.E. capillaries relative to that of a typical reference glass D.M.E. capillary was begun. The S/N of D.M.E. capillaries is a factor of interest relative to both the sensitivity and precision of polarography.

The first-derivative dc polarographic technique is the means selected to study the significance of the S/N of D.M.E. capillaries as a factor that limits the ultimate *sensitivity* of polarography. A 1 μM solution of $CdCl_2$ in 0.1 *M* KCl–1 m*M* HCl–0.01% gelatin is used. From the derivative polarograms, the S/N at the peak potential, E_p, is calculated from the following defining relationship:

$$(S/N)_{E_p} = \frac{\overline{(d\bar{i}_f/dt)}_{max}}{\Delta(d\bar{i}_r/dt) + \Delta(d\bar{i}_f/dt)},$$

where $\bar{i}_r$ is the residual current and $\bar{i}_f$ is the faradaic current.[17] The numerator and the ranges that appear in the denominator are measured at E_p from sets of at least five polarograms.

To establish the significance of the S/N in limiting the ultimate *precision* of polarography,

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[10]Helen P. Raaen, "Polarography in Hydrofluoric Acid and Other Glass-Corroding Media," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1964*, ORNL-3750, pp. 18–21.

[11]Helen P. Raaen, "Teflon Dropping-Mercury Electrode for Polarography in Hydrofluoric Acid and Other Glass-Corroding Media. Evaluation with the Thallium(I) $\rightleftharpoons$ Thallium Reaction," *Anal. Chem.* **36**, 2420 (1964).

[12]Helen P. Raaen, "Dropping-Mercury Electrode of Teflon for Polarography in Hydrofluoric Acid and Other Glass-Corroding Media. Evaluation with the Lead(II) $\rightleftharpoons$ Lead Reaction," *Anal Chem.* **37**, 677 (1965).

[13]Helen P. Raaen, "Polarography of Lead(II) in Aqueous Hydrochloric Acid (1 to 12*M*) with a Dropping-Mercury Electrode of Teflon," *Anal. Chem.* **37**, 1355 (1965).

[14]W. L. Belew and Helen P. Raaen, "Effect of Junction Potentials at the Salt Bridge on the Measured Values for the Half-Wave Potentials of the $Pb^{2+} \rightarrow Pb^0$ Reduction in Aqueous Solutions of Hydrochloric Acid and Potassium Chloride," *J. Electroanal. Chem.* **8**, 475 (1964).

[15]Analytical Instrumentation Group.

[16]Summer Student Trainee from Tuskegee Institute, Tuskegee Institute, Ala.

[17]D. J. Fisher, W. L. Belew, and M. T. Kelley, "Recent Developments in DC Polarography," pp. 89–134 in *Polarography 1964* (ed. by G. J. Hills), Macmillian, London, in press.

the subtractive-mode amperometric technique is used. This information will be of particular interest in the field of comparative polarography. The test solution chosen is 1 m*M* $CdCl_2$ in 0.1 *M* KCl–1 m*M* HCl–0.01% gelatin. The fluctuation of the compensated cell current with time at constant potential (–0.850 v vs S.C.E.) is observed on a sensitive current range for 5 to 10 min. The range of fluctuation of the compensated cell current is indicative of the noise under these conditions.

The horizontal-orifice D.M.E. capillaries studied thus far are a 72-μ-orifice-diameter reference (typical performance) glass capillary and 63-μ- and 72-μ-orifice-diameter Teflon capillaries. Residual oxygen present in very small concentrations in the test solution and also vibration and sporadic shocks appreciably decrease the S/N. The significant deduction that can be made from both the polarographic and amperometric data obtained to date is that the S/N of the glass and Teflon capillaries studied are of about the same order.

It is planned to continue the study with additional horizontal-orifice and also with vertical-orifice Teflon capillaries.

5.4 NEW AND MODIFIED METHODS

P. F. Thomason

5.4.a Determination of Titanium in Seawater

T. M. Florence[18]

The Chemical Technology Division has been investigating a method for concentrating uranium from seawater that involves adsorption of uranium on a column of titanium dioxide. A factor in the economics of the process could be loss of TiO_2 by dissolution in the seawater. To check this possibility an analytical method was required that could measure less than 5 ppb of titanium in seawater. This requirement was met by developing a procedure in which titanium in a 1-liter seawater sample is collected on ferric hydroxide carrier and is then separated by extracting the iron carrier and other impurities from 6.5 *M* HCl with a long-chain tertiary amine (Alamine 336). Titanium can then be determined polarographically in an oxalate supporting electrolyte. For a 1-liter sample, the limit of detection is 0.5 ppb, and *S* at the 10-ppb level is 5%.

[18]Alien Guest from Australian Atomic Energy Commission, Sutherland, New South Wales, Australia.

5.4.b Determination of Trace Metals in Niobium-Iron Alloy

T. M. Florence[18]

A sample of Nb(43%)-Fe(57%) alloy was analyzed for trace concentrations of Cu, Bi, Pb, and Zn. The sample was dissolved in the HNO_3-HF mixture, and the niobium was removed quantitatively by anion exchange from 18 *M* HF. Iron was then separated by extraction into methyl *iso*-butyl ketone from 6.5 *M* HCl solution. The trace elements were determined simultaneously by polarography in a CH_3COONH_4-CH_3COOH supporting electrolyte. The detection limits for Cu, Bi, Pb, and Zn in the alloy were 1 to 2 ppm.

5.4.c Spectrophotometric Determination of Uranium with Arsenazo III

T. M. Florence[18]

Arsenazo III is a sensitive reagent for U(VI) (ϵ = 51,600 at 660 mμ), but it lacks selectivity. The selectivity was improved by using cyclodiaminetetraacetic acid as a masking agent and by developing the color at pH 2.0 in a maleic acid buffer. The improved arsenazo III method can tolerate relatively large amounts of vanadium, an element that interferes seriously in many spectrophotometric methods for uranium. This tolerance can be used to advantage in the analysis of uranium ores, such as carnotite, that are high in vanadium. A sample of New Brunswick Laboratory carnotite (No. 4) that contained 0.17 wt % U_3O_8 was analyzed directly by the arsenazo III method after it was dissolved in $HClO_4$-HF; the result was 0.175 ± 0.005 wt % U_3O_8.

5.4.d Determination of Nitrite in a Neutral Nitrate-Containing Solution of Potassium Iodide

A. L. Travaglini

The Solid State Division needed to know whether nitrite was present in a nitrate-containing solution

of potassium iodide. In a neutral solution of iodide, nitrite, and nitrate, no oxidation of iodide by nitrite occurs. Addition of hydrochloric acid results in the immediate oxidation of iodide by nitrite but not by nitrate. The iodine liberated is determined by titration with a standard solution of sodium thiosulfate; from the titer, the amount of nitrite present is calculated. In the solution under study ~93 ppm of NO_2^- was present.

5.4.e Spectrophotometric Determination of Nitrated 4-*sec*-Butyl-2(α-methylbenzyl)phenol (BAMBP)

A. L. Travaglini

The Chemical Technology Division requested an analysis of 4-*sec*-butyl-2(α-methylbenzyl)phenol (BAMBP) to determine the degree of nitration by nitric acid that had occurred during an extraction of cesium with BAMBP. All samples of nitrated BAMBP show a strong absorption peak between 365 and 370 mμ. Similar compounds, such as 2,4-dinitrophenylphenol, exhibit no absorption in this region. BAMBP reacts with sodium nitrite in perchloric acid medium to give only nitrated BAMBP. This reaction was used to make a spectrophotometric calibration curve. The concentration of nitrated BAMBP in the standard solution was determined polarographically. The standards produced a straight-line calibration curve to an absorbance of 1.50; the curve passes through the origin. Depending on the nitration to which the BAMBP had been subjected, from 0 to 18 wt % nitrated BAMBP was found.

5.4.f Determination of Iodine and Fluorine in KIO_2F_2

A. L. Travaglini

The Chemistry Division requested the determination of iodine and fluorine in KIO_2F_2 before its crystal structure was studied. When dissolved in water, KIO_2F_2 hydrolyzes to KIO_3 and HF. The HF formed was determined by adding an aliquot of the unknown solution to an excess of a standard solution of base and then titrating the excess base with a standard solution of hydrochloric acid. Iodine was determined by adding excess potassium iodide and a little hydrochloric acid to an aliquot of the unknown solution. The liberated iodine was titrated with a standard solution of sodium thiosulfate. From this titer, the amount of iodine present in the compound was calculated. The amounts of I and F found experimentally were 98.9 and 99.3%, respectively, of the theoretical amounts based on the formula KIO_2F_2.

5.4.g Determination of Uranium(VI) in Uranium Oxide–Thorium Oxide Fuels

A. L. Travaglini

The Reactor Chemistry Division requested the analysis of UO_2-ThO_2 fuels for U^{6+}. The fuels were dissolved in a degassed equivolume mixture of concentrated sulfuric and phosphoric acids by heating to ~90°C under argon. The inert atmosphere prevents the oxidation of U^{4+} to U^{6+}. The U^{6+} was determined polarographically at an $E_{1/2}$ of −0.17 v vs S.C.E. by the method of Kubota.[19] The U^{6+} content of the various fuels ranged from 2.4 to 24 wt %.

5.4.h Spectrophotometric Thiocyanate Determination of Niobium in the Presence of Fluoride

D. C. Canada

Niobium in the presence of fluoride was determined rapidly by a modified thiocyanate method. Aluminum was used as the fluoride-complexing agent. The optimum range for the determination of niobium was extended to 35 μg per final 25-ml volume used for the spectrophotometric measurement. Interferences are MO^{3+}, W^{6+}, Ti^{3+}, V^{4+}, and $C_2O_4^{2-}$. The stable $NbOF_5^{2-}$ complex does not react with thiocyanate to produce the characteristic niobium-thiocyanate absorption spectrum. A plot of the Al^{3+}:F^- mole ratio vs the absorbance of the niobium-thiocyanate complex indicates that the formation of the thiocyanate complex is initiated at a ratio of 0.5 and is quantitatively complete at a ratio of 1.0. For 22 analyses, S is <2.3%.

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[19] H. Kubota, "Determination of the Stoichiometry of Uranium Dioxide. Polarographic Determination of Uranium(VI) in Uranium Dioxide," *Anal. Chem.* **32**, 610 (1960).

Table 5.2. Composition of Commercial Alkylbenzenesulfonate Detergent

Component		Analytical Method
Identity	Weight Percent	
Dodecylbenzenesulfonate	38.3	Ultraviolet and methylene blue spectrophotometry
Na^+	22.9	Flame spectrophotometry
Cl^-	13.2	Potentiometric titration with Ag^+
SO_4^{2-}	23.8	Volumetric titration with $BaCl_2$ and Thoron indicator
HCO_3^-	0.1	Volumetric titration with acid to bromocresol green and methyl red end points
H_2O	2.0	Azeotropic distillation with volume measurement
Total	100.3	

5.4.i Spectrophotometric Heteropoly-Blue Determination of Silicon

D. C. Canada

The spectrophotometric heteropoly-blue method for silicon was studied, because the color of the reduced silicomolybdic acid occasionally fades. According to the original method, an aqueous solution of stannous chloride reductant is added to an immiscible butyl alcohol extraction solution. The use of the miscible reductant, 1-amino-2-naphthol-4-sulfonic acid, has corrected the problem of color instability.

5.4.j Determination of Nitrate by Controlled-Potential Coulometric Titration

D. C. Canada

An investigation of accurate methods for determining nitrate included a study of a coulometric titrimetric method.[20] The titration is based on the catalytic reduction of nitrate to nitrogen by U^{3+}. The readout leads from a controlled-potential coulometric titrator are connected to the Y-input leads of an X-Y recorder. Time is plotted on the X axis. The titration is carried out in a solution of sodium perchlorate. Uranium(III) is added in the form of $UO_2(ClO_4)_2$. Prereduction is accomplished at -0.7 v vs S.C.E., and the titration is carried out at -1.0 v vs S.C.E. In the range from 0.1 to 0.5 mg of NO_3^- in the titration cell, the error is <0.5%; from 1 to 5 mg, it is <1%.

[20] L. R. Duncan, *Determination of Nitrate by Controlled-Potential Coulometry*, HW-SA-2168 (Feb. 23, 1963).

5.4.k Analysis of Alkylbenzenesulfonate Detergents

D. C. Canada

The determination of constituents in commercial detergents (alkylbenzenesulfonates) was investigated. The analysis for ionic sulfate in these materials is often inaccurate, because alkylbenzenesulfonates precipitate with barium. The presence of water in some of these detergents eliminates a Parr-bomb fusion for total sulfur content. Ethyl alcohol was used to separate Na_2SO_4, NaOH, $NaHCO_3$, and Na_2CO_3 as solids from the alkylbenzenesulfonate. The separated salts were then dissolved in water and were determined by standard analytical methods (Table 5.2). By use of this separation procedure, a standard was prepared from a commercial detergent. The analytical results are presented in Table 5.2.

5.4.l Determination of Oxidizing Power of Solutions of Hydrolyzed Xenon Hexafluoride

J. R. Lund A. L. Travaglini

Members of the Chemistry Division studied the change in the oxidizing power of a dilute solution of barium nitrate while it was in contact with a slowly hydrolyzing sample of xenon hexafluoride. Periodically, the oxidizing power of this solution was determined by treating a test portion with a quantity of hydrochloric acid and potassium iodide and then titrating the liberated iodine with a standard solution of thiosulfate. The oxidizing power of the first sample was of the order of 0.01 meq/ml (S = 2%); two months later the oxidizing power had increased to 0.05 meq/ml (S = 1%).

5.4.m Absorption Spectral Measurements

J. R. Lund

Solar-Cell Windows. – To study the effect of solar radiation on solar-cell windows and on their epoxy adhesives, members of NASA from Langley Field, Virginia, used the 86-in. cyclotron as their source of solar-type radiation. Initially, the spectra of the windows and adhesives were taken from 0.3 to 2.6 μ. Then the cyclotron was used to provide a dose of radiation equivalent to a year of exposure in space. The spectra were again recorded. Whereas some of the materials remained transparent over the 0.4-to-2.6-μ region, others became very opaque in the 0.8-to-2.6- and 0.4-to-0.45-μ regions.

Ribonuclease. – On fermentation, *Bacillus subtilis* produces ribonuclease and other proteins. Relative to foam separation studies being made in the Chemical Technology Division, the production of ribonuclease was followed by periodically recording the spectrum from 0.2 to 0.3 μ.

Alipal-LO-436. – In connection with desalination and related process studies, the buildup or depletion in the concentration of Alipal-LO-436, which is the ammonium salt of a sulfonated straight-chain alkylphenoxypoly(ethyleneoxy)ethanol, was followed by examining the spectrum from 0.2 to 0.45 μ.

5.4.n Technetium Studies

F. J. Miller

The chemistry of technetium in its lower valence states is the object of current study.

6. Analytical Biochemistry

J. C. White

6.1 TRANSFER RIBONUCLEIC ACID (tRNA) ANALYTICAL STUDIES

C. A. Horton

In July 1964 a program was initiated to aid the Biology and Chemical Technology Divisions with the analyses required in the production of purified specific transfer ribonucleic acids (tRNA) from *Escherichia coli* bacteria.[1]

The studies continued on methods to optimize, increase efficiency, and automate the ^{14}C-amino acid assay of column fractions by use of modifications of an earlier manual method.[2] The precision and ease of operation of various automatic and semiautomatic micro volumetric pipets and dispensers usable for the reagents of this assay were determined. The precisions attainable were compared with the precisions of Lang-Levy micropipets

[1] J. C. White, C. A. Horton, and I. B. Rubin, "Analytical Biochemistry," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1964*, ORNL-3750, p. 29.

[2] R. J. Mans and G. D. Novelli, "Measurement of the Incorporation of Radioactive Amino Acids into Protein by a Filter Paper Disc Method," *Arch. Biochem. Biophys.* **94**, 48 (1961).

and of ordinary graduated pipets used previously. Of those tested, the Labindustries Repipet (1-ml size with special reservoir, catalog No. 3001-SP, Labindustries, Berkeley, California) and the Servall-Sterling automatic pipet (1-ml liner, 40-rpm speed, catalog No. 4-3M, Ivan Sorvall, Norwalk, Connecticut) seem best; the relative standard errors of deliveries made with them were 2.8 to 15 and 0.74 to 1.26%, respectively, at the 95% confidence level. Various types of chromatographic papers available in rolls are being studied for use in a continuous washing and drying cycle. Of those tested, Whatman 3MM remains the best. The required spacing of various samples on paper strips to prevent overlap of radioactive areas was established with a Vanguard paper-radiochromatogram scanner, but the efficiency of this type detector is inadequate for ^{14}C for its use on actual samples. Attempts were made to carry out the complete aminoacylation reaction directly on the paper disks or strips instead of in solution; the results were erratic, even when the newest method suggested by Cherayil and Bock[3] was used.

Certain other possible methods for the assay of tRNA fractions were studied briefly. These included: direct and differential ultraviolet and visible spectrophotometry, infrared spectrophotometry of potassium bromide pellets of freeze-dried samples, optical rotatory dispersion of aqueous solutions, fluorescence of acidity-controlled aqueous solutions, conductivity measurements, and separation of hydroxamates formed from the aminoacyl-tRNA adducts by use of the Loftfield-Eigner[4] separation followed by paper chromatography of the hydroxamates. None of these techniques seemed promising.

The Lowry method[5] was unsatisfactory for determining traces of protein in tRNA; measurement of the absorbance at 280 mμ was not sufficiently sensitive.

Various nucleotides, polynucleotides, tRNA, and deoxyribonucleic acids form a ternary complex or adduct with the fluorescent aluminum-morin complex and thus cause increases and decreases in fluorescence depending on the types of heterocyclic bases present. A paper on this study is being prepared. Use of this effect to estimate various specific types of tRNA will be investigated soon.

6.2 STUDIES ON THE PHENYLALANYL TRANSFER RIBONUCLEIC ACID (tRNA) SYSTEM

I. B. Rubin

A number of the parameters involved in the phenylalanine-phenylalanyl tRNA charging reaction were studied to establish the optimum conditions under which the reaction occurs. This general method of determining specific tRNA's has been studied previously,[1] but essentially the same conditions had been used in the reactions to determine any of the individual tRNA's. This work was done in support of the Biology and Chemical Technology Divisions' project for the manufacture of crude tRNA and the subsequent separation of this product into its constituent parts. Kelmers, Novelli, and Stulberg[6] found that, by reverse-phase chromatography, phenylalanyl tRNA can be separated in a relatively uncontaminated state from a crude starting product; therefore, it was decided to study the phenylalanyl tRNA system to obtain the most nearly possible quantitative determination of this nucleic acid. Among the variables studied were time, pH, buffer type and concentration, concentration of various divalent cations, and concentration of sodium chloride.

The tRNA charging reaction in which a ^{14}C-labeled amino acid was used was first mentioned by Hoagland, Zamecnik, and Stephenson[7] and since has been modified a number of times, but most notably by Bollum,[8] who suggested the paper-disk method. The basic procedure is described by Kelmers, Novelli, and Stulberg.[6] Several of the variables are interrelated (e.g., time and divalent metal ion concentration), and although optimum

[3] J. D. Cherayil and R. M. Bock, "A Column Chromatographic Procedure for the Fractionation of s-RNA," *Biochemistry* 4, 1174 (1965).

[4] R. B. Loftfield and Elizabeth A. Eigner, "A Radioactive Hydroxamate Method for Determining Rates of Amino Acid Activation," *Biochim. Biophys. Acta* 72, 373 (1963).

[5] O. H. Lowry, N. J. Rosebrough, A. L. Farr, and R. J. Randall, "Protein Measurement with the Folin Phenol Reagent," *J. Biol. Chem.* 193, 265 (1951).

[6] A. Kelmers, G. Novelli, and M. Stulberg, "Separation of Transfer Ribonucleic Acids by Reverse Phase Chromatography," *J. Biol. Chem.* 240, 3979 (1965).

[7] M. B. Hoagland, P. C. Zamecnik, and Mary L. Stephenson, "Intermediate Reactions in Protein Biosynthesis," *Biochim. Biophys. Acta* 24, 215 (1957).

[8] F. J. Bollum, "Thermal Conversion of Nonpriming Deoxyribonucleic Acid to Primer," *J. Biol. Chem.* 234, 2733 (1959).

conditions for the phenylalanyl tRNA charging reaction were apparently found, a number of anomalies were also discovered. The optimum incubation time for the reaction was 25 to 30 min when the magnesium concentration in the reaction mixture was 0.018 to 0.02 *M*. A constant incubation temperature of 37°C was used in all these studies. The presence of manganese not only provided essentially the same results as did magnesium, but with manganese, time and concentration were not so critical. However, manganese tended to precipitate at the higher concentrations. Calcium gave results only about 20% as good as those with magnesium; results with cobalt were about the same as with calcium. No charging took place in the presence of nickel or copper, and iron and zinc both precipitated at the pH used. The tRNA charging reaction is normally carried out in a solution buffered with 0.10 *M* tris-(hydroxymethyl)-aminomethane at pH from 7.4 to 8.0. A study of the effects of pH on 0.02-*M*–acetate-buffered solutions in the pH range from 4.2 to 5.8 and on 0.01-*M*–phosphate-buffered solutions in the pH range from 6.2 to 7.8 indicated that in the latter range there were no essential differences when compared with control samples. In the acetate solutions, there was virtually no reaction at pH 4.6 or below; the reactions at pH 5.4 and above were only 90% complete. The effect of varying the acetate concentration was not studied, but changing the phosphate concentration from 0.001 to 0.01 *M* did not seem to have any effect. Since the phenylalanyl tRNA is eluted from the chromatographic column at a sodium chloride concentration of 0.70 to 0.80 *M* in the eluant, the effect of the concentration of this salt was also studied. As little NaCl as 0.25 *M* was inhibitory, yet as one of the anomalies of this study, results from dialyzed and undialyzed pooled fractions of the phenylalanyl tRNA chromatographic peak were inconsistent, depending on the quantity of ^{14}C-labeled amino acid added. Also for these pooled samples, a fourfold excess of ^{14}C-phenylalanine was needed, whereas for the peak fractions before they were pooled, only a twofold excess of the labeled amino acid was required. The amount of charging enzyme needed was also studied, but this amount seemed to be a property of the individual enzyme preparation. Although the same procedure is used each time in making the suspension of the crude enzyme, the product is not necessarily consistent.

Further work on this project will depend on the progress of the chromatographic separation procedure. Optimum conditions for the determination of other aminoacyl tRNA's will be established. It is also intended to study the analysis of RNA for other constituents – proteins and carbohydrates in particular.

6.3 GAS CHROMATOGRAPHY OF PYROLYTIC PRODUCTS OF PURINES AND PYRIMIDINES

C. B. Honaker[9]

The gas chromatography of the pyrolytic products of purines and pyrimidines was initiated in an attempt to develop a rapid method for determining the heterocyclic bases produced in the degradation of nucleic acids. The method of choice at present is an ion exchange separation followed by spectrophotometric determination – a time-consuming procedure that requires rather elaborate instrumentation. Three goals were set for the first phase of the pyrolysis–gas chromatographic study: to establish whether characteristic pyrochromatograms could be obtained for the different bases, to identify as many of the pyrolysis products as possible, and to determine a quantitative relationship between a major pyrolytic product and the parent compound.

The pyrolyzer used in this study was developed by C. B. Honaker[9] and A. D. Horton. It consists of a small tube furnace mounted directly on the sample-inlet port of a Micro-Tek GC-2500R chromatograph. The sample is inserted magnetically into the hot zone, and carrier gas flowing through the furnace sweeps the pyrolysis products onto the column. A thermal conductivity detector is used to determine inorganic gases; a hydrogen flame ionization detector is used for all other runs. Three columns were used. Carbon monoxide and carbon dioxide were determined on a 4-ft-long by ¼-in.-OD silica gel (high-activity) column. A 20-ft-long by ¼-in.-OD tricresyl phosphate column (20% on 42-to-60-mesh firebrick) was used for low boilers, and a 12-ft-long by ¼-in.-OD QF-1 column (15% on 60-to-80-mesh Chromosorb W) was used for less volatile substances. Pyrochromatograms

[9]Research Participant, summer 1965, from Department of Chemistry, Tennessee Wesleyan College, Athens, Tenn.

Table 6.1. Pyrolysis (880°C) Products of Heterocyclic Bases

Original Compound	Pyrolysis Products (wt %)						
	CO	CO_2	CH_4	C_2H_4	C_2H_2	CH_3CN	C_2H_5CN
Adenine	0.73		<0.1	<0.1		1.65	
Xanthine	7.21	21.37	0.24	0.46	0.10	1.00	
Guanine	5.31	4.73	<0.1			0.76	
Uracil	7.55	6.38	2.88	0.73	0.56	16.26	0.34
Cytosine	2.54	5.11	0.50	<0.1	0.44	8.19	
5-Methylcytosine hemihydrate	3.77	2.85	8.11	4.40	0.79	4.25	0.35

were determined for uracil, 5-methylcytosine hemihydrate, cytosine, adenine, guanine, and xanthine.

Seven pyrolysis products were identified and quantitatively determined. These are listed in Table 6.1. Most samples were pyrolyzed at a furnace temperature of 880°C. Some comparison runs, in which the QF-1 column was used, were made at a pyrolysis temperature of 680°C. No change was apparent in the nature of the products. However, the amounts of products coming through the column increased drastically as the pyrolysis temperature increased. This change was more marked with the purines than with the pyrimidines.

[10]G. F. Smith, "The Destruction of Viscera in Toxicological Investigations. Determination of Chromium," pp. 48–51 in *Mixed Perchloric, Sulfuric, and Phosphoric Acids and Their Applications in Analysis*, 2d ed., G. Frederick Smith Chemical Co., Columbus, Ohio, 1942.

6.4 DISSOLUTION OR DECOMPOSITION OF VARIOUS ORGANIC MATERIALS

J. R. Lund

Smith's "liquid fire" method[10] has been useful for decomposing enzymes, fats, and proteins prior to the determination of phosphorus and trace-metal impurities for the Biology Division. For the destruction of substances that were, or that contained, "fatty" material, the more time-consuming sulfuric acid–nitric acid treatment, followed by final decomposition with nitric acid–perchloric acid, was used. Practically all materials free of fats, such as organic extractants that contained phosphorus, were decomposed by the cautious use of nitric acid–perchloric acid alone.

7. X-Ray and Spectrochemical Analyses

7.1 X-RAY AND SPECTROCHEMICAL ANALYSES (X-10)

M. T. Kelley
Cyrus Feldman

7.1.a X-Ray Analysis

H. W. Dunn S. B. Lupica

Analysis of Solids by X-Ray Fluorescence. – With the aid of R. B. Bullock,[1] a CDC 1604-A code was written for analyzing multicomponent alloys and mixtures by x-ray fluorescence. The basic approach of Burnham, Hower, and Jones[2] was extended to cover eight-component alloys. Polished specimens of NBS steel and nickel-base alloys were used to provide the data for calculating coefficients of interaction between components (a_{ij}). When the entire group of alloys was used to obtain these coefficients, results accurate to within a few percent of the amount present were obtained for individual steels, nickel-base alloys, and pure specimens of each of the component metals. (Burnham, Hower, and Jones found it necessary to derive separate coefficients for each type of alloy, by use of appropriate standards, and could not provide for compositions outside the standard range.) Applicability of the method to powdered mixtures and rough-surfaced specimens is now being checked.

Electron Probe Analyzer. – The detection of very light elements (boron to calcium) by the electron probe analyzer depends on the use of a multilayer lead stearate film as an x-ray diffraction grating. The *d* spacing of the film supplied is not known exactly; therefore, the identification of these elements must be done on an empirical matching basis. The *d* spacing of the film has been measured to permit identifications to be made on the basis of absolute x-ray wavelengths.

Several computer codes for converting x-ray count rates directly into composition information are being examined.

Determination of Areal Density of Gold Coating on Silicon Radiation Detectors by Use of X-Ray Fluorescence. – Standards were prepared by depositing successive layers of gold leaf of known areal density ($\mu g/cm^2$) on a Mylar film, placing this film atop an uncoated silicon button, and measuring the x-ray fluorescent intensity of the radiation from the gold. The accuracy of this method of calibration for samples of vapor-deposited gold was checked by chemically dissolving the gold coating from a known area of sample and determining total gold by atomic absorption. The results of the two determinations (~ 50 $\mu g/cm^2$) agreed to within experimental error.

7.1.b Atomic Absorption

Cyrus Feldman N. Marion Ferguson

Determination of Gold, Silver, and Copper. – The atomic absorption of solutions of gold in dilute nitric acid was investigated using the 50-cm-long end-fed absorption tube, hollow cathode, and the detection equipment described previously.[3] Since the gold hollow cathode is very stable, a 2× scale expansion was used, and a sensitivity of 0.03 $\mu g/ml$ was achieved. However, nickel and iron present in the solution reduced the sensitivity of analysis, presumably by retarding the vaporization of gold.

In analogous tests with silver and copper, the detection limit was ~ 0.01 $\mu g/ml$ for each, that is, a concentration of ~ 0.01 $\mu g/ml$ gave an absorbance of 0.01, which is easily distinguishable from the "no absorption" condition.

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[1] Mathematics Division.

[2] H. D. Burnham, J. Hower, and L. C. Jones, "Generalized X-Ray Emission Spectrographic Calibration Applicable to Varying Compositions and Sample Forms," *Anal. Chem.* **29**, 1827 (1957).

[3] S. R. Koirtyohann and C. Feldman, "Atomic Absorption Spectroscopy Using Long Absorption Path Lengths and a Demountable Hollow Cathode Lamp," pp. 180–89 in *Developments in Applied Spectroscopy*, vol. 3, ed. by J. E. Forrette and E. Lanterman, Plenum, New York, 1964.

7.1.c Spectrographic Analysis of Radioactive Materials

Determination of Chromium in Radioactive MSRE Salt (W. R. Musick, Cyrus Feldman). – To determine chromium spectrographically in radioactive MSRE salt (a mixture of LiF, ZrF_4, BeF_2, UF_4, and impurities) at concentrations as low as 10 ppm, the chromium must be separated from the salt.[4] Extraction with tri-*n*-octylphosphine oxide (TOPO) appeared to be the best way to do this, but no way was known to back-extract the chromium from the organic layer.[5] None of the acid solutions tried for this purpose was consistently effective, but 0.5 *M* NaOH solution performed the task promptly and completely.

The procedure for determining chromium in MSRE salt consists in fusion with ammonium bisulfate, evaporation of the ammonium bisulfate, and dissolution of the residue in hydrochloric acid. Traces of iron and molybdenum are extracted from this solution and are determined spectrographically; zirconium and uranium are extracted and discarded. Next, chromium is extracted from the sample solution with TOPO, recovered with sodium hydroxide solution, and determined spectrographically.

Tests of the procedure were run with 1.5-g samples of chromium-free MSRE salt. Three samples of this material, to each of which 250 μg of chromium had been added, were analyzed as above; recoveries were 250, 250, and 256 μg.

Separation of Curium from Contaminant Metals (T. C. Rains,[6] W. R. Musick). – In the determination of contaminant elements in curium, it is desirable to separate the contaminants from curium before determining their concentration. This step makes the final determinations both safer and more sensitive than they would be if the original solution were analyzed directly. Extraction of curium with di-(2-ethylhexyl)phosphoric acid (HDEHP) was studied as a means of effecting this separation; ^{152,154}Eu tracer was used as a substitute for curium. The extractant was a 0.3 *M* solution of HDEHP in dodecane.[7,8] The data of Table 7.1 show the acid dependence of the extraction of ^{152,154}Eu in hydrochloric acid and in nitric acid.

Since chloride is incompatible with the sample solutions expected, distribution coefficients were obtained for a number of the contaminant metals extracted from 0.1 *N* HNO_3. Table 7.2 gives the results, which indicate that some of the contaminant metals would also be extracted into the organic layer if the rare-earth elements were

Table 7.1. Acid Dependence of the Extraction of ^{152,154}Eu Tracer by 0.3 *M* Di-(2-ethylhexyl)phosphoric Acid in Dodecane

Acid Concentration (*N*)	E_a^o For HCl	E_a^o For HNO_3
0.1	>1000	274
0.2	63	50
0.5	3.0	2.0
1.0	0.2	0.2

Table 7.2. Distribution Coefficients for the Extraction of Common Elements from 0.1 *N* HNO_3 by 0.3 *M* Di-(2-ethylhexyl)phosphoric Acid in Dodecane

Element	E_a^0
Pb	0.25
Mn	0.13
Ca	0.15
Al	0.48
Mg	0.01
Cu	0.04
Fe	>1000
Cr	0.026
Ni	0.028

[4]C. Feldman and W. R. Musick, "Spectrochemical Analysis of Radioactive Samples in a Remote-Control Facility," *Analytical Chemistry in Nuclear Reactor Technology, Second Conference, Gatlinburg, Tenn., Sept. 29–Oct. 1, 1958*, TID-7568 (Part 2), pp. 91–95.

[5]J. C. White and W. J. Ross, *Extraction of Chromium with Trioctylphosphine Oxide*, ORNL-2326 (July 22, 1957).

[6]Present address: National Bureau of Standards, Washington, D. C.

[7]T. V. Healy, "Rapid Solvent Extraction Methods for Fission Product Separation and Analysis," *Radiochim. Acta* 2(2), 52 (1963).

[8]J. J. McCown and R. P. Larsen, "Radiochemical Determination of Total Rare Earths by Liquid-Liquid Extraction," *Anal. Chem.* 33, 1003 (1961).

extracted under these conditions. However, the E_a^o values indicate that an increase of acidity to 0.2 *N* might provide a clean separation, except in the case of iron.

7.1.d Flame Photometry

T. C. Rains[6] N. Marion Ferguson
Cyrus Feldman

Collection of Barium from Tap Water. – The concentration of barium in natural waters is too low to permit its direct determination in the sample as received. A carrier compound was sought that, when precipitated, would coprecipitate all the barium present in 1 liter of water.

It was found that if 10 mg of Mg^{2+} is added to 1 liter of tap water and is precipitated as $MgNH_4PO_4$, 95 to 98% of the barium present is coprecipitated. The carrying efficiency is enhanced by the presence of calcium in the solution.

7.1.e Tissue Analysis

F. S. Jones

The program for the spectrographic analysis of human tissue was continued; the procedures[9] described previously were used. Analytical results for 544 soft-tissue samples analyzed for 23 elements were forwarded to the University of Tennessee for compilation in their computer program. The samples included some taken during autopsies done at Richmond, Virginia, and Baltimore as well as numerous small organs, such as lymph nodes and thyroid and adrenal glands, which types had not been analyzed previously because of their small size.

A way was needed to pulverize large samples of wet tissue so that a small analytical sample representative of the whole could be obtained. The sampling of large organs would thus be improved, and it would be possible to prepare aggregate samples large enough for distribution to different laboratories for comparison purposes. A hammer mill made by the Pulverizing Machinery Company of Summit, New Jersey, was installed and tested on beef fat, bone, and muscle that had been frozen and mixed with Dry Ice. Fat gave a white powder; muscle, a fine shredded fiber. As long as the product was kept frozen, it could be mixed and blended by use of simple equipment.

A blender was constructed that consists of an inclined rotating platform on which a plastic tub containing the pulverized tissue and chunks of Dry Ice is rotated at 20 rpm. Initial tests with pulverized fat that contained a fluorescent powder indicated adequate mixing. Contamination in the pulverizer and blender is negligible, because only carbon steel or plastic comes in contact with the sample. Analysis of the residue from sublimed Dry Ice revealed no significant contamination from that source.

Gamma-ray sterilization and freeze-drying are being investigated as ways to prevent spoilage and to facilitate the handling of the blended samples.

7.1.f Semiquantitative Spectrographic Analysis (Visual Interpolation)

Zell Combs C. A. Pritchard
Anna M. Yoakum

A survey of semiquantitative methods of spectrographic analysis was begun to determine by which method the best possible accuracy is obtainable without the use of separate standards for each type of sample. Three common universally applicable methods were tested: the graphite-dilution, Harvey,[10] and Kroonen-Vader Li_2CO_3 methods.[11] In the first, 1 mg of powdered sample is diluted with 10 mg of graphite; line intensities in the spectrum of this mixture are compared with those produced by standard mixtures. In the second, 10 mg of powdered sample is mixed with 10 mg of graphite. Quantitative estimates are made in terms of known threshold-concentration values for the appearance of a given impurity line in a

[9] S. R. Koirtyohann and C. Feldman, "The Spectrographic Determination of Trace Elements in Human Tissue," *Analytical Chemistry in Nuclear Reactor Technology, Fourth Conference, Gatlinburg, Tenn., Oct. 12–14, 1960*, TID-7606, pp. 51–63.

[10] C. E. Harvey, *A Method of Semi-Quantitative Spectrographic Analysis*, Applied Research Laboratories, Glendale, Calif., 1947.

[11] J. Kroonen and D. Vader, *Line Interference in Emission Spectrographic Analysis*, Elsevier, New York, 1963.

given matrix. The Kroonen-Vader method consists in dilution of the sample with graphite and Li_2CO_3 and comparison of the spectra of sample and standards.

Twelve standard samples were analyzed by each of the three methods. These samples included steels, other alloys, and minerals. Metals were dissolved and ignited to the oxides before analysis; other materials were analyzed as received. All concentrations were estimated visually.

The results (Table 7.3) were classified on the basis of the ratio of the concentration found to the true concentration, the larger value always being the numerator. For the samples tested, the Kroonen-Vader method is indicated to be superior to the other two.

Table 7.3. Comparison of Accuracy of Semiquantitative Spectrographic Methods by Use of Visual Estimation of Line Intensities

Method	Percent of Quantitative Estimates Accurate to Within a Factor of		
	2	3	4
Carbon-dilution	35	52	61
Harvey		48	55
Kroonen-Vader	48	75	87

The use of densitometric measurement will be investigated in connection with the above methods, and other universally applicable methods will be studied.

7.2 X-RAY AND SPECTROCHEMICAL ANALYSES (Y-12)

A. E. Cameron
J. A. Carter

7.2.a Determination of Lead, Bismuth, Cadmium, Tin, and Zinc in Airborne Dust Samples

J. A. Carter

A solution technique was established for determining microgram quantities of Bi, Cd, Pb, Sn, and Zn in airborne dust collected on 1-in.-diam filter paper. The paper is wet ashed, and the sample solution is analyzed on the Paschen Direct Reader. With the Sr 4077.71-A line as internal standard, calibration curves were prepared from standard solutions for the following element lines: Pb 2833.07 A, Pb 4057.82 A, Bi 3067.72 A, Cd 3261.06 A, Sn 3175.02 A, and Zn 3345.02 A. The equivalent concentration range encompassed by this technique is from 4 to 100 μg of metal per cubic meter of air sampled.

8. Mass Spectrometry

A. E. Cameron

8.1 SPARK-SOURCE MASS SPECTROMETRY

J. R. Sites J. A. Carter

The Associated Electrical Industries, Ltd. (AEI) MS-7 spark-source mass spectrometer (Fig. 8.1) was put into service in Building 9734 on November 1, 1964. This spectrometer is a Mattauch-type double-focusing instrument designed specifically for the analysis of inorganic materials. The instrument uses an rf spark ion source and photographic-plate ion detection. The spectrum from mass 7 to 240 or any lower range with a mass ratio of 35 : 1 can be recorded on a single exposure. The analyzer pressure is maintained at about 10^{-8} torr through the use of differential pumping, source-isolation valving for rapid sample changing, and photoplate magazine valving.

With a 5-mil slit, the resolution of the instrument is 1900 at mass 208. The instrument can detect boron in concentration as low as 2 ppb in high-purity silicon metal.

Fig. 8.1. Photograph of AEI MS-7 Spark-Source Mass Spectrometer Installed at ORNL.

This instrument has been used for survey and semiquantitative impurity analyses at the ppb and ppm levels on some 45 types of samples of pure metals, compounds, and alloys, which included: KCl, LiF, LuF_3, Sc_2O_3, ScF_3, Nd_2O_3, Sm_2O_3, Tb_2O_3, Tm_2O_3, TmF_3, La_2O_3, Eu_2O_3, EuF_3, Tb, Nd, Tb-Sc, Sn-Ce, Sn-Li, Sn-Li-Ce, Al-Li, AlN, Fe, Al, Re, Ta, W, Cr, Ni, Cu, Sn, Ag, C, Nb, AgCl, ZrO_2-UO_2, U, UO_2, Cerrobend alloy, INOR-8, stainless steel, Waspaloy, and Inconel.

Many of the samples are nonconductors and must, therefore, be mixed with a conducting material and pressed into a sample rod for analysis. To render a nonconducting sample conducting, the sample is mixed with carbon, gold, or silver, and the mixture is pressed into rods. A tantalum-lined die is used with carbon and gold at low pressures (500 to 1500 psi). For rare-earth compounds and refractory materials, isostatic compressing at higher pressures (20,000 to 40,000 psi) in a naphthalene mold is used; pure silver powder is the conductor-binder. A die was constructed from Teflon for making the naphthalene mold. A $\frac{1}{8}$-in.-diam rod forms the cavity in the naphthalene mold in which the silver-sample mixture is put. The loaded mold is sealed with hot naphthalene, covered with an evacuated plastic bag, and pressed. The naphthalene is then completely removed in hot acetone, and the sample is vacuum dried.

Figure 8.2 shows the die, the mold, and a pressed sample.

The ion-beam images on the exposed photographic plates are viewed on a densitometer. A simple qualitative method for estimating the amount of impurity is to determine visually the exposure for the weakest image of an isotope of the impurity

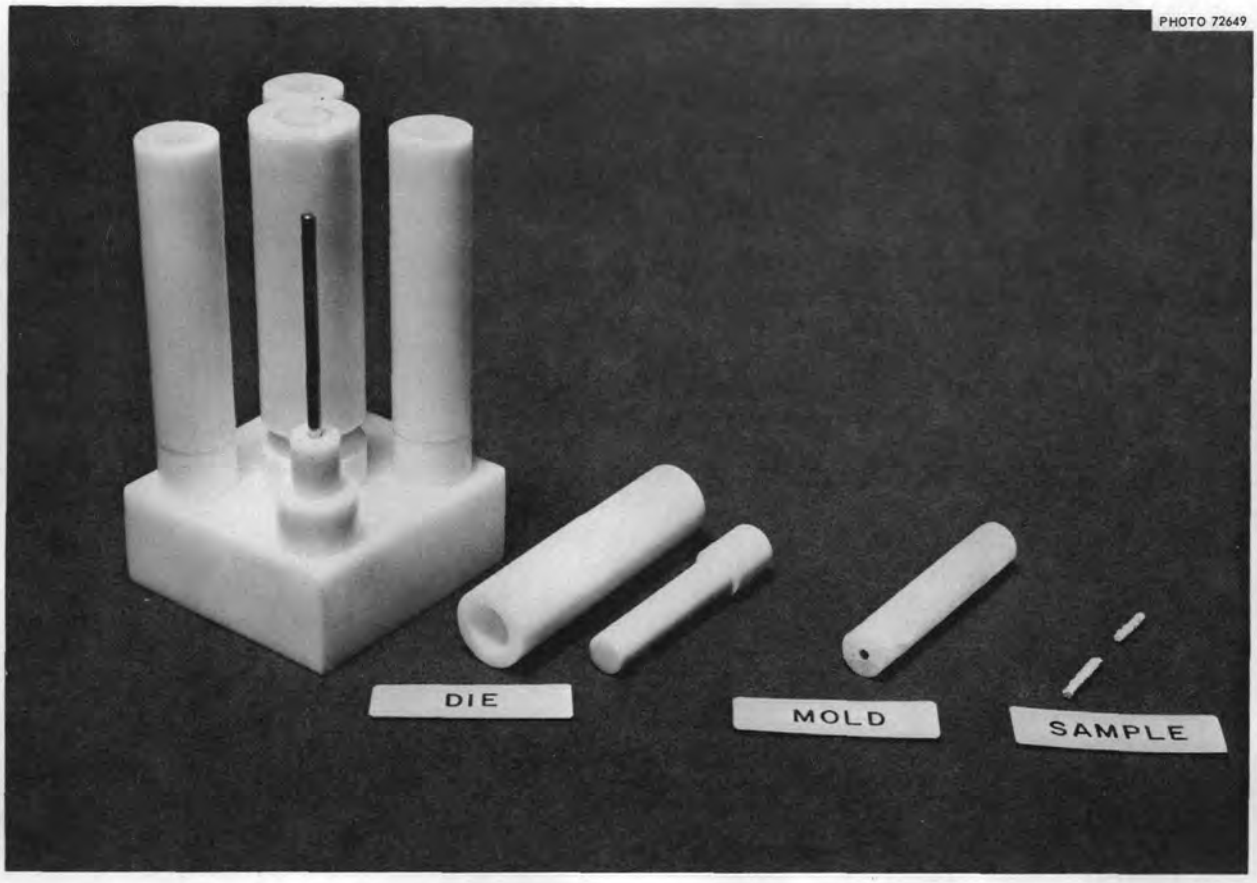

Fig. 8.2. Isostatic-Pressing Die, Mold, and Pressed Sample.

element. A more quantitative way is to make, for an isotope of each impurity, a plot of percent transmission vs exposure on seven-cycle semilogarithmic graph paper. The relative exposures at a chosen transmission are then used to calculate the impurity concentration. One also needs to take into account relative isotopic abundances of the internal standard and of the impurities, relative elemental emulsion sensitivities, mass effect along the emulsion, and relative areas under the peaks on the densitometer recorder trace.

A program for the ORNL CDC 1604 computer is used to reduce the time needed to calculate the impurity concentrations.

This laboratory participated in the MS-7-users cooperative analysis program on an unknown copper sample. The purpose of the test was to determine the reproducibility of the spark-source technique after having established relative sensitivities for seven impurities in a copper standard. Instrumentation parameters, sample-etching techniques, and photoplate data handling were specified briefly. Fourteen laboratories participated. Table 8.1 gives a summary of the results; their wide range is believed to be caused by sample inhomogeneity.

Samples of rare-earth metals, oxides, and halides have been analyzed. To eliminate the isomer problem of the mass spectra of the oxide ions of the lower rare earths being on top of those of the metal ions of the higher rare earths, data taken in the doubly ionized mass region are used. The limit of detection of contaminant rare earths in a pure rare-earth sample is 0.1 ppm or less.

Table 8.1. Summary of Results of MS-7-Users Copper Cooperative Analyses

Identity of Results	Metal Impurity (μg per gram of Cu)						
	Cr	Ga	Ag	Sn	Sb	Pb	Bi
Highest reported	130	135	145	202	160	244	370
Lowest reported	25	43	44	67	62	72	120
Certified[a]	60	80	150	120	120	100	130
ORNL	66	120	67	100	96	80	120

[a]Johnson, Matthey & Company, Ltd., standard sample CA-2.

8.2 TRANSURANIUM ELEMENT (TRU) MASS SPECTROMETRY

R. E. Eby

Isotopic analyses on uranium and transuranium samples are now done routinely in the TRU Laboratory. A total of 482 samples were analyzed during the year, and over 1400 results were reported. All transuranium elements through californium were analyzed. Samples also included "hot" europium for burnup calculations and various types of thorium samples. In conjunction with A. Chetham-Strode,[1] the half-lives of californium isotopes were checked by mass spectrometric analyses. Results obtained were in good agreement with published values.[2]

Effort has been directed toward reducing the possibility of spreading contamination and reducing costs of decontaminating source parts. Expendable filament insulators have been obtained, and a disposable stainless steel liner for the source chamber was designed and used. The source plates are now plated with nickel prior to use; after they are used, the plating can be easily dissolved to remove most of the contamination.

[1]Chemistry Division.

[2]R. E. Eby, "Isotopic Analysis of Transuranium Elements by Mass Spectrometry," presented at the Ninth Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Oct. 12–14, 1965.

8.3 SURFACE IONIZATION STUDIES

G. R. Hertel[3]

To learn more about thermal ionization and to determine the sources of discrimination in mass spectrometers used for analysis of solids, some investigations were initiated into ion optics and thermal ionization, both of previously deposited samples and of molecular beams impinging on a hot filament.

One interesting problem under study is a real, but very small, bias observed in uranium isotopic analyses of very small samples ($\lesssim$300 ng). The percent ^{235}U appears to increase as the sample size decreases. Available data indicated that a second variable – temperature – might also account for the observed bias. Therefore, a series of controlled experiments were made in which the sample size was held constant while the temperature was varied over a 300°C range. No bias was observed; thus, the possibility that temperature is the responsible parameter was eliminated. Although negative, this result is significant in that it implies the existence of complications in the process of surface ionization that are little understood. A new approach to this problem is under investigation.

In somewhat related studies, the ions produced when a molecular beam strikes a hot surface are being analyzed as a function of the temperature of the surface. Because of the strong influence exerted on the ion currents by trace impurities, especially hydrocarbons, investigations were delayed until the completion of an all-metal, VacIon-pumped vacuum system capable of maintaining a pressure of $\sim 2 \times 10^{-8}$ torr during experimental runs. At temperatures above $\sim$2200°K, the data obey the Saha-Langmuir equation, as expected. Below 2200°K, marked deviations, now always reproducible, are observed. A study of the low-temperature region is contemplated in which controlled amounts of "impurities" will be admitted, and their effects on the ion currents then studied.

In the course of this work, a survey of the available mass range revealed K_η^+ ions (where η = 1 to 9) with the odd-numbered species more abundant, in general, than the even-numbered species. The

[3]AEC Postdoctoral Fellow from the Johns Hopkins University, Baltimore, Md.

ion Na^+ was also observed but apparently does not polymerize easily. No molecular sodium ions were detected; Rb^+ was present in very small amounts, and a trace of Rb_2^+ was observed. Higher polymers were beyond the mass range and sensitivity of the instrument. These ions arise from the alkali-metal impurities in the filament materials.

A quadrupole lens was constructed, and several modifications of the existing Nier-type lens were tested in the mass spectrometer, but, to a first approximation, no great improvement over the present source was noted.

8.4 ISOTOPIC COMPARISON OF LEAD FROM TEKTITES AND CONTEMPORANEOUS IMPACT CRATERS

D. H. Smith J. M. Wampler[4]

An isotopic study of lead in tektites and in material from impact craters that are related in age to the tektites[5] was carried out to examine the hypothesis that tektites are formed from terrestrial materials as a result of the impact of asteroids or comets on the earth's surface.[6] Previous isotopic studies have shown that the major groups of tektites contain lead that is similar in isotopic composition to that typically found in the younger rocks of the earth's crust but provide no positive evidence that tektites are formed from terrestrial material.[7,8]

The Ivory Coast tektites are similar in age to glasses from the Lake Bosumtwi crater in Ghana. Since this crater occurs in Precambrian rocks, it was anticipated that lead from the local rocks might differ in isotopic composition from most terrestrial lead, and if similar lead were found in Ivory Coast tektites, a fairly positive correlation between the two materials would be established. The Rb-Sr relationships suggest an older source for the Ivory Coast tektites than for the major groups.[9] Isotopic analysis of lead from two Ivory Coast tektites shows a significantly high proportion of ^{204}Pb relative to lead from the other tektite groups, and also relative to most terrestrial lead having a similar $^{206}Pb/^{207}Pb$ ratio. Lead from a sample of phyllite that represents the most common rock type at Lake Bosumtwi is similar in isotopic composition, but lead from impact glass found at Lake Bosumtwi is enriched in the radiogenic isotopes relative to lead from the Ivory Coast tektites.

The isotopic composition of lead in Czechoslovakian tektites (moldavites) was compared with that of lead from raw and purified suevite (impact glass) from the Ries Kessel of Nordlingen, Germany. Lead from the raw sample was similar to lead in the moldavites, whereas lead from the purified suevite was enriched in the radiogenic isotopes.

Since there are notable chemical differences between the impact glasses and the associated tektites, the observed differences in lead isotopic composition are not inconsistent with the hypothesis that both were formed of terrestrial materials. The similarity of lead in moldavites and in raw suevite may be fortuitous, but the similarity between lead from Ivory Coast tektites and from Lake Bosumtwi phyllite suggests strongly that these tektites were formed of terrestrial material during the formation of the crater.

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[4]Research Participant from Georgia Institute of Technology, Atlanta, Ga.

[5]R. L. Fleischer, P. B. Price, and R. M. Walker, "On the Simultaneous Origin of Tektites and Other Natural Glasses," *Geochim. Cosmochim. Acta* **29**, 161 (1965).

[6]A. J. Cohen, "Asteroid – or Comet – Impact Hypothesis of Tektite Origin: The Moldavite Strewn-Fields," pp. 189–211 in *Tektites* (ed. by J. A. O'Keefe), University of Chicago Press, Chicago, 1963.

[7]G. R. Tilton, "Isotopic Composition of Lead from Tektites," *Geochim. Cosmochim. Acta* **14**, 323 (1958).

[8]I. E. Starik, E. V. Sobotovich, and M. M. Shats, "The Problem of the Origin of Meteorites and Tektites," *Geochemistry (USSR) (English Transl.)* 261 (1963).

[9]C. C. Schnetzler, W. H. Pinson, and H. W. Fairbairn, "Rb-Sr Analyses of Two Ivory Coast Tektites" (abstract P32), *Trans. Am. Geophys. Union* **46**, 118 (1965).

9. Infrared and Nuclear Magnetic Resonance Spectrometry

J. C. White
P. F. Thomason

9.1 INFRARED SPECTROMETRY

Lucy E. Scroggie M. M. Murray
J. R. Lund

Infrared spectrometry was used to examine over 420 samples of 64 types (Table 9.1). Most studies involved the detection and/or identification of impurities in commercial solvents, process streams, or compounds prepared at ORNL; the identification of commercial products; the characterization of organic compounds; or the identification of structural changes caused by irradiation or chemical degradation of both organic and inorganic compounds.

Several compounds prepared at ORNL, such as perfluorovinyl tin, perfluoroacrylic acid, 2,6-dioximinocyclohexanone, dipiperidyl, lauric acid chloride, 3,9-diethyltridecanone-6, 3,9-diethyltridecanamine-6, and ^{10}B diborane were examined for structure and purity. The formation and destruction of hydroxyl, carbonyl, carboxyl, nitro, nitroso, and nitrate-ester groups by irradiation or by nitric acid degradation of such compounds as di-*sec*-butylphenylphosphonate, tributyl phosphate, butyl-α-methylbenzylphenol, methanol–hydrochloric acid solutions, terpenes, and diethylbenzene were studied.

Commercial products such as solvents (diethylbenzene and *n*-butanol), lubricating oils, vacuum-pump oils, an epoxy paint component, a coating for protecting electrical equipment, strippable coatings used in decontamination work, dielectric films from radiation dosimeters, extractants (phosphorus compounds, sulfoxides, and amines), and surfactants were analyzed for impurities or to identify components. Smears of oil from different locations in machinery were studied to identify the type of oil and thus the source of the leak from which it originated. An organic contaminant found in drinking water was identified as the salt of an alkyl carboxylic acid. Alkyl phthalates were found in such diverse materials as a Vycor frit and an acetone leach of Tygon tubing. A silicone compound was identified in hydrofluoric acid solutions stored in polyethylene bottles.

Semiquantitative or quantitative determinations were made of ethylene glycol–water in engine oils and fuel oil, ketone carbonyl group of acetophenone in benzene and diethylbenzene solutions, phenolic -OH group in di-*sec*-butylphenylphosphonate solutions, and oil in water from the ORNL steam plant.

9.2 NUCLEAR MAGNETIC RESONANCE SPECTROMETRY

Lucy E. Scroggie

In June 1965 a Varian DP-60 (dual-purpose 60-Mc) nuclear magnetic resonance (NMR) spectrometer was made available to the Analytical Chemistry Division. The spectrometer system incorporates special modulation and detection equipment to obtain high sensitivity for broad-line spectra and yields a resolution of three cycles at 60 Mc for narrow line (high resolution) spectra. It is equipped with a fixed-frequency rf unit designed for studying ^{1}H nuclei and can be converted for studying ^{19}F nuclei. High resolution NMR operation permits identification of individual elements in a compound, chemical functional groups, the nature of molecular arrangements, and isotopic substitution sites. Molecular structure, potential barriers, degree of isotopic substitution, hydrogen bonding, chemical kinetics, tautomeric equilibria, and molecular conformations are examples of fields that can be investigated.

Before the DP-60 NMR spectrometer was put into operation, a Varian A-60 NMR spectrometer in the Chemistry Division was used to obtain the spectra of 15 mixtures of furfuryl alcohol and methyl alcohol, some of which contained additives

Table 9.1. Types of Samples Examined by Infrared Spectrometry

Organic	
Acetophenone-diethylbenzene	Commercial lubricating oils
Benzoic acid–diethylbenzene	Oils from MSRE pumps
Bromotrifluoroethylene	Oil smears
Soluble ribonucleic acids	Long-chain primary alcohol
Tributyl phosphate–Amsco	Engine oils
Ethyl hypobromite–CCl_4	High-temperature coating
Perfluorovinyl tin	Water-soluble coating
Perfluoroacrylic acid	Electrical-equipment coating
Iodomethane	Sorbitan monooleate–2-ethylhexanol
Butyl-α-methylbenzylphenol–Amsco	Nitrated diethylbenzene fractions
Bicyclic alcohols–CCl_4	Dielectrics from dosimeters
Fluorinated hydroxyhydrocarbons	Commercial cleanser (HP220)
2,6-Dioximinocyclohexanone	Silicone from HF solutions
Trioctylphosphoric triamide	Oil in steam-plant water
Cyclohexane-iodine mixtures	Fuel oil
Long-chain sulfoxides	Alkyl phthalate from Vycor frit
Diethylbenzene	Alkyl phthalate from Tygon tubing
Dipiperidyl	Terpenes
Amine O–2-ethylhexanol	*p*-Ethylbenzoic acid
Lauric acid chloride	Alkyl aldehyde
n-Butanol	Nonadecanamine-1
3,9-Diethyltridecanamine-6	Ion exchange resins (Li and H forms)
3,9-Diethyltridecanol-6	Commercial tertiary amine (Andogen 364)
3,9-Diethyltridecanone-6	
Di-sec-butylphenylphosphonate–diethylbenzene	
Alkyl carboxylic acid salt from drinking water	
Commercial epoxy paint component (Epothane C)	
Solids and $ZnBr_2$ solutions from HRLAF windows	
Inorganic	
$^{10}B_2H_6$	$CsBrO_3$ (irradiated)
LiOBr	$Ca(BrO_3)_2$ (irradiated)
Phosphate ions on alumina	$KClO_3$ (irradiated)
BF_3	Ge (single crystals)
Nb_2O_5	$Th(NO_3)_4$ solutions
Praseodymium germanium molybdate	Nd_2O_3
Dysprosium germanium molybdate	

being evaluated for their efficiencies in preventing the polymerization of the furfuryl alcohol at various temperatures. Some of the spectra exhibited weak peaks close to the bases of the main peaks and a decrease in the height of the peak of the proton at carbon-4 of the furan ring. Bridging evidently occurred between carbon-4 of one ring and the methylene on carbon-1 of another ring.

The spectra of 15 long-chain (8 to 18 carbons) glycerol monoethers were obtained for the ORINS Medical Division. Some of these ethers were studied at 50°C, because they were solid at room temperature.

Three fractions of 3,9-diethyltridecanone-6 and two fractions of methyl methanesulfonate, prepared by the Analytical Chemistry Division for the Chemical Technology Division and Biology Division, respectively, were studied by NMR spectrometry. Small amounts of the starting materials were detected in the ketone, and a trace of solvent was found in one sulfonate sample.

The NMR spectra of several column chromatographic fractions of nitrated *m*-diethylbenzene were obtained for the Chemical Technology Division. One sample was mainly *m*-(α-nitroethyl)ethylbenzene; some *m*-ethylacetophenone and at least one other compound were present as impurities. One of the minor impurities has been purified further by preparative-scale gas chromatography and derivative formation. Although only a small amount was available for analysis, it was identified as *m*-(α-hydroxyethyl)ethylbenzene. Infrared and NMR analyses were used earlier in the identification of a very pure sample of *p*-(α-nitroethyl)-ethylbenzene from the nitration products of *p*-diethylbenzene. Two very dilute (1% or less) solutions of *p*-ethylbenzoic acid in carbon tetrachloride were examined as part of this study of the nitration products of diethylbenzene.

The spectra of commonly used organic compounds are being recorded for reference and for comparison with published spectra.

10. Optical and Electron Microscopy

M. T. Kelley
T. E. Willmarth
T. G. Harmon H. W. Wright

10.1 ELECTRON MICROSCOPY OF RADIOACTIVE MATERIALS

10.1.a Electron Microscope Facility for Studying Radioactive Materials

The facility (in room 201, Building 3019) for the preparation and examination of radioactive materials by electron microscopy and diffraction began operation in December 1964. It has been possible to increase the maximum level of radioactivity of samples accepted and handled successfully, the limit now being that which conforms with the established ORNL Radiation Safety and Control Standards. The radiation limit of 100 r/hr per gram of powdered samples that contain beta or gamma activity, which was predicted at the beginning of the work, has been exceeded with no attendant difficulties. Radiation contamination of the sample-preparation laboratory and of the electron microscope (column and stage) has been so slight that decontamination was accomplished easily. There has been no radiation overexposure to operating personnel.

Studies have been made of material from fuel elements, urania-thoria powders, and irradiated and unirradiated magnesium oxide and aluminum oxide. A method is being developed for preparing thin foils from bulk samples of irradiated stainless steel for electron microscopy.

A facility for electron microscopy of highly radioactive alpha-emitting samples, including the

transuranium elements, is now being planned as one of the functions of the proposed High-Level Alpha Laboratory. Proper methods of handling and containment of such materials for microscopic examination, as practiced at other United States AEC installations, are being studied.

10.2 NUCLEAR SAFETY PROGRAM

Examination continued of particulates from experiments designed to simulate fallout of radioactive materials from possible accidents at experimental or industrial reactors. From the plasma-torch burnup of nuclear fuels, samples of particulates collected in the various sampling devices used in the Nuclear Safety Pilot Plant[1] were observed and photographed, for record, in the electron microscope. Similar studies were made for groups in the Reactor Chemistry Division doing experimental work to promote nuclear safety. Also, polyvinyl chloride filters were evaluated for use in collecting radioactive particulates from hot moist air.

10.3 METHODS DEVELOPMENT FOR ELECTRON MICROSCOPY

Methods were developed for: removing particulates collected on cellulose triacetate filters; extracting carbide precipitates from Nb-W-Zr alloys by a carbon-film technique[2] [Fig. 10.1 shows an (Nb-Zr)C precipitate extracted by the method]; removing inclusions (carbides, oxides, nitrides, etc.) from metals by gelatin extraction; direct carbon replication of powders that off-gas during dissolving; and etching and replicating surfaces of metals having a high oxidation rate.

An experiment is in progress to determine whether plate glass that has been exposed to a high-flux neutron source is harder and more durable than unirradiated plate glass. Initial hardness tests on

Fig. 10.1. Segmented Platelets of (Nb-Zr)C Extracted from Nb-W(10%)-Zr(1%)-C(0.1%) by the Carbon-Film Technique.

strips of plate glass exposed to a ^{60}Co source for several days indicate that the surface becomes harder. Other samples are now being exposed for longer times. If the irradiated glass is harder and more durable, it could be used in ultramicrotomes to replace the expensive diamond knives now used.

10.4 ELECTRON AND OPTICAL MICROSCOPY AND ELECTRON DIFFRACTION RESEARCH ASSISTANCE TO OTHER DIVISIONS

The Chemistry, Chemical Technology, Health Physics, Isotopes, Physics, Reactor, and Reactor Chemistry Divisions were assisted in research problems requiring optical and electron microscopy and electron diffraction. Materials of interest in nuclear technology that were studied included: Nb-W-Zr alloys, niobium, and stainless steels (304 and 316); aluminum films, fibers, flakes, powders, and amalgams; aluminum oxidized in air, in water, and in steam; iron oxide sols; iron oxide–thoria sols; calcium hypophosphate; sodium chloride; aerosols of stearic acid and of sulfur; glass microbeads; glass exposed to arc discharges; UO_2–stainless steel vaporized in a plasma torch; UO_2 vaporized by arc discharge in air, in helium, and in argon; Millipore Microweb filters; Metricel Acropor filters; silver membranes;

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[1] P. P. Holz and T. H. Row, "Sampling Devices Used in Nuclear Safety Pilot Plant," Aug. 2, 1965 (unpublished report).

[2] T. E. Willmarth, "Electron Optical Study of the Effect of Aging on Carbides in a Niobium-Tungsten-Zirconium Alloy," *Proceedings of the 19th AEC Metallography Group*, ORNL-TM-1161, in press.

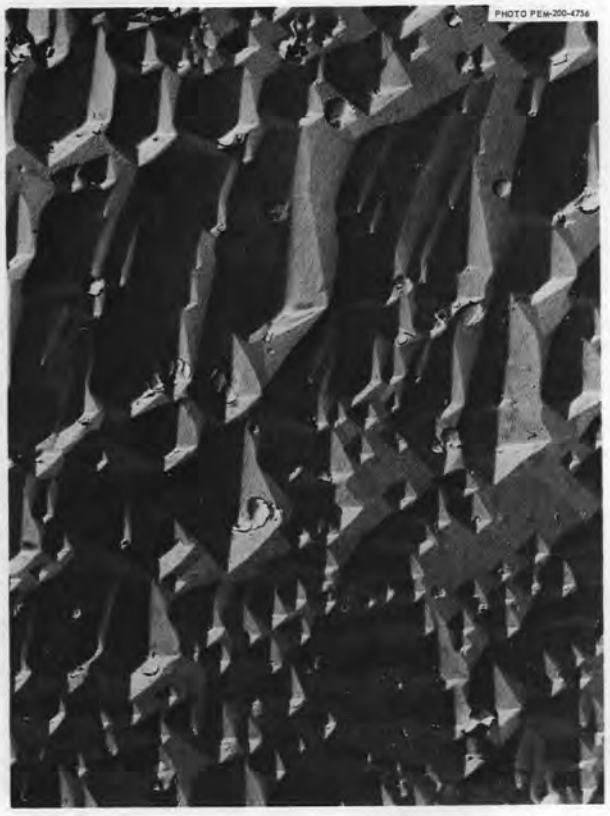

Fig. 10.2. Ultrastructure of Fibrous Platinum Membrane Used for the Adsorption of Iodine. Magnification, 9000×; photographic enlargement, 3×. Reduced 56%.

Teflon and glass capillaries; Teflon gaskets from desalination experiments; Xerox toners; Latex spheres; and platinum membranes. Figure 10.2 shows the replicated surface structure of a platinum membrane used for iodine adsorption.

10.5 PERSONNEL EDUCATION

To keep abreast of improvements in technologies directly related to optical and electron microscopy, members of the group attended classes conducted by industries in ultramicrotomy at New Orleans, Louisiana, and in ultrafiltration technology at Chattanooga, Tennessee.

11. Nuclear and Radiochemical Analyses

J. C. White
W. S. Lyon

11.1 NUCLEAR PROPERTIES OF RADIONUCLIDES

11.1.a ^{123}I

J. S. Eldridge

A cooperative effort was established with two groups in the Isotopes Division to characterize production-scale batches of various new isotopes. One such isotope is ^{123}I produced in the ORNL 86-in. cyclotron by the proton bombardment of ~200 mg of 79.2%-enriched ^{123}Te according to the reaction ^{123}Te$(p,n)^{123}$I. A previous attempt to produce ^{123}I from 49%-enriched ^{123}Te gave a product that contained too much ^{124}I and ^{126}I.[1]

Gamma-ray spectrometry was used to determine the total yield of ^{123}I. The first and second production runs gave ^{123}I in 220- and 170-mc yields, respectively. Decay measurements of the products indicated that the half-life of ^{123}I is 13.0 hr. No radioactive species other than iodine isotopes were detected in either product. The

[1] J. S. Eldridge, "I^{123}," *Anal. Chem. Div. Ann. Progr. Rept. Dec. 31, 1962*, ORNL-3750, pp. 85–86.

average amount of ^{124}I produced by the (p,n) reaction on the 5.1% ^{124}Te present in the target was ~0.85% of that of the ^{123}I. Similarly, the amounts of ^{125}I and ^{126}I were <0.08 and ~0.22% of that of the ^{123}I. The recovered tellurium target is low in residual radioactivity and is suitable for reirradiation.

Certain characteristics of ^{123}I, such as short half-life, electron-capture decay, and low-energy gamma emission, make it attractive for use in diagnostic studies of thyroid disorders. These preliminary production runs show that a product can be made which should be satisfactory for use in medical research when ^{123}I is advantageous.

This work was done in collaboration with J. E. Beaver[2] and H. L. Hupf.[2]

11.1.b ^{197}Hg

J. S. Eldridge W. S. Lyon

The properties of 24-hr ^{197m}Hg and 65-hr ^{197}Hg that affect the accuracy of ^{197}Hg radioassay were investigated because of assay discrepancies reported by several laboratories. At first, the primary method of production of ^{197}Hg consisted in the irradiation of 1 g of natural mercury in the LITR.[3] The reaction was $^{196}Hg(n,\gamma)^{197m,197}Hg$. This process gave an ^{197}Hg product whose specific activity was ~0.5 curie/g. Since natural mercury contains 0.46% ^{196}Hg and 29.8% ^{202}Hg, it was necessary to determine the 47-day ^{203}Hg content produced by neutron capture in ^{202}Hg. Interferences by both ^{197m}Hg and ^{197}Hg in the 279-kev photopeak of ^{203}Hg made this determination difficult. The establishment of gamma-ray energies and intensities for ^{197}Hg required the preparation of a source of ^{197}Hg that was free of ^{203}Hg.

Carrier-free ^{197}Hg was produced free of ^{203}Hg by the reaction $^{197}Au(\alpha,4n)^{197}Tl$, with subsequent electron-capture decay of the 2.8-hr ^{197}Tl to ^{197}Hg. This bombardment was accomplished in the ORIC. Additional samples of carrier-free $^{197m,197}Hg$ were produced by proton bombardment of gold in the ORNL 86-in. cyclotron according to the reaction $^{197}Au(p,n)^{197m,197}Hg$.

Decay measurements of a series of samples of both the ^{197m}Hg and the ^{197}Hg isomer were made with an NaI spectrometer system and a gross-gamma well-type detector. Table 11.1 shows the results of six independent decay studies made from different ^{197}Hg products and measured with different systems. The individual values shown are the results of a weighted least-squares fit of the decay data determined by use of the Brookhaven CLSQ Decay Curve Analysis Program.

Similar studies were done with cyclotron-produced ^{197m}Hg; a gamma-ray spectrometer was used to determine the individual peak counting rates in three independent determinations. Table 11.2 gives the results of these determinations.

The availability of 40%-enriched ^{196}Hg, coupled with an increased use of ^{197}Hg as an aid in locating brain tumors and in studies of kidney and spleen disorders, led to an alternative standard production method. An experimental production run of ^{197}Hg was made from 40%-enriched ^{196}Hg.

Table 11.1. Results of Determination of Half-Life of ^{197}Hg

^{197}Hg-Production Method	Measurement System	Half-Life (hr)
(p,n)	Gamma-ray spectrometer	64.12 ± 0.10
		64.23 ± 0.44
	Well counter	63.98 ± 0.17
(n,γ)	Well counter	64.19 ± 0.20
$(\alpha,4n)$	Well counter	63.94 ± 0.14
		64.20 ± 0.08
	Weighted mean and error	64.14 ± 0.05

Table 11.2. Results of Determinations of Half-Life of ^{197m}Hg

Photopeak Measured (kev)	Half-Life (hr)
130	23.72 ± 0.08
279	24.13 ± 0.24
279	23.99 ± 0.27
Weighted mean and error	23.8 ± 0.1

[2]Isotopes Division.

[3]ORNL Isotopes Catalog, 4th Rev., p. 43, Oak Ridge National Laboratory (April 1963).

The target was 0.1 mg of HgO; it was irradiated for 61 hr at a flux of 2×10^{14} neutrons cm^{-2} sec^{-1} in the ORR. By gamma-ray spectral and decay measurements, the product was shown to have the requisite radiochemical purity. In addition, the ^{203}Hg content of the product was reduced to $<1 \times 10^{-2}$% of the ^{197}Hg. The specific activity of ^{197}Hg produced by this new method is ~2000 curies/g.

11.1.c Gamma-Ray Branchings in ^{237}U

J. S. Eldridge

Several high-specific-activity samples of ^{237}U were obtained from the Fluoride Volatility Pilot Plant. The absolute disintegration rate, as well as the photon emission for the prominent gamma rays, was measured. It was thus possible to calculate gamma-ray branchings for ^{237}U. A lithium-drifted germanium diode detector[4] was used to isolate the 374-kev gamma ray, which is not separated by NaI spectrometry. Decay measurements are being made on four samples of this material to determine the half-life of ^{237}U.

11.1.d Half-Lives of Long-Lived Beta Emitters

Gerald Goldstein

A liquid-scintillation method for the absolute counting of beta emitters[5,6] was used to measure the half-lives of long-lived beta emitters. Determination of the half-life of ^{99}Tc was described previously.[5]

^{36}Cl. – The partial half-life for beta decay of ^{36}Cl was determined by the specific-activity method with high-specific-activity ^{36}Cl (32.6 at. %) obtained from the Isotopes Division. A solution of the ^{36}Cl was prepared and was analyzed chemically by micropotentiometric titration and radiochemically by the liquid-scintillation method and by 4π beta counting in a proportional counter. The measured half-life values were 3.06 (±0.02) $\times 10^5$ years (liquid-scintillation counting) and 3.10 (±0.04) $\times 10^5$ years (4π beta counting). These values are in excellent agreement with the presently accepted value, 3.08 (±0.03) $\times 10^5$ years. A report on this measurement will be published.[7]

^{10}Be. – The half-life of ^{10}Be was last measured in 1947[8] with a sample having extremely low specific radioactivity (0.005 at. %). This half-life value is of some significance, because ^{10}Be is produced in meteorites by cosmic radiation and is used to calculate such values as radiation ages and age of deep-sea sediments.

A piece of beryllium reflector was obtained that had been used in the Oak Ridge Research Reactor for some months and should therefore contain ^{10}Be produced by the $^{9}Be(n,\gamma)^{10}Be$ reaction. After extensive chemical purification of the beryllium reflector, a BeO sample was prepared from the reflector which showed radioactive impurities in the beryllium that amounted to only 4 dis min^{-1} mg^{-1}. This sample was analyzed chemically and was counted by the liquid-scintillation method; the Be content was 8.72 mg/ml and the beta radioactivity was 9.39 (±0.08) $\times 10^4$ dis min^{-1} ml^{-1}. From the mass spectrometry value of 0.026 (±0.002) at. % of ^{10}Be, the half-life was calculated to be 1.91 (±0.15) $\times 10^6$ years; the value previously measured is 2.6×10^6 years. We are presently trying to obtain a more precise value for the atom fraction of ^{10}Be.

11.1.e Nuclear Spectroscopy of Neutron-Deficient Hafnium and Rare-Earth Radionuclides

T. H. Handley

To obtain more evidence on the systematic behavior of nuclear-energy levels, a number of neutron-deficient radionuclides in the rare-earth region

[4] J. S. Eldridge, "Gamma-Ray Spectroscopy with Lithium-Drifted Germanium Diode Detectors," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1964*, ORNL-3750, pp. 43–44.

[5] G. Goldstein, "Absolute Counting of Beta Emitters by the Liquid-Scintillation Method," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1964*, ORNL-3750, pp. 51–53.

[6] G. Goldstein, "Absolute Liquid-Scintillation Counting of Beta Emitters," *Nucleonics* 23(3), 67 (1965).

[7] G. Goldstein, "Partial Half-Life for Beta Decay of ^{36}Cl," accepted for publication in the *Journal of Inorganic & Nuclear Chemistry*.

[8] E. M. McMillan, "Energy and Half-Life of the Be^{10} Radioactivity," *Phys. Rev.* 72, 591 (1947).

were studied with internal-conversion spectrographs. Measurements that confirm and extend previous results on the decays of ^{173}Tm, ^{157}Eu, and ^{149,151}Gd were obtained. A partial scheme of nuclear-energy levels was constructed for the electron capture of ^{133}La (4 hr) to ^{133}Ba. A systematic study of excited states in Lu isotopes ($168 \leq A \leq 175$) indicates regularities in nuclear-energy-level properties characteristic of deformed nuclei. A paper on this subject has been submitted for publication;[9] this work was done jointly with B. Harmatz.[10]

11.1.f Studies of Rare-Earth Alpha-Emitting Radionuclides

T. H. Handley

Rare-earth alpha-emitting radionuclides were produced by alpha-particle bombardment of natural or isotopically enriched europium in the ORIC. Following the separation of the radionuclides by conventional ion exchange methods, sources were prepared by electrodeposition. The alpha radiations from the sources were examined with an Si(Au) surface-barrier semiconductor detector. The gamma spectra were studied with a Ge(Li)-crystal spectrometer.

The alpha radioactivity from ^{149}Tb, ^{151}Tb, ^{152}Tb, and the daughter products ^{148}Gd and ^{149}Gd was studied; energies and α/EC branching ratios were assigned. Gamma-ray spectra of ^{151,152,153,154m,154,155,156}Tb, ^{149,151,153}Gd, and ^{149}Eu were studied in order to make measurements of energy and relative intensity. The data, when combined with published internal-conversion intensity data, made possible the assignment of multipolarities. These studies were done jointly with K. S. Toth[10] and I. R. Williams.[10] The results will be submitted to the *Physical Review* for publication.

[9] T. H. Handley and B. Harmatz, "Nuclear Spectroscopy of Neutron-Deficient Hafnium and Rare Earth Activities," submitted for publication in *Nuclear Physics*.

[10] Electronuclear Division.

11.2 COOPERATIVE ISOTOPES PROGRAM

11.2.a Reviews and Technical Assistance

S. A. Reynolds

The Isotopes Division and the Analytical Chemistry Division are jointly developing a program on isotope characterization and analysis. The program includes reviewing analytical methods, effecting economies of time and effort where possible, troubleshooting, improving data on nuclear properties, and establishing standards and specifications for radioisotope preparations. Cooperative work of this type has been done for many years on specific problems, but the new program is wider in scope, involving assistance in routine radioisotope processing as well as research and development in related areas. Already, revision has been made to the list of radioisotope-assay methods, decay schemes, and half-lives given in the ORNL Isotopes Catalog. The nuclear properties of several power-source nuclides were reviewed; the nuclides included ^{90}Sn, ^{137}Cs, ^{144}Ce, ^{147}Pm, ^{210}Po, ^{238}Pu, ^{242}Cm, and ^{244}Cm. Reviews will be made continually of all isotope products.

Several short-term problems were studied; these include a method of producing ^{18}F, analyses and interpretations with respect to ^{58}Co preparations, availability of ionium (^{230}Th), and effects of target self-shadowing in the production of radioisotopes. Discrepancies in the results of determinations of inactive components in shipments of ^{90}Sr from Hanford were investigated; in collaboration with those performing the analyses, difficulties in determining magnesium content were corrected. The cause of uncertainties in analysis of ^{198}Au products for ^{199}Au was found to be inadequate simulation of the unknowns by the standards used. Measurements made in the last five years by the Radioisotopes-Radiochemistry Laboratory and the Nuclear and Radiochemical Analyses Group on standards submitted by IAEA, NBS, and Nuclear-Chicago Corporation were reviewed. The overall mean deviation between the suppliers' and ORNL's results was 0.8% for 59 samples of 17 different nuclides.

11.2.b ^{131}I Products

S. A. Reynolds F. N. Case[2]

Review of analyses being requested on ^{131}I products showed that operating experience and changes in the ^{131}I-production process made some of the analyses unnecessary; they were eliminated accordingly.

The test for foreign, or "noniodine," radioactivity in ^{131}I products is important, because 99.9% minimum radiochemical purity is required. A newly devised test involves evaporation on a 1-in.-diam watch glass under an infrared lamp as is done in a gross-beta determination. Dilutions of representative products were used in all the experiments. Acid reducing solutions do not permit removal of a large enough fraction of the iodine; therefore, acid oxidizing media were studied. Bromine was the best oxidant tried; once-repeated evaporation produces a decontamination of 1×10^4 to 5×10^4, which is sufficient to demonstrate product purity of 99.99% or better.

Repeated treatments after the initial one usually effected little additional decontamination. With the idea that iodination of organic material on the watch glass might cause retention of radioactivity, various pretreatments were tried. These included soaking or pre-evaporation with trichloroethylene, carbon tetrachloride with and without iodine, bromine water, aqua regia, butanol-chloroform, and hydriodic acid. All these agents lessened retention, but it was about equally effective simply to add bromine water to the watch glass before the sample was added. The addition of a little carrier, as HI, further improved decontamination. Obviously, the evaporation procedure would not result in the loss of alkali, alkaline-earth, or rare-earth elements. The recovery of typical mixtures of long-lived fission products was 100.2 ± 1.4%. The contaminant most likely to be lost is ruthenium, which is known to be slightly volatile from nitric acid solution. Therefore, ^{106}Ru tracer was treated by the procedure; after one, two, and three evaporations the amount of the original ^{106}Ru remaining was 97.3, 96.5, and 95.6%, respectively. The procedure is thus satisfactory for detecting ruthenium contamination.

11.2.c ^{14}C Products

S. A. Reynolds J. S. Eldridge

Discrepancies were noted between measurements of ^{14}C content in $Ba^{14}CO_3$ products by liquid-scintillation counting and by mass spectrometry. It was found that a standard had deteriorated; therefore, new standards were obtained. However, a disagreement remains that is larger than the expected analytical uncertainties. Various chemical tests and spectrographic analyses are in progress, but these are difficult because of the high isotopic abundance – and therefore high radioactivity – of ^{14}C.

With regard to radiochemical purity, it was suggested that some of the by-product tritium (as water) might contaminate ^{14}C products. Data were obtained by distillation of water from a Versene solution of a ^{14}C product and liquid-scintillation counting. The tritium content was less than 0.0014% of the ^{14}C content. Routine inspection by gamma spectrometry and determination of nonvolatile radioactivity ensures the absence of significant amounts of other radiocontaminants.

11.2.d ^{144}Ce Product

S. A. Reynolds

A purchaser inquired about the possibility of alpha contamination in a batch of ^{144}Ce-^{144}Pr. Alpha tracks were reported to be present in an autoradiograph of a tissue section from a sheep that had been given 10 mc of this product. The Radioisotopes-Radiochemistry Laboratory had reported <0.06 μc per curie of ^{144}Ce. A small volume of the normal saline solution of the product used for the injection was returned. By gamma spectrometry the ^{144}Ce content of the solution was found to be 2 mc/ml, a value in agreement with the purchaser's value. The gross alpha content was determined to be ~ 0.02 μc per curie of ^{144}Ce. To make certain that alpha radiation

was being counted and not beta "pileup,"[11] applied voltage on the proportional counter was varied. A sheep dosed with 10 mc of the material would thus receive only $\sim 4 \times 10^2$ dis/min of alpha radioactivity. It does not appear that any measurable alpha radioactivity in a tissue section could have come from the ^{144}Ce-^{144}Pr preparation. Incidental to this work was a perfunctory check of the stability of ^{144}Ce in normal saline solution. Agreement between the intended concentration and that observed is significant. Further, a portion of the solution was centrifuged, and no decrease in radioactivity was observed.

11.2.e Trace High-Energy Gamma Radioactivity in Isotope Products

S. A. Reynolds

Gamma spectrometry was used to detect the 2.6-Mev gamma radiation of ^{208}Tl in an ^{237}Np preparation. In such a sample, ^{208}Tl is maintained by its parent ^{228}Th. The spectrometric technique developed to measure traces of ^{232}U in ^{233}U and ^{235}U[12] consists in absorbing most of the low-energy gamma radiation in a lead absorber and also suppressing the first 20-odd channels of the spectrometer. Dead times are thus made tolerable, and it is possible to measure low levels of high-energy gamma radionuclides. The radioactivity of ^{237}Np and its ^{233}Pa daughter in equilibrium was about 1.4 mc. The ^{228}Th content was 9×10^4 dis/min in the 1-g sample. It is believed that ^{228}Th was not associated with its parent, ^{232}U, as it is in ^{233}U and ^{235}U preparations. Probably the ^{228}Th present in irradiated uranium (natural or enriched) accompanies neptunium to some degree in the separation process. No such gamma radiation was found in a 2-curie ^{241}Am source. The same method was used to measure ^{232}U at a level of 0.2 ppb in ^{233}U preparations.

[11] S. A. Reynolds, "Effects of Low-Energy Beta Activity on Alpha Counting," *Anal. Chem. Div. Ann. Progr. Rept. Dec. 31, 1957*, ORNL-2453, pp. 19–21.

[12] S. A. Reynolds, "Determinations of ^{232}U by Gamma Spectrometry," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1964*, ORNL-3750, p. 45.

11.2.f $^{85}Rb(n,2n)$ Cross Section

S. A. Reynolds

A purchaser[13] of an ^{86}Rb isotope product detected the presence of annihilation radiation by gamma-gamma coincidence counting. Chemical tests and further spectrometric examinations resulted in its identification as that from ^{84}Rb, produced by the $(n,2n)$ reaction on the ^{85}Rb target. The value for the $(n,2n)$ cross section was calculated to be 0.2 mb for fission-spectrum neutrons, which agrees very well with the computed value, 0.23.[14]

11.2.g Release of Tritium by Tritiated Paint

T. H. Handley

A study is being made for the Isotopes Division to determine the amount and chemical form of tritium released from tritiated paint. The paint consists of a tritiated organic polymer, binder, and thinner, which are mixed before application. Samples of the paint were obtained from commercial suppliers. Results show that at elevated temperatures tritium is released chiefly in the form of tritiated water. For the conditions studied, loss of tritium by the paint was roughly exponential with respect to temperature. Tests to determine release of tritium at room temperature during the initial drying period are in progress.

11.2.h ^{210}Pb in Auto Exhaust Fumes and in Natural Materials

S. A. Reynolds

Consideration was given to an inquiry received by the Isotopes Division about the possible hazard associated with ^{210}Pb in products of the combustion of gasoline that contains lead tetraethyl.

[13] W. Pierson, Ford Motor Co., Dearborn, Mich., personal communication to J. E. Ratledge, Isotopes Division, September 1965.

[14] S. Pearlstein, *Analysis of (n,2n) Cross Sections for Nuclei of Mass A > 30*, BNL-897 (December 1964).

The highest concentration of ^{210}Pb found in commercial lead was 36 pc/g.[15] Its maximum permissible concentration in air is 4 pc/m^3,[16] which corresponds to 110 mg/m^3 of the 36-pc/g material and to a larger quantity of less radioactive material. The maximum allowable concentration for lead is 0.2 mg/m^3;[16] therefore, the chemical-toxicity hazard is completely predominant.

Interesting differences in specific radioactivities of lead in natural materials have been observed. These materials included ocean water, precipitation, and human tissues, the specific radioactivity of the latter being intermediate between that of water and that of metallic lead. The observations can be explained in terms of two sources of lead: radiogenic lead (high-specific-activity) from radon in the air and from radium in food, and fossil lead (low-specific-activity) from lead minerals in which any ^{210}Pb originally present has decayed. The intermediate specific radioactivity in human tissues is caused by the intake of lead from both sources. Much of the fossil lead is said to be due to automobile exhaust fumes.[17]

11.3 MEASUREMENT OF RADIOACTIVITY

11.3.a Preparation by Electrodeposition of Mounts for 4π Beta Counting

Gerald Goldstein

The conventional sample mount for 4π beta counting in a proportional counter and for 4π beta-gamma coincidence counting consists of an aluminum disk (~1¼ in. in diameter) with a central hole (~½ in. in diameter). A thin, self-supporting plastic film is spread across the disk, and the film is rendered conducting by a suitable metal surface vacuum-evaporated onto the film. A 20- to 50-μg/cm^2 layer of gold is typical. Sources are prepared by evaporating solutions on the mount. Although this type of backing eliminates mount-absorption and backscatter difficulties, samples have a tendency to concentrate into a dense spot on the film unless a spreading agent is also added. Ludox, a colloidal suspension of silica, is commonly employed to spread the sample. Autoradiographs of several ^{99}Tc mounts (Fig. 11.1) clearly illustrate these tendencies. Counting efficiencies were 90 and 97% without and with Ludox, respectively; these results indicate that self-absorption is still a problem with soft-beta emitters. Since a chemically inert and conducting surface is available, the possibility was explored of performing an electrodeposition on this surface to prepare a uniform, homogeneous, and essentially weightless source.

A small electrolytic cell (Fig. 11.2) was fabricated from Lucite and consists of identical halves, each of which will hold 2 ml of solution. The 4π beta mount is placed between two Teflon gaskets that expose only the area over the central hole to the solution and provide a watertight seal. For electrical contact, a small strip of gold foil was inserted on the gold-plated side of the mount. The gasketed mount was placed between the Lucite halves, which were then screwed together. A 2-ml sample was pipetted into the compartment that opens onto the gold-plated side of the mount, and an equal volume of electrolyte was placed on the other side to balance the pressure on the film. All depositions were made at constant current (10 ma) for 1 to 2 hr with ^{99}Tc.

The results showed it is possible to electrodeposit metals onto a 4π beta mount provided that the ring is a nonconducting material such as a nylon; the deposits appear to be uniform. With aluminum rings, deposition occurred only around the edges nearest the ring (Fig. 11.3). Although it was not possible to deposit ^{99}Tc quantitatively, this technique might be useful with elements that are more easily reduced.

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[15] S. A. Reynolds and C. L. Burros, "Low-Level Analyses," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1964*, ORNL-3750, pp. 50–51.

[16] N. I. Sax, *Dangerous Properties of Industrial Materials*, 2d ed., pp. 116 and 928, Reinhold, New York, 1963.

[17] T. J. Chow and M. S. Johnstone, "Lead Isotopes in Gasoline and Aerosols of Los Angeles Basin, California," *Science* 147, 502 (1965).

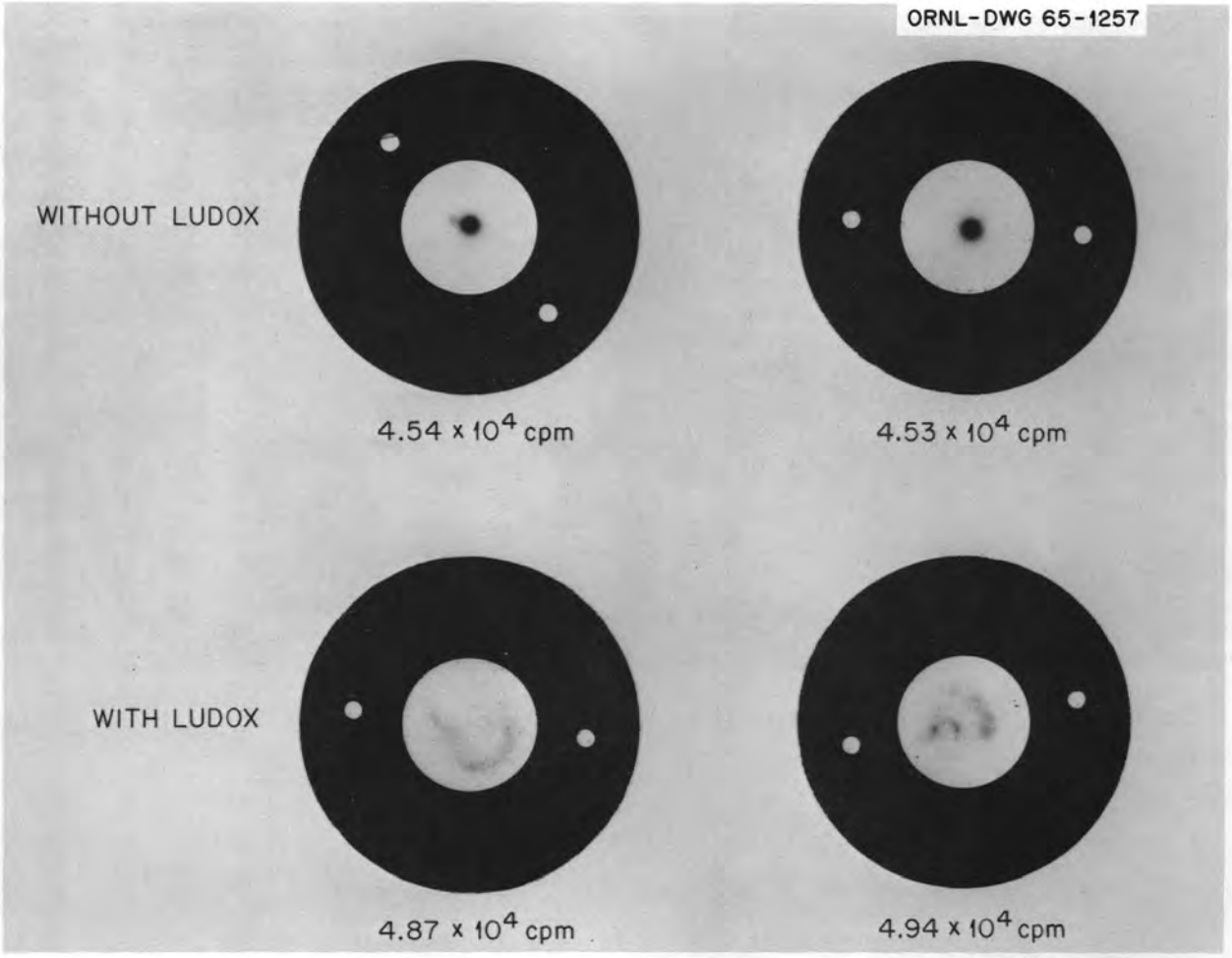

Fig. 11.1. Autoradiographs of 4π Beta Mounts Prepared by Conventional Techniques.

11.3.b Detection of Natural Radionuclides by Well-Type Scintillation Counters

S. A. Reynolds

Predictions were made of the response of a standard well-type scintillation counter[18] to the common natural radionuclides of the uranium series, thorium series, and potassium. The predictions were compared with measurements of sources of the individual radionuclides and of Redstone granite, which contains a significant amount of all these activities. Radioactive equilibrium normally exists in natural materials, and the equilibrium radioactivities of uranium, thorium, and their daughters are much greater than the radioactivities of the elements themselves. This information has possible usefulness in low-level work and in estimating the upper limit of these materials in natural samples. Table 11.3 shows the specific radioactivities of ^{238}U, ^{238}Th, and K and of the radionuclides associated with them in equilibrium. Predictions are given for count rate with the lead absorber, and experimental findings by use of 1-g samples are given for Pb-, Al-, and no-absorber cases. For the latter two cases, no predictions were attempted because of the uncertainties in weak-gamma, x-ray, and beta contributions. Table 11.4 gives the predicted activities of the Redstone granite sample.

[18] S. A. Reynolds, "Gamma Scintillation Counter," Method Nos. 2 00365, 5 00365, and 9 00365 (5-17-63), *ORNL Master Analytical Manual;* TID-7015, Suppl. 6 (June 1964).

Fig. 11.2. Exploded View of Electrodeposition Cell.

Fig. 11.3. Electrodeposited ^{99}Tc on Aluminum and Nylon 4π Beta Mounts.

Table 11.3. Radioactivities of Standards

Parent Nuclide	Specific Radioactivity (dis min^{-1} mg^{-1})	Total Radioactivity (dis min^{-1} mg^{-1})	Gamma Radioactivity (counts min^{-1} mg^{-1})			
			Pb Absorber		Al Absorber, Observed	No Absorber, Observed
			Predicted	Observed		
^{238}U	74_0	1.0×10^4	24_0	22_0	74_0	81_0
^{232}Th	24_0	2.4×10^3	102	103 ± 7	35_0	36_0
K[a]	1.68 beta 0.207 gamma	1.68 beta 0.207 gamma	0.052	0.06 ± 0.02		

[a]No daughters.

Table 11.4. Radioactivities of Redstone Granite

Component	Percent	Total Radioactivity (dis min^{-1} g^{-1})	Total Radioactivity ($\mu c/g$)	Predicted Gamma Radioactivity (counts min^{-1} g^{-1})[a] Pb Absorber	Predicted Gamma Radioactivity (counts min^{-1} g^{-1})[a] Al Absorber
			$\times 10^4$		
Th	0.010	240	1.1	10	35
U	0.0015	156	0.7	3	11
K	4.5	75	0.34	2	2
				Total 15[b]	48[c]

[a]Based on data of Table 11.3.
[b]Observed counting rate, 15 ± 4 counts min^{-1} g^{-1}.
[c]Observed counting rate, 49 ± 4 counts min^{-1} g^{-1}.

11.3.c $^{33}S(n,p)$ Cross Section

S. A. Reynolds R. E. Lewis[2]

A preliminary experiment[19] related to production of ^{33}P involved the two-day irradiation of small samples of sulfur moderately enriched in ^{33}S. The samples were irradiated in the ANL CP-5 reactor in a region of well-thermalized neutrons. The thermal-neutron cross section estimated from this work was ~5 to 6 mb. Two subsequent irradiations of larger, more highly enriched samples for longer periods gave values of 2.2 and 2.3 mb. A Co-Al alloy dilute in Co was used to monitor the thermal-neutron flux. The ^{33}P (and accompanying ^{32}P) was measured by liquid-scintillation and end-window proportional counting. The finding is in agreement with the value 2.3 mb reported previously;[20] the reason for the disagreement with the preliminary value (above) is not known.

[19]C. M. Steinberg, H. R. Gwinn, R. C. von Borstel, E. W. McDaniel, and S. A. Reynolds, "Thermal Cross Section for the Reaction $^{33}S(n,p)^{33}P$," *Biol. Div. Semiann. Progr. Rept. Feb. 15, 1965*, ORNL-3768, pp. 39–40.

[20]T. Westermark, "Production of P^{33} with Thermal Neutrons," *Phys. Rev.* **88**, 573 (1952).

11.3.d Half-Life Measurements

S. A. Reynolds

The measurement of the half-life of ^{137}Cs by direct gamma decay has continued; the decay of four sources has been followed for six years. The current tentative value is 30.2 ± 0.3 years; this value supersedes last year's value, 30.6 ± 0.7 years.[21] Uncertainties in new results are standard deviations. In another ORNL study,[22] the mean of three values measured by independent methods was 30.3 ± 0.3 years. Twelve published values, which range from 29.4 to 30.9 years, have an unweighted mean of 30.17 ± 0.13 years. Plots on probability paper indicate a normal distribution about the mean, which is not changed significantly by the addition of the two unpublished values.

The half-life of ^{153}Sm was 46.8 ± 0.1 hr, in agreement with that reported previously[23] and near the average of published values.

[21]S. A. Reynolds, "Half-Life Measurements," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1964*, ORNL-3750, p. 42.

[22]J. H. Gillette, *Review of Radioisotopes Program, 1964*, ORNL-3802 (May 1965); esp. p. 11.

[23]E. I. Wyatt, S. A. Reynolds, T. H. Handley, W. S. Lyon, and H. A. Parker, "Half-Lives of Radionuclides – II," *Nucl. Sci. Eng.* 11, 74 (1961).

Three samples of Ge were bombarded to produce ^{77}As, and the data were analyzed separately. The calculated values for the half-life of ^{77}As were 38.76 ± 0.10, 38.83 ± 0.09, and 38.89 ± 0.09 hr, the mean of the values being 38.83 ± 0.05 hr; the value 38.7 ± 0.1 was published earlier.[24]

The half-life observed for ^{198}Au was 64.63 ± 0.11 hr, which agrees with the mean of published values.

The decay curve of a mixture of fission product iodines, ^{131}I, ^{133}I, and ^{135}I, was resolved by CLSQ, and the half-life of ^{133}I was found to be 20.9 ± 0.1 hr, a value very near the accepted value, 21 hr.

The half-life of ^{33}P was found to be 25.30 ± 0.05 days, which confirms an earlier one, 25.2 days.[25]

11.3.e Experimental Study of Artifacts and Errors Encountered in Gamma-Ray Spectrometry for Radiochemistry and Activation Analysis

W. S. Lyon J. S. Eldridge
Peter Crowther[26]

A number of artifacts and errors have been observed to occur in the measurement of gamma-ray spectra. Some of the more serious were studied in an effort to understand the origin, prevention, or minimization of such phenomena. Effects dependent on source-detector geometry, such as 180° Compton-annihilation gamma-ray summing and cascade gamma-ray summing, have been evaluated.

Positron emitters may be assayed by measurement of the 0.51-Mev annihilation radiation; however, changes in the position at which the positrons are annihilated relative to the detector can cause serious discrepancies in the observed photopeak. The effects of production of annihilation radiation through interaction of high-energy gamma rays with walls, shields, etc., and backscatter from material in proximity to the source can also introduce artifacts and errors. Preparation and canning of NaI crystals can markedly affect both the shape of observed low-energy gamma-ray photopeaks and the resolution of the detector system. Experimental results are presented that show the effects of different crystal-preparation techniques; a recommended procedure to yield optimum spectral shape and resolution is given.

An article on this subject has been published.[27]

11.3.f Artifacts in Gamma-Ray Spectrometry

S. A. Reynolds

In connection with work on the ^{85}Rb(n,2n) cross section (see Sect. 11.2.f), the possibility was considered that the annihilation radiation might be an artifact. Such an effect occurs in gamma-ray spectrometry when the energy of gamma radiation exceeds 1.02 Mev.[27] From observed ratios[27] of artifact annihilation radiation to primary gamma radiation and from the assumption that the cross section for pair production "rises monotonically from zero at threshold, varying approximately as E . . . ,"[28] it was estimated that the highest ratio for the 1.08-Mev gamma of ^{86}Rb would be ~0.002. Failure to find an annihilation peak in the normal (single-detector) gamma spectrum of ^{86}Rb indicated that the ratio 0.002 is realistically low.

11.4 RADIOCHEMICAL STUDIES

11.4.a N,N′,N″-Trihexylphosphoric Triamide (THPA) as an Extractant for Metal Ions

T. H. Handley

The extraction properties of the trialkylphosphoric triamides appear to be intermediate between

[24] M. E. Bunker, R. J. Prestwood, and J. W. Starner, "Scintillation Study of As^{77} and Br^{77}," *Phys. Rev.* 91, 1021 (1953).

[25] Ingrid Fogelström-Fineman and T. Westermark, "On the Half-Life of ^{33}P," *Acta Chem. Scand.* 14, 2046 (1960).

[26] Alien Guest, 1962–63, Temporary Alien Employee, 1963–64; from South African Atomic Energy Board, Pelindaba, Pretoria, South Africa.

[27] W. S. Lyon, J. S. Eldridge, and P. Crowther, "Experimental Study of Artifacts and Errors Encountered in Gamma-Ray Spectrometry for Radiochemistry and Activation Analysis," in *Radiochemical Methods of Analysis*, vol. II, pp. 33–46, IAEA, Vienna, 1965.

[28] J. H. Hubbell and M. J. Berger, *Photon Attenuation and Energy Transfer Coefficients. Tabulations and Discussion*, N.B.S. Rept. 8681 (May 10, 1965).

those of the oxygenated slightly basic solvents and those of the highly basic amines. The extractability of trivalent lanthanides and actinides from concentrated aqueous solutions of chloride, nitrate, and thiocyanate contrasts strongly with their nonextractability from the corresponding acids. In this respect, N,N′,N″-trihexylphosphoric triamide (THPA) is related to the basic anion resins and amines. A 0.01 *M* solution of THPA in cyclohexane (5 ml) will quantitatively extract 3.5 mg of uranium from an aqueous phase that contains $LiNO_3$. A study of D_U vs uranium initially present in the aqueous phase shows that the extracted species is $UO_2(NO_3)_2 \cdot 2THPA$. Uranium is also quantitatively extracted from an aqueous phase 1 *N* in HNO_3, H_2SO_4, HCl, or H_3PO_4. Uranium is not backextracted by washing the organic phase with distilled water. A 0.01 *M* solution of THPA in cyclohexane will quantitatively extract 5 μg to 1 mg of Co, Zn, or Fe from an aqueous phase that contains thiocyanate. In this respect, THPA is related to the carbonyl compounds.

11.4.b Review of Organic Compounds That Contain Phosphorothioate, P=S, and Phosphorodithioate, P(S)SH, Groups as Separation and Analytical Reagents

T. H. Handley

Although considerable information appears in the literature concerning organic compounds with P=S and P(S)SH groups, many problems still exist for investigation. Also, utilization of these versatile reagents has only just begun. A review article has been published[29] that should stimulate further evaluation and development of these reagents.

11.4.c Rapid Separation of Technetium from Fission Products

Gerald Goldstein

A previously described[30] procedure for the solvent extraction of technetium, as pertechnetate ion, with cyclohexanone proved to be unusually rapid, efficient, and selective. This separation was applied to the determination of the number of fissions occurring in a short irradiation of uranium by measurement of ^{99m}Tc and to the separation of ^{99}Tc from long-lived fission products for burnup analysis. A report on this work is to be published.[31]

11.4.d High-Voltage Paper Electrophoresis

J. F. Wild[32]

High-voltage paper electrophoresis was investigated to determine its usefulness in effecting rapid separations of inorganic ions. Also, a search was made for phenomena, such as discontinuities or inflections in migration-velocity-vs-voltage curves, believed to exist at high potential gradients. Ten anionic and cationic systems were selected for initial studies. The results of the investigation indicate that many simple mixtures can be separated efficiently and with great speed (30 sec or less).

11.4.e Separation of Trace Metals from Uranium by Chromatography on a Cellulose Column

L. C. Bate R. A. A. Muzzarelli[33]

The study of the separation of trace metals from uranium by chromatography on a cellulose column[34] was completed. A paper on the work has been published.[35]

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[29] T. H. Handley, "A Review of Organic Compounds Containing P=S and P(S)SH Groups as Separatory and Analytical Reagents," *Talanta* **12**, 893 (1965).

[30] G. Goldstein, "Rapid Separation and Determination of Technetium," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1964*, ORNL-3750, pp. 47–48.

[31] G. Goldstein, "Rapid Separation of Technetium from Fission Products," accepted for publication in *Radiochimica Acta*.

[32] Summer Student Trainee; graduate student, Chemistry Department, Massachusetts Institute of Technology, Cambridge, Mass.

[33] Alien Guest, 1963–64; from Communauté Européenne de L'Energie Atomique, EURATOM, Bureau Central de Mesures Nucléaires, Geel, Belgium.

[34] L. C. Bate and R. A. A. Muzzarelli, "Separation of Trace Metals from Uranium by Cellulose Column Chromatography," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1964*, ORNL-3750, p. 48.

[35] R. A. A. Muzzarelli and L. C. Bate, "Separation of Traces of Metals from Uranium by Chromatography on Cellulose Column," *Talanta* **12**, 823 (1965).

11.5 LIQUID SCINTILLATORS

11.5.a N,N′,N″-Trimethylborazine as a Liquid-Scintillator Solvent for Thermal-Neutron Detection

H. H. Ross H. L. Holsopple

A paper has been published that describes the various characteristics and properties of N,N′,N″-trimethylborazine as a liquid-scintillator solvent for thermal-neutron detection.[36]

11.5.b Color-Quench Correction in Liquid-Scintillator Systems by Use of an Isolated Internal Standard

H. H. Ross

A new technique of color-quench correction by use of an isolated internal standard was developed. When this technique is used in conjunction with any of the other methods for total-quench correction, the separate effects of color and chemical quenching are easily resolved. The technique is based on the absorption of light photons in a color-quenched liquid-scintillator sample. However, in contrast to the spectrophotometric method, the light source consists of a small ampul that contains an unquenched liquid scintillator spiked with the desired isotope. The photons emitted by the standard have the same intensity and spectral distribution as those produced in the sample itself. An article on the technique has been published.[37]

11.6 APPLICATION OF COMPUTERS

11.6.a Electronic Gamma-Ray Spectral Resolution

J. S. Eldridge

Test results of ten cases of computer-performed gamma-ray spectral resolution were analyzed. Binary, tertiary, and quaternary mixtures of ^{46}Sc, ^{51}Cr, ^{59}Fe, and ^{134}Cs were resolved with the gamma-ray fitting program[38] and were analyzed by an independent assay procedure. Table 11.5 gives typical results, which show that the library of standard spectra, as well as the computer program, functions satisfactorily for the complex problem of gamma-ray spectral resolution.

Digital data from several of the nuclides included in the library were obtained in a paper-tape format and were used in a test of the concept of applying this library in an absolute assay technique. Several radioactive sources were prepared and were assayed independently by absolute gamma-ray spectrometry. These sources were measured in a spectrometer system at the Isotopes Division. The paper-tape-library data for the measured nuclides were then read into the spectrometer system, where a direct comparison of the source and tape data was made by use of the electronic summation feature. Table 11.6 gives the results of this assay. These results show that the paper-tape format of the library data will function satisfactorily in this form of absolute assay. The negative bias of the results is due to the uncertainty in determining the position of the NaI crystal beneath the can in the detectors used for the comparisons. This discrepancy may be partially removed by normalizing the library data with an independent source in conjunction with the library data.

This project is now complete, and the termination report is being prepared.

11.6.b Evaluation of Computer Programs for Gamma-Ray Spectrometry in Activation Analysis

J. F. Emery T. D. Alexander[39]
F. F. Dyer Ernest Schonfeld[40]

At the 1965 International Conference on Activation Analysis, a paper was given on the application of computers. The paper will be published

[36] H. H. Ross and H. L. Holsopple, "N,N,N, Trimethylborazine as a Liquid Scintillator Solvent for Thermal Neutron Detection," *Nucl. Instr. Methods* 33, 194 (1965).

[37] H. H. Ross, "Color Quench Correction in Liquid Scintillator Systems Using an Isolated Internal Standard," *Anal. Chem.* 37, 621 (1965).

[38] J. S. Eldridge, "Computer Applications in Nuclear Analyses," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1963*, ORNL-3537, p. 85.

[39] Research participant, summer of 1964; from Department of Chemistry, Georgetown College, Georgetown, Ky.

[40] Chemical Technology Division.

Table 11.5. Results of Determination of Radionuclides in Mixtures by a Computer Program for Gamma-Ray Spectral Resolution

Nuclide	Amount Added (dis/sec)	Amount Found (dis/sec)		
		Binary Mixture	Tertiary Mixture	Quaternary Mixture
	$\times 10^4$	$\times 10^4$	$\times 10^4$	$\times 10^4$
^{46}Sc	1.44 ± 0.01		1.43 ± 0.02	1.45 ± 0.03
^{51}Cr	3.20 ± 0.05	3.04 ± 0.08	3.13 ± 0.11	3.11 ± 0.16
^{59}Fe	2.19 ± 0.01	2.20 ± 0.02	2.22 ± 0.04	2.23 ± 0.05
^{134}Cs	2.32 ± 0.02		2.31 ± 0.03	2.34 ± 0.02

Table 11.6. Results of Tape-Comparison Assay

Nuclide	Amount (dis/sec)		Deviation (%)
	Added	Found	
	$\times 10^4$	$\times 10^4$	$\times 10^4$
^{59}Fe	2.05	2.01	−2.2
^{124}Sb	2.31	2.29	−0.9
^{137}Cs	3.66	3.55	−3.1
^{198}Au	8.12	8.04	−1.0

in the proceedings of the conference; the abstract of it follows.

Three operational computer programs for resolving gamma-ray spectral data were evaluated. Some problems considered are: ability to make gain-shift corrections, effect of zero-activity nuclides on other nuclides present, and significance and reliability of computed standard deviations. Computer programs for resolving gamma-ray spectra require a number of computer-control data, such as photopeak channel, gamma-ray energies, gain shift, etc. The number and complexity of this control information depend largely on the versatility of the program and on the purpose for which the program was written. Each program was evaluated for simplicity of use with a large number of samples and for cost per analysis.

11.6.c Computer Program for Calculating Correlation Coefficients

F. F. Dyer

Recent studies in the forensic problem of comparing materials (e.g., hair and opium) by their trace-element content led to an interest in the degree of correlation among the concentrations of trace elements. If the concentrations of two elements are highly correlated, then on the average, the concentration of one can be predicted from that of the other, and very little information is gained by measuring both elements.

A computer program was written to calculate correlation coefficients for all pairs of elements in a set of measurements. The input information required is the names of the elements and their concentrations. The output includes input data, standard deviations for the concentrations of each element, and correlation coefficients identified by the names of the elements. Calculations made with data from the analysis of samples of head hair taken from 99 different humans showed no significant correlations, but a number of significant correlations existed for 107 samples of opium.

11.6.d Use of Computers in Applied Activation Analysis

J. F. Emery

A subroutine was written for Cumming's CLSQ Decay Curve Analysis Program[41] that allows data accumulated in the time mode or multiscaler mode of a multichannel analyzer to be analyzed by CLSQ. The subroutine will accept data from any multichannel scaler. A constant background or

[41] J. B. Cumming, "CLSQ, the Brookhaven Decay Curve Analysis Program," pp. 25–33 in *Radiochemical Techniques. Applications of Computers to Nuclear and Radiochemistry*, ed. by G. D. O'Kelley, NAS-NS-3107 (March 1963).

a continually varying background may be subtracted from each channel.

A program LIMIT was written for the CDC 1604-A to calculate the detection limits for the elements by neutron activation. Any irradiation time or neutron flux intensity may be used. The detection limits calculated depend on the detector geometry, the efficiency for the particular gamma ray used for assay, the decay scheme of the particular radionuclide, and the background of the detector for any given count interval.

LIMIT may also be used to compute upper limits of concentration for a large number of elements in a high-purity matrix; for example, upper limits of 46 elements were computed for semiconductor-grade silicon metal that had been irradiated for 2.75 hr in the ORR pneumatic tube. These concentrations were: for 13 elements, $\leq 10^{-4}$ ppm; for 20 elements, $\leq 10^{-2}$ ppm; for 9 elements, ≤ 1 ppm; and for only 3, ≥ 1 ppm. These upper limits were high because of the poor sensitivity of neutron activation analysis for these elements.

A program ABSNAA was written for the CDC 1604-A computer for the absolute neutron activation analysis of materials. Use of the program requires the irradiation of two neutron-flux monitors, preferably one monitor with a large resonance integral and one monitor with a small one. These two monitors are then used to calculate the thermal- and resonance-neutron fluxes.

A complex gamma-ray spectrum is first resolved by ALPHA[42] into disintegration rates of the various components at reactor discharge time. ALPHA normally outputs this information on punched cards. These cards are used as input data for ABSNAA. The only additional information needed is the duration of irradiation of the samples. ABSNAA then selects the proper element, thermal-neutron cross section, resonance integral, isotopic abundance, etc., and by use of the previously calculated computer values of the thermal and resonance flux calculates the weight of the element present.

An advantage of this program is that only two monitor samples need be irradiated for any and all elements that might be determined.

[42]E. Schonfeld, *Alpha-A Computer Program for the Determination of Radioisotopes by Least-Squares Resolution of the Gamma-Ray Spectra*, ORNL-3810 (July 1965).

11.6.e Program NETAREA

R. L. Hahn Enzo Ricci

The code NETAREA was written for the IBM 7090 computer to calculate photopeak areas of gamma-ray spectra. The computation consists of fitting a Gaussian shape to the peak. Two sets of three points of the peak are used to calculate exactly two sets of parameters for a Gaussian curve; these parameters are the magnitude and position of the maximum of the Gaussian curve and its half-width at half maximum. The two sets of parameters form the basis of a series of successive approximations which lead to a final set of parameters that best fit all six points. The area under this calculated Gaussian curve is then computed. Two calculational options are available in the program: subtraction from the photopeak of a calculated exponential approximation to a Compton background and calculation of the Gaussian area by subtracting an exponential curve tangent to the valleys from the sum of counts under the gamma-ray peak.

The program will normally print as its output the final values of the three Gaussian parameters, a figure of merit for the fit to the Gaussian, and the Gaussian area with its standard deviation. Two additional modes of output can be selected: a diagnostic output that prints the results of all intermediate calculations and a punched-card output in a format compatible with the input requirements of the CLSQ least-squares decay-curve analysis program.[41]

11.6.f Computer Program for Calculating Neutron Flux

F. F. Dyer

When neutron-flux monitors are irradiated for long periods of time during which the reactor power changes a number of times, calculation of neutron flux from the induced radioactivity is difficult to do manually. Usually, assumptions are made about the operating history of the reactor which simplify the calculations but which introduce errors in the computed fluxes. A computer program was written that will make the calculations rapidly and exactly. The program, written in FORTRAN 63 for the CDC 1604, requires as

input data: identification numbers, absolute activities and weights of samples, and operating history of the reactor. The operating history consists of two one-dimensional arrays which list the operating times and powers of the reactor for each operating period. Calculations are based on the assumption that neutron flux is linearly dependent on reactor power. The program gives as output: all input quantities, power-averaged (weighted) neutron flux, neutron flux corresponding to full-power operation of the reactor, and neutron dose. Major power changes of the reactor, which information is of interest in mechanical-design studies, are also outputed.

If the reactor operates nearly continuously at full power, corrections are made for thermal-neutron burnup of the induced radionuclide. The program is being modified to correct for burnup under all operating conditions.

11.7 APPLICATION OF RADIOISOTOPES TO ANALYTICAL CHEMISTRY

11.7.a A New Concept in Precision Photometric Analysis: Use of a Radioisotopic Light Source

H. H. Ross

Although absorption photometry is one of the most valuable techniques available to analytical chemists, its use is generally restricted to the determination of materials in trace quantities. This limitation is easy to understand if one recognizes that the precision of measurement with conventional photometric instruments cannot compete with that of standard gravimetric and titrimetric procedures. Müller discusses this problem with considerable insight.[43]

The initial phase of a program to develop a unique theoretical and experimental approach for making precise photometric measurements has been completed. A new system has been demonstrated that uses a radioisotopic light source; the system provides a new physical basis of measurement for absorption studies. The experimental results are in agreement with a simple mathematical model and are more precise than those obtained with conventional absorption instruments. Among the additional advantages of the method are controlled sensitivity, simplicity, low power requirements, and direct digital results. A paper on this development will be published.[44]

11.7.b Radio-Release Methods for the Evaluation of Atmospheric Pollution: Sulfur Dioxide

H. H. Ross

A method for the determination of sulfur dioxide in the atmosphere that uses a radio-release technique has been published.[45]

11.7.c Measurement of Wear Rates in Automotive Engines by Liquid-Scintillation Counting of ^{55}Fe

H. H. Ross R. P. Gardner[46]
J. W. Dunn III[47]

A new radiotracer technique for determining wear rates of selected automotive-engine parts was demonstrated for piston rings; ^{55}Fe instead of ^{59}Fe is used as the radiotracer. A liquid-scintillation method for counting ^{55}Fe is necessary, since ^{55}Fe decays by electron capture and emits only the Mn characteristic x ray of 5.9 kev. A simple method for extracting the wear particles from the engine oil and getting the iron into the liquid-scintillation mixture is described. Counting yields of 8 to 9% are obtained.

The ^{55}Fe technique of wear measurement does not compete directly with the existing ^{59}Fe technique; when ^{55}Fe is used the sensitivity is slightly lower and the sample preparation is longer. However, the much longer half-life (45 vs 2.6 days) and the lower radiation energy (5.9 kev vs >1 Mev) allow the ^{55}Fe technique to

[43] R. H. Müller, "Precision Photometry," *Anal. Chem.* 37(10), 123A (1965).

[44] H. H. Ross, "A New Concept in Precision Photometric Analysis Using a Radioisotopic Light Source," to be published in *Analytical Chemistry*.

[45] H. H. Ross and W. S. Lyon, "Radio-Release Methods for the Evaluation of Atmospheric Pollution: Sulphur Dioxide," pp. 285–93 in *Radiochemical Methods of Analysis*, vol. II, IAEA, Vienna, 1965.

[46] Measurement and Controls Laboratory, Research Triangle Institute, Durham, N.C.

[47] Research Assistant, Department of Mathematics, North Carolina State University, Raleigh.

be used for long-term wear studies, for double-tracer studies, and for studies of large engine parts.[48]

11.7.d Radiotracer Method for the Determination of Lead

L. E. Mattison[49]

A new, sensitive, and rapid method for the determination of lead is based on the use of ^{131}I tracer. Lead is extracted from dilute hydriodic acid (1:20) with 5 v/v % pyridine in benzene solution. Although the structure of the extracted species is not known, the lead extracted is proportional to the ^{131}I radioactivity in the organic phase. The counting rate is linear over the range from 0 to 70 μg of lead. These conclusions were confirmed by neutron activation analysis. The procedure is applicable to the determination of cadmium. Thallium(I) does not extract.

Work is continuing on the study of interferences.

11.8 ACTIVATION ANALYSIS

11.8.a Studies of ^{3}He Nuclear Reactions

^{3}He Activation Analysis (Enzo Ricci, R. L. Hahn). – The data obtained at the 5.5-Mev Van de Graaff accelerator[50] were analyzed. A general mathematical treatment was developed that reduces charged-particle activation of thick samples to almost the simplicity of neutron activation. This treatment is based on the definition of an average cross section, $\bar{\sigma}$, which is shown to be approximately independent of the nature of the target material:

$$\bar{\sigma} = \frac{\int_0^R \sigma(t)\, dt}{\int_0^R dt} \approx \frac{\int_0^{E_i} \sigma(E)\, E\, dE}{\int_0^{E_i} E\, dE} .$$

Table 11.7. Sensitivities for Oxygen Determination in Thick ZrO_2 Targets[a]

Reaction	Product Half-Life (min)	Sensitivity (dis sec^{-1} ppb^{-1})
$^{16}O(^3He,\alpha n)^{14}O$	1.2	0.047 ± 0.004
$^{16}O(^3He,\alpha)^{15}O$	2.07	20 ± 1
$^{16}O(^3He,2\alpha)^{11}C$	20.5	1.9 ± 0.2
$^{16}O(^3He,p)^{18}F$ + $^{16}O(^3He,n)^{18}Ne \rightarrow {}^{18}F$	110	43 ± 2

[a]Values calculated for irradiation intervals of one half-life and a 10-Mev ^{3}He-beam current of 100 μa.

Here, σ is the cross section of the given reaction at depth t or for particle energy E; E_i is the initial energy of the charged particles, and R is their range in the thick target. The activation-analysis sensitivity is proportional to $\bar{\sigma}R$; Table 11.7 lists sensitivities for several reactions that could be used for determining oxygen. The neutron outputs[50] from reactions of 10-Mev ^{3}He particles with lithium, beryllium, and boron compare favorably with those from (d + t) reactions in conventional neutron generators. Furthermore, the neutron yields from ^{3}He irradiations do not decrease with target usage. The yield of high-energy gamma rays (~20 Mev) is also significant. These results suggest that a small cyclotron, built for ^{3}He activation analysis, could be used effectively for fast-neutron and photon activation analysis. All the work mentioned above is described in detail in a published article.[51]

In recent experiments at the tandem Van de Graaff accelerator, thick targets of Be, B, C, N, O, F, Na, Mg, Al, Si, P, S, Cl, K, and Ca were irradiated with 18-Mev ^{3}He particles to obtain activation-analysis sensitivities for these elements. Gamma-ray spectrometry was used to assay the many radionuclides produced. The experience gained at the 5.5-Mev Van de Graaff accelerator made possible a drastic reduction in the time required for a comparable number

[48]H. H. Ross, R. P. Gardner, and J. W. Dunn III, "Wear Rates in Automotive Engines by Liquid Scintillation Counting of Fe^{55}," *Nucl. Sci. Eng.* **20**, 521 (1964).

[49]Research Participant, summer of 1964; from Department of Chemistry, King College, Bristol, Tenn.

[50]E. Ricci and R. L. Hahn, "Helium-3 Activation Analysis," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1964*, ORNL-3750, pp. 58–59.

[51]E. Ricci and R. L. Hahn, "Theory and Experiment in Rapid, Sensitive Helium-3 Activation Analysis. Helium-3 Reactions as Neutron Sources," *Anal. Chem.* **37**, 742 (1965).

of experiments at the tandem Van de Graaff accelerator.

Excitation Functions for ^{3}He-Induced Nuclear Reactions (R. L. Hahn, Enzo Ricci). – Knowledge of the excitation functions for nuclear reactions induced by ^{3}He particles in low-Z elements is necessary in evaluating the role of interferences in ^{3}He activation analysis and avoiding them. Also, these data may be useful in elucidating the mechanisms of these reactions. The data obtained during the irradiation of foils at the 5.5-Mev Van de Graaff accelerator[50] were used to calculate excitation functions up to 10 Mev for several ^{3}He reactions. Table 11.8 lists these reactions and their cross sections at various energies. Recent nuclear physical experiments support the idea that some of the ^{3}He-induced reactions proceed by direct interaction. We are currently attempting to compare the excitation functions predicted from the distorted-wave theory of direct nuclear reactions[52] with our results.

Thick-target yields for boron, nitrogen, and sodium were measured recently at the tandem Van de Graaff accelerator. The ^{3}He energies ranged from 6 to 18 Mev in intervals of 1.5 Mev. Excitation functions for the reactions observed will be calculated by differentiating the curves of yield vs energy.

11.8.b Study of Precision and Accuracy in Neutron Activation Analysis

F. F. Dyer J. F. Emery

The study of the precision and accuracy of activation analysis for elements that yield short-lived radionuclides was completed.[53] Measurements were made with single-component samples and with mixtures of the radionuclides ^{20}F, ^{52}V, ^{77}Se, ^{104}Rh, and ^{110}Ag. The major source of error was found to be loss of counts caused by dead time of the multichannel analyzer. Expressions were derived that precisely and accurately correct for this effect for both single-component and multicomponent samples. All measurements were made on samples that contained known amounts of elements. Relative standard errors for single-component samples ranged from ±1 to ±5% at the 95% confidence level. In most instances, deviations between weights taken and weights found in mixtures were <10%. A paper that describes this work is near completion, and publication is planned.

11.8.c Determination of ^{99}Tc by Neutron Activation Analysis

Gerald Goldstein J. F. Emery

Certain problems in geochemistry require the determination of extremely small quantities (of the order of 10^{-10} g) of technetium. Of the analytical methods available, only thermal-neutron activation analysis has, at least potentially, the necessary sensitivity.

Sample mounts of technetium were prepared by evaporation of standardized solutions on 1-mil-thick polystyrene foil that had been thoroughly washed. The samples were irradiated for 20 sec at the pneumatic facility of the ORR. The beta decay of the samples was followed with a well-type plastic phosphor and a multichannel analyzer operated in the time mode. From these data we attempted to measure 17-sec ^{100}Tc either by resolving the decay curve into its components by the CLSQ decay-curve-analysis program or by spectrum-stripping analysis by means of synthetic decay curves calculated for the known components as library spectra. A detailed report of this work has been published.[54] Essentially, we were not able to determine less than 10^{-8} g of ^{99}Tc with acceptable precision, because silver is an impurity in the polystyrene mount. On irradiation, 24-sec ^{110}Ag is produced, which cannot be distinguished from ^{100}Tc. Other materials have been examined for use as sample mounts, but we have not yet found one that is better than polystyrene.

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[52] R. H. Bassel, R. M. Drisko, and G. R. Satchler, *The Distorted-Wave Theory of Direct Nuclear Reactions. I: "Zero-Range" Formalism Without Spin-Orbit Coupling, and the Code SALLY*, ORNL-3240 (Jan. 26, 1962).

[53] F. F. Dyer and J. F. Emery, "Study of Precision in Activation Analysis," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1964*, ORNL-3750, p. 55.

[54] G. Goldstein and J. F. Emery, "Thermal-Neutron Activation Analysis of Technetium," *Chem. Div. Ann. Progr. Rept. May 20, 1965*, ORNL-3832, pp. 20–31.

Table 11.8. Experimental Cross Sections (mb) for Several ^{3}He Nuclear Reactions

Mid-Energy (Mev)	Energy Interval (Mev)	$^9Be(^3He,n)^{11}C$	$^{12}C(^3He,n)^{14}O$	$^{12}C(^3He,pn)^{13}N$	$^{12}C(^3He,\alpha)^{11}C$	$^{16}O(^3He,p)^{18}F$ + $^{16}O(^3He,n)^{18}Ne \rightarrow ^{18}F$	$^{16}O(^3He,\alpha)^{15}O$	$^{19}F(^3He,\alpha)^{18}F$	$^{19}F(^3He,\alpha n)^{17}F$
2.05	3.00–1.11	87.7 ± 17.0							
2.40	3.02–1.77		11.2 ± 1.4		118 ± 14				
2.90	3.35–2.44		6.99 ± 0.74		103 ± 7	40.3 ± 4.0	24.4 ± 2.9		
3.22	4.00–2.43	102 ± 10							
3.71	4.18–3.24		4.50 ± 0.48		135 ± 21			6.87 ± 0.69	4.52 ± 0.81
4.09	4.40–3.77		8.66 ± 0.92		153 ± 11	108 ± 11	60.4 ± 6.4		
4.34	5.00–3.67	113 ± 11							
4.92	5.31–4.52		12.8 ± 1.5		256 ± 18			18.6 ± 1.9	18.7 ± 2.1
5.19	5.49–4.88		13.4 ± 1.3		182 ± 13	206 ± 21	115 ± 12		
5.38	6.00–4.75	98.6 ± 9.9							
6.04	6.37–5.71		11.3 ± 1.3	32.9 ± 3.9	268 ± 19			20.5 ± 2.1	28.6 ± 3.1
6.31	6.55–6.06		16.5 ± 1.8	46.2 ± 6.4	218 ± 27	436 ± 44	147 ± 16		
6.51	7.00–6.01	93.0 ± 9.3							
6.63	7.14–6.11		13.9 ± 1.4	59.6 ± 8.4	278 ± 39	401 ± 47	169 ± 17		
7.14	7.43–6.84		8.60 ± 0.97	56.6 ± 5.0	329 ± 23			22.1 ± 2.0	44.0 ± 4.4
7.17	8.00–6.34					388 ± 58			
7.57	8.00–7.14	78.0 ± 7.8							
7.76	8.22–7.30		12.1 ± 1.3	56.5 ± 8.4	289 ± 29	356 ± 36	128 ± 24		
8.24	8.51–7.96		13.6 ± 1.6	81.1 ± 6.9	366 ± 26			21.0 ± 2.1	50.4 ± 5.0
8.42	9.06–7.77					321 ± 50			
8.60	9.00–8.20	65.8 ± 6.6							
8.88	9.29–8.46		10.2 ± 1.0	61.8 ± 8.8	349 ± 26	274 ± 27	112 ± 18		
9.30	9.55–9.05		7.72 ± 0.83	60.8 ± 6.0	331 ± 23			15.0 ± 1.5	45.2 ± 4.5
9.48	9.67–9.29			98.9 ± 12.2	308 ± 31	320 ± 32			
9.64	10.00–9.27	58.7 ± 5.9							

11.8.d Neutron Activation Analysis of Human Hair: Forensic Applications

L. C. Bate F. F. Dyer

The study of trace-element content of human hair for forensic applications[55] was terminated, and the results were reported.[56,57] The work led to the following conclusions. Hair should be cleaned thoroughly of surface contamination before trace elements are measured. It can be cleaned satisfactorily by washing with either a nonionic detergent or an organic solvent followed by distilled water. Although the hair can be washed either before or after irradiation, washing before irradiation may be preferable because of the destructive effects of irradiation. Determinations of sodium, bromine, and probably chlorine have little value in matching hair samples, because their concentrations in an individual's hair likely fluctuate rapidly with time. Many elements are very tightly bound in hair and cannot be removed easily by washing. Therefore, determinations of these elements in clean hair should be the determinations most useful for forensic investigations. Adsorption from the environment is a major source of trace elements in hair. Concentrations of trace elements in hair vary greatly with distance from the scalp. Therefore, in selecting samples for comparison it is probably necessary to obtain hair of equal lengths and equal distances from the scalp. No correlation exists among concentrations of trace elements in hair. Diversity of backgrounds of persons (age, sex, occupation, etc.) contributes to diversity in the trace-element concentrations of their hair.

Service work was done in two rape cases for the Criminal Investigation Division of Fort Bragg, North Carolina. No study had been made of single hairs, but such information was necessary in these cases. Consequently, samples of three to five hairs were obtained from ten persons other than the suspects, and each hair was analyzed separately to aid in interpreting results from the Fort Bragg samples. Duplicate samples were taken from one of the individuals, and an additional sample was obtained three months later from another. The results were evaluated by determining the number of elements whose concentrations matched among all the samples. Two samples were said to match in an element if the ranges of concentration of the element, as measured in several single hairs from each sample, overlapped. Because of the large variation in the concentration of each element (20 to 100%) among hairs from the same person, samples from a few individuals matched in more elements (five) than did the duplicates, which matched in only four elements. The two samples taken from the same person three months apart were very dissimilar in trace-element concentrations. It was concluded that the variation of element concentrations among hairs from the same person is probably too large to permit comparisons to be made from only three- or four-hair samples. From this work no evidence of guilt was found in the rape cases perhaps because of insufficient samples and because five months had elapsed between the time samples were taken from the crime site and the time samples were taken from the suspects.

[55] L. C. Bate and W. B. Healy, "Hair Analysis," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1964*, ORNL-3750, p. 57.

[56] L. C. Bate and F. F. Dyer, "The Use of Neutron Activation Analysis to Determine the Trace Elements in Hair," presented at Fourth Meeting of the Society for Applied Spectroscopy, Denver, Colo., Aug. 30–Sept. 3, 1965.

[57] L. C. Bate and F. F. Dyer, "Trace Elements in Human Hair," *Nucleonics* 23(10), 74 (1965).

11.8.e Applied Neutron Activation Analysis

J. F. Emery

Single cells of biological specimens were analyzed for Na, K, and Cl. The cells were mounted between 1-mil-thick polystyrene foils and were irradiated for 20 min in the pneumatic tube of the ORR. The amount of Na, K, and Cl for one cell was 1.15, $\leq$0.01, and 1.36 μg, respectively, and for the other cell, 0.14, $\leq$0.04, and 0.28 μg, respectively.

Bismuth metal from the bismuth-shielded facility in the BSR was analyzed for Ag, Al, Co, and Hf. The Co and Hf contents were each less than 0.01 ppm, whereas the Ag content was 0.01 ppm, and the Al content was 0.11 ppm.

Three series of electron-beam zone-refined Nb were analyzed for Ta and W. The samples were

from experiments in which niobium rods were passed 1, 6, and 12 times through the zone-melting process. The Ta concentration was constant at 367 ppm, whereas the W content after the first two experiments was 225 ppm and 242 ppm after the third. In the third experiment, W was apparently picked up from the electrodes.

A segment of corroded stainless steel pipe from the Bonus Reactor was analyzed for chloride. An upper limit of 0.3 ppm/cm^2 was found.

Other materials analyzed were: fission product solutions and filters for ^{235}U; ZrF_4 and ZrO_2 for Hf; INOR-8 for Al and V; HgO for Rh, Al, V, and Ag; Pb for natural radioactivity, Cu, Sb, and Ag; and Cs_2CO_3 for Tl.

11.8.f Gold Atoms Sputtered from Neutron-Irradiated Gold Foils

J. F. Emery

Polystyrene catcher foils that were located immediately behind gold foils irradiated in the beam hole of the ORR were analyzed for induced radionuclides, ^{198}Au, and stable ^{197}Au. The radioactivity of ^{198}Au (11%) found on the polystyrene foil agreed with that reported from earlier work,[58] which indicated that 13% of the neutron-capture and prompt-gamma deexcitation that occurred at one face of the gold foil produced recoil energies great enough to cause ^{198}Au atoms to escape from the surface of the foil. The amount of stable ^{197}Au sputtered onto the polystyrene foil was 3.47×10^{-11} g/cm^2. An additional experiment to determine the extent of interference from gamma irradiation was performed, and the amount of ^{197}Au found on the polystyrene foil was 4.6×10^{-12} g/cm^2. These experiments were performed by members of the Solid State Division.

11.8.g Determination of Uranium in Seawater by Neutron Activation Analysis

W. J. Ross

Submicrogram amounts of uranium were determined by neutron activation analysis in aqueous systems that contained macro amounts of interfering elements, such as those found in seawater. Potentially overshadowing gamma radioactivities were eliminated by preseparating uranium by solvent extraction with tri-*n*-octylphosphine oxide (TOPO). Essentially specific extraction of nanogram amounts of uranium is obtained by equilibrating 0.5 *M* HNO_3 solutions with 0.005 *M* solutions of TOPO in cyclohexane. The cyclohexane diluent evaporates readily; therefore, only a very small residue is activated to produce ^{239}U (and its daughter ^{239}Np) from ^{238}U. The maximum sensitivity of the method is limited to 0.1 μg of ^{238}U by the bremsstrahlung of 14-day ^{32}P produced by activation of TOPO. Efforts to eliminate this interference so that the limit of detection can be reduced are continuing.

11.8.h Neutron Activation Analysis of River Water

W. J. Ross

A method was developed for determining ppb concentrations of Cu, Zn, Ni, and Cr in river water of variable purity. The method consists in neutron activation analysis and a sequential chemical-separation procedure that permits the determination of as little as 7×10^{-5} μg of Cu, 9×10^{-3} μg of Zn, 4×10^{-2} μg of Ni, and 5×10^{-2} μg of Cr.

11.8.i Wool Analysis

L. C. Bate W. B. Healy[59]

A paper that describes the neutron activation analysis of wool[60] has been published.[61]

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[58] S. Yosim and T. Davies, "Recoil Atoms from Slow Neutron Capture by Gold and Indium Surfaces," *J. Phys. Chem.* **56**, 599 (1952).

[59] Alien Guest, 1963–64; from Soil Bureau, Department of Scientific and Industrial Research, Lower Hutt, New Zealand.

[60] L. C. Bate and W. B. Healy, "Wool Analysis," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1964*, ORNL-3750, p. 56.

[61] W. B. Healy, L. C. Bate, and T. G. Ludwig, "Micro-Element Content of Wool from Twin Wethers Raised on Two Soils as Determined by Neutron Activation Analysis," *New Zealand J. Agr. Res.* **7**, 603 (1964).

11.9 14-Mev NEUTRON GENERATOR

11.9.a Catalog of 14-Mev Neutron Reactions

J. E. Strain W. J. Ross

Efforts to catalog nuclear reactions obtained with 14.7-Mev neutrons[62] were continued. A compilation of these reactions and their cross sections, as well as of gamma spectra of the reaction products, was completed for the lighter elements ($Z < 30$).[63] Data are now being collected from irradiations of the heavier natural elements. Although the catalog is designed primarily for those interested in practical activation analysis, that is, irradiation periods of <20 min, the scope of the investigation has been extended to include the identification of reactions that result from longer periods (2 to 4 hr) of neutron bombardment.

11.9.b Studies of Neutron-Producing Targets

J. E. Strain W. J. Ross

Neutron-producing targets, such as Ti-^{3}H and Er-^{3}H, were studied to determine why targets that are presumably identical differ in neutron yield and useful life. The methods of target preparations, purity of the materials used, and neutron yield of targets were studied extensively. A final report on this work is being prepared. E. H. Kobisk[2] supplied the targets and is summarizing the methods used to prepare them.

11.9.c Standardization of Neutron-Flux Measurements

Enzo Ricci

A method for standardizing neutron-flux measurements performed at 14-Mev neutron generators was suggested by a group of scientists at the 1965 International Conference on Modern Trends in Activation Analysis, College Station, Texas, April 19–22.[64] This technique uses the reaction $^{63}Cu(n,2n)^{62}Cu$, which has a threshold energy of 11.0 Mev. To test the validity of the proposed method, this reaction, as well as the reactions $^{27}Al(n,p)^{27}Mg$ and $^{28}Si(n,p)^{28}Al$, were induced simultaneously at the neutron generator on targets of copper, aluminum, and silicon. These targets were irradiated first bare and then encased in ~0.5 in. of Lucite. The activity ratios $^{27}Mg/^{62}Cu$ and $^{28}Al/^{62}Cu$ were 7% higher for the Lucite irradiation than in the bare experiment; the standard deviations of the ratios were all smaller than 1.5%. This result clearly indicates that the copper reaction cannot be used alone to characterize a given facility, if one recalls that plastics and other materials are used extensively at neutron-generator rabbit installations. The results obtained can be easily explained and, in fact, were expected in the light of former studies.[65] The Lucite causes degradation of the neutron energy; the copper reaction cannot be induced by neutrons of energies lower than 11.0 Mev, whereas the reactions on aluminum and silicon (thresholds 1.9 and 4.0 Mev, respectively) can still occur. An experimental study is being made to improve and complete the standardization procedure proposed at the Texas conference.

11.9.d Activation of ^{64}Ni with 14-Mev Neutrons

J. E. Strain W. J. Ross

The reactions between ^{64}Ni and 14-Mev neutrons were investigated comprehensively. These reactions and the characterization of the resulting products have assumed special interest because of the wide discrepancies between our data and those that appear in the literature.[66] A paper is

[62] J. E. Strain and W. J. Ross, "Catalog of 14-Mev Neutron Reactions," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1964*, ORNL-3750, p. 59.

[63] J. E. Strain and W. J. Ross, *14-Mev Neutron Reactions*, ORNL-3672 (January 1965).

[64] Margaret B. Glos, "Activation Analysis Ready for Routine Use," *Nucleonics* 23(6), 62 (1965).

[65] E. Ricci, "Output Spectrum from 14-Mev Neutron Generators. Rapid Estimation and Influence in Cross-Section Measurements and Activation Analysis," *J. Inorg. Nucl. Chem.* 27, 41 (1965).

[66] I. L. Preiss and R. W. Fink, "New Isotopes of Cobalt; Activation Cross-Sections of Nickel, Cobalt, and Zinc for 14.8 Mev Neutrons," *Nucl. Phys.* 15, 326 (1960).

Table 11.9. Cross Sections for the Reactions of 14-Mev Neutrons with ^{99}Tc

Reaction	Product	Cross Section (mb)	
		Measured[a]	Calculated[b]
(n,n')	^{99m}Tc	81, 73	124 ± 24
(n,α)	^{96}Nb	7.3, 7.7	5 ± 5
(n,p)	^{99}Mo	~5	5 ± 5
$(n,2n)$	^{98}Tc	c	2100

[a]Two runs.

[b]L. Dresner, *EVAP-Fortran Program for Calculating the Evaporation of Various Particles from Excited Compound Nuclei* (Dec. 19, 1961) (unpublished report).

[c]^{98}Tc was not detected; half-life of ^{98}Tc is 10^6 years.

being written in which we classify the reaction products on the basis of our nuclear and chemical results.

11.9.e Activation Cross Sections for the Reactions of 14-Mev Neutrons with ^{99}Tc

Gerald Goldstein

To ascertain whether activation analysis with 14.8-Mev neutrons is a useful analytical method for technetium, cross sections for reactions were determined. A 50-mg sample of ^{99}Tc, as the metal, was irradiated for 1 hr together with aluminum flux monitors. Gamma spectra of the irradiated technetium were determined at intervals for several days. Table 11.9 shows the results of these measurements. An article on this work is to be published.[67]

[67]G. Goldstein, "Activation Cross Sections for the Reactions of 14.8-Mev Neutrons with ^{99}Tc," accepted for publication in the *Journal of Inorganic & Nuclear Chemistry*.

11.9.f Monitoring of Leached Fuel Elements with a Neutron Generator

W. J. Ross J. E. Strain
J. W. Landry[40]

In the program to recover spent reactor fuel by the shear-leach method,[68] it is important to know that essentially all the fissionable material has been removed from the short sections of chopped fuel pins by the acid leach. Because the stainless steel cladding is highly radioactive and because large quantities of fuel must be handled, a method had to be developed whereby batches of leached segments or "hulls" could be inspected for residual fuel within a processing hot cell. Based on the techniques of thermal-neutron activation analysis and delayed-neutron counting,[69] an apparatus was constructed that can detect as little as 1 mg of ^{235}U in the presence of the several hundred curies of radioactivity associated with the spent fuel. The moderated neutron flux from a pulsed Cockcroft-Walton accelerator irradiates the sample; a high-efficiency (~5%) $^{10}BF_3$ thermal-neutron detector monitors the delayed-neutron emission. In operation, the neutron generator is pulsed on for 10 sec; 1 sec after the end of the irradiation, the neutron emission is measured for 10 sec. In the present system a large basket of hulls is lowered in 10-cm increments through the radiation and counting moderator; a neutron count is made at each position.

Final evaluation of a full-scale working model of the apparatus is in progress. The apparatus is also being evaluated with respect to the Al_2O_3 residues from the Fluoride Volatility Process.[70]

[68]C. D. Watson, B. C. Finney, B. A. Hannaford, and G. A. West, *The Shear Leach Process for Spent Nuclear Fuel*, ORNL-3625 (to be published).

[69]F. F. Dyer, J. F. Emery, and G. W. Leddicotte, *Comprehensive Study of the Neutron Activation Analysis of Uranium by Delayed-Neutron Counting*, ORNL-3342 (July 27, 1962).

[70]M. A. Breazeale, F. M. Carr, and R. P. Milford, "A Proposal for an ORNL Fluid-Bed Volatility Pilot Plant" (March 31, 1964) (unpublished report).

12. Inorganic Preparations

M. T. Kelley
D. E. LaValle
R. B. Quincy

The routine preparation of anhydrous fused salts continued and usually consisted of 100-g quantities of alkali, alkaline-earth, and rare-earth-metal halides. Of these salts, $MgCl_2$ and $ZnCl_2$ were prepared for the Ion Spectroscopy Laboratory of the Metals and Ceramics Division. The $MgCl_2$ was obtained by the action of NH_4Cl on $MgCl_2 \cdot 6H_2O$ to produce $MgNH_4Cl_3$, which was decomposed by heating in a vacuum; the resulting $MgCl_2$ was purified by sublimation. The $ZnCl_2$ was prepared by causing chlorine to react with high-purity zinc metal. Very pure CsCl was made by multiple recrystallization of $CsICl_2$, which was finally decomposed to CsCl by heating in a vacuum. The last traces of the complex chloroiodide ion were removed by fusing the CsCl and bubbling hydrogen through the melt.

Compounds, other than fused salts, prepared for the High Temperature and Structural Chemistry Group of the Chemistry Division were 30-g quantities of Na_2O and K_2O. These compounds were made in a helium-atmosphere glove box. The alkali metal, contained in a stainless steel beaker, was melted on a small hot plate. The melt was stirred manually while pure oxygen was metered into the helium atmosphere in an amount insufficient to react with all the metal. The resulting spongy mass was transferred to a nickel boat, which was then placed in a quartz tube; the excess metal was removed by sublimation.

A small sample (~3 g) of $LiOBr \cdot 2H_2O$ was prepared for the Radiation Chemistry Group of the Chemistry Division. The method followed that of Williamson,[1] in which bromine is added slowly to a solution of LiOH at 0°C to form LiOBr and LiBr. A stoichiometric amount of freshly precipitated Ag_2O is added to precipitate the bromide ion; in the process half the amount of the original LiOH is regenerated. The treatment with Ag_2O is repeated once, and finally the last of the LiOH is removed by wet ion exchange resin in the hydrogen form. The $LiOBr \cdot 2H_2O$ is crystallized from the solution by evaporation at reduced pressure and low temperature. An attempt to prepare barium bromite monohydrate, $Ba(BrO_2)_2 \cdot H_2O$, according to the method of Kircher and Periat[2] did not produce a pure product.

For the Nuclear Chemistry Group of the Chemistry Division, the preparation of Na_2ReCl_6 was attempted and was abandoned, because the high solubility of the salt prevented its crystallization from solution. Instead, 50 g of K_2ReCl_6 was prepared by the method[3] in which hypophosphorous acid is used to reduce the perrhenate.

The work for the Solid State Division fell into three categories. The heat treatment of alloys of the rare-earth metals with other metals and of alloys of transition metals (such as Cr, Re, Ta, W, and Fe) with each other comprised most of the work for the Neutron Diffraction Group. The routine preparation of high-purity KCl continued for the Research and Development of Pure Materials Group. For the Neutron Diffraction Studies Group, the investigation of the holmium-sulfur system was essentially completed. It was desired to obtain a simple cubic structure in this system, and very little information on holmium sulfides existed in the literature. The preparation of Ho_2S_3 was accomplished in a double-chambered evacuated tube that contained sulfur at 500°C in one chamber and holmium metal at 700°C in the other. A dull-red material was obtained, which was heated further in vacuum at several temperatures. Between 800 and 900°C the dull-red form changed to a dull-green form stable to 1300°C. At 1400°C a black form was

[1] S. M. Williamson, "An Investigation into the Preparation of Crystalline Hypobromites," *Technical Report of the Physical Research Laboratory*, The Dow Chemical Co., Midland, Mich. (Oct. 10, 1960).

[2] R. Kircher and R. Periat, "Preparation du bromite de baryum cristallisé," *Ind. Chim. Belge*, Suppl. 1, 877 (1959).

[3] G. W. Watt and R. J. Thompson, "51. Potassium Hexachlororhenate(IV) and Potassium Hexabromorhenate(IV)," pp. 189–92 in *Inorganic Syntheses*, vol. VII, ed. by J. Kleinberg, McGraw-Hill, New York, 1963.

obtained. The three forms gave different x-ray patterns of very complicated structure; the patterns are not like those of the neighboring rare-earth-metal sulfides. (Since this work was completed, Collins and Loriers[4] have prepared the dull-green form of Ho_2S_3 and have determined that its structure is monoclinic.) The attempt to prepare HoS by the reduction of Ho_2S_3 with HoH_2 yielded a yellow product 80% of which was the cubic HoS phase; the remainder was an unidentified phase. However, with the adjacent rare-earth metal, erbium, an attempt to produce the analogous lower erbium sulfide, ErS, in a single phase was successful. However, to avoid the problem of undecomposed hydride, ErS was made by combining the elements in a double-chambered tube as described above.

By the same technique, thulium phosphide, thulium nitride, and holmium nitride were also prepared. Unlike earlier syntheses of nitrides achieved by action of ammonia on the metal hydride, these nitrides were obtained by heating the finely divided metal in a static atmosphere of nitrogen. It was found that some hydrides of rare-earth metals are not completely decomposed even at 1000°C in vacuum, and it was hoped to achieve higher purities by this alternative method.

Also for the Neutron Diffraction Group, a series of preparations of potassium superoxide, KO_2, was made. In general, the method of Kazarnovskii and Raikhshmein[5] was followed, in which a mixture of nitrogen with a gradually increasing percentage of oxygen is passed over the metal while it is heated slowly to 370°C. This principle, used in all the preparations of KO_2, but with some variation in equipment, yielded products that all approach 100% in purity but differ widely in magnetic properties. Rubidium superoxide, RbO_2, was also prepared by the same method, but an attempt to prepare sodium superoxide, NaO_2, which cannot be made at normal pressures, by a bomb method[6] did not yield a pure product.

For the Special Electromagnetic Separations of Heavy Elements Group of the Isotopes Division, work was begun on a program to prepare solid solutions of uranium trihalides in lanthanum halides. Initially, 50-g quantities of UCl_3 and UI_3 were made from uranium depleted in ^{235}U for preliminary studies. These products were about 98% pure. To prepare UCl_3, an apparatus similar to that described by Johnson, Butler, and Newton[7] was used; their procedure of passing HCl gas through UH_3 at 250°C was followed. The trichloride is formed, but no single point of temperature and duration of reaction time were found at which 100% UCl_3 can be obtained. Before all the UH_3 is decomposed, some UCl_4 appears from the reaction of the HCl gas on the newly formed UCl_3. The disappearance of the UH_3 is followed best by x-ray examination. The UCl_4 is eliminated by reduction with hydrogen at a temperature not greater than 550°C, since UCl_3 shows some evidence of disproportionation at a temperature as low as 525°C. However, at 550°C the rate of reduction of UCl_4 is very slow; 10-g quantities may require as long as 24 hr. The stringent purity requirement thereby imposed on the hydrogen was met by passing the gas through the same prepurifying quantity of UCl_3 at 300°C that was used for the purification of the HCl gas. Disappearance of the UCl_4 is followed best by assay of the UCl_3 by the hydrogen-evolution method.[8]

Since gaseous HI is now available from the Matheson Company, Inc., it was convenient to attempt to prepare UI_3 in the same manner as the UCl_3 was prepared. However, the action of HI on UH_3 produces UI_4 almost exclusively. Reduction of UI_4 with hydrogen yields a mixture of substances in which the valence of uranium is lower than in UI_3. However, the UI_4 was decomposed successfully to UI_3 by heating it in a vacuum at 500°C. Since part of the UI_4 decomposes and part sublimes, the sublimate must be treated further for a higher yield.

About 27 g of $^{235}UCl_3$ was made from 97% uranium enriched in ^{235}U and was incorporated in

[4] G. Collins and J. Loriers, "Les oxysulfures et sesquisulfures de terbium, holmium, thulium, et lutétium," *Compt. Rend.* **260**(19), 5043 (1965).

[5] I. A. Kazarnovskii and S. I. Raikhshmein, chap. 11, "Inorganic Peroxides. Upper (sic) Potassium Oxides" (English Transl.), *Zh. Fiz. Khim.* **21**, 245 (1947).

[6] S. E. Stephanou, W. H. Schechter, W. J. Argersinger, Jr., and J. Kleinberg, "The Absorption of Oxygen by Sodium Peroxide: Preparation and Magnetic Properties of Sodium Superoxide," *J. Am. Chem. Soc.* **71**, 1819 (1949).

[7] O. Johnson, T. Butler, and A. S. Newton, "Preparation, Purification, and Properties of Anhydrous Uranium Chlorides," paper 1, p. 2, in *Chemistry of Uranium. Collected Papers*, ed. by J. J. Katz and E. Rabinowitch, TID-5290, Book 1 (1958).

[8] D. L. Manning, W. K. Miller, and R. Rowan, Jr., *Methods of Determination of Uranium Trifluoride*, ORNL-1279 (Apr. 25, 1952).

77.6 g of anhydrous sublimed $LaCl_3$ as an ~20 mole % solid solution. The intimate mixture of the two substances was fused at 900°C in an alundum crucible contained in a quartz tube in an atmosphere of argon. The fused product was identified by x-ray examination as a single phase with x-ray parameters intermediate between those of UCl_3 and $LaCl_3$; chemical analysis showed 25.68 wt % UCl_3 (18.96 mole %), 0.32 wt % UCl_4, and 1.33 wt % $UOCl_2$.

Work for the Physics Division was confined to the Low Temperature, Nuclear, and Solid State Physics Group. A small sample of $AuAl_2$ was prepared, and a series of alums of the type $AlNH_4(SO_4)_2 \cdot 12H_2O$ in which some of the Al atoms are replaced by atoms of ^{57}Fe in amounts varying from 3 to 10 atom %. Also, in one case a quantity of $FeNH_4(SO_4)_2 \cdot 12D_2O$ was made by dissolving the anhydrous alum[9] in D_2O and recrystallizing it. In the water and ammonia groups, 91 and 52%, respectively, of the hydrogen atoms were replaced by deuterium.

For the Small Potassium Systems Group of the Reactor Division, sodium chromite, $NaCrO_2$, a known compound, was easily prepared by the reaction of Na_2CO_3 with chromium sesquioxide, Cr_2O_3, at 900°C. Numerous attempts to prepare the potassium analog were unsuccessful. In a similar problem for the High Temperature Materials–Materials Compatibility Group of the Metals and Ceramics Division, attempts to prepare potassium niobates that contained quadrivalent niobium were only partly successful, although the sodium series is known to exist. The synthesis of the reported[10] $K_2Nb_2O_5$ by the reaction of K_2CO_3 with niobium dioxide, NbO_2, could not be duplicated. However, the reaction of potassium metal with niobium pentoxide, Nb_2O_5, in a sealed niobium tube yielded a substance of composition very nearly that indicated by the formula K_2NbO_3.

[9] N. V. Shishkin, "Anhydrous Ferric Alums," *J. Gen. Chem. USSR (English Transl.)* 10, 271 (1940).

[10] E. I. Krylov and A. A. Sharnin, "Synthesis and Properties of Niobium Bronzes," *J. Gen. Chem. USSR (English Transl.)* 25, 1637 (1955).

13. Organic Preparations

J. C. White
P. F. Thomason
H. L. Holsopple

Several organic compounds were custom synthesized for research use in the Analytical Chemistry, Biology, Chemical Technology, Metals and Ceramics, and Physics Divisions. These compounds, prepared and purified on request, were not readily available from commercial sources.

13.1 COMPOUNDS PREPARED

13.1.a Quaternary Phosphonium Halides

Benzyltributylphosphonium bromide (375 g) and benzyltributylphosphonium chloride (400 g) were prepared for the Metals and Ceramics Division according to the reactions

$$(C_4H_9)_3P + C_6H_5CH_2Br \rightarrow [(C_4H_9)_3PCH_2C_6H_5]Br ,$$

$$(C_4H_9)_3P + C_6H_5CH_2Cl \rightarrow [(C_4H_9)_3PCH_2C_6H_5]Cl .$$

Results of elemental analysis were in good agreement with theoretical values (Table 13.1) based on the formulas indicated above.

13.1.b 2,6-Dioximinocyclohexanone

A new and unusual class of compounds that contain a monovalent metal ion, an organic anion, and 2,6-dioximinocyclohexanone in a 1:1:1 mole

Table 13.1. Compositions of Two Quaternary Phosphonium Halides

Component	Weight Percent			
	In Benzyltributylphosphonium Bromide		In Benzyltributylphosphonium Chloride	
	Theoretical[a]	Found	Theoretical[b]	Found
Carbon	61.1	61.1	69.4	69.3
Bromide	21.4	21.2		
Chloride			10.8	10.3

[a] $C_{19}H_{34}PBr$.
[b] $C_{19}H_{34}PCl$.

ratio has been discovered.[1] A quantity of 2,6-dioximinocyclohexanone was requested by the Nuclear and Radiochemical Analyses Group of the Analytical Chemistry Division for use in testing the suitability of these coordination compounds as extractants for sodium. The preparation, to be described in a formal report, consists essentially in the reaction of methyl nitrite with cyclohexanone in an acid ethereal solution. The dioximino ketone (25 g) was synthesized; elemental analysis of the product gave the following results (in weight percent):

Component	Theoretical[a]	Found
Carbon	46.2	46.2
Hydrogen	5.16	5.14
Nitrogen	18.0	17.8

[a] $C_6H_8N_2O_3$.

13.1.c ^{10}B Diborane

Essentially pure ^{10}B diborane, $^{10}B_2H_6$, was prepared for the Physics Division by the incremental addition of $^{10}BF_3$-etherate to a diethyl ether solution of lithium aluminum hydride. The product was purified by fractional condensation, and its purity was established from its infrared absorption spectrum and mass spectrometric analysis. The details of this preparation are given elsewhere.[2]

13.1.d Methyl Methanesulfonate

Methyl methanesulfonate (25 g) was synthesized for research members of the Biology Division. This compound, used as a carcinogen in animal studies, was prepared by the reaction of methyl iodide with silver methanesulfonate. Silver methanesulfonate was prepared by treating silver oxide with methanesulfonic acid in acetonitrile. The purity of the product was established by elemental analysis and by nuclear-magnetic-resonance spectrometry; the results of the elemental analysis (in weight percent) are:

Component	Theoretical[a]	Found
Carbon	21.8	21.9
Hydrogen	5.5	6.1
Sulfur	29.1	27.6

[a] $C_2H_6O_3S$.

13.1.e Heptadecylamine

Heptadecylamine (500 g) was prepared for the Chemical Technology Division according to Barnes'[3] three-step process in which heptadecanol and 2-ethylhexylamine are used.

13.1.f Azo Dyes

The literature gives several new sensitive spectrophotometric procedures in which azo dyes are

[1] A. F. Ferris and O. L. Salerni, "Compounds Containing a Monovalent Metal Cation, an Organic Anion, and an α,α'-Dioximino Cyclic Ketone," presented at the Fifteenth Annual Kansas City Chemistry Conference, Kansas City, Mo., Nov. 15, 1963.

[2] H. L. Holsopple and Lucy E. Scroggie, *Synthesis and Infrared Absorption Spectrum of Diborane-^{10}B*, ORNL-TM-1061 (Feb. 19, 1965).

[3] R. K. Barnes, Union Carbide, Chemicals Division, South Charleston, W.Va., personal communication to F. G. Seeley, Chemical Technology Division, March 1965.

used. Two of these dyes, Calcichrome and "chlorophosphonazo III," were prepared for developmental use by the Analytical Chemistry Division.

Calcichrome was prepared by diazotizing 1-amino-8-naphthol-3,6-disulfonic acid followed by self-coupling in strong sodium hydroxide solution. The reaction of this chromogenic reagent with calcium ions in alkaline solution is highly sensitive.

Chlorophosphonazo III was synthesized in a three-step process. First, *o*-nitroaniline diazoniumfluoborate was prepared by diazotizing the reaction product of *o*-nitroaniline and fluoboric acid. A mixture of this compound, cuprous chloride, and phosphorus trichloride was reacted to produce a copper complex from which a small amount of 2-amino-5-chlorobenzenephosphoric acid was recovered with difficulty. The amino compound was then diazotized and coupled with chromotropic acid (1,8-dihydroxy-3,6-disulfonic acid) to yield the chromogenic reagent, chlorophosphonazo III.

Part B. Service Analyses

A summary of the service analyses made by the laboratories of the Analytical Chemistry Division is given in the table below.

Summary of Analytical Service Work

Group Making Analyses	Average Number in Group		Number of Results Reported																									
			For ORNL																			For Others						
	Scientists	Technicians	Analytical Chemistry	Biology	Chemical Technology	Chemistry	Electronuclear	Health	Health Physics	Inspection Engineering	Instrumentation and Controls	Isotopes	Metals and Ceramics	Neutron Physics	Operations	Physics	Plant and Equipment	Reactor	Reactor-Chemistry	Solid State	Thermonuclear	General Electric	K-25	Miscellaneous	ORINS	Y-12	Total
Mass Spectrometry Laboratory	2	4			1,858	42						8,280	2,182	18	14	310		5,220	2,972				18	4563		310	25,793
Optical and Electron Microscopy	3	2	48		174	47			4			30	156			50		427	256							2	1,199
Process Analyses																											
General Analyses Laboratory	13	25	134	58	34,860	857			978			172	9,027	501	687	27	732	5,753	5,299	723				605			60,451
High-Level Alpha Radiation Laboratory	3	4			7,615	2			10			359	30		10				10						27	17	8,087
High-Radiation-Level Analytical Laboratory	11	14			12,398	1			22			1,139	597		4,706			1,676	1,363	4						5	21,936
Radioisotopes-Radiochemistry Laboratory	10	19	12	2	11,203	12			4,262	16	41	10,032	811	93	7,259	14		10,272	19,424	1232		1066				16	65,796
Spectrochemical Analyses Laboratory (Y-12)	1	2	247	9	1,416	282	146	11,101				10,538	772	156				1,788	2,004	900	117					394	29,873
TRU Mass Spectrometry	1	1	660		307	42						98	138	38					127							46	1,458
X-Ray and Spectrochemical Analyses	9	6	9	14	4,911	352			13,592			547	2,824	447	43	905		782	775	623				2925			28,764
Total	53	77	1110	83	74,742	1637	146	11,101	18,868	16	41	31,195	16,537	1253	12,719	1306	732	25,918	32,230	3482	117	1066	18	8093	27	790	243,357

14. Quality Control

L. T. Corbin G. R. Wilson

During the 12-month period July 1964 through June 1965, the overall quality level of the analytical work remained about the same as in the previous 12-month period. The decrease in the quality level of the General Hot Analyses Laboratory is due mostly to the decrease in the number of control tests run, particularly coulometric determinations of uranium.

Several changes and additions have been made that caused a decrease in the Quality Control Program. The Materials Testing Laboratory and the Reactor Engineering Services Laboratory were consolidated with the General Hot Analyses and the General Analyses Laboratories. The quarterly quality-control calculations and plotting of data were programmed for the computer. In June a quality-control program for the MSRE analyses done in the General Hot Analyses Laboratory was started. This program includes the determinations of Zr, Be, Fe, Cr, Ni, and U.

Table 14.1 shows the distribution of the control tests by laboratory; Table 14.2 gives the number of control tests and their specific characteristics.

Table 14.1. Distribution by Laboratories of Control Tests for July 1964 Through June 1965

Laboratory	Number of Control Results		Quality Level (%)[a]	
	Total	Outside Fixed Limits	1964	1965
General Hot Analyses	733	57	95.6	92.2
General Analyses	3240	86	96.8	97.3
	Total 3973	Total 143	Av 96.5	Av 96.4

[a]Control results within prescribed 2*S* limits.

Table 14.2. Distribution by Methods of Control Results for July 1964 Through June 1965

Type of Method	Constituent	Number of Control Programs		Number of Control Results	
Colorimetric (spectrophotometric)	Aluminum	1		170	
	Chromium	2		207	
	Iron	2		249	
	Molybdenum	1		46	
	Nickel	2		199	
	Niobium	1		36	
	Thorium	3		354	
	Uranium	2	14	584	1845
Coulometric	Uranium	2	2	918	918
Fluorometric	Uranium	2	2	365	365
Gravimetric	Carbon	2	2	538	538
Potentiometric	Uranium	1	1	20	20
Volumetric	Aluminum	1		155	
	Nitrate	1		114	
	Sulfate	1	3	18	287
		Total	24		3973

15. Mass Spectrometric Analysis

A. E. Cameron
J. R. Sites

The Mass Spectrometry Laboratory had numerous requests for certified analyses of cylinders of helium, argon, and nitrogen. Frequently, these were on an emergency basis and, at times, necessitated overtime work. To better supply the needs of ORNL and Y-12, the stores departments at X-10 and Y-12 now stock a supply of analyzed cylinders. This arrangement evens out the work load in the laboratory for gas analyses and provides much better customer service.

Supporting analyses were continued for the sweep gas in the ORR loop, fission product off-gases, thermal-diffusion products, and special gas mixtures. The problem of an impure cover gas in a large dry box in the welding shop was solved. Several mixtures of ^{3}He, CH_4, and Xe that are used in a counter were analyzed.

Thirty-five samples of enriched isotopes of Te, Se, Zn, Sn, Cd, and Si were analyzed for the Harwell Laboratory of the United Kingdom Atomic Energy Research Establishment. These elements are among the most difficult to analyze mass spectrometrically.

Filaments for the standard-solids mass spectrometer were placed in calutron receivers along the *Z* direction of the ion beam. For the nanogram amounts of strontium collected, this technique eliminates possible contamination by chemistry and handling. Data taken on several strontium samples are now being evaluated by the Research and Development Section of the Stable Isotopes Separation Group of the Isotopes Division.

The increased rate of submission of separated stable isotopes was matched by a 40% higher analysis rate. A total of 1315 samples were analyzed for 37 elements: Li, B, C, Mg, Si, S, Cl, K, Ca, Ti, Fe, Ni, Zn, Ga, Se, Br, Rb, Sr, Zr, Mo, Ru, Pd, Cd, In, Sn, Te, Ba, Ce, Eu, Yb, Lu, W, Ir, Pt, Hg, Tl, and Pb.

A number of radioactive samples were analyzed for the Isotopes Division. Calculations from mass spectrometric data indicate that ^{36}Cl may have a much lower neutron cross section than the accepted value of $\sim$90 barns. If the lower value is confirmed, it may be possible to produce ^{36}Cl in a much higher neutron flux than is now used.

Mass analysis of ^{40}K samples indicates that the accepted neutron-cross-section value of $\sim$70 barns for this isotope may be much too high. If further analyses of unirradiated and irradiated samples prove that this lower cross section is more reliable, then the price ($1400/mg) for highly enriched ^{40}K may be reduced considerably.

16. Spectrochemical Analysis Laboratory

A. E. Cameron
J. A. Carter

A total of 29,870 results were reported on some 1800 samples, which is a 50% increase in the sample load. About one-third of the results were reported to the Isotopes Division on stable isotopes of B, Ba, Br, C, Ca, Cd, Cl, Cr, Cu, Eu, Ga, Ge, Hf, Hg, Fe, K, Li, Lu, Pb, Pd, Pt, Mg, Mo, Ni, Ru, S, Se, Si, Sn, Sr, Ti, W, Yb, and Zn; one-third on environmental air samples to the Health Division; and the remaining third to 12 other divisions. On some 150 reactor construction parts, certification analyses were made for Cr, Mn, Mo, Nb, Ni, Si, Ti, and V; some 50 MSRE samples were analyzed for rare earths.

Modified methods and techniques were used to analyze several new types of sample. For example, it was necessary to prepare synthetic standards and to establish calibration curves employing the Paschen Direct Reader to analyze superstrength Ta-W-Hf alloys and Au-Dy, Au-Tb, Tb-Sc, Sn-Li, Sn-Ce, Sn-Ce-Li, and Pb-Ce alloys. Procedures were also established to determine 12 major and minor constituents of the modified INOR-8 alloy.

17. Process Analyses

L. T. Corbin

17.1 HIGH-LEVEL ALPHA RADIATION LABORATORY

J. H. Cooper

The High-Level Alpha Radiation Laboratory reported 8080 results – 94% for the Chemical Technology Division and 5% for the Isotopes Division.

An increase in the alpha radioactivity of enriched uranium oxide prepared by the sol-gel process necessitated that analysis be done in the High-Level Alpha Radiation Laboratory. Methods conventional for inactive materials were used; O/U ratio, nitrate, and carbon were determined.

The Chemical Technology Division, in its study of plutonium fluoride volatility, required the analysis of many types of samples for plutonium. Initially, the determination was made by counting the gamma radioactivity of an entire sample. However, at low levels of radioactivity, this method is unreliable because of high backgrounds. The samples were dissolved, and an aliquot of the solution was counted for plutonium alpha radioactivity. Samples of various metal fluorides were dissolved in mineral acid that contained boric acid to complex the fluoride. Samples that contained aluminum oxide were fused with sodium carbonate–sodium tetraborate, and the melt was dissolved in nitric acid or hydrochloric acid.

Americium alpha radioactivity was determined in samples in which the ratio of curium radioactivity to americium radioactivity was greater than 1000. The americium was oxidized to the fluoride-soluble hexavalent state, and the curium was then carried down on lanthanum fluoride. Americium alpha radioactivity was determined in the supernatant solution. Alpha pulse-height analysis showed that less than 0.01% of the curium remained in solution with the americium. Tests on standard solutions showed that ~90% of the americium was recovered.

17.2 GENERAL ANALYSES LABORATORY

W. R. Laing

A total of 60,413 results were reported by the General Analyses Laboratory. Several new or modified methods were placed into service. A modification of the method of Holt and Stoessel[1] was used to determine oxygen in U_3O_8, Al_2O_3, ThO_2, and Nb_2O_5. A Leco inert-gas-fusion apparatus was used, and the resulting CO_2 was adsorbed on Ascarite and weighed. The ORNL model Q-1728 velocity servo titrator was used to titrate benzoic acid and nitric acid present in solutions in the 0.0001 to 0.02 *N* range. The compatibility of motor oils and antifreezes with each other was tested. A Shatterbox grinder was used to crush 20 g of sintered Nb_2O_5 ash to <300-mesh size in 5 min. A 2 *N* H_2SO_4–H_2O_2 solution was found to dissolve sol-gel UO_2 for nitrate determination. Existing methods were modified to permit the determination of C, O, and F in vapor-deposited tungsten at the <10-ppm level. The

[1] B. D. Holt and J. E. Stoessel, "Macrodetermination of Oxygen in Metal Oxides by Inert Gas Fusion," *Anal. Chem.* 36, 1320 (1964).

Technicon Auto Analyzer was used with a flow cell in a Beckman model B spectrophotometer to determine U, Th, Al, and Fe. In each of these determinations the sampler was operated at a rate of 60 samples/hr. Samples of algae that weighed ~5 mg were analyzed for C, H, N, and ash.

A new vacuum-fusion analyzer that will include an uninsulated crucible and a gas chromatograph is being built. These modifications should result in increased sensitivity and speed of analysis.

Computer programs for quality control and for measurements of surface area, helium density, and coating density have been completed.

17.3 HIGH-RADIATION-LEVEL ANALYTICAL LABORATORY (HRLAL)

C. E. Lamb

The General Hot Analyses Laboratory's operations were transferred into the Analytical Chemistry Division's new High-Radiation-Level Analytical Laboratory (HRLAL) (Fig. 17.1) located in Building 2026.[2] Equipment was installed throughout the building to provide the analytical chemistry support necessary to programs involved in the study of highly irradiated materials. The shielding integrity of the HRLAL work cells was tested with a 1400-curie ^{60}Co source; the radiation penetration measured <1 mr/hr along the outer surfaces. The first radioactive sample to be received in the HRLAL, a 50-curie ^{147}Pm source, was analyzed on December 17, 1964.

A personnel training program on the operation of the HRLAL was initiated. Eight sessions covered topics on heat and ventilation systems, off-gas systems, cell-door and unloading-cell operations, and emergency systems and procedures.

[2] L. T. Corbin, W. R. Winsbro, C. E. Lamb, and M. T. Kelley, "Design and Construction of ORNL High-Radiation-Level Analytical Laboratory," *Trans. Am. Nucl. Soc.* 6(2), 440 (1963).

Fig. 17.1. Photograph of the High-Radiation-Level Analytical Laboratory Building.

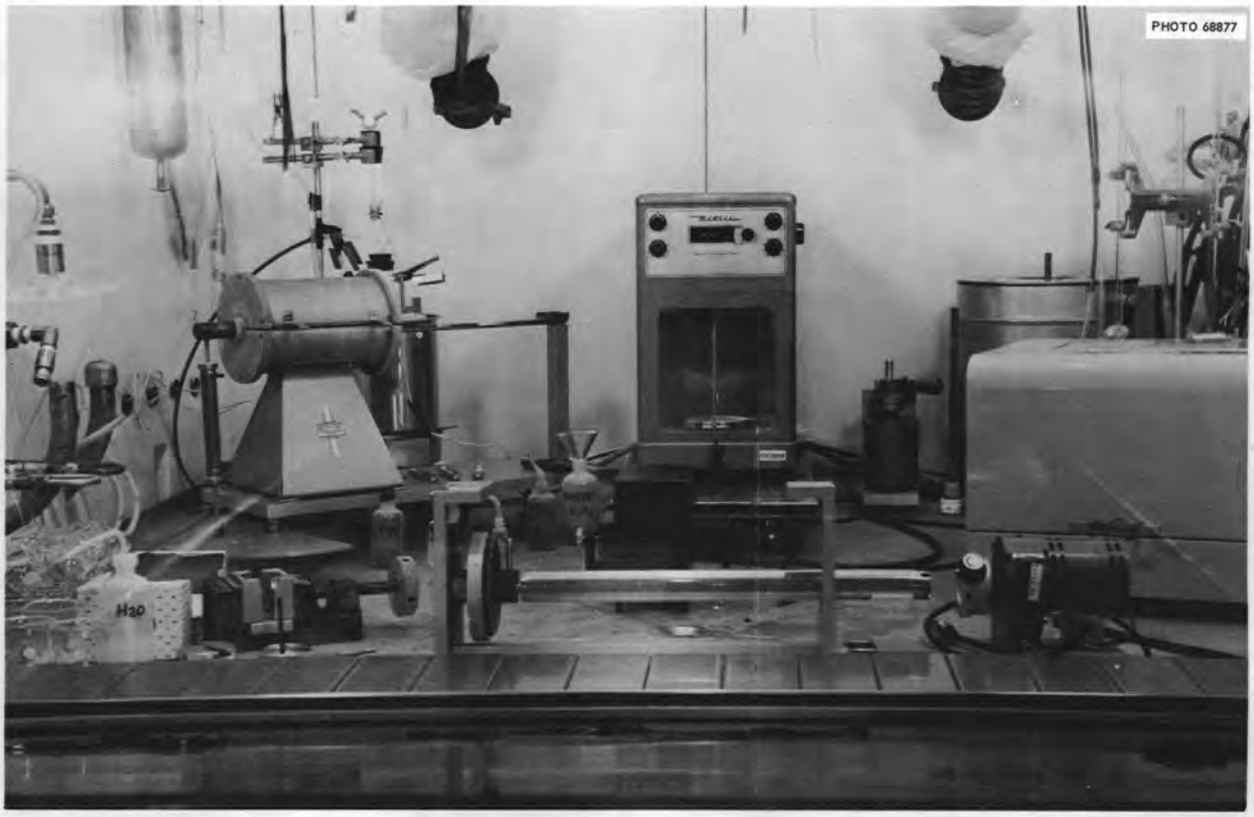

Fig. 17.2. In-Cell Equipment for the Physical Treatment of Undissolved MSRE Salt Samples.

A quality-control program was established for use with MSRE fuel-salt samples. Synthetic solutions that contained the same components as solutions of MSRE salt were prepared for analysis along with each dissolved sample. Three hot cells were used to contain the equipment required for handling and analyzing MSRE samples. One cell (Fig. 17.2) was equipped for the physical treatment of the sample (ladle unloading, salt crushing, weighing, etc.), whereas the other two cells (Fig. 17.3) were equipped for chemical analyses. Samples were received almost daily from the MSRE during precritical and zero-power experiments. They were analyzed[3] for U, Zr, Li, Fe, Ni, Mo, Be, and F^-. The results[4] were within the expected limitations of the methods except for a slight bias in those for U, Li, and Be. The biases are being studied to determine their causes and the measures necessary for their corrections.

In the two sealed cells of the HRLAF and in three of the glove boxes of the Alpha Facility, treatment of samples from the Curium Recovery Program[5] was continued for the determination of Al, Cl^-, NO_3^-, free acid, and rare-earth elements. Radiolytic formation of gas in the samples that contained $>10^{11}$ dis/min of ^{242}Cm created a problem in the aliquot-removal step but was controlled by rapid dilution. Waste removal from the cells required that persons enter the cells as many as three times a week and that arduous "bag-out" procedures in conformance with safety regulations be used.

[3]R. B. Briggs, "Development and Evaluation of Equipment for Analyzing Radioactive MSRE Fuel Samples," *MSR Program Semiann. Progr. Rept. Feb. 28, 1965*, ORNL-3812, pp. 155–60.

[4]R. E. Thoma, "MSRE Salt Chemistry During Precritical and Zero-Power Experiments," unpublished MSRE memorandum to P. N. Haubenreich (Aug. 2, 1965).

[5]C. E. Lamb, "General Hot Analyses Laboratory," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1964*, ORNL-3750, pp. 71–72.

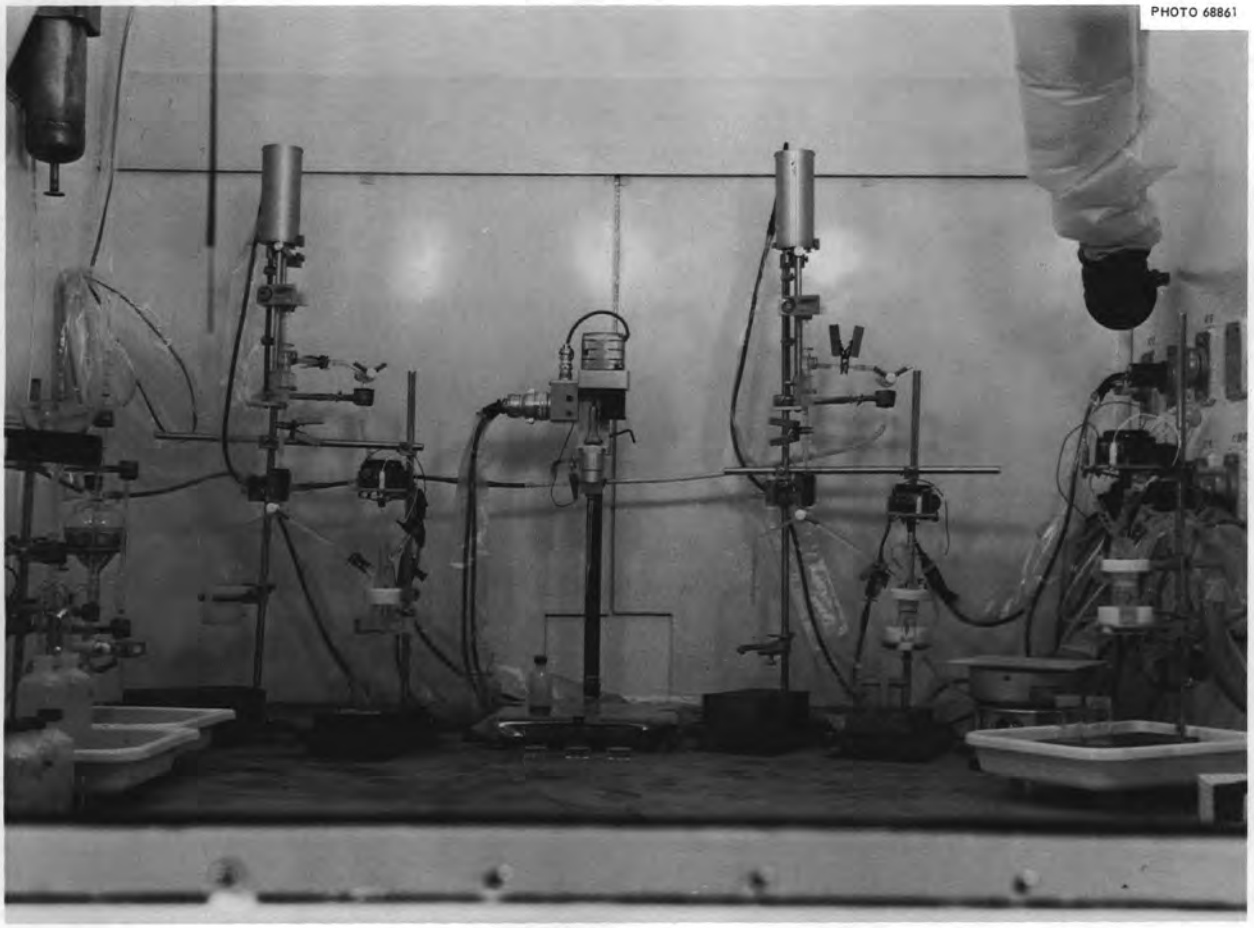

Fig. 17.3. In-Cell Equipment for the Chemical Analysis of Dissolved MSRE Samples.

Dissolution of unusual and highly irradiated materials was continued as an important phase of the work. Among the unusual materials received and dissolved were rubber O-rings, fiber-glass filter paper, Dacron, platinum, thorium oxide–stainless steel, plutonium oxide–aluminum, zirconium oxide–calcium oxide, thorium oxide–uranium oxide–stainless steel, europium molybdate–aluminum, and europium oxide–aluminum. The dissolved materials were usually analyzed for uranium and fission products.

Samples to be analyzed by gas chromatography were received from eight programs in the Chemical Technology Division. In addition to the usual constituents, deuterium was identified and measured in samples received from a program of study on deuterated hydrocarbons.

17.4 RADIOISOTOPES-RADIOCHEMISTRY LABORATORY

E. I. Wyatt

The Radioisotopes-Radiochemistry Laboratory increased the number of analyses performed during the year by 35%. This increase came from the Reactor Chemistry Division's experiments with fuel meltdown and with fission product transport in graphite and in gas mixtures and from the Reactor Division's Nuclear Safety Pilot Plant (NSPP). Most of the samples from the NSPP were assayed by gamma-ray spectrometry and computer codes. As many as 15,000 determinations on 1500 samples from a single NSPP fuel-meltdown

experiment were performed in this way. An automatic IBM card-punching machine was acquired on a rental basis for converting information stored on punched tape to punched cards.

The Chemical Technology Division asked that a standard test be devised to measure the ease and completeness of decontamination of various available corrosion-resistant structural materials. It was requested that the aqueous mixture 0.4 *M* oxalic acid–0.05 *M* sodium fluoride–0.30 *M* hydrogen peroxide be used as the decontaminant. Coupons of 30 structural and coating materials of fixed size and shape were treated with a neutral solution of fission products and were allowed to air-dry. They were then decontaminated in a uniform manner. The residual radioactivity was identified and measured by gamma-ray spectrometry. New structural materials will be tested, and the results will be given in an unpublished report.

A large part of the analytical work of the group was transferred from Building 3550 to Building 3019. This move provided additional facilities for hot-cell work and for the handling of alpha radioactivity in high levels.

The study of the mass distribution of fission products in the epicadmium-induced fission of ^{237}Np was described.[6]

17.5 HIGH-RADIATION-LEVEL ANALYTICAL FACILITY (HRLAF)

L. G. Farrar

About 70% of the work done in the High-Radiation-Level Analytical Facility (HRLAF) has involved high-level alpha-emitting materials in the presence of fission products. Containment efforts associated with cell maintenance, solid-waste removal, and manipulator repairs continue to be of prime importance. The handling and storage of curium samples whose levels of alpha and gamma radioactivity are 10^{13} and 10^{9} dis min^{-1} ml^{-1}, respectively, have made it necessary to enclose the access drawers of the work cells with a glove-box-type cubicle to permit the removal of alpha-contaminated sample dilutions for transfer to glove boxes. Hot cells sealed in glove-box fashion for work with high-level alpha-emitting materials were operated continuously over the past year and have maintained a contamination differential of about 10^{7} over adjacent cells.

An effort is being made to improve the method of solid-waste disposal. Multiple sample dilutions on alpha-emitting materials have caused a twofold increase in the volume of solid waste removed from hot cells for burial. Several methods of volume reduction and packaging for solid waste are being tried.

Twelve new manipulators in Building 2026 and eight in Building 3019 were equipped with two-piece manipulator boots. Since most boot failures occur either in the lower section or in the gauntlet, the new manipulator boots feature a remotely replaceable lower section that has improved resistance to attack by heat and acids.

The liner material of the zinc bromide viewing windows in the hot cells of the HRLAF is showing accelerated breakdown. It recently became necessary to reprocess the zinc bromide solution and to clean the viewing surfaces of Cell No. 3. The solution was removed, filtered, and restabilized, but it was not possible to remove all the iron from the corroded liner. The interior window surfaces were cleaned and dried with a high-pressure spray of water followed by ethyl alcohol. The window is back in service; a continuous argon purge is now used to prevent additional liner oxidation. It is expected that five more of the windows will soon require reconditioning.

Routine hot-cell maintenance has included repair to 156 manipulators and decontamination of 14 work cells. In Cell No. 1 of the HRLAF, a new acid fume hood with scrubber was installed; in Cell No. 6, the arc stand and filter system for the emission spectrograph were replaced.

Replacement of the overhead cell off-gas duct was completed in March 1965. Drawings for the enclosure of the loading dock attached to the west side of the HRLAF were completed. Construction of this enclosure is needed to improve the containment in the cell-access area.

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[6] R. R. Rickard, H. A. Parker, and E. I. Wyatt, *Mass-Fission Yield Curve for ^{237}Np*, Dec. 24, 1964 (unpublished report).

Part C. ORNL Master Analytical Manual

18. ORNL Master Analytical Manual

M. T. Kelley
Helen P. Raaen

18.1 CUMULATIVE INDEXES TO THE *ORNL MASTER ANALYTICAL MANUAL*

The cumulative indexes to the *ORNL Master Analytical Manual* were updated to indicate the changes made in the content of the *Manual* during 1964. The updated cumulative indexes are titled "Indexes to the *Oak Ridge National Laboratory Master Analytical Manual* (1953–1964), TID-7015 (Indexes), Revision 2." The document is available from the Clearinghouse for Federal Scientific and Technical Information, National Bureau of Standards, U.S. Department of Commerce, Springfield, Virginia; the price is $1.75. The indexes were issued as a companion document to Supplement 7 of TID-7015.

The computer work required for the preparation of the updated indexes was supervised by Ann S. Haas.[1]

[1]Technical Information Division.

18.2 MAINTENANCE OF THE *ORNL MASTER ANALYTICAL MANUAL*

The seventh supplement to the reprinted form of the *ORNL Master Analytical Manual* was published. It contains the new methods and the revisions issued in 1964. This supplement, designated TID-7015 (Suppl. 7), is available from the Clearinghouse for Federal Scientific and Technical Information, National Bureau of Standards, U.S. Department of Commerce, Springfield, Virginia, at a price of $5.25.

Altogether, 20 new methods were added to the *Manual*; 6 of these were for the purpose of record only. Revisions were made to 19 methods; 8 obsolete methods were discontinued. Also, the Table of Contents for the *Manual* was updated. (See "Presentation of Research Results.")

A survey was made to determine what methods now contained in the *Manual* are no longer used, what revisions to existing methods are needed, and what new methods are required for current or anticipated analytical work. On the basis of the survey, method-writing assignments were made to persons in the Analytical Chemistry Division.

Part D. Activities Related to Educational Institutions

19. Activities Related to Educational Institutions

Certain activities come into existence when the interests, purposes, and problems of educational institutions and of the Oak Ridge National Laboratory overlap in the area of analytical chemistry. The more formal of these activities include discussions with consultants, who are also university faculty members, and thesis-research programs for graduate students. By less formal arrangements, faculty members engage in research in the Division under the ORINS-ORNL Research Participant Program, and students participate in the Summer Student Trainee Program and in the Loanee Program for special work. Many of the Alien Guests in residency for work in the Division are from foreign educational institutions or from foreign laboratories whose programs intersect those of educational institutions.

The nature and the mutual benefits of these activities are indicated throughout this report. Of the approximately 220 formal presentations of the research of the Division made during the past year, about 15% reflect cooperative ventures with educational institutions. These presentations are enumerated in the Presentation of Research Results section, and the work is discussed in the sections of the report indicated.

19.1 CONSULTANTS

The consultants who have worked under subcontract in collaboration with the Analytical Chemistry Division are:

J. A. Dean
University of Tennessee

P. H. Emmett
Johns Hopkins University

N. H. Furman (deceased)
Princeton University

H. V. Malmstadt
University of Illinois

Gleb Mamantov
University of Tennessee

G. H. Morrison
Cornell University

M. L. Moss
Institute of Muscle Disease

A. O. Nier
University of Minnesota

L. B. Rogers
Purdue University

W. B. Schaap
Indiana University

H. H. Willard
University of Michigan

19.2 RESEARCH PARTICIPANTS

The Research Participants with us in the summer of 1965 were:

Research Participant	Division Group	See Section
C. B. Honaker Professor and Chairman Chemistry Department Tennessee Wesleyan College Athens	Reactor Projects Group	6.3
L. E. Mattison Professor of Chemistry King College Bristol, Tenn.	Nuclear and Radiochemical Analyses Group	11.7.d
D. G. Peters Assistant Professor of Chemistry Indiana University Bloomington	Analytical Instrumentation Group	1.2
J. M. Wampler Assistant Professor School of Ceramic Engineering Georgia Institute of Technology Atlanta	Mass Spectrometry Section	8.4

19.3 AEC POSTDOCTORAL PROGRAM

Under the AEC Postdoctoral Program, *G. R. Hertel* (Ph.D. in chemistry, The Johns Hopkins University, 1964) worked in the Mass Spectrometry Research Group (see Sect. 8.3).

19.4 GRADUATE THESIS RESEARCH PROGRAMS

Under three separate programs, five graduate students either have completed or are doing their thesis research in the Analytical Chemistry Division; three (*C. F. Goeking, Gerald Goldstein, W. D. Shults*) are members of the Division.

19.4.a Educational Assistance – Ph.D. Program

W. D. Shults is a candidate for the Ph.D. degree in chemistry from Indiana University. Except for the final oral examination, he has completed the requirements for the degree, including the writing of his thesis, which is titled "Controlled-Potential DC Differential Polarography" (see Sect. 1.15). *M. T. Kelley* is a member of W. D. Shults' Ph.D. Advisory Committee.

19.4.b Oak Ridge Resident Graduate Program of the University of Tennessee

Gerald Goldstein completed the Ph.D. program during 1965 and was awarded the Ph.D. degree in chemistry at the end of the spring quarter. His thesis is titled "Analytical Radiochemistry of Technetium" (see Sects. 11.1.d, 11.3.a, 11.4.c, 11.8.c, and 11.9.e).[1-6]

Also, **C. F. Goeking** has completed the course work for the M.S. degree in chemistry. At the Laboratory in January 1966, he will begin his thesis research in the field of radiochemistry.

19.4.c Oak Ridge Graduate Fellowship Programs

Two recipients of predoctoral fellowships from the ORNL Graduate Fellow Selection Panel are now working in the Nuclear and Radiochemical Analyses Group.

W. J. Armento, a graduate student from the Georgia Institute of Technology, Atlanta, has almost completed his graduate research and is now writing his Ph.D. thesis, which is tentatively titled "The Chemistry of Several Chlorohydroxoplatinates(IV) and Their Use in the Isolation of Carrier-Free ^{197}Pt." *J. C. White* is Research Supervisor of Mr. Armento's thesis research.

J. R. Stokely, Jr., has completed the experimental work and is now writing his thesis to obtain a Ph.D. degree from Clemson University. The tentative title of his thesis is "Solvent Extraction and Gas Chromatography of Metal Fluoroacetylacetonates." *J. C. White* is the Laboratory Advisor to Mr. Stokely.

19.5 LOANEES

J. F. Steenbergen, a graduate student in bacteriology at Indiana University, spent a short time in the X-Ray and Spectrochemical Analyses Group under *Cyrus Feldman's* supervision. He came to learn the techniques of atomic absorption spectrometry for determining zinc relative to his study of metabolism in bacteria.

H. W. Jenkins, a graduate student and assistant in chemistry with *Professor Gleb Mamantov* at the University of Tennessee, is now working in the Research Group with *D. L. Manning*. He is comparing the behaviors of reference electrodes described thus far for use in the electroanalytical chemistry of fluoride melts and is working to develop a new and more reliable reference electrode for such use. He is using the unique dry box–furnace available here for electroanalytical research on fluoride melts. His research is being done toward an M.S. degree in chemistry.

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[1] G. Goldstein, "Rapid Separation and Determination of Technetium," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1964*, ORNL-3750, p. 47.

[2] G. Goldstein, "Absolute Counting of Beta Emitters by the Liquid Scintillation Method," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1964*, ORNL-3750, p. 51.

[3] G. Goldstein, "Specific Activity of ^{99}Tc by Liquid Scintillation Counting," *Chem. Div. Ann. Progr. Rept. June 20, 1964*, ORNL-3679, p. 19.

[4] G. Goldstein and J. F. Emery, "Thermal-Neutron Activation Analysis of Technetium," *Chem. Div. Ann. Progr. Rept. May 20, 1965*, ORNL-3832, p. 29.

[5] G. Goldstein, "Activation Cross Sections for the Reactions of 14.8-Mev Neutrons with ^{99}Tc," *Chem. Div. Ann. Progr. Rept. May 20, 1965*, ORNL-3832, p. 31.

[6] G. Goldstein and J. A. Dean, "Rapid Separations of Technetium from Fission Products," *Chem. Div. Ann. Progr. Rept. May 20, 1965*, ORNL-3832, p. 32.

19.6 SUMMER STUDENT TRAINEE PROGRAM

In the summer of 1965, seven students participated in the Summer Student Trainee Program; they were:

Student	Educational Institution	Division Group	See Section
Graduate			
F. L. Whiting	Wofford College	Reactor Projects Group	
J. F. Wild	Massachusetts Institute of Technology	Nuclear and Radiochemical Analyses Group	11.4.d
Undergraduate			
Linda A. Billington	University of North Carolina	Division Administration	
R. L. Caldwell	Tennessee Technological University	Mass Spectrometry Service Laboratory	
Bonnie C. Johnson	Tuskegee Institute	Methods Development Group	5.3.b
J. B. Pressly	University of Tennessee	Radioisotopes-Radiochemistry Laboratory	
Serena Savage	Rice University	Methods Development Group	1.13

19.7 ALIEN GUESTS IN RESIDENCY

Seven scientists from six countries either were during the year or now are guests in the Analytical Chemistry Division:

Guest	Sponsor	Division Group	See Section
M. M. Al-Kayssi College of Education, Baghdad, Iraq	International Atomic Energy Agency	Nuclear and Radiochemical Analyses Group	
F. S. Balestic Centre d'Etudes, Nucléaires de Cadarache, France	Centre d'Etudes Nucléaires de Cadarache	Radioisotopes-Radiochemistry Laboratory	
T. M. Florence Australian Atomic Energy Commission Research Establishment, Sutherland, New South Wales	Australian Atomic Energy Commission	Methods Development Group	5.4.a 5.4.b 5.4.c
Jun-Lan Wang (*Mrs. Kian-Chu Li*) Institute of Nuclear Science, National Tsing Hua University, Hsinchu, Taiwan, Republic of China	International Atomic Energy Agency	Radioisotopes-Radiochemistry Laboratory	

Lothar Koch EURATOM, Transuranium Institute, Karlsruhe, Germany	European Atomic Energy Commission	Radioisotopes-Radiochemistry Laboratory	
Max Peisach Southern Universities Nuclear Institute, Faure, C.P., Republic of South Africa	Southern Universities Nuclear Institute	Nuclear and Radiochemical Analyses Group	
Dietrich Thiele Kernforschungszentrum Karlsruhe, Institut für Heisse Chemie, Karlsruhe, Germany	Kernreaktor Bau- und Betriebs-Gesellschaft m.b.H.	Analytical Instrumentation Group, Radioisotopes-Radiochemistry Laboratory, General Analyses Laboratory, High-Level Alpha Radiation Laboratory, and General Hot Analyses Laboratory	1.3

Presentation of Research Results

Several of the presentations listed below were made jointly with members of other divisions. In these cases the member(s) of the other division(s) is indicated by a single asterisk.

PUBLICATIONS

Books, Theses, Monographs

	AUTHOR(S)	TITLE	PUBLISHER
1	Cameron, A. E., C. M. Stevens[1]	"Mass Spectrometry," chap. 13 in *Analysis of Essential Nuclear Reactor Materials*, ed. by C. J. Rodden	U.S. Govt. Printing Office, Washington, D.C., 1964
2	Goldstein, G.	*Analytical Radiochemistry of Technetium* (Ph.D. thesis)	University of Tennessee, June 1965
3	Lyon, W. S., J. S. Eldridge, P. Crowther[2]	"Experimental Study of Artifacts and Errors Encountered in Gamma-Ray Spectrometry for Radiochemistry and Activation Analysis" (SM-55/47), pp. 33–46 in *Radiochemical Methods of Analysis* (Proceedings of the Symposium on Radiochemical Methods of Analysis Held by the International Atomic Energy Agency at Salzburg, 19–23 October 1964), vol. II	International Atomic Energy Agency, Vienna, 1965
4	Reynolds, S. A.	"Radiochemical and Nuclear Methods," chap. 14 in *Analysis of Essential Nuclear Reactor Materials*, ed. by C. J. Rodden	U.S. Govt. Printing Office, Washington, D.C., 1964
5	Ross. H. H., W. S. Lyon	"Radio-Release Methods for the Evaluation of Atmospheric Pollution: Sulfur Dioxide" (SM-55/49), pp. 285–93 in *Radiochemical Methods of Analysis* (Proceedings of the Symposium on Radiochemical Methods of Analysis Held by the International Atomic Energy Agency at Salzburg, 19–23 October 1964), vol. II	International Atomic Energy Agency, Vienna, 1965

[1] Argonne National Laboratory, Argonne, Ill.

[2] Alien Guest, 1962–63; Temporary Alien Employee, 1963–64; from South African Atomic Energy Board, Pretoria, South Africa.

6 Strain, J. E., W. S. Lyon	"The Use of Isotopic Neutron Sources for Chemical Analysis" (SM-55/48), pp. 245–61 in *Radiochemical Methods of Analysis* (Proceedings of the Symposium on Radiochemical Methods of Analysis Held by the International Atomic Energy Agency at Salzburg, 19–23 October 1964), vol. I	International Atomic Energy Agency, Vienna, 1965

Articles

AUTHOR(S)	TITLE	PUBLICATION
7 Attrill, J. E., C. M. Boyd, A. S. Meyer, Jr.	"Gas Chromatographic Determination of Permanent Gases in Helium at Reduced Sample Pressures"	*Anal. Chem.* **37**, 1543 (1965)
8 Bate, L. C.	"The Use of Activation Analysis in Procedures for the Removal and Characterization of the Surface Contaminants of Hair"	*J. Forensic Sci.* **10**, 60 (1965)
9 Bate, L. C., F. F. Dyer	"Trace Elements in Human Hair"	*Nucleonics* **23**(10), 74 (1965)
10 Belew, W. L., Helen P. Raaen	"Effect of Junction Potentials at the Salt Bridge on the Measured Values for the Half-Wave Potentials of the $Pb^{2+} \rightarrow Pb^{0}$ Reduction in Aqueous Solutions of Hydrochloric Acid and Potassium Chloride"	*J. Electroanal. Chem.* **8**, 475 (1964)
11 Crowther, P.,[2] J. S. Eldridge	"Decay of ^{99}Mo-^{99m}Tc"	*Nucl. Phys.* **66**, 472 (1965)
12 Eldridge, J. S., W. S. Lyon	"Measurement of ^{197}Hg"	*J. Nucl. Med.* **6**, 343 (1965)
13 Emery, J. F.	"A New Isomer of Bromine: ^{82m}Br"	*J. Inorg. Nucl. Chem.* **27**, 903 (1965)
14 Goldstein, G.	"Absolute Liquid-Scintillation Counting of Beta Emitters"	*Nucleonics* **23**(3), 67 (1965)
15 Handley, T. H.	"Trialkyl- and Hexaalkylphosphorothioic Triamides as Extractants for Metal Ions. Selective Extraction of Mercury (II), Silver(I), and Copper(I)"	*Anal. Chem.* **36**, 2467 (1964)
16	"Use of Metal Di-*n*-butyl Phosphorodithioates as Extractants of Metals"	*Anal. Chem.* **37**, 311 (1965)
17	"A Review of Organic Compounds Containing P=S and P(S)SH Groups as Separatory and Analytical Reagents"	*Talanta* **12**, 893 (1965)
18 Healy, W. B.,[3] L. C. Bate	"Determination of Molybdenum in Hair and Wool by Neutron Activation Analysis"	*Anal. Chim. Acta* **33**, 443 (1965)

[3]Alien Guest, 1963–64; from Soil Bureau, Department of Scientific and Industrial Research, Private Bag, Lower Hutt, New Zealand.

19	Healy, W. B.,[3] L. C. Bate, T. G. Ludwig[4]	"Micro-Element Content of Wool from Twin Wethers Raised on Two Soils as Determined by Neutron Activation Analysis"	*New Zealand J. Agr. Res.* 7, 603 (1964)
20	Jones, H. C., W. D. Shults, J. M. Dale	"High-Sensitivity Controlled-Potential Coulometric Titrator. Controlled-Potential Coulometric Determination of Milli- and Microgram Quantities of Uranium and Iron"	*Anal. Chem.* 37, 680 (1965)
21	Kochanny, G. L., Jr.[5]	"Effect of Cobalt-60 Gamma Radiation on the Determination of Uranium(VI) in Phosphoric Acid Solutions of Uranium(IV) Oxide"	*Anal. Chim. Acta* 33, 76 (1965)
22	Kubota, H., D. A. Costanzo	"Reactions in Concentrated Lithium Chloride Solution. Determination of Free Acid and Hydrolyzable Cation"	*Anal. Chem.* 36, 2454 (1964)
23	Kubota, H., F. M. Empson*	"Problems in Disposal of Radioactive Liquid Wastes in Salt"	*Chem. Eng. Progr., Symp. Ser.* 60(53) (Nuclear Engineering, Part XIII), 68 (1964)
24	Lyon, W. S.	"Availability and Use of Radioactivity Standards"	*Anal. Chem.* 36, 808 (1964)
25		Book review of: *The Theory and Practice of Scintillation Counting*, J. B. Birks, Pergamon, London; Macmillan, New York, 1964	*Anal. Chem.* 37(12), 79A (1965)
26	Maddox, W. L.	"Analytical Laboratory Hot-Cell Equipment: III. A Remotely Operated Filter Photometer"	*Proceedings of the 13th Conference on Remote Systems Technology, 1965*, p. 184, American Nuclear Society, Inc., Hinsdale, Ill.
27	Maddox, W. L., F. E. Haga[6]	"Analytical Laboratory Hot-Cell Equipment: I. Sample Preparation Apparatus for the Molten Salt Reactor Experiment"	*Proceedings of the 13th Conference on Remote Systems Technology, 1965*, p. 179, American Nuclear Society, Inc., Hinsdale, Ill.
28	Maddox, W. L., F. E. Haga[6] D. J. Fisher	"Analytical Laboratory Hot-Cell Equipment: II. A Remotely Controlled Corrosion Resistant Pipetter"	*Proceedings of the 13th Conference on Remote Systems Technology, 1965*, p. 182, American Nuclear Society, Inc., Hinsdale, Ill.
29	Mamantov, G.,[7] D. B. Freeman,[8] F. J. Miller, H. E. Zittel	"Film Formation on Pyrolytic Graphite Electrodes"	*J. Electroanal. Chem.* 9, 305 (1965)

[4]Dental Research Unit, Medical Council, Wellington, New Zealand.

[5]Present address: Dow Chemical Company, Midland, Mich.

[6]Present address: Southern Railway Co., Atlanta, Ga.

[7]Consultant; Assistant Professor of Chemistry, University of Tennessee, Knoxville.

[8]Graduate student, Department of Chemistry, University of Tennessee, Knoxville.

30	Mamantov, G.,[7] D. L. Manning, J. M. Dale	"Reversible Deposition of Metals on Solid Electrodes by Voltammetry with Linearly Varying Potential"	*J. Electroanal. Chem.* **9**, 253 (1965)
31	Moore, F. L.	"Liquid-Liquid Extraction of Metal Ions from Aqueous Solutions of Organic Acids with High-Molecular-Weight Amines. The Trivalent Actinide-Lanthanide Elements"	*Anal. Chem.* **37**, 1235 (1965)
32	Moore, F. L., W. T. Mullins	"Rapid Removal of Transuranium Elements from Aqueous Solutions Prior to the Determination of Total Lanthanide Fission Products"	*Anal. Chem.* **37**, 419 (1965)
33		"Separation of Berkelium from Other Elements. Application to Purification and Radiochemical Determination of Berkelium"	*Anal. Chem.* **37**, 687 (1965)
34	Mountcastle, W. R., Jr.,[9] Louise B. Dunlap, P. F. Thomason	"Controlled-Potential Coulometric Tiltration of Uranium(VI) in Aqueous Solutions of Sodium Fluoride"	*Anal. Chem.* **37**, 336 (1965)
35	Mueller, T. R.	"Arbitrary-Set, Proportional Temperature Controller"	*Anal. Chem.* **37**, 172 (1965)
36	Mueller, T. R., R. W. Stelzner, D. J. Fisher, H. C. Jones	"High-Sensitivity, Direct-Reading, Linear, Recording Conductometric Titrator with Automatic Temperature Compensation or Proportional Temperature Control"	*Anal. Chem.* **37**, 13 (1965)
37	Muzzarelli, R. A. A.,[10] L. C. Bate	"Separation of Traces of Metals from Uranium by Chromatography on Cellulose Column"	*Talanta* **12**, 823 (1965)
38	Perone, S. P.,[11] T. R. Mueller	"Application of Derivative Techniques to Stationary Electrode Polarography"	*Anal. Chem.* **37**, 2 (1965)
39	Raaen, Helen P.	"Teflon Dropping-Mercury Electrode for Polarography in Hydrofluoric Acid and Other Glass-Corroding Media. Evaluation with Thallium(I) $\rightleftharpoons$ Thallium Reaction"	*Anal. Chem.* **36**, 2420 (1964)
40		"Dropping-Mercury Electrode of Teflon for Polarography in Hydrofluoric Acid and Other Glass-Corroding Media. Evaluation with the Lead(II) $\rightleftharpoons$ Lead Reaction"	*Anal. Chem.* **37**, 677 (1965)
41		"Polarography of Lead(II) in Aqueous Hydrofluoric Acid (1 to 12*M*) with a Dropping-Mercury Electrode of Teflon"	*Anal. Chem.* **37**, 1355 (1965)
42	Reynolds, S. A.	Book review of: *Radioactivation Analysis*, H. J. M. Bowen and D. Gibbons, Clarendon, Oxford, 1963	*J. Chem. Educ.* **41**, A964 (1964)

[9]Research Participant, summers of 1961 and 1962; Temporary Employee, summer of 1963; from Chemistry Department, Birmingham-Southern College, Birmingham, Ala.

[10]Alien Guest, 1963–64; from Communauté Européenne de L'Energie Atomique, EURATOM, Bureau Central de Mesures Nucléaires, Geel, Belgium.

[11]Assistant Professor of Chemistry, Purdue University, Lafayette, Ind.

43	Ricci, E.	"Output Spectrum from 14-Mev Neutron Generators. Rapid Estimation and Influence in Cross-Section Measurements and Activation Analysis"	*J. Inorg. Nucl. Chem.* **27**, 41 (1965)
44		"Output Spectra of 14-Mev Neutron Generators – Rapid Estimation and Influence in Activation Analysis"	*Trans. Am. Nucl. Soc.* **7**, 203 (1964)
45	Ricci, E., R. L. Hahn	"Theory and Experiment in Rapid, Sensitive Helium-3 Activation Analysis. Helium-3 Reactions as Neutron Sources"	*Anal. Chem.* **37**, 742 (1965)
46	Rickard, R. R., C. F. Goeking, E. I. Wyatt	"Mass–Fission-Yield Curve for Americium-241"	*Nucl. Sci. Eng.* **23**, 115 (1965)
47	Ross, H. H.	"Color Quench Correction in Liquid Scintillator Systems Using an Isolated Internal Standard"	*Anal. Chem.* **37**, 621 (1965)
48	Ross, H. H., R. P. Gardner,[12] J. W. Dunn III[13]	"Wear Rates in Automotive Engines by Liquid Scintillation Counting of Fe^{55}"	*Nucl. Sci. Eng.* **20**, 521 (1964)
49	Ross, H. H., H. L. Holsopple	"N,N,N, Trimethylborazine as a Liquid Scintillator Solvent for Thermal Neutron Detection"	*Nucl. Instr. Methods* **33**, 194 (1965)
50	Ross, W. J.	"Gravimetric Determination of Sodium with Radiotracers"	*Anal. Chem.* **37**, 168 (1965)
51	Shults, W. D., F. E. Haga,[6] T. R. Mueller, H. C. Jones	"Chronopotentiometer with Compensation for Extraneous Currents"	*Anal. Chem.* **37**, 1415 (1965)
52	Susano, C. D., L. J. Brady, G. Goldberg, W. R. Laing, W. S. Lyon	"Analytical Chemistry in Nuclear Technology"	*Science* **147**, 523 (1965)
53		"Eighth Conference on Analytical Chemistry in Nuclear Technology"	*Nucl. News* **7**(12), 30 (1964)
54		"A Résumé of the Eighth Conference on Analytical Chemistry in Nuclear Technology"	*J. Electroanal. Chem.* **9**, 328 (1965)
55	Terry, Anne A.[14]	"Integrated Assembly for Generation, Storage, and Delivery of Standard Chromium(II) Solutions"	*Chemist-Analyst* **53**, 63 (1964)

[12]Measurement and Controls Laboratory, Research Triangle Institute, Durham, N.C.

[13]Research Assistant, Department of Mathematics, North Carolina State University, Raleigh.

[14]Research Participant, summers of 1962 and 1963; from Department of Chemistry, Texas Woman's University, Denton.

56	Toth, K. S.,* T. H. Handley, E. Newman,* I. R. Williams*	"New Rare-Earth Alpha Emitter, ^{148}Eu"	*Phys. Rev.* **136**, B1233 (1964)
57	White, J. C.	Book review of: *Analytical Chemistry of Thallium*, I. M. Korenman, Program for Scientific Translations, Jerusalem, 1964	*Anal. Chem.* **37**(1), 71A (1965)
58		Book review of: *The Solvent Extraction of Metal Chelates*, by J. Stary, Pergamon, London; Macmillan, New York, 1964	*Nucl. Sci. Eng.* **23**, 200 (1965)
59	Young, J. P.	Book review of: *Spectroscopic Properties of Rare Earths*, by Brian G. Wybourne, Interscience, New York, 1965	*Anal. Chem.* **37**(11), 65A (1965)
60	Zittel, H. E.	Book review of: *Amperometric Titrations*, by John T. Stock, Interscience, New York, 1965	*J. Chem. Educ.* **42**, 521 (1965)
61	Zittel, H. E., F. J. Miller	"A Glassy-Carbon Electrode for Voltammetry"	*Anal. Chem.* **37**, 200 (1965)

Reports

	AUTHOR(S)	TITLE	REPORT NO.
62	Browning, W. E., Jr.,* R. E. Adams,* J. E. Attrill	"Gas Chromatographic Identification of Radioiodine Vapors" in *Semiannual Progress Report for Period Ending December 31, 1964, Nuclear Safety Program*	ORNL-3776, p. 7 (March 1965)
63		"Effects of Surface Materials on Radioiodine Vapors" in *Semiannual Progress Report for Period Ending December 31, 1964, Nuclear Safety Program*	ORNL-3776, p. 19 (March 1965)
64		"Gas Chromatographic Identification of Radioiodine Vapors" in *Semiannual Progress Report for Period Ending June 30, 1965, Nuclear Safety Program*	ORNL-3843, p. 171 (September 1965)
65		"Reaction of Methyl Iodide with Chemically Impregnated Charcoal" in *Semiannual Progress Report for Period Ending June 30, 1965, Nuclear Safety Program*	ORNL-3843, p. 191 (September 1965)
66	Browning, W. E., Jr.,* R. D. Ackley,* A. F. Roemer, Jr.	"Investigation of Radioiodine Behavior Under Moist Conditions in May Packs by Means of Composite Diffusion Tubes" in *Semiannual Progress Report for Period Ending December 31, 1964, Nuclear Safety Program*	ORNL-3776, p. 23 (March 1965)

67		"Investigation of Airborne Radioiodine Species by Means of Composite Diffusion Tubes" in *Semiannual Progress Report for Period Ending December 31, 1964, Nuclear Safety Program*	ORNL-3776, p. 12 (March 1965)
68		"Investigation of Chemical Reactivity of Nonelemental Radioiodine Vapor by Means of Composite Diffusion Tubes" in *Semiannual Progress Report for Period Ending June 30, 1965, Nuclear Safety Program*	ORNL-3843, p. 173 (September 1965)
69		"Chemical Behavior of the Radioiodine Species Found on Rubber Sections of Composite Diffusion Tubes" in *Semiannual Progress Report for Period Ending June 30, 1965, Nuclear Safety Program*	ORNL-3843, p. 184 (September 1965)
70	Cameron, A. E.	*Report on Foreign Travel of A. E. Cameron June 30–July 10, 1965*	July 27, 1965 (unpublished report)
71	Cameron, A. E., J. R. Walton	*The Isotopic Composition of Atmospheric Neon*	ORNL-3857 (September 1965)
72	Feldman, C.	*Report on Foreign Travel of Cyrus Feldman June 25 to July 30, 1965*	Sept. 13, 1965 (unpublished report)
73	Goldberg, G.	*The Rapid Determination of Low Concentrations of Oxygen and Hydrogen in Alkali Materials by a Modified Amalgamation Technique*	CONF 721-4 (Oct. 30, 1964)
74	Goldstein, G.	*Equilibrium Distribution of Metal-Ion Complexes*	ORNL-3620 (November 1964)
75		"Activation Cross Sections for the Reactions of 14.8-Mev Neutrons with ^{99}Tc" in *Chemistry Division Annual Progress Report for Period Ending May 20, 1965*	ORNL-3832, p. 31 (September 1965)
76	Goldstein, G., J. A. Dean[15]	"Rapid Separation of Technetium from Fission Products" in *Chemistry Division Annual Progress Report for Period Ending May 20, 1965*	ORNL-3832, p. 32 (September 1965)
77	Goldstein, G., J. F. Emery	"Inorganic and Nuclear Chemistry of Technetium. Thermal Neutron Activation Analysis of Technetium" in *Chemistry Division Annual Progress Report for Period Ending May 20, 1965*	ORNL-3832, p. 29 (September 1965)
78	Goldstein, G., S. A. Reynolds	*Half-Lives and Specific Activities of Common Radionuclides*	ORNL-TM-1318 (Nov. 8, 1965)
79	Holsopple, H. L., Lucy E. Scroggie	*Synthesis and Infrared Absorption Spectrum of Diborane-^{10}B*	ORNL-TM-1061 (Feb. 19, 1965)

[15]Consultant; Professor of Chemistry, University of Tennessee, Knoxville.

80	Jones, H. C.	*Check Out and Test Procedure for Q-2792 Controlled-Potential DC Polarograph-Voltammeter*	ORNL Test Specification St-293 (Apr. 9, 1965)
81	Kelley, M. T.	*Statistical Quality Control Report, April Through June, 1965, Analytical Chemistry Division*	July 22, 1965 (unpublished report)
82		*Statistical Quality Control Report, January Through March, 1965, Analytical Chemistry Division*	May 3, 1965 (unpublished report)
83		*Statistical Quality Control Report, Analytical Chemistry Division, January Through December, 1964*	Jan. 25, 1965 (unpublished report)
84		*Statistical Quality Control Report, January Through September, 1964, Analytical Chemistry Division*	Dec. 14, 1964 (unpublished report)
85		*Report on Foreign Travel of Myron T. Kelley September 2, 1965, to October 6, 1965*	Nov. 2, 1965 (unpublished report)
86	Lee, T.,[16] J. A. Carter	*Direct Reading Spectrographic Determination of the Ratios of Three Metals*	K-L-1916 (Dec. 4, 1964)
87	Lyon, W. S.	*Report of Foreign Travel of W. S. Lyon October 10 to October 28, 1964*	Nov. 11, 1964 (unpublished report)
88	Norris, J. A.[17]	*The Analysis of Promethium-147*	ORNL-TM-922 (Aug. 19, 1964)
89	Orr, P. B.,* R. S. Pressly,* E. J. Spitzer	*Evidence of the Absence of Long-Lived Isotopes of Promethium from Fission of Uranium, and the Purification of Promethium for the Establishment of a Primary Spectrographic Standard*	ORNL-3631 (January 1965)
90	Quincy, R. B., Jr., D. E. LaValle	*High Purity Potassium Chloride*	ORNL-TM-1071 (March 1965)
91	Raaen, Helen P., ed.	*Annual Progress Report for Period Ending November 15, 1964, Analytical Chemistry Division*	ORNL-3750 (January 1965)
92		*Oak Ridge National Laboratory Master Analytical Manual, Supplement 7*	TID-7015, suppl. 7 (March 1965)
93	Raaen, Helen P., Ann S. Klein,* eds.	*Indexes to the Oak Ridge National Laboratory Master Analytical Manual (1953–1964)*	TID-7015, Indexes, revision 2 (March 1965)
94	Rickard, R. R., H. A. Parker, E. I. Wyatt	*Mass-Fission Yield Curve for ^{237}Np*	Dec. 24, 1964 (unpublished report)

[16]ORGDP, Oak Ridge, Tenn.

[17]Present address: Jarrel-Ash Corp., Waltham, Mass.

95	Ross, H. H.	*Report on Foreign Travel of H. H. Ross May 15 to June 9, 1965*	July 1, 1965 (unpublished report)
96	Savage, Serena,[18] W. D. Shults	*PØLARGRM: A Computer Program for Analysis of Polarograms*	ORNL-TM-1253 (Aug. 31, 1965)
97		*CHRNØGRM: A Computer Program for Analysis of Chronopotentiograms*	ORNL-TM-1254 (Aug. 31, 1965)
98	Stelzner, R. W.	"Comments on the paper by Lauer, Brück, and LeDuigou, 'Precise Determinations of Uranium. A Comparison of Different Methods'" in *Round Table on High Precision Chemical Analysis of Substances of Interest to Nuclear Energy*	EANDC-42"S", p. 181 (Jan. 18–22, 1965)
99	Strain, J. E.	*Report on Foreign Travel of J. E. Strain October 19 Through October 30, 1964*	Nov. 18, 1964 (unpublished report)
100	Strain, J. E., W. J. Ross	*14-Mev Neutron Reactions*	ORNL-3672 (January 1965)
101	Thomason, P. F.	*Report on Foreign Travel of P. F. Thomason January 16 to February 20, 1965*	March 15, 1965 (unpublished report)
102		"Methods Used at ORNL for the Precise Determination of Uranium" in *Round Table on High Precision Chemical Analysis of Substances of Interest to Nuclear Energy*	EANDC-42"S", p. 163 (Jan. 18–22, 1965)
103		"Methods Used at ORNL for the Precise Determination of Plutonium" in *Round Table on High Precision Chemical Analysis of Substances of Interest to Nuclear Energy*	EANDC-42"S", p. 231 (Jan. 18–22, 1965)
104	White, J. C.	*Analytical Chemistry Research and Development Group Monthly Summary – November 1964*	Dec. 11, 1964 (unpublished report)
105		*Analytical Chemistry Research and Development Group Monthly Summary – December 1964*	Jan. 13, 1965 (unpublished report)
106		*Analytical Chemistry Research and Development Group Monthly Summary – January 1965*	Feb. 11, 1965 (unpublished report)
107		*Analytical Chemistry Research and Development Group Monthly Summary – February 1965*	Mar. 12, 1965 (unpublished report)
108		*Analytical Chemistry Research and Development Group Monthly Summary – March 1965*	Apr. 8, 1965 (unpublished report)
109		*Analytical Chemistry Research and Development Group Monthly Summary – April 1965*	May 7, 1965 (unpublished report)
110		*Analytical Chemistry Research and Development Group Monthly Summary – May 1965*	June 10, 1965 (unpublished report)
111		*Analytical Chemistry Research and Development Group Monthly Summary – June 1965*	July 14, 1965 (unpublished report)
112		*Analytical Chemistry Research and Development Group Monthly Summary – July 1965*	Aug. 11, 1965 (unpublished report)

[18]Summer Student Trainee, summer 1965; from Rice University, Houston, Tex.

113		*Analytical Chemistry Research and Development Group Monthly Summary – August 1965*	Sept. 10, 1965 (unpublished report)
114		*Analytical Chemistry Research and Development Group Monthly Summary – September 1965*	Oct. 15, 1965 (unpublished report)
115		*Analytical Chemistry Research and Development Group Monthly Summary – October 1965*	Nov. 9, 1965 (unpublished report)
116	Wright, R. R.,[16] W. S. Pappas,[16] J. A. Carter, C. W. Weber[16]	*Preparation and Recovery of Cesium Compounds for Density Gradient Solutions*	K-L-6037 (Mar. 3, 1965)
117	Anonymous	"Controlled-Potential and Derivative Polarograph, Q-1988 A" in *Engineering Materials List*, TID-4100 (Supplement 28)	CAPE-1001 (June 1965)
118		"High-Sensitivity Coulometric Titrator/Q-2564/" in *Engineering Materials List*, TID-4100 (Supplement 28)	CAPE-1196 (June 1965)
119		"Pipetters" in *Engineering Materials List*, TID-4100 (Supplement 28)	CAPE-1220 (June 1965)
120		"Decappers" in *Engineering Materials List*, TID-4100 (Supplement 29)	CAPE-1221 (September 1965)

New Methods Issued to *ORNL Master Analytical Manual*

	AUTHOR(S)	TITLE	NUMBER(S)	DATE
121	Apple, R. F.	"Uranium in Molten-Salt Reactor Experiment (MSRE) Fuel, Automatic, Controlled-Potential, Coulometric Titration Method"	9 021205	3-4-65
122		"Uranium in the Molten-Salt Reactor Experiment (MSRE) Fuel, High-Sensitivity, Automatic, Controlled-Potential Coulometric Titration Method"	9 021206	3-17-65
123	Costanzo, D. A.	"Nitrate in Transuranium (TRU) Solutions, Azo Dye Filter Photometric Method"	9 062201	3-5-65
124	Haga, F. E.,[6] D. J. Fisher	"Pipetters, Remotely Servo-Controlled, Corrosion-Resistant, ORNL Models Q-1348A, Q-1348B, and Q-1348C"	1 003102 9 003102	3-26-65
125	Jones, H. C.	"Coulometric Titrator, Controlled-Potential, High-Sensitivity, ORNL Model Q-2564"	1 003025 9 003025	8-20-65
126	Kubota, H.	"Chloride, Distillation–Indirect Iodometric Titration Method"	1 212073	9-14-65
127		"Strong and Weak Acids in Their Mixtures, Potentiometric Sodium Hydroxide Titration Method"	1 220021 9 00720021	9-10-65
128	Meyer, H. C.	"Other *ORNL Master Analytical Manual* Methods Needed in the Transuranium (TRU) Process"	9 0600	8-20-65

129		"Molybdenum Radioactivity in Transuranium (TRU) Process Solutions, 8-Hydroxyquinoline Precipitation—Beta Counting Method"	9 062312	9-21-65
130	Moore, F. L.	"Californium-252 in Transuranium (TRU) Process Solutions, Measurement of Prompt Gamma Rays from Spontaneous Fission"	9 062311	3-5-65
131	Morton, J. E.	"Carbon-14, Liquid-Scintillation-Counting Method"	9 0733173	3-16-65
132	Stringer, C. D.	"Nitrate, Phenoldisulfonic Acid Spectrophotometric Method"	1 215612 9 00715612	3-22-65
133	Vaughan, W. F.	"Iron, 1,10-Phenanthroline Spectrophotometric Method"	1 214113 9 00714113	9-24-65
134		"Nickel, in Molten-Salt Reactor Experiment (MSRE) Fuel, Spectrophotometric Dimethylglyoxime (Persulfate Oxidation) Method"	9 021207	9-10-65

Revised Methods Issued to *ORNL Master Analytical Manual*

	AUTHOR(S)	TITLE	NUMBER(S)	DATE
135	Apple, R. F.	"Zirconium in MSRE Fuel, Amperometric Cupferron Titration Method"	9 021201	R. 4-12-65
136		"Chromium in MSRE Fuel, Amperometric Ferrous Sulfate Titration Method"	9 021202	R. 4-12-65
137		"Fluoride in MSRE Fuel, Pyrolysis Method"	9 021203	R. 4-12-65
138		"Uranium in MSRE Fuel, Polarographic Method"	9 021204	R. 4-12-65
139	Burros, C. L., R. E. Brooksbank,* E. I. Wyatt	"Barium Radioactivity in Aqueous or Organic Solutions"	2 21081	R. 4-26-65
140	Druschel, R. E.,* E. I. Wyatt	"Strontium Radioactivity and/or Barium Radioactivity in Aqueous or Organic Solutions"	2 21082 2 21801	R. 4-26-65
141		"Cerium Radioactivity in Aqueous or Organic Solutions"	2 21181	R. 4-26-65
142		"Niobium Radioactivity in Aqueous or Organic Solutions"	2 21551	R. 4-26-65
143		"Ruthenium Radioactivity in Aqueous Solutions"	2 21731	R. 4-26-65
144		"Tellurium Radioactivity in Aqueous or Organic Solutions"	2 21841	R. 4-26-65
145		"Zirconium Radioactivity in Aqueous or Organic Solutions"	2 21981	R. 4-26-65
146	Jones, H. C.	"Automatic Coulometric Titrator, ORNL Model Q-2005, Electronic, Controlled-Potential"	1 003029	R. 8-30-65
147	Reynolds, S. A., E. I. Wyatt	"Alpha Proportional Counter"	2 00351 9 00351	R. 4-26-65

148		"Apparatus, General"	2 0010 5 10110	R. 4-26-65
149	Rickard, R. R., E. I. Wyatt	"Iodine Radioactivity in Aqueous or Organic Solutions"	2 21391	R. 4-26-65
150	Walker, R. L., E. I. Wyatt	"Gross Alpha"	9 002301 9 0733000	R. 4-26-65
151	Wyatt, E. I., C. L. Ghann	"Cesium Radioactivity in Aqueous Solutions, Chlorostannate Method"	2 21195	R. 4-26-65
152	Wyatt, E. I.	"Carbon-14, Product Analysis Guide"	9 0733171	R. 5-26-65
153		"Iodine-131, Product Analysis Guide"	9 0733391	R. 5-17-65

Record Copies Issued to *ORNL Master Analytical Manual*

	AUTHOR(S)	TITLE	NUMBER(S)	DATE
154	Bate, L. C.	"Particle-Size Distribution in Uranium Dioxide, Neutron Activation—Sedimentation Method"	5 10201	9-22-65
155	Boyd, C. M.	"Uranium(IV)-to-Uranium(VI) Ratio in Thorium Oxide—Uranium Oxide Mixtures, Controlled-Potential Coulometric Titration Method"	1 219226 9 00719226	3-2-65
156	Hill, F. M.	"Di-sec-Butyl Phenylphosphonate in Di-sec-Butyl Phosphonate—Diethylbenzene Solutions, Dielectric Constant Method"	1 100032 9 00607	9-17-65
157	Kubota, H.	"Boron, Spectrophotometric Curcumin Method"	1 21121	3-3-65
158		"Oxalate in Eluates from Irradiated Ion-Exchange Resins, Indirect Iodometric Titration Method"	1 221007 9 00721007	3-1-65
159		"Magnesium in Process Water, Spectrophotometric Magon Method"	9 00714712	9-20-65

ORAL PRESENTATIONS

Ninth Conference on Analytical Chemistry in Nuclear Technology

Some 300 scientists, including eleven representatives from seven foreign countries, attended the the Ninth Conference on Analytical Chemistry in Nuclear Technology, which was held on October 12–14, 1965, in Gatlinburg, Tennessee. The ORNL Analytical Chemistry Division continued its sponsorship of this annual event.

The Conference Committee for this meeting was comprised of nine ORNL staff members: M. T. Kelley, Director of the Division; C. D. Susano, Associate Director; and J. A. Carter, L. T. Corbin, R. L. Hahn, H. L. Holsopple, C. E. Lamb, F. L. Moore, and J. C. White. Fifty-one papers were presented at the meeting, ten of which were given by members of the ORNL staff.

In addition to the program of the Ninth Conference, meetings were also held simultaneously by members of the Southeastern Section of the Society for Applied Spectroscopy in its regular fall meeting and by another group representing emission spectroscopists from the U.S. Atomic Energy Commission (USAEC) and related contractor groups.

The format of this meeting was essentially the same as has been the case in the past, except that two symposia on subjects of current importance in space and nuclear technology were held simultaneously with two sessions on a variety of subjects of general interest in nuclear matters, including bioanalytical techniques. In the symposium on the Analytical Chemistry of the Transuranium Elements, 16 papers were read at three sessions; in the symposium on The Role of Analytical Chemistry in Pure Materials Research, 21 papers were presented at four sessions.

In lieu of proceedings, tape recordings were made of all presentations except paper No. 37, which unfortunately was not recorded due to technical difficulties. This is the third year that tape recordings have been made in lieu of proceedings. Reproductions are made available generally to any one requesting the loan of any of these recordings. In addition to being available from ORNL, copies of the tape recordings have been deposited at the Library of Congress and can be secured therefrom on a loan basis simply by contacting that agency.

The Tenth Conference in this series is tentatively scheduled to be held at the Mountain View Hotel, Gatlinburg, Tennessee, on September 27–29, 1966.

At Meetings of Professional Societies

	AUTHOR(S)	TITLE	PRESENTED AT
160	Belew, W. L.,[19] D. J. Fisher, M. T. Kelley, J. A. Dean[15]	"Determination of Trace Quantities of Uranium by Controlled-Potential DC Polarography in a Tri-Octylphosphine Oxide Extract"	International Symposium on Microchemical Techniques – 1965, American Microchemical Society and International Union of Pure and Applied Chemistry, Pennsylvania State University, University Park, Pa., Aug. 22–27, 1965
161	Bell, J. T.,[19]* R. E. Biggers	"The Absorption Spectrum of the Uranyl Ion in Perchlorate Media. Part I. Mathematical Resolution of the Overlapping Band Structure and Studies of the Environmental Effects"	Symposium on Molecular Structure and Spectroscopy, Department of Physics and Astronomy, Ohio State University, Columbus, June 14–18, 1965
162	Browning, W. E., Jr.,[19]* R. E. Adams,* R. D. Ackley,* M. E. Davis,* J. E. Atrill	"Identity, Character and Chemical Behavior of Vapor Forms of Radioiodine"	International Symposium on Fission Product Release and Transport Under Accident Conditions, Oak Ridge, Tenn., Apr. 5–7, 1965
163	Cameron, A. E.	"The Present Status of the Atomic Weights"	13th Annual Conference on Mass Spectrometry and Allied Topics, ASTM Committee E-14, St. Louis, May 16–21, 1965
164	Cook, J. L.,* W. R. Laing, R. L. Beatty[19]*	"Determination of the Coating Density of Pyrolytic Carbon Coated Nuclear Fuel Particles"	7th Biennial Conference on Carbon, Cleveland, June 21–25, 1965
165	Costanzo, D. A.,[19] R. D. Baybarz,* J. E. Bigelow*	"Alpha Radiation Effects on Concentrated Solutions of Lithium Chloride Containing Hydrochloric Acid"	9th Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Oct. 12–14, 1965

[19]Speaker.

166	Dunn, H. W.	"Quantitative Analysis by the X-Ray Absorption Edge Technique"	9th Annual Meeting, Analytical Subcommittee, UCC Research Liaison Committee, Oak Ridge, Tenn. (ORNL), May 24–25, 1965
167	Eby, R. E.	"Isotopic Analysis of Transuranium Elements by Mass Spectrometry"	9th Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Oct. 12–14, 1965
168	Eldridge, J. S.	"Electronic Resolution of Gamma-Ray Spectra"	6th Annual Contractor's Meeting, Division of Isotopes Development, U.S. Atomic Energy Commission, Washington, D.C., Nov. 9–10, 1964
169	Eldridge, J. S.,[19] P. Crowther,[2] W. S. Lyon	"Gamma-Ray Branchings in the Decay of ^{116}In, ^{141}Ce, and ^{147}Nd"	149th Meeting, American Chemical Society, Detroit, Mich., Apr. 4–9, 1965
170	Eldridge, J. S.,[19] W. S. Lyon	"Measurement of ^{197}Hg"	12th Annual Meeting, Society of Nuclear Medicine, Bal Harbour, Fla., June 17–19, 1965
171	Emery, J. F.,[19] F. F. Dyer, T. D. Alexander,[20] E. Schonfeld*	"The Evaluation of Computer Programs for Gamma-Ray Spectrometry in Activation Analysis"	International Conference on Modern Trends in Activation Analysis, College Station, Tex., Apr. 19–22, 1965
172	Feldman, C.	"Spectrometry in Analytical Chemistry"	Summer Seminar, Knoxville–Oak Ridge Section, American Institute of Chemical Engineers, Oak Ridge, Tenn., Aug. 18–19, 1965
173		"The Quantitative Analysis of Highly Radioactive Materials: Spectrochemical and X-Ray Techniques"	20th Congress, International Union for Pure and Applied Chemistry, Moscow, USSR, July 12–18, 1965
174		"The Laser in Metallurgical Spectroscopy"	19th AEC Metallography Group Meeting, Oak Ridge, Tenn., Apr. 20–22, 1965
175	Goldberg, G.	"The Effects of Trace Impurities of Moisture, Oxygen, Hydrogen, Nitrogen, and Carbon Dioxide in Glove Box Atmospheres on the Analysis of the Alkali Metals"	Joint AEC-NASA Meeting, "Second Round-Robin Analysis of Oxygen in Sodium and Potassium," Lewis Research Center, NASA, Cleveland, Sept. 22–23, 1965
176	Hahn, R. L.	"Activation Analysis with 14-MeV Neutrons and with Helium-3 Ions"	9th Annual Meeting, Analytical Subcommittee, UCC Research Liasion Committee, Oak Ridge, Tenn. (ORNL), May 24–25, 1965
177		"Radio and Nuclear Chemistry"	Summer Seminar, Knoxville–Oak Ridge Section, American Institute of Chemical Engineers, Oak Ridge, Tenn., Aug. 18–19, 1965
178		"Particle Accelerators in Pure Materials Analyses"	9th Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Oct. 12–14, 1965

[20]Research Participant, summer of 1964; from Department of Chemistry, Georgetown College, Georgetown, Ky.

179		"Interactions of Cadmium-106 with Alpha Particles"	149th Meeting, American Chemical Society, Detroit, Mich., Apr. 4–9, 1965
180	Horton, A. D., A. S. Meyer, M. T. Kelley[19]	"Applications of Gas Chromatography in Nuclear Technology"	Journées Internationales de la Séparation Immediate et de la Chromatographie, Athens, Greece, Sept. 19–24, 1965
181	Kelley, M. T.,[19] W. L. Belew, G. G. V. Pierce, W. D. Shults, H. C. Jones, D. J. Fisher	"Controlled-Potential Polarography and Coulometry as Micro-Analytical Techniques"	International Symposium on Microchemical Techniques—1965, American Microchemical Society and International Union of Pure and Applied Chemistry, Pennsylvania State University, University Park, Aug. 22–27, 1965
182	Laing, W. R.	"Analytical Services for Chemical Engineers"	Summer Seminar, Knoxville—Oak Ridge Section, American Institute of Chemical Engineers, Oak Ridge, Tenn., Aug. 18–19, 1965
183	Laing, W. R.,[19] E. C. Lynn	"Alkylbenzene Sulfonate (ABS) Control for the Foam Separation Process"	Technicon Symposium on Automation in Analytical Chemistry, New York, Sept. 8–10, 1965
184			Paris, Frankfurt, Rome, London, and Copenhagen, fall and winter, 1965 (read by representative of Technicon)
185	Lyon, W. S.	"Application of Neutron Activation Analysis to Forensic Science"	Southeast Regional National Science Teachers Association Conference, Hollywood, Fla., Nov. 27, 1964
186		"Caution and Precaution in Forensic Applications"	Advanced Seminar on Activation Analysis, San Diego, Calif., Apr. 28, 1965
187		"Radioactivation Analysis as an Analytical Tool"	Conference on Purification of Materials, New York Academy of Science, New York, May 6–8, 1965
188		"Absolute Counting of Gamma Activity"	Advanced Isotope Technology Course, ORINS, Oak Ridge, Tenn., July 28, 1965
189		"Activation Analysis with Particles Other than Neutrons"	Advanced Isotope Technology Course, ORINS, Oak Ridge, Tenn., Aug. 26, 1965
190		"Applications of Radioisotopes"	Isotopes Development Center Program Review Meeting, Oak Ridge, Tenn. (ORNL), May 14, 1965
191	Mamantov, G.,[7] D. L. Manning, J. M. Dale[19]	"Reversible Deposition of Metals on Solid Electrodes by Voltammetry with Linearly Varying Potential"	149th Meeting, American Chemical Society, Detroit, Mich., Apr. 4–9, 1965

192	McKown, H. S.,[19] A. E. Cameron, W. H. Christie, A. S. Trundle III[21]	"A Programmable High Speed Data Acquisition System for Mass Spectrometric Application"	13th Annual Conference on Mass Spectrometry and Allied Topics, ASTM Committee E-14, St. Louis, May 16–21, 1965
193	Miller, F. J.	"Voltammetry of Iodide, Iodine, and Iodate with the Pyrolytic Graphite Indicating Electrode"	Southwestern Regional American Chemical Society Meeting, Shreveport, La., Dec. 3–5, 1964
194		"Electrochemistry with the Pyrolytic Graphite Electrode"	9th Annual Meeting, Analytical Subcommittee, UCC Research Liaison Committee, Oak Ridge, Tenn. (ORNL), May 24–25, 1965
195	Raaen, Helen P.	"Instrumentation for Polarography of Glass-Corroding Media"	Instrument Society of America 1st Joint International Symposium on Analysis Instrumentation and Chemical and Petroleum Instrumentation, Montreal, Canada, May 26–28, 1965
196	Reynolds, S. A.	"Recent Findings in Radiochemistry"	Taft Sanitary Engineering Center, Public Health Service, Cincinnati, Ohio, Feb. 11, 1965
197	Ricci, E.	"Activation Analysis. Reactor Neutron Fluxes and Interferences"	Advanced Isotope Technology Course, ORINS, Oak Ridge, Tenn., Aug. 23, 1965
198	Ricci, E.,[19] R. L. Hahn, J. E. Strain, F. F. Dyer	"He^3 Activation Analysis"	International Conference on Modern Trends in Activation Analysis, College Station, Tex., Apr. 19–22, 1965
199	Ross, H. H.	"Theoretical and Experimental Aspects of Quenching Variables from Biomedical Samples in Liquid Scintillator System"	International Atomic Energy Agency's Symposium on Radioisotope Sample Measurement Techniques in Medicine and Biology, Vienna, Austria, May 24–28, 1965
200		"Gas Chromatography"	Summer Seminar, Knoxville–Oak Ridge Section, American Institute of Chemical Engineers, Oak Ridge, Tenn., Aug. 18–19, 1965
201	Sites, J. R.	"Applications of ORNL Spark Source Mass Spectrograph to High Purity Materials Analysis"	9th Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Oct. 12–14, 1965
202	Stelzner, R. W.	"Comments on the paper by Lauer, Brück, and LeDuigou, 'Precise Determination of Uranium. A Comparison of Different Methods'"	Round Table on High Precision Chemical Analysis of Substances of Interest to Nuclear Energy, European-American Nuclear Data Committee, Brussels, Belgium, Jan. 18–22, 1965

[21]Present address: Edgerton, Germeshausen and Grier, Santa Barbara, Calif.

203		"A Survey of Analytical Instrumentation Development at Oak Ridge National Laboratory"	Seminar, Institut für Heisse Chemie, Kernforschungszentrum, Karlsruhe, W. Germany, Oct. 4, 1965
204	Susano, C. D.	"Management Looks at Analytical Chemistry in a National Laboratory"	16th Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, Pittsburgh, Pa., Mar. 1–5, 1965
205	Thomason, P. F.	"Methods Used at ORNL for the Precise Determination of Uranium"	Round Table on High Precision Chemical Analysis of Substances of Interest to Nuclear Energy, European-American Nuclear Data Committee, Brussels, Belgium, Jan 18–22, 1965
206		"Methods Used at ORNL for the Precise Determination of Plutonium"	Round Table on High Precision Chemical Analysis of Substances of Interest to Nuclear Energy, European-American Nuclear Data Committee, Brussels, Belgium, Jan 18–22, 1965
207	Vissers, D. R.,[19,*,22] N. G. Anderson,* J. E. Attrill, J. G. Green*	"Automated Nucleotide Analyzer"	9th Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Oct. 12–14, 1965
208	Walton, J. R.,[19] A. E. Cameron	"The Isotopic Composition of Atmospheric Neon"	13th Annual Conference on Mass Spectrometry and Allied Topics, ASTM Committee E-14, St. Louis, May 16–21, 1965
209	White, J. C.	"Modern Analytical Chemistry"	Summer Seminar, Knoxville–Oak Ridge Section, American Institute of Chemical Engineers, Oak Ridge, Tenn., Aug. 18–19, 1965
210	Willmarth, T. E.	"Electron Optical Study of the Effect of Aging on Carbides in a Niobium-Tungsten-Zirconium Alloy"	19th AEC Metallography Group Meeting, Oak Ridge, Tenn., Apr. 20–22, 1965
211			Southeastern Electron Microscopy Conference, Georgia Institute of Technology, Atlanta, May 14–15, 1965
212	Yoakum, Anna M.	"Recent Advances in Analytical Emission Spectrometry"	16th Mid-America Symposium on Spectroscopy, Chicago, June 14–17, 1965
213			St. Louis Chapter, Society for Applied Spectroscopy, St. Louis, Sept. 16, 1965
214	Young, J. P.,[19] G. W. Clark*	"Absorbance Spectra of Single Crystals"	9th Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Oct. 12–14, 1965

[22]Present address: Argonne National Laboratory, Chicago, Ill.

Under the ORNL Traveling Lecture Program

	AUTHOR(S)	TITLE	PRESENTED AT
215	Cameron, A. E.	"Geological Age Determination by Isotopic Measurement"	Auburn University, Auburn, Ala., Feb. 24, 1965
216			Knoxville College, Knoxville, Tenn., Apr. 28, 1965
217			University of South Carolina, Columbia, Mar. 15, 1965
218			North Carolina Academy of Science, Chapel Hill, May 7, 1965
219			Sam Houston State College, Huntsville, Tex., Oct. 18, 1965
220			Lamar State College, Beaumont, Tex., Oct. 19, 1965
221	White, J. C.	"The New Image of Analytical Chemistry in Science"	Glenville State College, Glenville, W.Va., Nov. 1, 1965
222			Bethany College, Bethany, W.Va., Nov. 3, 1965

PATENTS

	AUTHOR(S)	TITLE	PATENT NO(S).	ISSUED
223	Moore, F. L.	"Method for Separating Transplutonium Elements from Rare Earth Fission Products"	3,178,256	Apr. 13, 1965

Translations

	TRANSLATOR	ORIGINAL REFERENCE			TRANSLATION		
		Author (Transliterated)	Source	Language	Language	Translated Title	ORNL-tr Number
1	Kelley, M. T.	L. Němec	*Collection Czech. Chem. Commun.* **25**, 3085–96 (1960)	German	English	"Influence of the Recording Arrangement on the Form of Polarographic Curves"	555
2		S. B. Tsfasman	*Zavodsk. Lab.* **22**, 131–40 (1956)	Russian	English	"Differential Polarography on an Electronic Self-Recording Integro-Differentiating Polarograph"	562
3		F. G. Will	*Z. Electrochem.* **63**, 689–94 (1959)	German	English	"Transient Processes with Potentiostatic Regulating Circuits"	563
4		S. B. Tsfasman	*Elektronnye Polarography*, chap. II, Topic 11, Metalurgizdat, Moscow, 1960	Russian	English	"Speed of Response of a Polarograph"	564
5		P. Valenta and J. Vogel	*Chem. Listy* **54**, 1279–98 (1960)	Czechoslovakian	English	"Polarography with a Triangular Potential Impulse"	565
6		H. Gerischer and K. E. Staubach	*Z. Electrochem.* **61**, 789–94 (1957)	German	English	"The Electronic Potentiostat and Its Use for the Investigation of Rapid Electrode Reactions"	573
7		M. Breiter and F. G. Will	*Z. Electrochem.* **61**, 1177–83 (1957)	German	English	"The Inner Resistance of Potentiostatic Regulating Circuits and Examples of the Use of a Potentiostat"	574

	TRANSLATOR	ORIGINAL REFERENCE			TRANSLATION		
		Author (Transliterated)	Source	Language	Language	Translated Title	ORNL-tr Number
8	Kubota, H.	T. Uchida	*Tokyo Kogyo Shikensho Hokoku* **59**, 507–10 (1964)	Japanese	English	"Prediction of the Solubility and Temperature Dependence of Dissociation Constant of Carbonic Acid in Concentrated NaCl Solution"	738
9		S. Kaga and T. Hayashi	*Nippon Nogeikagaku Kaishi* (Noka) **37**(2), 116–17 (1963)	Japanese	English	"The Rapid Determination of L-Leucine, L-Isoleucine, and L-Valine in a Mixture by Infrared Spectrometry"	841
10		T. Shigematsu, M. Tabushi, and T. Tarumoto	*Nippon Kagaku Zasski* **84**, 131–34 (1963)	Japanese	English	"Solvent Extraction of Beryllium, Copper, Nickel, Cobalt, and Zinc as Dibenzoylmethane Complexes"	942
11	Miller, F. J.	L. Y. Kheifets	*Zh. Analit. Khim.* **20**(3), 388–91 (1965)	Russian	English	"Concerning the Method of Determining the Nature of the Polarographic Current in Relation to the Height of a Mercury Column"	772

Indexes

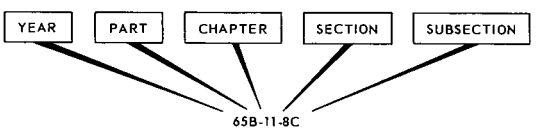
Helen P. Raaen Ann S. Haas[1]

Indexes are a part of the Analytical Chemistry Division annual report for the second time. The numbering system used has made possible the preparation of cumulative indexes to the annual progress reports from the Division. This section of the report contains a key-word index and an author index to this report. The cumulative indexes for the years 1964 and 1965 are bound separately and are designated ORNL-3904.

The Key Word Index is prepared both from the numbered headings that appear within the report and from the titles of the entries in the Presentation of Research Results and in the Translations. To increase the depth of indexing of the work reported, supplementary words were added to some of the headings. These words appear in parentheses, together with the heading, in the key-word index; they do not appear elsewhere in the report.

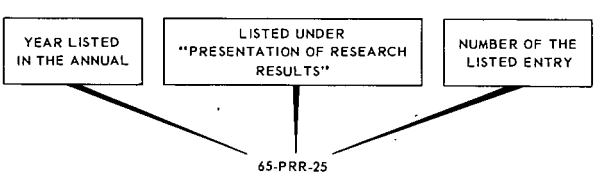
The Author Index is an alphabetical listing of authors, together with number entries that specify the material to which each author contributed. This index includes the authors of material in the body of the report, the authors of the entries listed in the Presentation of Research Results, and the translators of the entries listed in Translations. In the cases of co-authorships, the member of another ORNL division is so indicated by an asterisk which precedes that author's name. If, for any of a number of reasons, an author is not a permanent member of ORNL, the symbol = appears before his name.

An example of the numbering system used to refer to material in the main body of the report is 65B-11-8C, where 65 indicates the year of publication of the annual report, B designates a major part or division of the report, 11 is the chapter number, 8 is a primary section within the chapter, and C is a subsection (designated in the subsection heading with c instead of C); for example,



In the use of the index to locate material in the body of the report, only the last two groups of numbers are needed, because the chapters are numbered consecutively.

For reference to the Presentation of Research Results, the numbering system is of the type 65-PRR-25, where 65 designates the year in which the entry was listed in the annual report, PRR identifies the authored work as being listed in the Presentation of Research Results part of the annual report, and 25 is the number of the entry listed in that part of the report; for example,



In the use of the Index to locate an entry in the Presentation of Research Results part of the report, only the last group of numbers is needed, because the entries are numbered consecutively.

If the entry is in the Translations part of the report, the entry will be identified by the letters TR in the same position as the letters PRR.

[1]Technical Information Division.

Machine limitations necessitate that some entries be in an unusual form. Because special type cannot be used, entries that otherwise would require it are written out; for example, chemical symbols, chemical formulas, Greek letters, *o*-, *m*-, *p*-, etc. Superscript and subscript numbers cannot be printed; hence, valence is designated by a Roman numeral following the name of the element or ion, and an isotope mass number is indicated by a hyphen and an Arabic number following the name of the element. The lower-case *m*, used with an isotope mass number to designate a metastable state, appears as a capital *M* in the listing.

Additional useful entries are provided by dividing certain words; for example, ethylenediamine is divided into ethylene, di, and amine. *Chemical Titles* is used as a guide in making such divisions.

Key Word Index

DETERMINATION OF IODINE AND FLUORINE IN ,KIO2F2 65A-05-04F
/CNTAIN PHOSPHORO THIOATE, ,P=S AND PHOSPHORO DI THIOATE, ,P(S)SH GROUPS AS SEPARATION AND ANALYTICAL REA/ 65A-11-04B
/AINING PHOSPHORO THIOATE, ,P=S AND PHOSPHORO DI THIOATE, ,P(S)SH GROUPS AS SEPARATORY AND ANALYTICAL REA/ 65PRR-017
/IEW OF ORGANIC COMPOUNDS THAT CONTAIN PHOSPHORO THIOATE, ,P=S AND PHOSPHORO DI THIOATE, ,P(S)SH GROUPS A/ 65A-11-04B
/EVIEW OF ORGANIC COMPOUNDS CONTAINING PHOSPHORO THIOATE, ,P=S AND PHOSPHORO DI THIOATE, ,P(S)SH GROUPS A/ 65PRR-017
SULFUR-33 (N,P) CROSS SECTION 65A-11-03C
RUBIDIUM-85 (N,2N) CROSS SECTION 65A-11-02F
ALKYL BENZENE SULFONATE (ABS) CONTROL FOR THE FOAM SEPARATION PROCESS 65PRR-183
M FISSION OF URANIUM, AND THE PURIFICATI/ EVIDENCE OF THE ABSENCE OF LONG-LIVED ISOTOPES OF PROMETHIUM FRO 65PRR-089
ABSOLUTE COUNTING OF GAMMA ACTIVITY 65PRR-188
MITTERS ABSOLUTE LIQUID SCINTILLATION COUNTING OF BETA E 65PRR-014
ABSORBANCE SPECTRA OF SINGLE CRYSTALS 65PRR-214
PPER) ATOMIC ABSORPTION (DETERMINATION OF GOLD SILVER AND CO 65A-07-018
LID MATERIALS ABSORPTION AND DIFFUSE REFLECTANCE SPECTRA OF SO 65A-02-01C
LITHIUM FLUORIDE - BERYLLIUM FLUORIDE - ZIRCONIUM FLUOR/ ABSORPTION AND REFLECTANCE SPECTRA OF IRRADIATED 65-02-01A
QUANTITATIVE ANALYSIS BY THE X-RAY ABSORPTION EDGE TECHNIQUE 65PRR-166
NDOWS / RIBO NUCLEASE / BACILLUS-SUBTILIS / ALIPAL-LC-43/ ABSORPTION SPECTRAL MEASUREMENTS (SOLAR CELL WI 65A-05-04M
MOLTEN SALT SYSTEMS ABSORPTION SPECTROPHOTOMETRIC STUDIES RELATED TO 65A-02-01
FLUORIDE - SODIUM FLUORIDE - POTASSIUM FLUORIDE ABSORPTION SPECTRUM OF COBALT(II) ION IN LITHIUM 65A-02-01B
SYNTHESIS AND INFRARED ABSORPTION SPECTRUM OF DI BORANE - BORON-10 65PRR-079
ORATE MEDIA I. MATHEMATICAL RESOLUTION OF THE OVERL/ THE ABSORPTION SPECTRUM OF THE URANYL ION IN PER CHL 65PRR-161
PARTICLE ACCELERATORS IN PURE MATERIALS ANALYSES 65PRR-178
IME EFFECTS) STUDY OF PRECISION AND ACCURACY IN NEUTRON ACTIVATION ANALYSIS (DEAD T 65A-11-08B
/ROMETRIC DC POLAROGRAPHY - A NEW HIGH PRECISION AND HIGH ACCURACY METHOD (ELECTROCHEMISTRY / MODEL-Q-19/ 65A-01-03
OF GAMMA IRRADIATED MIXTURES OF METHANOL AND HYDROCHLORIC ACID GAS CHROMATOGRAPHIC ANALYSIS 65A-03-02
TED SOLUTIONS OF LITHIUM CHLORIDE CONTAINING HYDROCHLORIC ACID ALPHA RADIATION EFFECTS ON CONCENTRA 65PRR-165
TRANSFER RIBO NUCLEIC ACID (TRNA) ANALYTICAL STUDIES 65A-06-01
STUDIES ON THE PHENYL ALANYL TRANSFER RIBO NUCLEIC ACID (TRNA) SYSTEM 65A-06-02
TRODE O/ POLAROGRAPHY OF LEAD(II) IN AQUEOUS HYDROFLUORIC ACID (1-12-MOLAR) WITH A DROPPING MERCURY ELEC 65PRR-041
/ PER CHLORATE / CHRONOPOTENTIOMETRIC OXIDATION OF OXALIC ACID / CHRONOPOTENTIOMETRIC OXIDATION OF FERROU/ 65A-01-02
NTRATED LITHIUM CHLORIDE SOLUTION - DETERMINATION OF FREE ACID AND HYDROLYZABLE CATION REACTIONS IN CONCE 65PRR-022
/PPING MERCURY ELECTRODE FOR POLAROGRAPHY IN HYDROFLUORIC ACID AND OTHER GLASS CORRODING MEDIA - EVALUATI/ 65PRR-039
/URY ELECTRODE OF TEFLON FOR POLAROGRAPHY IN HYDROFLUORIC ACID AND OTHER GLASS CORRODING MEDIA - EVALUATI/ 65PRR-040
N OF THE HORIZONTAL ORIFICE/ POLAROGRAPHY IN HYDROFLUORIC ACID AND OTHER GLASS CORRODING MEDIA (EVALUATIO 65A-05-03A
X REACTION REDUCTION IN AQUEOUS SOLUTIONS OF HYDROCHLORIC ACID AND POTASSIUM CHLORIDE / TO LEAD METAL REDO 65PRR-010
GAMMA RADIOLYSIS OF ACID CHLORIDE SOLUTIONS 65A-03-01
MPERATURE DEPENDENCE OF DISSOCIATION CONSTANT OF CARBONIC ACID IN CONCENTRATED SODIUM CHLORIDE SOLUTION /E 65TR-08
DIATION ON THE DETERMINATION OF URANIUM(VI) IN PHOSPHORIC ACID SOLUTIONS OF URANIUM(IV) OXIDE /60 GAMMA RA 65PRR-021
NITRATE, PHENOL DI SULFONIC ACID SPECTROPHOTOMETRIC METHOD 65PRR-132
/EAD TO LEAD METAL REDOX REACTION IN AQUEOUS HYDROFLUORIC ACID WITH HORIZONTAL ORIFICE TEFLON DROPPING ME/ 65A-05-03A
YDROXIDE TITRATION METHOD STRONG AND WEAK ACIDS IN THEIR MIXTURES, POTENTIOMETRIC SODIUM H 65PRR-127
/TRACTION OF METAL IONS FROM AQUEOUS SOLUTIONS OF ORGANIC ACIDS WITH HIGH MOLECULAR WEIGHT AMINES - THE T/ 65PRR-031
ATION A PROGRAMMABLE HIGH SPEED DATA ACQUISITION SYSTEM FOR MASS SPECTROMETRIC APPLIC 65PRR-192
C ACIDS WITH HIGH MOLECULAR WEIGHT AMINES - THE TRIVALENT ACTINIDE - LANTHANIDE ELEMENTS /UTIONS OF ORGANI 65PRR-031
PARTICLE SIZE DISTRIBUTION IN URANIUM(IV) OXIDE, NEUTRON ACTIVATION - SEDIMENTATION METHOD 65PRR-154
USE OF COMPUTERS IN APPLIED ACTIVATION ANALYSIS 65A-11-06D
ACTIVATION ANALYSIS 65A-11-08
DETERMINATION OF TECHNETIUM-99 BY NEUTRON ACTIVATION ANALYSIS 65A-11-08C
APPLIED NEUTRON ACTIVATION ANALYSIS 65A-11-08E
DETERMINATION OF URANIUM IN SEAWATER BY NEUTRON ACTIVATION ANALYSIS 65A-11-08G
DETERMINATION OF MOLYBDENUM IN HAIR AND WOOL BY NEUTRON ACTIVATION ANALYSIS 65PRR-018
HELIUM-3 ACTIVATION ANALYSIS 65PRR-198
UATION OF COMPUTER PROGRAMS FOR GAMMA-RAY SPECTROMETRY IN ACTIVATION ANALYSIS EVAL 65A-11-06B
UATION OF COMPUTER PROGRAMS FOR GAMMA-RAY SPECTROMETRY IN ACTIVATION ANALYSIS THE EVAL 65PRR-171
EV NEUTRON GENERATORS - RAPID ESTIMATION AND INFLUENCE IN ACTIVATION ANALYSIS OUTPUT SPECTRA OF 14-M 65PRR-044
ETERMINATION OF OXYGEN IN ALKALI METALS BY 14-MEV NEUTRON ACTIVATION ANALYSIS) /N HYDROGEN AND OXYGEN / D 65A-04-03A
RON S/ THEORY AND EXPERIMENT IN RAPID, SENSITIVE HELIUM-3 ACTIVATION ANALYSIS - HELIUM-3 REACTIONS AS NEUT 65PRR-045
INTERFERENCES ACTIVATION ANALYSIS - REACTOR NEUTRON FLUXES AND 65PRR-197
STUDY OF PRECISION AND ACCURACY IN NEUTRON ACTIVATION ANALYSIS (DEAD TIME EFFECTS) 65A-11-08B
UNCTION/ STUDIES OF HELIUM-3 NUCLEAR REACTIONS (HELIUM-3 ACTIVATION ANALYSIS / SENSITIVITY / EXCITATION F 65A-11-08A
TWIN WETHERS RAISED ON TWO SOILS AS DETERMINED BY NEUTRON ACTIVATION ANALYSIS /EMENT CONTENT OF WOOL FROM 65PRR-019
STIMATION AND INFLUENCE IN CROSS SECTION MEASUREMENTS AND ACTIVATION ANALYSIS /EUTRON GENERATORS - RAPID E 65PRR-043
OUNTERED IN GAMMA-RAY SPECTROMETRY FOR RADIOCHEMISTRY AND ACTIVATION ANALYSIS /CF ARTIFACTS AND ERRORS ENC 65A-11-03E
OUNTERED IN GAMMA-RAY SPECTROMETRY FOR RADIOCHEMISTRY AND ACTIVATION ANALYSIS /OF ARTIFACTS AND ERRORS ENC 65PRR-003
L AND CHARACTERIZATION OF THE SURFACE CONTAMI/ THE USE OF ACTIVATION ANALYSIS IN PROCEDURES FOR THE REMOVA 65PRR-008
LICATIONS NEUTRON ACTIVATION ANALYSIS OF HUMAN HAIR - FORENSIC APP 65A-11-08D
NEUTRON ACTIVATION ANALYSIS OF RIVER WATER 65A-11-08H
NIC AND NUCLEAR CHEMISTRY OF TECHNETIUM - THERMAL NEUTRON ACTIVATION ANALYSIS OF TECHNETIUM INORGA 65PRR-077
APPLICATION OF NEUTRON ACTIVATION ANALYSIS TO FORENSIC SCIENCE 65PRR-185
UTRONS ACTIVATION ANALYSIS WITH PARTICLES OTHER THAN NE 65PRR-189
H HELIUM-3 IONS ACTIVATION ANALYSIS WITH 14-MEV NEUTRONS AND WIT 65PRR-176
4-MEV NEUTRONS WITH TECHNETIUM-99 ACTIVATION CROSS SECTIONS FOR THE REACTIONS OF I 65A-11-09E
4.8-MEV NEUTRONS WITH TECHNETIUM-99 ACTIVATION CROSS SECTIONS FOR THE REACTIONS OF I 65PRR-075
ACTIVATION OF NICKEL-64 WITH 14-MEV NEUTRONS 65A-11-09D
RECENT ADVANCES IN ANALYTICAL EMISSION SPECTROMETRY 65PRR-212

OLAROGRAPHIC DETECTION LIMITS FOR URANIUM / CONCENTRATION ADVANTAGE / SEAWATER) /LOSS IN POLAROGRAPHY / P 65A-01-06
GEOLOGICAL AGE DETERMINATION BY ISOTOPIC MEASUREMENT 65PRR-215
CNIUM ALLOY . ELECTRON OPTICAL STUDY OF THE EFFECT OF AGING ON CARBIDES IN A NIOBIUM - TUNGSTEN - ZIRC 65PRR-210
DETERMINATION OF LEAD, BISMUTH, CADMIUM, TIN, AND ZINC IN AIRBORNE DUST SAMPLES 65A-07-02A
ITE DIFFUSION TUBES INVESTIGATION OF AIRBORNE RADIO IODINE SPECIES BY MEANS OF COMPOS 65PRR-067
M STUDIES ON THE PHENYL ALANYL TRANSFER RIBO NUCLEIC ACID (TRNA) SYSTE 65A-06-02
/SOLAR CELL WINDOWS / RIBO NUCLEASE / BACILLUS-SUBTILIS / ALIPAL-LO-436 / AMMONIUM SALT OF A SULFONATED S/ 65A-05-04M
/MINATION OF LOW CONCENTRATIONS OF OXYGEN AND HYDROGEN IN ALKALI MATERIALS BY A MODIFIED AMALGAMATION TEC/ 65PRR-073
DETERMINATION OF OXYGEN IN ALKALI METAL/ ANALYSIS OF THE ALKALI METALS (FOR CARBON HYDROGEN AND OXYGEN / 65A-04-03A
DI OXIDE IN GLOVE BOX ATMOSPHERES ON THE ANALYSIS OF THE ALKALI METALS /N, HYDROGEN, NITROGEN, AND CARBON 65PRR-175
/ CARBON HYDROGEN AND OXYGEN / DETERMINATION OF OXYGEN IN ALKALI METALS BY 14-MEV NEUTRON ACTIVATION ANAL/ 65A-04-03A
AS EXTRACTANTS FOR METAL IONS - SELECTIVE EXTRACTIO/ TRI ALKYL AND HEXA ALKYL PHOSPHORO THIOIC TRI AMIDES 65PRR-015
FOAM SEPARATION PROCESS ALKYL BENZENE SULFONATE (ABS) CONTROL FOR THE 65PRR-183
ANALYSIS OF ALKYL BENZENE SULFONATE DETERGENTS 65A-05-04K
PAL-LO-436 / AMMONIUM SALT OF A SULFONATED STRAIGHT CHAIN ALKYL PHENOXY POLY (ETHYLENE OXY) ETHANOL) /I 65A-05-04M
FOR METAL IONS - SELECTIVE EXTRACTIO/ TRI ALKYL AND HEXA ALKYL PHOSPHORO THIOIC TRI AMIDES AS EXTRACTANTS 65PRR-015
DETERMINATION OF TRACE METALS IN NIOBIUM - IRON ALLOY 65A-05-04B
OF AGING ON CARBIDES IN A NIOBIUM - TUNGSTEN - ZIRCONIUM ALLOY ELECTRON OPTICAL STUDY OF THE EFFECT 65PRR-210
GROSS ALPHA 65PRR-150
NEW RARE EARTH ALPHA EMITTER, EUROPIUM-148 65PRR-056
STUDIES OF RARE EARTH ALPHA EMITTING RADIONUCLIDES 65A-11-01F
CTROPHOTOMETRIC DETERMINATION OF NITRATED 4 SEC BUTYL 2 (ALPHA METHYL BENZYL) PHENOL (BAMBP) SPE 65A-05-04E
INTERACTIONS OF CADMIUM-106 WITH ALPHA PARTICLES 65PRR-179
ALPHA PROPORTIONAL COUNTER 65PRR-147
S OF LITHIUM CHLORIDE CONTAINING HYDROCHLORIC ACID ALPHA RADIATION EFFECTS ON CONCENTRATED SOLUTION 65PRR-165
HIGH LEVEL ALPHA RADIATION LABORATORY 65B-17-01
OF OXYGEN AND HYDROGEN IN ALKALI MATERIALS BY A MODIFIED AMALGAMATION TECHNIQUE /ON OF LOW CONCENTRATIONS 65PRR-073
MASS FISSION YIELD CURVE FOR AMERICIUM-241 65PRR-046
N,N,N TRI HEXYL PHOSPHORIC TRI AMIDE (THPA) AS AN EXTRACTANT FOR METAL IONS 65A-11-04A
EXTRACTIO/ TRI ALKYL AND HEXA ALKYL PHOSPHORO THIOIC TRI AMIDES AS EXTRACTANTS FOR METAL IONS - SELECTIVE 65PRR-015
HEPTA DECYL AMINE 65A-13-01E
/US SOLUTIONS OF ORGANIC ACIDS WITH HIGH MOLECULAR WEIGHT AMINES - THE TRIVALENT ACTINIDE - LANTHANIDE EL/ 65PRR-031
/WS / RIBO NUCLEASE / BACILLUS-SUBTILIS / ALIPAL-LO-436 / AMMONIUM SALT OF A SULFONATED STRAIGHT CHAIN AL/ 65A-05-04M
/ OXALIC ACID / CHRONOPOTENTIOMETRIC OXIDATION OF FERROUS AMMONIUM SULFATE / CHRONOPOTENTIOMETRY WITH PLA/ 65A-01-02
ZIRCONIUM IN MSRE FUEL, AMPEROMETRIC CUPFERRON TITRATION METHOD 65PRR-135
ION AND HIGH ACCURACY METHOD (ELECTROCHEMIS/ COMPARATIVE AMPEROMETRIC DC POLAROGRAPHY - A NEW HIGH PRECIS 65A-01-03
CHROMIUM IN MSRE FUEL, AMPEROMETRIC FERROUS SULFATE TITRATION METHOD 65PRR-136
/GRAPHY / PRECISION OF DC POLAROGRAPHY / SUBTRACTIVE MODE AMPEROMETRIC POLAROGRAPHY / COMPARATIVE POLAROG/ 65A-05-03A
REVIEW OF AMPEROMETRIC TITRATIONS 65PRR-060
ANALYTICAL BIOCHEMISTRY 65A-06
APPLICATION OF RADIOISOTOPES TO ANALYTICAL CHEMISTRY 65A-11-07
SPECTROMETRY IN ANALYTICAL CHEMISTRY 65PRR-172
MODERN ANALYTICAL CHEMISTRY 65PRR-209
STATISTICAL QUALITY CONTROL REPORT, APR.-JUNE,1965, ANALYTICAL CHEMISTRY DIVISION 65PRR-081
STATISTICAL QUALITY CONTROL REPORT, JAN.-MAR.,1965, ANALYTICAL CHEMISTRY DIVISION 65PRR-082
STATISTICAL QUALITY CONTROL REPORT, JAN.-SEPT.,1964, ANALYTICAL CHEMISTRY DIVISION 65PRR-084
ANNUAL PROGRESS REPORT FOR PERIOD ENDING NOV.15,1964, ANALYTICAL CHEMISTRY DIVISION 65PRR-091
STATISTICAL QUALITY CONTROL REPORT, ANALYTICAL CHEMISTRY DIVISION, JAN.-DEC.,1964 65PRR-083
ANALYTICAL CHEMISTRY FOR REACTOR PROJECTS 65A-04
MANAGEMENT LOOKS AT ANALYTICAL CHEMISTRY IN A NATIONAL LABORATORY 65PRR-204
ANALYTICAL CHEMISTRY IN NUCLEAR TECHNOLOGY 65PRR-052
EIGHTH CONFERENCE ON ANALYTICAL CHEMISTRY IN NUCLEAR TECHNOLOGY 65PRR-053
A RESUME OF THE EIGHTH CONFERENCE ON ANALYTICAL CHEMISTRY IN NUCLEAR TECHNOLOGY 65PRR-054
THE NEW IMAGE OF ANALYTICAL CHEMISTRY IN SCIENCE 65PRR-221
REVIEW OF ANALYTICAL CHEMISTRY OF THALLIUM 65PRR-057
OUP MONTHLY SUMMARY - NOV.,1964 / DEC.,1964 / JAN.,1965 / ANALYTICAL CHEMISTRY RESEARCH AND DEVELOPMENT GR 65PRR-104
RECENT ADVANCES IN ANALYTICAL EMISSION SPECTROMETRY 65PRR-212
HIGH RADIATION LEVEL ANALYTICAL FACILITY (HRLAF) 65B-17-05
ANALYTICAL INSTRUMENTATION 65A-01
A SURVEY OF ANALYTICAL INSTRUMENTATION DEVELOPMENT AT ORNL 65PRR-203
HIGH RADIATION LEVEL ANALYTICAL LABORATORY (HRLAL) 65B-17-03
PLE PREPARATION APPARATUS FOR THE MOLTEN SALT REACTOR EX/ ANALYTICAL LABORATORY HOT CELL EQUIPMENT I. SAM 65PRR-027
REMOTELY CONTROLLED CORROSION RESISTANT PIPETTER ANALYTICAL LABORATORY HOT CELL EQUIPMENT II. A 65PRR-028
REMOTELY OPERATED FILTER PHOTOMETER ANALYTICAL LABORATORY HOT CELL EQUIPMENT III. A 65PRR-026
ORNL MASTER ANALYTICAL MANUAL 65C-18
CUMULATIVE INDEXES TO THE ORNL MASTER ANALYTICAL MANUAL 65C-18-01
MAINTENANCE OF THE ORNL MASTER ANALYTICAL MANUAL 65C-18-02
ORNL MASTER ANALYTICAL MANUAL 65C
INDEXES TO THE ORNL MASTER ANALYTICAL MANUAL (1953-1964) 65PRR-093
NIUM (TRU) PROCESS OTHER ORNL MASTER ANALYTICAL MANUAL METHODS NEEDED IN THE TRANSURA 65PRR-128
ORNL MASTER ANALYTICAL MANUAL, SUPPLEMENT 7 65PRR-092
EFFECTS OF RADIATION ON ANALYTICAL METHODS 65A-03
/ ASSEMBLY FOR COBALT-60 SOURCE (EFFECTS OF RADIATION ON ANALYTICAL METHODS / REMOTELY CONTROLLED INSTRU/ 65A-01-12
ANALYTICAL RADIOCHEMISTRY OF TECHNETIUM 65PRR-002
ND PHOSPHORO DI THIOATE, ,P(S)SH GROUPS AS SEPARATORY AND ANALYTICAL REAGENTS /G PHOSPHORO THIOATE, ,P=S A 65PRR-017
ND PHOSPHORO DI THIOATE, ,P(S)SH GROUPS AS SEPARATION AND ANALYTICAL REAGENTS /N PHOSPHORO THIOATE, ,P=S A 65A-11-04B
ANALYTICAL RESEARCH 65A
ANALYTICAL SERVICES FOR CHEMICAL ENGINEERS 65PRR-182
TRANSFER RIBO NUCLEIC ACID (TRNA) ANALYTICAL STUDIES 65A-06-01
ANALYTICAL STUDIES OF MOLTEN SALT SYSTEMS 65A-02
CONTROLLED POTENTIAL POLAROGRAPHY AND COULOMETRY AS MICRO ANALYTICAL TECHNIQUES 65PRR-181
/OLAROGRAPHY AND CONTROLLED POTENTIAL COULOMETRY AS MICRO ANALYTICAL TECHNIQUES (DETERMINATION OF URANIU/ 65A-01-07
RADIOACTIVATION ANALYSIS AS AN ANALYTICAL TOOL 65PRR-187
AUTOMATED NUCLEOTIDE ANALYZER 65PRR-207
/NALYSIS OF SOLIDS BY X-RAY FLUORESCENCE / ELECTRON PROBE ANALYZER / DETERMINATION OF AREAL DENSITY OF GO/ 65A-07-01A
1964, ANALYTICAL CHEMISTRY DIVISION ANNUAL PROGRESS REPORT FOR PERIOD ENDING NOV.15, 65PRR-091
MSRE SAMPLE PREPARATION APPARATUS 65A-04-01A
/CAL LABORATORY HOT CELL EQUIPMENT 1. SAMPLE PREPARATION APPARATUS FOR THE MOLTEN SALT REACTOR EXPERIMEN/ 65PRR-027
APPARATUS, GENERAL 65PRR-148
/.,1964 / DEC.,1964 / JAN.,1965 / FEB.,1965 / MAR.,1965 / APR.,1965 / MAY,1965 / JUNE,1965 / JULY,1965 / / 65PRR-104

STATISTICAL QUALITY CONTROL REPORT, APR.-JUNE,1965, ANALYTICAL CHEMISTRY DIVISION 65PRR-081
CROPPING MERCURY ELECTRODE O/ POLAROGRAPHY OF LEAD(II) IN AQUEOUS HYDROFLUORIC ACID (1-12-MOLAR) WITH A 65PRR-041
/OGRAPHY OF DIVALENT LEAD TO LEAD METAL REDOX REACTION IN AQUEOUS HYDROFLUORIC ACID WITH HORIZONTAL ORIFI/ 65A-05-03A
BARIUM RADIOACTIVITY IN AQUEOUS OR ORGANIC SOLUTIONS 65PRR-139
STRONTIUM RADIOACTIVITY AND / OR BARIUM RADIOACTIVITY IN AQUEOUS OR ORGANIC SOLUTIONS 65PRR-140
CERIUM RADIOACTIVITY IN AQUEOUS OR ORGANIC SOLUTIONS 65PRR-141
NIOBIUM RADIOACTIVITY IN AQUEOUS OR ORGANIC SOLUTIONS 65PRR-142
TELLURIUM RADIOACTIVITY IN AQUEOUS OR ORGANIC SOLUTIONS 65PRR-144
ZIRCONIUM RADIOACTIVITY IN AQUEOUS OR ORGANIC SOLUTIONS 65PRR-145
IODINE RADIOACTIVITY IN AQUEOUS OR ORGANIC SOLUTIONS 65PRR-149
RUTHENIUM RADIOACTIVITY IN AQUEOUS SOLUTIONS 65PRR-143
/ DIVALENT LEAD TO LEAD METAL REDOX REACTION REDUCTION IN AQUEOUS SOLUTIONS OF HYDROCHLORIC ACID AND POTA/ 65PRR-010
ECULAR WEIGH/ LIQUID-LIQUID EXTRACTION OF METAL IONS FROM AQUEOUS SOLUTIONS OF ORGANIC ACIDS WITH HIGH MOL 65PRR-031
TROLLED POTENTIAL COULOMETRIC TITRATION OF URANIUM(VI) IN AQUEOUS SOLUTIONS OF SODIUM FLUORIDE CON 65PRR-034
TOTAL LANTHA/ RAPID REMOVAL OF TRANSURANIUM ELEMENTS FROM AQUEOUS SOLUTIONS PRIOR TO THE DETERMINATION OF 65PRR-032
CESIUM RADIOACTIVITY IN AQUEOUS SOLUTIONS, CHLORO STANNATE METHOD 65PRR-151
ER ARBITRARY-SET, PROPORTIONAL TEMPERATURE CONTROLL 65PRR-035
PROGRAM NETAREA (GAMMA-RAY PHOTOPEAK AREA) 65A-11-06E
/LUORESCENCE / ELECTRON PROBE ANALYZER / DETERMINATION OF AREAL DENSITY OF GOLD COATING ON SILICON RADIAT/ 65A-07-01A
INFLUENCE OF THE RECORDING ARRANGEMENT ON THE FORM OF POLAROGRAPHIC CURVES 65TR-01
SPECTROPHOTOMETRIC DETERMINATION OF URANIUM WITH ARSENAZO-III 65A-05-04C
33) HALF-LIFE MEASUREMENTS (CESIUM-137 / SAMARIUM-153 / ARSENIC-77 / GOLD-198 / IODINE-133 / PHOSPHORUS- 65A-11-03D
ECTROMETRY FOR RADIOCHEMISTRY AND / EXPERIMENTAL STUDY OF ARTIFACTS AND ERRORS ENCOUNTERED IN GAMMA-RAY SP 65A-11-03E
ECTROMETRY FOR RADIOCHEMISTRY AND / EXPERIMENTAL STUDY OF ARTIFACTS AND ERRORS ENCOUNTERED IN GAMMA-RAY SP 65PRR-003
ARTIFACTS IN GAMMA-RAY SPECTROMETRY 65A-11-03F
MODIFICATIONS TO THE COBALT-60 SOURCE ASSEMBLY 65A-03-04
TION ON ANALYTICAL METHODS / REMOTELY/ SAMPLE IRRADIATION ASSEMBLY FOR COBALT-60 SOURCE (EFFECTS OF RADIA 65A-01-12
F STANDARD CHROMIUM(II) SOLUTIONS INTEGRATED ASSEMBLY FOR GENERATION, STORAGE, AND DELIVERY O 65PRR-055
F IODINE WITH CYCLOHEXANE AND WITH BENZENE AT 300C AND 20 ATMOSPHERES /S OF THE PRODUCTS OF THE REACTION O 65A-04-04C
/EN, HYDROGEN, NITROGEN, AND CARBON DI OXIDE IN GLOVE BOX ATMOSPHERES ON THE ANALYSIS OF THE ALKALI METAL/ 65PRR-175
THE ISOTOPIC COMPOSITION OF ATMOSPHERIC NEON 65PRR-071
THE ISOTOPIC COMPOSITION OF ATMOSPHERIC NEON 65PRR-208
RADIO RELEASE METHODS FOR THE EVALUATION OF ATMOSPHERIC POLLUTION - SULFUR DI OXIDE 65PRR-005
RADIO RELEASE METHODS FOR THE EVALUATION OF ATMOSPHERIC POLLUTION - SULPHUR DI OXIDE 65A-11-07B
AND COPPER) ATOMIC ABSORPTION (DETERMINATION OF GOLD SILVER 65A-07-01B
THE PRESENT STATUS OF THE ATOMIC WEIGHTS 65PRR-163
LS GOLD ATOMS SPUTTERED FROM NEUTRON IRRADIATED GOLD FOI 65A-11-08F
AR.,1965 / APR.,1965 / MAY,1965 / JUNE,1965 / JULY,1965 / AUG.,1965 / SEPT.,1965 / OCT.,1965 /EB.,1965 / M 65PRR-104
LEAD-210 IN AUTO EXHAUST FUMES AND IN NATURAL MATERIALS 65A-11-02H
AUTOMATED NUCLECTIDE ANALYZER 65PRR-207
5, ELECTRONIC, CONTROLLED POTENTIAL AUTOMATIC COULOMETRIC TITRATOR, ORNL MODEL-Q-200 65PRR-146
/ READING, LINEAR, RECORDING CONDUCTOMETRIC TITRATOR WITH AUTOMATIC TEMPERATURE COMPENSATION OR PROPORTIO/ 65PRR-036
/SALT REACTOR EXPERIMENT (MSRE) FUEL, HIGH SENSITIVITY, AUTOMATIC, CONTROLLED POTENTIAL COULOMETRIC TIT/ 65PRR-122
URANIUM IN MOLTEN SALT REACTOR EXPERIMENT (MSRE) FUEL, AUTOMATIC, CONTROLLED POTENTIAL, COULOMETRIC TIT 65PRR-121
ING OF IRON-55 MEASUREMENT OF WEAR RATES IN AUTOMOTIVE ENGINES BY LIQUID SCINTILLATION COUNT 65A-11-07C
ING OF IRON-55 WEAR RATES IN AUTOMOTIVE ENGINES BY LIQUID SCINTILLATION COUNT 65PRR-048
AVAILABILITY AND USE OF RADIOACTIVITY STANDARDS 65PRR-024
NITRATE IN TRANSURANIUM (TRU) SOLUTIONS, AZO DYE FILTER PHOTOMETRIC METHOD 65PRR-123
) AZO DYES (CALCICHROME / CHLORO PHOSPHONAZO-III 65A-13-01F
/TRAL MEASUREMENTS (SOLAR CELL WINDOWS / RIBO NUCLEASE / BACILLUS-SUBTILIS / ALIPAL-LO-436 / AMMONIUM SA/ 65A-05-04M
F NITRATED 4 SEC BUTYL 2 (ALPHA METHYL BENZYL) PHENOL (BAMBP) SPECTROPHOTOMETRIC DETERMINATION O 65A-05-04E
/ATE MEDIA I. MATHEMATICAL RESOLUTION OF THE OVERLAPPING BAND STRUCTURE AND STUDIES OF THE ENVIRONMENTAL/ 65PRR-161
/II) CHLORIDE IN LANTHANUM CHLORIDE / SODIUM SUPEROXIDE / BARIUM BROMITE MONO HYDRATE / SODIUM HEXA CHLOR/ 65A-12
FLAME PHOTOMETRY (COLLECTION OF BARIUM FROM TAP WATER) 65A-07-01D
IONS BARIUM RADIOACTIVITY IN AQUEOUS OR ORGANIC SOLUT 65PRR-139
IONS STRONTIUM RADIOACTIVITY AND / OR BARIUM RADIOACTIVITY IN AQUEOUS OR ORGANIC SOLUT 65PRR-140
ROL OF DROPPING MERCURY ELECTRODE DROP TIME / LINEAR TIME BASE / SMOLER DROPPING MERCURY ELECTRODE) /CONT 65A-01-04
BBER SECTIONS OF COMPOSITE DIFFUSION TUBES CHEMICAL BEHAVIOR OF THE RADIO IODINE SPECIES FOUND ON RU 65PRR-069
IDENTITY, CHARACTER AND CHEMICAL BEHAVIOR OF VAPOR FORMS OF RADIO IODINE 65PRR-162
MEANS OF COMPOSITE DIFFUSI/ INVESTIGATION OF RADIO IODINE BEHAVIOR UNDER MOIST CONDITIONS IN MAY PACKS BY 65PRR-066
IC STUDY OF THE STABILITY OF IODINE IN CYCLOHEXANE AND IN BENZENE SPECTROPHOTOMETR 65A-04-04B
DUCTS OF THE REACTION OF IODINE WITH CYCLOHEXANE AND WITH BENZENE AT 300C AND 20 ATMOSPHERES /S OF THE PRO 65A-04-04C
PHENYL PHOSPHONATE IN DI SEC BUTYL PHOSPHONATE - DI ETHYL BENZENE SOLUTIONS, DIELECTRIC CONSTANT METHOD / 65PRR-156
EPARATION PROCESS ALKYL BENZENE SULFONATE (ABS) CONTROL FOR THE FOAM S 65PRR-183
ANALYSIS OF ALKYL BENZENE SULFONATE DETERGENTS 65A-05-04K
TION OF BERYLLIUM, COPPER, NICKEL, COBALT, AND ZINC AS DI BENZOYL METHANE COMPLEXES SOLVENT EXTRAC 65TR-10
IC DETERMINATION OF NITRATED 4 SEC BUTYL 2 (ALPHA METHYL BENZYL) PHENOL (BAMBP) SPECTROPHOTOMETR 65A-05-04E
I BUTYL PHOSPHONIUM CHL/ QUATERNARY PHOSPHONIUM HALIDES (BENZYL TRI BUTYL PHOSPHONIUM BROMIDE / BENZYL TR 65A-13-01A
SPHONIUM HALIDES (BENZYL TRI BUTYL PHOSPHONIUM BROMIDE / BENZYL TRI BUTYL PHOSPHONIUM CHLORIDE) /ARY PHO 65A-13-01A
CATION TO PURIFICATION AND RADIOCHEMICAL DETERMINATION OF BERKELIUM /BERKELIUM FROM OTHER ELEMENTS - APPLI 65PRR-033
URIFICATION AND RADIOCHEMICAL DETERMINATIO/ SEPARATION OF BERKELIUM FROM OTHER ELEMENTS - APPLICATION TO P 65PRR-033
/AND REFLECTANCE SPECTRA OF IRRADIATED LITHIUM FLUORIDE - BERYLLIUM FLUORIDE - ZIRCONIUM FLUORIDE - THORI/ 65-C2-01A
/LT SYSTEMS (SILVER / IRON / NICKEL / LITHIUM FLUORIDE - BERYLLIUM FLUORIDE / LITHIUM FLUORIDE - SODIUM / 65A-C2-02
I BENZOYL METHANE COMPLEXES SOLVENT EXTRACTION OF BERYLLIUM, COPPER, NICKEL, COBALT, AND ZINC AS D 65TR-10
HALF-LIVES OF LONG-LIVED BETA EMITTERS (CHLORINE-36 BERYLLIUM-10) 65A-11-01D
PREPARATION BY ELECTRODEPOSITION OF MOUNTS FOR 4 PI BETA COUNTING 65A-11-03A
) PROCESS SOLUTIONS, 8 HYDROXY QUINOLINE PRECIPITATION - BETA COUNTING METHOD /VITY IN TRANSURANIUM (TRU 65PRR-129
ABSOLUTE LIQUID SCINTILLATION COUNTING OF BETA EMITTERS 65PRR-014
HALF-LIVES OF LONG-LIVED BETA EMITTERS (CHLORINE-36 BERYLLIUM-10) 65A-11-01D
ANALYTICAL BIOCHEMISTRY 65A-06
/CAL AND EXPERIMENTAL ASPECTS OF QUENCHING VARIABLES FROM BIOMEDICAL SAMPLES IN LIQUID SCINTILLATOR SYSTE/ 65PRR-199
SAMPLES DETERMINATION OF LEAD, BISMUTH, CADMIUM, TIN, AND ZINC IN AIRBORNE DUST 65A-07-02A
/RNL MODEL-Q-2564 (POTENTIOSTAT / CONTROLLED POTENTIAL / BLANK CORRECTIONS IN CONTROLLED POTENTIAL COULO/ 65A-01-05
/IC METHOD / CONTROLLED POTENTIAL COULOMETRIC TITRATION / BLANK CORRECTIONS IN COULOMETRY / POTENTIOSTATI/ 65A-01-07
BORON-10 DI BORANE 65A-13-01C
SYNTHESIS AND INFRARED ABSORPTION SPECTRUM OF DI BORANE - BORON-10 65PRR-079
ERMAL NEUTRON DETECTION N,N,N TRI METHYL BORAZINE AS A LIQUID SCINTILLATOR SOLVENT FOR TH 65A-11-05A
ERMAL NEUTRON DETECTION N,N,N, TRI METHYL BORAZINE AS A LIQUID SCINTILLATOR SOLVENT FOR TH 65PRR-049
BORON, SPECTROPHOTOMETRIC CURCUMIN METHOD 65PRR-157
SYNTHESIS AND INFRARED ABSORPTION SPECTRUM OF DI BORANE - BORON-10 65PRR-079
BORON-10 DI BORANE 65A-13-01C

/OXYGEN, HYDROGEN, NITROGEN, AND CARBON DI OXIDE IN GLOVE BOX ATMOSPHERES ON THE ANALYSIS OF THE ALKALI M/ 65PRR-175
I, AND NEODYMIUM-147 GAMMA-RAY BRANCHINGS IN THE DECAY OF INDIUM-116, CERIUM-14 65PRR-169
GAMMA-RAY BRANCHINGS IN URANIUM-237 65A-11-01C
POTENTIALS OF / EFFECT OF JUNCTION POTENTIALS AT THE SALT BRIDGE ON THE MEASURED VALUES FOR THE HALF-WAVE 65PRR-010
/RNARY PHOSPHONIUM HALIDES (BENZYL TRI BUTYL PHOSPHONIUM BROMIDE / BENZYL TRI BUTYL PHOSPHONIUM CHLORIDE/ 65A-13-01A
A NEW ISOMER OF BROMINE - BROMINE-82M 65PRR-013
A NEW ISOMER OF BROMINE - BROMINE-82M 65PRR-013
/CRIDE IN LANTHANUM CHLORIDE / SODIUM SUPEROXIDE / BARIUM BROMITE MONO HYDRATE / SODIUM HEXA CHLORO RHENA/ 65A-12
ANIUM - A COMPARISON OF / COMMENTS ON THE PAPER BY LAUER, BRUCK, AND LEDUIGOU, PRECISE DETERMINATION OF UR 65PRR-202
RANIUM - A COMPARISON OF/ COMMENTS ON THE PAPER BY LAUER, BRUCK, AND LEDUIGOU, PRECISE DETERMINATIONS OF U 65PRR-098
NATE - DI ETHYL BENZENE SOLUTIONS, DIELECTRIC CON/ DI SEC BUTYL PHENYL PHOSPHONATE IN DI SEC BUTYL PHOSPHO 65PRR-156
DIELECTRIC CON/ DI SEC BUTYL PHENYL PHOSPHONATE IN DI SEC BUTYL PHOSPHONATE - DI ETHYL BENZENE SOLUTIONS, 65PRR-156
SPHONIUM CHL/ QUATERNARY PHOSPHONIUM HALIDES (BENZYL TRI BUTYL PHOSPHONIUM BROMIDE / BENZYL TRI BUTYL PHO 65A-13-01A
LIDES (BENZYL TRI BUTYL PHOSPHONIUM BROMIDE / BENZYL TRI BUTYL PHOSPHONIUM CHLORIDE) /ARY PHOSPHONIUM HA 65A-13-01A
TALS USE OF METAL DI N BUTYL PHOSPHORO DI THIOATES AS EXTRACTANTS OF ME 65PRR-016
SPECTROPHOTOMETRIC DETERMINATION OF NITRATED 4 SEC BUTYL 2 (ALPHA METHYL BENZYL) PHENOL (BAMBP) 65A-05-04E
/APHY / POTENTIOSTATIC OC POLAROGRAPHY / DETERMINATION OF CADMIUM / DIFFERENTIAL POLAROGRAPHY / DIFFERENT/ 65A-01-03
DETERMINATION OF LEAD, BISMUTH, CADMIUM, TIN, AND ZINC IN AIRBORNE DUST SAMPLES 65A-07-02A
INTERACTIONS OF CADMIUM-106 WITH ALPHA PARTICLES 65PRR-179
AZO DYES (CALCICHROME / CHLORO PHOSPHONAZO-III) 65A-13-01F
COMPUTER PROGRAM FOR CALCULATING CORRELATION COEFFICIENTS 65A-11-06C
COMPUTER PROGRAM FOR CALCULATING NEUTRON FLUX 65A-11-06F
SOLUTIONS, MEASUREMENT OF PROMPT GAMMA-RAYS FROM SPONTAN/ CALIFORNIUM-252 IN TRANSURANIUM (TRU) PROCESS 65PRR-130
/AVE POLAROGRAPH WITH SOLID STATE CIRCUITS / DOUBLE LAYER CAPACITANCE MEASUREMENT / TEFLON DROPPING MERCU/ 65A-01-14
OS (S/N) OF GLASS AND TEFLON DROPPING MERCURY ELECTRODE CAPILLARIES SIGNAL TO NOISE RATI 65A-05-03B
CY ELECTRON OPTICAL STUDY OF THE EFFECT OF AGING ON CARBIDES IN A NIOBIUM - TUNGSTEN - ZIRCONIUM ALL 65PRR-210
DETERMINATION OF THE COATING DENSITY OF PYROLYTIC CARBON COATED NUCLEAR FUEL PARTICLES 65PRR-164
/ IMPURITIES OF MOISTURE, OXYGEN, HYDROGEN, NITROGEN, AND CARBON DI OXIDE IN GLOVE BOX ATMOSPHERES ON THE/ 65PRR-175
HE PYROLYTIC GRAPHITE ELECTRODE (P.G.E.) AND THE GLASSY CARBON ELECTRODE (G.C.E.) /L TITRIMETRY WITH T 65A-05-02C
A GLASSY CARBON ELECTRODE FOR VOLTAMMETRY 65PRR-061
YGEN IN ALKALI METAL/ ANALYSIS OF THE ALKALI METALS (FOR CARBON HYDROGEN AND OXYGEN / DETERMINATION OF OX 65A-04-03A
GLASSY CARBON INDICATOR ELECTRODE (G.C.E.) 65A-05-02A
CARBON-14 PRODUCTS 65A-11-02C
CARBON-14, LIQUID SCINTILLATION COUNTING METHOD 65PRR-131
CARBON-14, PRODUCT ANALYSIS GUIDE 65PRR-152
/ECTROGRAPHIC ANALYSIS (VISUAL INTERPOLATION) (LITHIUM CARBONATE DILUTION METHOD OF SPECTROGRAPHIC ANA/ 65A-07-01F
/Y AND TEMPERATURE DEPENDENCE OF DISSOCIATION CONSTANT OF CARBONIC ACID IN CONCENTRATED SODIUM CHLORIDE S/ 65TR-08
METHYL METHANE SULFONATE (CARCINOGEN) 65A-13-01D
CATALOG OF 14-MEV NEUTRON REACTIONS 65A-11-09A
DE SOLUTION - DETERMINATION OF FREE ACID AND HYDROLYZABLE CATION REACTIONS IN CONCENTRATED LITHIUM CHLORI 65PRR-022
CAUTION AND PRECAUTION IN FORENSIC APPLICATIONS 65PRR-186
/ALYTICAL DETERMINATION OF URANIUM / 0.5-ML POLAROGRAPHIC CELL / ELECTROCHEMISTRY / MODEL-Q-1988-FES POLA/ 65A-01-06
FOR THE MOLTEN SALT REACTOR EX/ ANALYTICAL LABORATORY HOT CELL EQUIPMENT I. SAMPLE PREPARATION APPARATUS 65PRR-027
ION RESISTANT PIPETTER ANALYTICAL LABORATORY HOT CELL EQUIPMENT II. A REMOTELY CONTROLLED CORROS 65PRR-028
PHOTOMETER ANALYTICAL LABORATORY HOT CELL EQUIPMENT III. A REMOTELY OPERATED FILTER 65PRR-026
/ ALIPAL-LO-43/ ABSORPTION SPECTRAL MEASUREMENTS (SOLAR CELL WINDOWS / RIBO NUCLEASE / BACILLUS-SUBTILIS 65A-05-04M
ATION OF TRACE METALS FROM URANIUM BY CHROMATOGRAPHY ON A CELLULOSE COLUMN SEPAR 65A-11-04E
ION OF TRACES OF METALS FROM URANIUM BY CHROMATOGRAPHY ON CELLULOSE COLUMN SEPARAT 65PRR-037
ICNS CERIUM RADIOACTIVITY IN AQUEOUS OR ORGANIC SOLUT 65PRR-141
GAMMA-RAY BRANCHINGS IN THE DECAY OF INDIUM-116, CERIUM-141, AND NEODYMIUM-147 65PRR-169
CERIUM-144 PRODUCT 65A-11-02D
/ANIC PREPARATIONS (MAGNESIUM CHLORIDE / ZINC CHLORIDE / CESIUM CHLORIDE / SODIUM OXIDE / POTASSIUM OXID/ 65A-12
PREPARATION AND RECOVERY OF CESIUM COMPOUNDS FOR DENSITY GRADIENT SOLUTIONS 65PRR-116
O STANNATE METHOD CESIUM RADIOACTIVITY IN AQUEOUS SOLUTIONS, CHLOR 65PRR-151
8 / IODINE-133 / PHOSPHORUS-33) HALF-LIFE MEASUREMENTS (CESIUM-137 / SAMARIUM-153 / ARSENIC-77 / GOLD-19 65A-11-03D
// ALIPAL-LO-436 / AMMONIUM SALT OF A SULFONATED STRAIGHT CHAIN ALKYL PHENOXY POLY (ETHYLENE OXY) ETHAN/ 65A-05-04M
L ANALYSIS, VOL. III. (THEORY OF COULOME/ PREPARATION OF CHAPTER FOR WELCHERS STANDARD METHODS OF CHEMICA 65A-01-10
F RADIO IODINE IDENTITY, CHARACTER AND CHEMICAL BEHAVIOR OF VAPOR FORMS O 65PRR-162
/OF ACTIVATION ANALYSIS IN PROCEDURES FOR THE REMOVAL AND CHARACTERIZATION OF THE SURFACE CONTAMINANTS OF/ 65PRR-008
REACTION OF METHYL IODIDE WITH CHEMICALLY IMPREGNATED CHARCOAL 65PRR-065
NTROLLED POTENTIAL DC POLAROGRAPH VOLTAMMETER CHECK OUT AND TEST PROCEDURE FOR MODEL-Q-2792 CO 65PRR-080
REVIEW OF THE SOLVENT EXTRACTION OF METAL CHELATES 65PRR-058
THE USE OF ISOTOPIC NEUTRON SOURCES FOR CHEMICAL ANALYSIS 65PRR-006
/ PREPARATION OF CHAPTER FOR WELCHERS STANDARD METHODS OF CHEMICAL ANALYSIS, VOL. III. (THEORY OF COULOME 65A-01-10
UND ON RUBBER SECTIONS OF COMPOSITE DIFFUSION TUBES CHEMICAL BEHAVIOR OF THE RADIO IODINE SPECIES FO 65PRR-069
IDENTITY, CHARACTER AND CHEMICAL BEHAVIOR OF VAPOR FORMS OF RADIO IODINE 65PRR-162
HIGH TEMPERATURE CHEMICAL DOSIMETRY 65A-03-03
ANALYTICAL SERVICES FOR CHEMICAL ENGINEERS 65PRR-182
VAPOR BY MEANS OF COMPOSITE DIFFUSION / INVESTIGATION OF CHEMICAL REACTIVITY OF NONELEMENTAL RADIO IODINE 65PRR-068
REACTION OF METHYL IODIDE WITH CHEMICALLY IMPREGNATED CHARCOAL 65PRR-065
APPLICATION OF RADIOISOTOPES TO ANALYTICAL CHEMISTRY 65A-11-07
SPECTROMETRY IN ANALYTICAL CHEMISTRY 65PRR-172
RADIO AND NUCLEAR CHEMISTRY 65PRR-177
MODERN ANALYTICAL CHEMISTRY 65PRR-209
STICAL QUALITY CONTROL REPORT, APR.-JUNE,1965, ANALYTICAL CHEMISTRY DIVISION STATI 65PRR-081
STICAL QUALITY CONTROL REPORT, JAN.-MAR.,1965, ANALYTICAL CHEMISTRY DIVISION STATI 65PRR-082
TICAL QUALITY CONTROL REPORT, JAN.-SEPT.,1964, ANALYTICAL CHEMISTRY DIVISION STATIS 65PRR-084
PROGRESS REPORT FOR PERIOD ENDING NOV.15,1964, ANALYTICAL CHEMISTRY DIVISION ANNUAL 65PRR-091
STATISTICAL QUALITY CONTROL REPORT, ANALYTICAL CHEMISTRY DIVISION, JAN.-DEC.,1964 65PRR-083
ANALYTICAL CHEMISTRY FOR REACTOR PROJECTS 65A-04
MANAGEMENT LOOKS AT ANALYTICAL CHEMISTRY IN A NATIONAL LABORATORY 65PRR-204
ANALYTICAL CHEMISTRY IN NUCLEAR TECHNOLOGY 65PRR-052
EIGHTH CONFERENCE ON ANALYTICAL CHEMISTRY IN NUCLEAR TECHNOLOGY 65PRR-053
A RESUME OF THE EIGHTH CONFERENCE ON ANALYTICAL CHEMISTRY IN NUCLEAR TECHNOLOGY 65PRR-054
THE NEW IMAGE OF ANALYTICAL CHEMISTRY IN SCIENCE 65PRR-221
TION ANALYSIS OF TECHNETIUM INORGANIC AND NUCLEAR CHEMISTRY OF TECHNETIUM - THERMAL NEUTRON ACTIVA 65PRR-077
REVIEW OF ANALYTICAL CHEMISTRY OF THALLIUM 65PRR-057
SUMMARY - NOV.,1964 / DEC.,1964 / JAN.,1965 / ANALYTICAL CHEMISTRY RESEARCH AND DEVELOPMENT GROUP MONTHLY 65PRR-104
/I CYANIDE / CHRONOPOTENTIOMETRIC REDUCTION OF FERRIC PER CHLORATE / CHRONOPOTENTIOMETRIC OXIDATION OF OX/ 65A-01-02
E OVERL/ THE ABSORPTION SPECTRUM OF THE URANYL ION IN PER CHLORATE MEDIA I. MATHEMATICAL RESOLUTION OF TH 65PRR-161
HIGH PURITY POTASSIUM CHLORIDE 65PRR-090
BUTYL PHOSPHONIUM BROMIDE / BENZYL TRI BUTYL PHOSPHONIUM CHLORIDE) /ARY PHOSPHONIUM HALIDES (BENZYL TRI 65A-13-01A

YDRATE / SODIUM HEXA CHLORO RHENATE(IV) / URANIUM-235 TRI CHLORIDE) /M SUPEROXIDE / BARIUM BROMITE MONO H 65A-12
SSIUM/ INORGANIC PREPARATIONS (MAGNESIUM CHLORIDE / ZINC CHLORIDE / CESIUM CHLORIDE / SODIUM OXIDE / POTA 65A-12
/EPARATIONS (MAGNESIUM CHLORIDE / ZINC CHLORIDE / CESIUM CHLORIDE / SODIUM OXIDE / POTASSIUM OXIDE / LIT/ 65A-12
/ / SOLID SOLUTIONS OF URANIUM(III) CHLORIDE IN LANTHANUM CHLORIDE / SODIUM SUPEROXIDE / BARIUM BROMITE M/ 65A-12
N IN AQUEOUS SOLUTIONS OF HYDROCHLORIC ACID AND POTASSIUM CHLORIDE / TO LEAD METAL REDOX REACTION REDUCTIO 65PRR-010
/OTASSIUM SUPEROXIDE / RUBIDIUM SUPEROXIDE / URANIUM(III) CHLORIDE / URANIUM(III) IODIDE / SOLID SOLUTION/ 65A-12
IUM OXIDE / POTASSIUM/ INORGANIC PREPARATIONS (MAGNESIUM CHLORIDE / ZINC CHLORIDE / CESIUM CHLORIDE / SOD 65A-12
FA RADIATION EFFECTS ON CONCENTRATED SOLUTIONS OF LITHIUM CHLORIDE CONTAINING HYDROCHLORIC ACID ALP 65PRR-165
/ / URANIUM(III) IODIDE / SOLID SOLUTIONS OF URANIUM(III) CHLORIDE IN LANTHANUM CHLORIDE / SODIUM SUPEROX/ 65A-12
ND HYDROLYZABLE CATION REACTIONS IN CONCENTRATED LITHIUM CHLORIDE SOLUTION - DETERMINATION OF FREE ACID A 65PRR-022
OCIATION CONSTANT OF CARBONIC ACID IN CONCENTRATED SODIUM CHLORIDE SOLUTION /EMPERATURE DEPENDENCE OF DISS 65TR-08
GAMMA RADIOLYSIS OF ACID CHLORIDE SOLUTIONS 65A-03-01
RATION METHOD CHLORIDE, DISTILLATION - INDIRECT IODOMETRIC TIT 65PRR-126
HALF-LIVES OF LONG-LIVED BETA EMITTERS (CHLORINE-36 BERYLLIUM-10) 65A-11-01D
AZO DYES (CALCICHROME / CHLORO PHOSPHONAZO-III) 65A-13-01F
// POTASSIUM OXIDE / LITHIUM HYPOBROMITE / POTASSIUM HEXA CHLORO RHENATE(IV) / HOLMIUM SULFIDE / ERBIUM S/ 65A-12
UM SUPEROXIDE / BARIUM BROMITE MONO HYDRATE / SODIUM HEXA CHLORO RHENATE(IV) / URANIUM-235 TRI CHLORIDE) 65A-12
CESIUM RADIOACTIVITY IN AQUEOUS SOLUTIONS, CHLORO STANNATE METHOD 65PRR-151
RONOPOTENTIOGRAMS CHRONOGRM - A COMPUTER PROGRAM FOR ANALYSIS OF CH 65PRR-097
NDPOTENTIOGRAMS (HEYROVSKY-ILKOVIC EQUATION / POLARGRM / CHRONOGRM / KARADGLANOFF EQUATION) /AMS AND CHRO 65A-01-13
TURES OF METHANOL AND HYDROCHLORIC ACID GAS CHROMATOGRAPHIC ANALYSIS OF GAMMA IRRADIATED MIX 65A-03-02
REACTION OF IODINE WITH CYCLOHEXANE AND WITH BENZENE/ GAS CHROMATOGRAPHIC ANALYSIS OF THE PRODUCTS OF THE 65A-04-04C
IN HELIUM AT REDUCED SAMPLE PRESSURES GAS CHROMATOGRAPHIC DETERMINATION OF PERMANENT GASES 65PRR-007
CTRIC FILM PYROLYSIS AND GAS CHROMATOGRAPHIC DETERMINATION OF STYROFOAM DIELE 65A-05-01B
APORS GAS CHROMATOGRAPHIC IDENTIFICATION OF RADIO IODINE V 65PRR-062
APORS GAS CHROMATOGRAPHIC IDENTIFICATION OF RADIO IODINE V 65PRR-064
GAS CHROMATOGRAPHY 65A-05-01
GAS CHROMATOGRAPHY 65PRR-200
NUCLEAR SAFETY PROGRAM (IODINE / METHYL IODIDE / GAS CHROMATOGRAPHY) 65A-04-04A
APPLICATIONS OF GAS CHROMATOGRAPHY IN NUCLEAR TECHNOLOGY 65PRR-180
AND PYRIMIDINES GAS CHROMATOGRAPHY OF PYROLYTIC PRODUCTS OF PURINES 65A-06-03
SYSTEM RADIO GAS CHROMATOGRAPHY OF THE TRITIUM - WATER - METHANE 65A-05-01A
SEPARATION OF TRACE METALS FROM URANIUM BY CHROMATOGRAPHY ON A CELLULOSE COLUMN 65A-11-04E
SEPARATION OF TRACES OF METALS FROM URANIUM BY CHROMATOGRAPHY ON CELLULOSE COLUMN 65PRR-037
ATE TITRATION METHOD CHROMIUM IN MSRE FUEL, AMPEROMETRIC FERROUS SULF 65PRR-136
/HIC ANALYSIS OF RADIOACTIVE MATERIALS (DETERMINATION OF CHROMIUM IN RADIOACTIVE MSRE SALT / SEPARATION / 65A-07-01C
SSEMBLY FOR GENERATION, STORAGE, AND DELIVERY OF STANDARD CHROMIUM(II) SOLUTIONS INTEGRATED A 65PRR-055
VOLTAMMETRY / SUBTRACTIVE POLAROGRAPHY / POTENTIAL-SWEEP CHRONOAMPEROMETRY) /L VOLTAMMETRY / COMPARATIVE 65A-01-03
/PH VOLTAMMETER, ORNL MODEL-Q-2792 (CYCLIC VOLTAMMETRY / CHRONOAMPEROMETRY / HAMMER-CONTROL OF DROPPING / 65A-01-04
CHRONOGRM - A COMPUTER PROGRAM FOR ANALYSIS OF CHRONOPOTENTIOGRAMS 65PRR-097
/ POL/ COMPUTER PROGRAMS FOR ANALYSIS OF POLAROGRAMS AND CHRONOPOTENTIOGRAMS (HEYROVSKY-ILKOVIC EQUATION 65A-01-13
EOUS CURRENTS (VOLTAMMETRY AT CONTROLLED CURRENT) CHRONOPOTENTIOMETER WITH COMPENSATION FOR EXTRAN 65A-01-01
EOUS CURRENTS CHRONOPOTENTIOMETER WITH COMPENSATION FOR EXTRAN 65PRR-051
/LORATE / CHRONOPOTENTIOMETRIC OXIDATION OF OXALIC ACID / CHRONOPOTENTIOMETRIC OXIDATION OF FERROUS AMMON/ 65A-01-02
/ CHRONOPOTENTIOMETRIC REDUCTION OF FERRIC PER CHLORATE / CHRONOPOTENTIOMETRIC OXIDATION OF OXALIC ACID // 65A-01-02
/CNOPOTENTIOMETRIC REDUCTION OF POTASSIUM FERRI CYANIDE / CHRONOPOTENTIOMETRIC REDUCTION OF FERRIC PER CH/ 65A-01-02
/RANEOUS CURRENTS (VOLTAMMETRY WITH CONTROLLED CURRENT / CHRONOPOTENTIOMETRIC REDUCTION OF POTASSIUM FER/ 65A-01-02
D CURRENT / DERIVATIVE VOLTAMMETRY WITH CONTR/ DERIVATIVE CHRONOPOTENTIOMETRY (VOLTAMMETRY WITH CONTROLLE 65A-01-09
CHRONOPOTENTIOMETRY OF THE IODINE SYSTEM 65A-05-02E
EOUS CURRENTS (VOLTAMMETRY W/ EVALUATION OF TECHNIQUE OF CHRONOPOTENTIOMETRY WITH COMPENSATION FOR EXTRAN 65A-01-02
CNOPOTENTIOMETRIC OXIDATION OF FERROUS AMMONIUM SULFATE / CHRONOPOTENTIOMETRY WITH PLATINUM ELECTRODES) / 65A-01-02
(DERIVATIVE COMPUTER / INSTRUMENT DESI/ DIFFERENTIATING CIRCUIT FOR THE CONTROLLED POTENTIAL VOLTAMMETER 65A-01-11
TRANSIENT PROCESSES WITH POTENTIOSTATIC REGULATING CIRCUITS 65TR-03
/(DESIGN OF NEW SQUARE-WAVE POLAROGRAPH WITH SOLID STATE CIRCUITS / DOUBLE LAYER CAPACITANCE MEASUREMENT/ 65A-01-14
AT THE INNER RESISTANCE OF POTENTIOSTATIC REGULATING CIRCUITS AND EXAMPLES OF THE USE OF A POTENTIOST 65TR-07
DETERMINATION OF THE COATING DENSITY OF PYROLYTIC CARBON COATED NUCLEAR FUEL PARTICLES 65PRR-164
AR FUEL PARTICLES DETERMINATION OF THE COATING DENSITY OF PYROLYTIC CARBON COATED NUCLE 65PRR-164
/ PROBE ANALYZER / DETERMINATION OF AREAL DENSITY OF GOLD COATING ON SILICON RADIATION DETECTORS BY USE O/ 65A-07-01A
SOLVENT EXTRACTION OF BERYLLIUM, COPPER, NICKEL, COBALT, AND ZINC AS DI BENZOYL METHANE COMPLEXES 65TR-10
F URANIUM(VI) IN PHOSPHORIC ACID SOLUTIONS OF / EFFECT OF COBALT-60 GAMMA RADIATION ON THE DETERMINATION O 65PRR-021
TICAL METHODS / REMOTELY/ SAMPLE IRRADIATION ASSEMBLY FOR COBALT-60 SOURCE (EFFECTS OF RADIATION ON ANALY 65A-01-12
MODIFICATIONS TO THE COBALT-60 SOURCE ASSEMBLY 65A-03-04
RIDE - POTASSIUM FLUORIDE ABSORPTION SPECTRUM OF COBALT(II) ION IN LITHIUM FLUORIDE - SODIUM FLUO 65A-02-01B
COMPUTER PROGRAM FOR CALCULATING CORRELATION COEFFICIENTS 65A-11-06C
FLAME PHOTOMETRY (COLLECTION OF BARIUM FROM TAP WATER) 65A-07-01D
YSTEMS BY USE OF AN ISOLATED INTERNAL STANDARD COLOR QUENCH CORRECTION IN LIQUID SCINTILLATOR S 65A-11-05B
YSTEMS USING AN ISOLATED INTERNAL STANDARD COLOR QUENCH CORRECTION IN LIQUID SCINTILLATOR S 65PRR-047
RACE METALS FROM URANIUM BY CHROMATOGRAPHY ON A CELLULOSE COLUMN SEPARATION OF T 65A-11-04E
CES OF METALS FROM URANIUM BY CHROMATOGRAPHY ON CELLULOSE COLUMN SEPARATION OF TRA 65PRR-037
AROGRAPHIC CURRENT IN RELATION TO THE HEIGHT OF A MERCURY COLUMN /HOD OF DETERMINING THE NATURE OF THE POL 65TR-11
HALF-LIVES AND SPECIFIC ACTIVITIES OF COMMON RADIONUCLIDES 65PRR-078
CHRONOPOTENTIOMETER WITH COMPENSATION FOR EXTRANEOUS CURRENTS 65PRR-051
RY AT CONTROLLED CURRENT) CHRONOPOTENTIOMETER WITH COMPENSATION FOR EXTRANEOUS CURRENTS (VOLTAMMET 65A-01-01
RY W/ EVALUATION OF TECHNIQUE OF CHRONOPOTENTIOMETRY WITH COMPENSATION FOR EXTRANEOUS CURRENTS (VOLTAMMET 65A-01-02
/RDING CONDUCTOMETRIC TITRATOR WITH AUTOMATIC TEMPERATURE COMPENSATION OR PROPORTIONAL TEMPERATURE CONTRO/ 65PRR-036
EQUILIBRIUM DISTRIBUTION OF METAL - ION COMPLEXES 65PRR-074
M, COPPER, NICKEL, COBALT, AND ZINC AS DI BENZOYL METHANE COMPLEXES SOLVENT EXTRACTION OF BERYLLIU 65TR-10
NVESTIGATION OF AIRBORNE RADIO IODINE SPECIES BY MEANS OF COMPOSITE DIFFUSION TUBES - I 65PRR-067
R OF THE RADIO IODINE SPECIES FOUND ON RUBBER SECTIONS OF COMPOSITE DIFFUSION TUBES CHEMICAL BEHAVIO 65PRR-069
BEHAVIOR UNDER MOIST CONDITIONS IN MAY PACKS BY MEANS OF COMPOSITE DIFFUSION TUBES /ATION OF RADIO IODINE 65PRR-066
REACTIVITY OF NONELEMENTAL RADIO IODINE VAPOR BY MEANS OF COMPOSITE DIFFUSION TUBES /TIGATION OF CHEMICAL 65PRR-068
THE ISOTOPIC COMPOSITION OF ATMOSPHERIC NEON 65PRR-071
THE ISOTOPIC COMPOSITION OF ATMOSPHERIC NEON 65PRR-208
/IT FOR THE CONTROLLED POTENTIAL VOLTAMMETER (DERIVATIVE COMPUTER / INSTRUMENT DESIGN / ELECTROCHEMISTRY/ 65A-01-11
RAMS CHRONOGRM - A COMPUTER PROGRAM FOR ANALYSIS OF CHRONOPOTENTIOG 65PRR-097
POLARGRM - A COMPUTER PROGRAM FOR ANALYSIS OF POLAROGRAMS 65PRR-096
FFICIENTS COMPUTER PROGRAM FOR CALCULATING CORRELATION COE 65A-11-06C
COMPUTER PROGRAM FOR CALCULATING NEUTRON FLUX 65A-11-06F
D CHRONOPOTENTIOGRAMS (HEYROVSKY-ILKOVIC EQUATION / POL/ COMPUTER PROGRAMS FOR ANALYSIS OF POLAROGRAMS AN 65A-01-13
ACTIVATION ANALYSIS EVALUATION OF COMPUTER PROGRAMS FOR GAMMA-RAY SPECTROMETRY IN 65A-11-06B
ACTIVATION ANALYSIS THE EVALUATION OF COMPUTER PROGRAMS FOR GAMMA-RAY SPECTROMETRY IN 65PRR-171
APPLICATION OF COMPUTERS 65A-11-06

USE OF COMPUTERS IN APPLIED ACTIVATION ANALYSIS 65A-11-06D
LAROGRAPHY / POLAROGRAPHIC DETECTION LIMITS FOR URANIUM / CONCENTRATION ADVANTAGE / SEAWATER) /LOSS IN PO 65A-01-06
OF A RADIOISOTOPIC LIGHT SOURCE A NEW CONCEPT IN PRECISION PHOTOMETRIC ANALYSIS - USE 65A-11-07A
RE C/ HIGH SENSITIVITY, DIRECT READING, LINEAR, RECORDING CONDUCTOMETRIC TITRATOR WITH AUTOMATIC TEMPERATU 65PRR-036
CHNOLOGY EIGHTH CONFERENCE ON ANALYTICAL CHEMISTRY IN NUCLEAR TE 65PRR-053
CHNOLOGY A RESUME OF THE EIGHTH CONFERENCE ON ANALYTICAL CHEMISTRY IN NUCLEAR TE 65PRR-054
MIUM IN RADIOACTIVE MSRE SALT / SEPARATION OF CURIUM FROM CONTAMINANT METALS) /LS (DETERMINATION OF CHRO 65A-07-01C
CURES FOR THE REMOVAL AND CHARACTERIZATION OF THE SURFACE CONTAMINANTS OF HAIR /TIVATION ANALYSIS IN PROCE 65PRR-008
ISOTOPIC COMPARISON OF LEAD FROM TEKTITES AND CONTEMPORANEOUS IMPACT CRATERS 65A-08-04
QUALITY CONTROL 65B-14
ATIC TEMPERATURE COMPENSATION OR PROPORTIONAL TEMPERATURE CONTROL /DING CONDUCTOMETRIC TITRATOR WITH AUTOM 65PRR-036
ALKYL BENZENE SULFONATE (ABS) CONTROL FOR THE FOAM SEPARATION PROCESS 65PRR-183
AN.-DEC.,1964 STATISTICAL QUALITY CONTROL REPORT, ANALYTICAL CHEMISTRY DIVISION, J 65PRR-083
STRY DIVISION STATISTICAL QUALITY CONTROL REPORT, APR.-JUNE,1965, ANALYTICAL CHEMI 65PRR-081
STRY DIVISION STATISTICAL QUALITY CONTROL REPORT, JAN.-MAR.,1965, ANALYTICAL CHEMI 65PRR-082
ISTRY DIVISION STATISTICAL QUALITY CONTROL REPORT, JAN.-SEPT.,1964, ANALYTICAL CHEM 65PRR-084
ANALYTICAL LABORATORY HOT CELL EQUIPMENT II. A REMOTELY CONTROLLED CORROSION RESISTANT PIPETTER 65PRR-028
OEL-Q-1348B REMOTELY SERVO CONTROLLED CORROSION RESISTANT PIPETTER, ORNL MO 65A-04-01B
ITH COMPENSATION FOR EXTRANEOUS CURRENTS (VOLTAMMETRY AT CONTROLLED CURRENT) CHRONOPOTENTIOMETER W 65A-01-01
TRY WITH CONTROLLED CURRENT / DERIVATIVE VOLTAMMETRY WITH CONTROLLED CURRENT) /NOPOTENTIOMETRY (VOLTAMME 65A-01-09
/ COMPENSATION FOR EXTRANEOUS CURRENTS (VOLTAMMETRY WITH CONTROLLED CURRENT / CHRONOPOTENTIOMETRIC REDUC/ 65A-01-02
CONTR/ DERIVATIVE CHRONOPOTENTIOMETRY (VOLTAMMETRY WITH CONTROLLED CURRENT / DERIVATIVE VOLTAMMETRY WITH 65A-01-09
E (EFFECTS OF RADIATION ON ANALYTICAL METHODS / REMOTELY CONTROLLED INSTRUMENT) /BLY FOR COBALT-60 SOURC 65A-01-12
ATIC COULOMETRIC TITRATOR, ORNL MODEL-Q-2005, ELECTRONIC, CONTROLLED POTENTIAL AUTOM 65PRR-146
/COULOMETRIC TITRATOR, ORNL MODEL-Q-2564 (POTENTIOSTAT / CONTROLLED POTENTIAL / BLANK CORRECTIONS IN CON/ 65A-01-05
MODEL-Q-1988A CONTROLLED POTENTIAL AND DERIVATIVE POLAROGRAPH, 65PRR-117
/ SENSITIVITY CONTROLLED POTENTIAL COULOMETRIC TITRATOR - CONTROLLED POTENTIAL COULOMETRIC DETERMINATION / 65PRR-020
DETERMINATION OF NITRATE BY CONTROLLED POTENTIAL COULOMETRIC TITRATION 65A-05-04J
/4 COULOMETRIC TITRATOR / I-VERSUS-Q COULOMETRIC METHOD / CONTROLLED POTENTIAL COULOMETRIC TITRATION / BL/ 65A-01-07
/R EXPERIMENT (MSRE) FUEL, HIGH SENSITIVITY, AUTOMATIC, CONTROLLED POTENTIAL COULOMETRIC TITRATION METH/ 65PRR-122
/IUM(VI) RATIO IN THORIUM OXIDE - URANIUM OXIDE MIXTURES, CONTROLLED POTENTIAL COULOMETRIC TITRATION METH/ 65PRR-155
ANIUM(VI) IN A SYNTHETIC MSRE FUEL CONTROLLED POTENTIAL COULOMETRIC TITRATION OF UR 65A-04-01D
ANIUM(VI) IN AQUEOUS SOLUTIONS OF SODIUM FLUORIDE CONTROLLED POTENTIAL COULOMETRIC TITRATION OF UR 65PRR-034
ROLLED POTENTIAL COULOMETRIC DETERMINAT/ HIGH SENSITIVITY CONTROLLED POTENTIAL COULOMETRIC TITRATOR - CONT 65PRR-020
OTENTIOSTAT / CONTROLLED POTENTIAL / BLANK CORRECTIONS IN CONTROLLED POTENTIAL COULOMETRY) /EL-Q-2564 (P 65A-01-05
TERMINATION OF PLUTONIUM) UTILITY OF I-VERSUS-Q PLOTS IN CONTROLLED POTENTIAL COULOMETRY (COULOMETRIC DE 65A-01-08
/DERIVATIVE DC POLAROGRAPHY / DETERMINATION OF URANIUM BY CONTROLLED POTENTIAL COULOMETRY / DETERMINATION/ 65A-01-07
/LED POTENTIAL COULOMETRY / DETERMINATION OF PLUTONIUM BY CONTROLLED POTENTIAL COULOMETRY / DETERMINATION/ 65A-01-07
/NTROLLED POTENTIAL COULOMETRY / DETERMINATION OF IRON BY CONTROLLED POTENTIAL COULOMETRY / MODEL-Q-2564 / 65A-01-07
CAL / CONTROLLED POTENTIAL DERIVATIVE DC POLAROGRAPHY AND CONTROLLED POTENTIAL COULOMETRY AS MICRO ANALYTI 65A-01-07
/GH ACCURACY METHOD (ELECTROCHEMISTRY / MODEL-Q-1988-FES CONTROLLED POTENTIAL DC POLAROGRAPH / CONTROLLE/ 65A-01-03
CHECK OUT AND TEST PROCEDURE FOR MODEL-Q-2792 CONTROLLED POTENTIAL DC POLAROGRAPH VOLTAMMETER 65PRR-080
ORNL MODEL-Q-2792 (CYCLIC VOLTAMMETRY / CHRONOAMPEROME/ CONTROLLED POTENTIAL DC POLAROGRAPH VOLTAMMETER, 65A-01-04
// MODEL-Q-1988-FES CONTROLLED POTENTIAL DC POLAROGRAPH / CONTROLLED POTENTIAL DC POLAROGRAPHY / POTENTIO/ 65A-01-03
/ECTIONS IN COULOMETRY / POTENTIOSTATIC DC POLAROGRAPHY / CONTROLLED POTENTIAL DC POLAROGRAPHY / POTENTIO/ 65A-01-07
TYL PHOS/ DETERMINATION OF TRACE QUANTITIES OF URANIUM BY CONTROLLED POTENTIAL DC POLAROGRAPHY IN A TRI OC 65PRR-160
- APPLICATION TO DETERMINATION OF MICROGRAM QUANTITIES O/ CONTROLLED POTENTIAL DERIVATIVE DC POLAROGRAPHY 65A-01-06
AND CONTROLLED POTENTIAL COULOMETRY AS MICRO ANALYTICAL / CONTROLLED POTENTIAL DERIVATIVE DC POLAROGRAPHY 65A-01-07
Y (COMPARATIVE POLAROGRAPHY / DELTA-E-DIFFERENTIAL POLA/ CONTROLLED POTENTIAL DIFFERENTIAL DC POLAROGRAPH 65A-01-15
AS MICRO ANALYTICAL TECHNIQUES CONTROLLED POTENTIAL POLAROGRAPHY AND COULOMETRY 65PRR-181
MPUTER / INSTRUMENT DESI/ DIFFERENTIATING CIRCUIT FOR THE CONTROLLED POTENTIAL VOLTAMMETER (DERIVATIVE CO 65A-01-11
/MOLTEN SALT REACTOR EXPERIMENT (MSRE) FUEL, AUTOMATIC, CONTROLLED POTENTIAL, COULOMETRIC TITRATION MET/ 65PRR-121
EL-Q-2564 COULOMETRIC TITRATOR, CONTROLLED POTENTIAL, HIGH SENSITIVITY, ORNL MOD 65PRR-125
48A, MODEL-Q-1348B, AND MODEL-/ PIPETTERS, REMOTELY SERVO CONTROLLED, CORROSION RESISTANT, ORNL MODEL-Q-13 65PRR-124
ARBITRARY-SET, PROPORTIONAL TEMPERATURE CONTROLLER 65PRR-035
COOPERATIVE ISOTOPES PROGRAM 65A-11-02
ATOMIC ABSORPTION (DETERMINATION OF GOLD SILVER AND COPPER) 65A-07-01B
ETHANE COMPLEXES SOLVENT EXTRACTION OF BERYLLIUM, COPPER, NICKEL, COBALT, AND ZINC AS DI BENZOYL M 65TR-10
CNS - SELECTIVE EXTRACTION OF MERCURY(II), SILVER(I), AND COPPER(I) /TRI AMIDES AS EXTRACTANTS FOR METAL I 65PRR-015
OF AN ISOLATED INTERNAL STANDARD COLOR QUENCH CORRECTION IN LIQUID SCINTILLATOR SYSTEMS BY USE 65A-11-05B
AN ISOLATED INTERNAL STANDARD COLOR QUENCH CORRECTION IN LIQUID SCINTILLATOR SYSTEMS USING 65PRR-047
/DEL-Q-2564 (POTENTIOSTAT / CONTROLLED POTENTIAL / BLANK CORRECTIONS IN CONTROLLED POTENTIAL COULOMETRY / 65A-01-05
/MOD / CONTROLLED POTENTIAL COULOMETRIC TITRATION / BLANK CORRECTIONS IN COULOMETRY / POTENTIOSTATIC DC P/ 65A-01-07
INSTRUMENTATION FOR POLAROGRAPHY OF GLASS CORRODING MEDIA 65PRR-195
/DE FOR POLAROGRAPHY IN HYDROFLUORIC ACID AND OTHER GLASS CORRODING MEDIA - EVALUATION WITH MONOVALENT TH/ 65PRR-039
/CN FOR POLAROGRAPHY IN HYDROFLUORIC ACID AND OTHER GLASS CORRODING MEDIA - EVALUATION WITH THE DIVALENT / 65PRR-040
RIFICE/ POLAROGRAPHY IN HYDROFLUORIC ACID AND OTHER GLASS CORRODING MEDIA (EVALUATION OF THE HORIZONTAL O 65A-05-03A
LABORATORY HOT CELL EQUIPMENT II. A REMOTELY CONTROLLED CORROSION RESISTANT PIPETTER ANALYTICAL 65PRR-028
REMOTELY SERVO CONTROLLED CORROSION RESISTANT PIPETTER, ORNL MODEL-Q-1348B 65A-04-01B
-1348B, AND MODEL-/ PIPETTERS, REMOTELY SERVO CONTROLLED, CORROSION RESISTANT, ORNL MODEL-Q-1348A, MODEL-Q 65PRR-124
/EO POTENTIAL COULOMETRIC TITRATOR - CONTROLLED POTENTIAL COULOMETRIC DETERMINATION OF MILLIGRAM AND MICR/ 65PRR-020
OF I-VERSUS-Q PLOTS IN CONTROLLED POTENTIAL COULOMETRY (COULOMETRIC DETERMINATION OF PLUTONIUM) UTILITY 65A-01-08
/LOMETRY / MODEL-Q-2564 COULOMETRIC TITRATOR / I-VERSUS-Q COULOMETRIC METHOD / CONTROLLED POTENTIAL COULO/ 65A-01-07
VOL. III. (THEORY OF COULOMETRIC METHODS / TECHNIQUE OF COULOMETRIC METHODS / COULOMETRY) /AL ANALYSIS, 65A-01-10
/DARD METHODS OF CHEMICAL ANALYSIS, VOL. III. (THEORY OF COULOMETRIC METHODS / TECHNIQUE OF COULOMETRIC / 65A-01-10
DETERMINATION OF NITRATE BY CONTROLLED POTENTIAL COULOMETRIC TITRATION 65A-05-04J
/R / I-VERSUS-Q COULOMETRIC METHOD / CONTROLLED POTENTIAL COULOMETRIC TITRATION / BLANK CORRECTIONS IN CO/ 65A-01-07
) FUEL, HIGH SENSITIVITY, AUTOMATIC, CONTROLLED POTENTIAL COULOMETRIC TITRATION METHOD /EXPERIMENT (MSRE 65PRR-122
RIUM OXIDE - URANIUM OXIDE MIXTURES, CONTROLLED POTENTIAL COULOMETRIC TITRATION METHOD /M(VI) RATIO IN THO 65PRR-155
XPERIMENT (MSRE) FUEL, AUTOMATIC, CONTROLLED POTENTIAL, COULOMETRIC TITRATION METHOD /TEN SALT REACTOR E 65PRR-121
TIC MSRE FUEL CONTROLLED POTENTIAL COULOMETRIC TITRATION OF URANIUM(VI) IN A SYNTHE 65A-04-01D
SOLUTIONS OF SODIUM FLUORIDE CONTROLLED POTENTIAL COULOMETRIC TITRATION OF URANIUM(VI) IN AQUEOUS 65PRR-034
CMETRIC DETERMINAT/ HIGH SENSITIVITY CONTROLLED POTENTIAL COULOMETRIC TITRATOR - CONTROLLED POTENTIAL COUL 65PRR-020
/F IRON BY CONTROLLED POTENTIAL COULOMETRY / MODEL-Q-2564 COULOMETRIC TITRATOR / I-VERSUS-Q COULOMETRIC M/ 65A-01-07
HIGH-SENSITIVITY COULOMETRIC TITRATOR MODEL-Q-2564 65PRR-118
SENSITIVITY, ORNL MODEL-Q-2564 COULOMETRIC TITRATOR, CONTROLLED POTENTIAL, HIGH 65PRR-125
NIC, CONTROLLED POTENTIAL AUTOMATIC COULOMETRIC TITRATOR, ORNL MODEL-Q-2005, ELECTRO 65PRR-146
IOSTAT / CONTROLLED POTENTIAL / BLANK C/ HIGH SENSITIVITY COULOMETRIC TITRATOR, ORNL MODEL-Q-2564 (POTENT 65A-01-05
COULOMETRIC METHODS / TECHNIQUE OF COULOMETRIC METHODS / COULOMETRY) /AL ANALYSIS, VOL. III. (THEORY OF 65A-01-10
LED POTENTIAL / BLANK CORRECTIONS IN CONTROLLED POTENTIAL COULOMETRY) /EL-Q-2564 (POTENTIOSTAT / CONTROL 65A-01-05
IUM) UTILITY OF I-VERSUS-Q PLOTS IN CONTROLLED POTENTIAL COULOMETRY (COULOMETRIC DETERMINATION OF PLUTON 65A-01-08
/TRY / DETERMINATION OF PLUTONIUM BY CONTROLLED POTENTIAL COULOMETRY / DETERMINATION OF IRON BY CONTROLLE/ 65A-01-07

/RAPHY / DETERMINATICN OF URANIUM BY CONTRCLLED POTENTIAL COULCMETRY / DETERMINATION OF PLUTONIUM BY CONT/ 65A-01-07
Y / CONTROLLED POTENTIAL DC POLARCGRAPHY / POTENTIOSTATIC COULCMETRY / ELECTROCHEMISTRY) / DC POLAROGRAPH 65A-01-07
/ULOMETRY / DETERMINATICN CF IRON BY CCNTRCLLED POTENTIAL COULCMETRY / MCCEL-Q-2564 COULOMETRIC TITRATOR / 65A-01-07
/C POTENTIAL COULCMETRIC TITRATICN / BLANK CCRRECTICNS IN COULCMETRY / PCTENTIOSTATIC DC POLAROGRAPHY / C/ 65A-01-07
CCNTROLLED POTENTIAL PCLARCGRAPHY AND COULCMETRY AS MICRO ANALYTICAL TECHNIQUES 65PRR-181
/TIAL DERIVATIVE CC POLAROGRAPHY ANO CONTRCLLED POTENTIAL COULCMETRY AS MICRO ANALYTICAL TECHNIQUES (DET/ 65A-01-07
ALPHA PROPORTICNAL COUNTER 65PRR-147
CTION OF NATURAL RADICNUCLIDES BY WELL TYPE SCINTILLATION CCUNTERS DETE 65A-11-038
PREPARATION BY ELECTRODEPCSITION OF MOUNTS FOR 4 PI BETA COUNTING 65A-11-03A
REVIEW OF THE THECRY AND PRACTICE OF SCINTILLATION COUNTING 65PRR-025
CARBON-14, LIQUID SCINTILLATION COUNTING METHOC 65PRR-131
OCESS SOLUTICNS, 8 HYCROXY QUINOLINE PRECIPITATICN - BETA COUNTING METHOC /VITY IN TRANSURANIUM (TRU) PR 65PRR-129
ABSOLUTE LIQUID SCINTILLATION COUNTING OF BETA EMITTERS 65PRR-014
ABSCLUTE COUNTING OF GAMMA ACTIVITY 65PRR-188
WEAR RATES IN AUTOMCTIVE ENGINES BY LIQUID SCINTILLATION COUNTING OF IRCN-55 65PRR-048
WEAR RATES IN AUTOMCTIVE ENGINES BY LIQUID SCINTILLATION COUNTING OF IRCN-55 MEASUREMENT OF 65A-11-07C
MPARISCN OF LEAD FRCM TEKTITES ANC CCNTEMPORANEOUS IMPACT CRATERS ISCTOPIC CO 65A-08-04
RUBIDIUM-85 (N,2N) CRCSS SECTICN 65A-11-02F
SULFUR-33 (N,P) CRCSS SECTICN 65A-11-03C
/V NEUTRON GENERATORS - RAPID ESTIMATICN AND INFLUENCE IN CRCSS SECTICN MEASUREMENTS AND ACTIVATICN ANALY/ 65PRR-043
TATICN FUNCTICNS FOR HELIUM-3 INDUCED NUCLEAR REACTICNS / CRCSS SECTIONS) / ANALYSIS / SENSITIVITY / EXCI 65A-11-08A
TRONS WITH TECHNETIUM-99 ACTIVATION CRCSS SECTICNS FOR THE REACTICNS OF 14.8-MEV NEU 65PRR-075
CNS WITH TECHNETIUM-99 ACTIVATION CRCSS SECTICNS FOR THE REACTIONS OF 14-MEV NEUTR 65A-11-09E
ABSORBANCE SPECTRA OF SINGLE CRYSTALS 65PRR-214
MANUAL CUMULATIVE INDEXES TO THE ORNL MASTER ANALYTICAL 65C-18-01
ZIRCCNIUM IN MSRE FUEL, AMPERCMETRIC CUPFERRON TITRATION METHOD 65PRR-135
BCRCN, SPECTROPHCTOMETRIC CURCUMIN METHOC 65PRR-157
TION OF CHROMIUM IN RADICACTIVE MSRE SALT / SEPARATICN OF CURIUM FROM CONTAMINANT METALS) /LS (DETERMINA 65A-07-01C
ATION FOR EXTRANEOUS CURRENTS (VOLTAMMETRY AT CCNTROLLED CURRENT) CHRONOPOTENTIOMETER WITH COMPENS 65A-01-01
NTROLLED CURRENT / DERIVATIVE VOLTAMMETRY WITH CCNTROLLED CURRENT) /NOPCTENTIOMETRY (VOLTAMMETRY WITH CO 65A-01-09
/CN FOR EXTRANEOUS CURRENTS (VOLTAMMETRY WITH CCNTROLLED CURRENT / CHRCNCPOTENTIOMETRIC REDUCTICN OF PCT/ 65A-01-02
/VATIVE CHRONOPOTENTICMETRY (VOLTAMMETRY WITH CCNTROLLED CURRENT / DERIVATIVE VOLTAMMETRY WITH CONTROLLE/ 65A-01-09
/HE METHOD OF DETERMINING THE NATURE OF THE POLAROGRAPHIC CURRENT IN RELATION TO THE HEIGHT OF A MERCURY / 65TR-11
CHRONOPOTENTICMETER WITH COMPENSATICN FOR EXTRANECUS CURRENTS 65PRR-051
CHRONOPOTENTICMETER WITH COMPENSATICN FCR EXTRANEOUS CURRENTS (VOLTAMMETRY AT CONTROLLED CURRENT) 65A-01-01
/ OF CHRONOPOTENTICMETRY WITH COMPENSATICN FOR EXTRANECUS CURRENTS (VOLTAMMETRY WITH CONTROLLED CURRENT / 65A-01-02
MASS FISSICN YIELD CURVE FOR AMERICIUM-241 65PRR-046
MASS FISSICN YIELD CURVE FOR NEPTUNIUM-237 65PRR-094
OF THE RECORDING ARRANGEMENT ON THE FORM OF POLAROGRAPHIC CURVES INFLUENCE 65TR-01
/RENT / CHRONOPOTENTICMETRIC REDUCTICN OF POTASSIUM FERRI CYANIDE / CHRCNCPOTENTIOMETRIC REDUCTICN OF FER/ 65A-01-02
/OTENTIAL DC POLAROGRAPH VOLTAMMETER, ORNL MODEL-Q-2792 (CYCLIC VOLTAMMETRY / CHRONOAMPEROMETRY / HAMMER/ 65A-01-04
SPECTROPHOTOMETRIC STUDY OF THE STABILITY OF IODINE IN CYCLOHEXANE ANC IN BENZENE 65A-04-048
/ ANALYSIS OF THE PRODUCTS OF THE REACTICN OF IODINE WITH CYCLCHEXANE ANC WITH BENZENE AT 300C ANC 20 ATM/ 65A-04-04C
2,6 DI OXIMINC CYCLCHEXANONE 65A-13-018
STUCIES WITH THE TEFLCN DROPPING MERCURY ELECTRODE (D.M.E.) 65A-05-03
/ERCURY ELECTRODE FOR OBTAINING FUNDAMENTAL POLAROGRAPHIC DATA / MONOVALENT THALLIUM TO THALLIUM METAL RE/ 65A-05-03A
PPLICATION A PROGRAMMABLE HIGH SPEED DATA ACQUISITICN SYSTEM FOR MASS SPECTRCMETRIC A 65PRR-192
/ELECTROCHEMISTRY / MODEL-Q-1988-FES CONTRCLLED POTENTIAL DC PCLAROGRAPH / CONTROLLEC POTENTIAL DC POLARO/ 65A-01-03
AND TEST PROCEDURE FOR MODEL-Q-2792 CONTRCLLED POTENTIAL DC FCLAROGRAPH VOLTAMMETER CHECK OUT 65PRR-080
CYCLIC VOLTAMMETRY / CHRCNOAMPERCME/ CONTRCLLED POTENTIAL OC POLAROGRAPH VOLTAMMETER, ORNL MODEL-C-2792 (65A-01-04
ACCURACY METHOD (ELECTROCHEMIS/ COMPARATIVE AMPEROMETRIC OC PCLAROGRAPHY - A NEW HIGH PRECISION AND HIGH 65A-01-03
F MICROGRAM QUANTITIES O/ CONTROLLEO POTENTIAL OERIVATIVE DC PCLAROGRAPHY - APPLICATION TO DETERMINATION O 65A-01-06
TA-E-DIFFERENTIAL POLA/ CCNTROLLEC POTENTIAL DIFFERENTIAL OC POLAROGRAPHY (COMPARATIVE POLAROGRAPHY / DEL 65A-01-15
/ATICN / BLANK CORRECTICNS IN COULCMETRY / POTENTICSTATIC OC FCLAROGRAPHY / CONTROLLED POTENTIAL DC POLAR/ 65A-01-07
/ / CONTROLLED PCTENTIAL DC POLARCGRAPHY / POTENTICSTATIC DC PCLAROGRAPHY / DETERMINATION OF CADMIUM / DI/ 65A-01-03
/ICAL TECHNIQUES (DETERMINATION CF URANIUM BY OERIVATIVE DC FCLAROGRAPHY / DETERMINATION OF URANIUM BY C/ 65A-01-07
/ / POTENTIOSTATIC OC POLAROGRAPHY / CONTRCLLED PCTENTIAL DC PCLAROGRAPHY / POTENTIOSTATIC COULOMETRY / E/ 65A-01-07
/NTROLLEC POTENTIAL CC POLAROGRAPH / CONTROLLED POTENTIAL DC FCLAROGRAPHY / POTENTIOSTATIC DC POLAROGRAPH/ 65A-01-03
/ POLARCGRAPH / MODEL-Q-2792 POLARCGRAPH / PCTENTIOSTATIC DC FCLAROGRAPHY / SOLVENT EXTRACTICN PRECEEDING/ 65A-01-06
/ALUES / SENSITIVITY LIMIT OF POLAROGRAPHY / PRECISICN OF DC POLAROGRAPHY / SUBTRACTIVE MODE AMPERCMETRIC/ 65A-05-03A
TRY AS MICRO ANALYTICAL / CONTROLLED FOTENTIAL DERIVATIVE DC POLAROGRAPHY AND CONTROLLEC POTENTIAL COULOME 65A-01-07
/N OF TRACE QUANTITIES OF URANIUM BY CONTRCLLED POTENTIAL OC PCLAROGRAPHY IN A TRI OCTYL PHOSPHINE OXIOE / 65PRR-160
F PRECISION AND ACCURACY IN NEUTRCN ACTIVATION ANALYSIS (DEAD TIME EFFECTS) STUDY O 65A-11-08B
/ARCH ANC DEVELOPMENT GROUP MONTHLY SUMMARY - NOV.,1964 / DEC.,1964 / JAN.,1965 / FEB.,1965 / MAR.,1965 // 65PRR-104
OECAPPERS 65PRR-120
47 GAMMA-RAY BRANCHINGS IN THE DECAY CF INCIUM-116, CERIUM-141, AND NECDYMIUM-1 65PRR-169
DECAY OF MOLYBDENUM-99 - TECHNETIUM-99M 65PRR-011
DISSOLUTICN OR DECCMPOSITON OF VARIOUS ORGANIC MATERIALS 65A-06-04
HEPTA OECYL AMINE 65A-13-01E
NUCLEAR SPECTROSCOPY OF NEUTRON DEFICIENT HAFNIUM AND RARE EARTH RADIONUCLIDES 65A-11-01E
INTEGRATEC ASSEMBLY FOR GENERATION, STORAGE, AND DELIVERY OF STANCARD CHROMIUM(II) SOLUTICNS 65PRR-055
/IFFERENTIAL CC POLAROGRAPHY (COMPARATIVE POLAROGRAPHY / DELTA-E-OIFFERENTIAL POLAROGRAPHY / SUBTRACTIVE/ 65A-01-15
PREPARATION AND RECOVERY OF CESIUM COMPCUNDS FOR DENSITY GRADIENT SOLUTIONS 65PRR-116
/CENCE / ELECTRCN PROBE ANALYZER / DETERMINATICN OF AREAL DENSITY OF GOLC COATING ON SILICON RADIATION DE/ 65A-07-01A
PARTICLES OETERMINATION OF THE COATING DENSITY OF PYRCLYTIC CARBON COATEO NUCLEAR FUEL 65PRR-164
AMMETRY WITH LINEARLY VARYING POTENTIAL REVERSIBLE DEPCSITION OF METALS ON SOLID ELECTRODES BY VOLT 65A-02-02A
AMMETRY WITH LINEARLY VARYING POTENTIAL REVERSIBLE DEPOSITION OF METALS CN SOLID ELECTRODES BY VOLT 65PRR-030
AMMETRY WITH LINEARLY VARYING POTENTIAL REVERSIBLE OEPOSITION OF METALS ON SOLID ELECTRODES BY VOLT 65PRR-191
H CONTROLLED CURRENT / DERIVATIVE VOLTAMMETRY WITH CCNTR/ OERIVATIVE CHRCNOPOTENTIOMETRY (VOLTAMMETRY WIT 65A-01-09
/ATING CIRCUIT FOR THE CONTROLLED POTENTIAL VOLTAMMETER (OERIVATIVE COMPUTER / INSTRUMENT DESIGN / ELECT/ 65A-01-11
RMINATION OF MICROGRAM QUANTITIES O/ CONTRCLLED POTENTIAL DERIVATIVE DC PCLAROGRAPHY - APPLICATION TO DETE 65A-01-06
/ICRO ANALYTICAL TECHNIQUES (DETERMINATION OF URANIUM BY DERIVATIVE CC PCLAROGRAPHY / DETERMINATICN OF U/ 65A-01-07
IAL COULOMETRY AS MICRO ANALYTICAL / CONTRCLLEO POTENTIAL DERIVATIVE DC PCLAROGRAPHY ANC CONTROLLEO POTENT 65A-01-07
CONTROLLED POTENTIAL AND DERIVATIVE POLAROGRAPH, MODEL-Q-1988A 65PRR-117
LAROGRAPHY APPLICATICN OF DERIVATIVE TECHNIQUES TO STATIONARY ELECTRODE PO 65PRR-038
/CNOPOTENTIOMETRY (VOLTAMMETRY WITH CONTRCLLED CURRENT / DERIVATIVE VOLTAMMETRY WITH CONTROLLED CURRENT / 65A-01-09
/POTENTIAL VOLTAMMETER (DERIVATIVE COMPUTER / INSTRUMENT DESIGN / ELECTRCCHEMISTRY / ANALYSIS OF MOLTEN / 65A-01-11
STATE CIRCUITS / DOUBLE LAY/ A SQUARE-WAVE POLAROGRAPH (DESIGN CF NEW SCUARE-WAVE POLAROGRAPH WITH SOLID 65A-01-14
ZINE AS A LIQUID SCINTILLATOR SOLVENT FOR THERMAL NEUTRON DETECTION N,N,N TRI METHYL BORA 65A-11-05A
ZINE AS A LIQUID SCINTILLATOR SOLVENT FOR THERMAL NEUTRON OETECTION N,N,N, TRI METHYL BCRA 65PRR-049
// ELIMINATICN OF IR LOSS IN POLAROGRAPHY / POLAROGRAPHIC OETECTION LIMITS FOR URANIUM / CONCENTRATION AO/ 65A-01-06
SCINTILLATION COUNTERS DETECTION OF NATURAL RADIONUCLIDES BY WELL TYPE 65A-11-038

ICN OF AREAL DENSITY OF GOLD COATING CN SILICON RADIATION DETECTORS BY USE OF X-RAY FLUORESCENCE) /RMINAT 65A-07-01A
ANALYSIS OF ALKYL BENZENE SULFCNATE DETERGENTS 65A-05-04K
CONTENT OF WOCL FROM TWIN WETHERS RAISED CN TWO SOILS AS DETERMINED BY NEUTRON ACTIVATION ANALYSIS /EMENT 65PRR-019
RACTICN OF BERYLLIUM, COPPER, NICKEL, COBALT, AND ZINC AS DI BENZOYL METHANE COMPLEXES SOLVENT EXT 65TR-1C
BCRCN-1C DI BCRANE 65A-13-01C
SYNTHESIS AND INFRARED ABSORPTICN SPECTRUM OF DI BCRANE - BORCN-1C 65PRR-079
/C BUTYL PHENYL PHOSPHCNATE IN DI SEC BUTYL PHCSPHCNATE - DI ETHYL BENZENE SOLUTIONS, DIELECTRIC CONSTANT/ 65PRR-156
/ALT REACTOR EXPERIMENT (MSRE) FUEL, SPECTROPHOTOMETRIC DI METHYL GLYOXIME (PER SULFATE OXIDATION) ME/ 65PRR-134
OF METALS USE OF METAL DI N BUTYL PHOSPHORO DI THIOATES AS EXTRACTANTS 65PRR-016
HODS FOR THE EVALUATICN OF ATMOSPHERIC POLLUTICN - SULFUR DI OXICE RADIO RELEASE MET 65PRR-005
ODS FOR THE EVALUATICN OF ATMOSPHERIC POLLUTICN - SULPHUR DI OXICE RADIO RELEASE METH 65A-11-07B
/TIES OF MOISTURE, OXYGEN, HYDROGEN, NITROGEN, AND CARBON DI OXICE IN GLOVE BOX ATMOSPHERES ON THE ANALYS/ 65PRR-175
2,6 DI OXIMINO CYCLOHEXANCNE 65A-13-01B
PHOSPHCNATE - DI ETHYL BENZENE SOLUTICNS, DIELECTRIC CCN/ DI SEC BUTYL PHENYL PHOSPHCNATE IN DI SEC BUTYL 65PRR-156
TIONS, DIELECTRIC CON/ DI SEC BUTYL PHENYL PHOSPHCNATE IN DI SEC BUTYL PHCSPHONATE - DI ETHYL BENZENE SOLU 65PRR-156
NITRATE, PHENOL DI SULFONIC ACID SPECTROPHOTOMETRIC METHOD 65PRR-132
/OUNDS THAT CCNTAIN PHOSPHORO THICATE, ,P=S AND PHOSPHORO DI THIOATE, ,P(S)SH GROUPS AS SEPARATION AND AN/ 65A-11-04B
/MPOUNDS CONTAINING PHOSPHORO THICATE, ,P=S AND PHOSPHORO DI THICATE, ,P(S)SH GROUPS AS SEPARATORY AND AN/ 65PRR-017
USE OF METAL DI N BUTYL PHCSPHCRO DI THIOATES AS EXTRACTANTS OF METALS 65PRR-016
IN DI SEC BUTYL PHOSPHCNATE - DI ETHYL BENZENE SOLUTICNS, DIELECTRIC CCNSTANT METHOD / PHENYL PHOSPHONATE 65PRR-156
CLYSIS AND GAS CHROMATOGRAPHIC DETERMINATICN OF STYROFCAM DIELECTRIC FILM PYR 65A-05-01B
OGRAPHY / DELTA-E-DIFFERENTIAL POLA/ CONTRCLLED POTENTIAL DIFFERENTIAL DC POLAROGRAPHY (COMPARATIVE POLAR 65A-01-15
/ENTIOSTATIC DC POLAROGRAPHY / DETERMINATICN OF CADMIUM / DIFFERENTIAL POLAROGRAPHY / DIFFERENTIAL VOLTAM/ 65A-01-03
RECORDING INTEGRO - DIFFERENTIATING POLAROGRAPH DIFFERENTIAL POLAROGRAPHY ON AN ELECTRONIC SELF- 65TR-02
ITE ELECTRODE (P.G.E.) AND THE CLASSY CARBON ELECTRODE/ DIFFERENTIAL TITRIMETRY WITH THE PYROLYTIC GRAPH 65A-05-02C
// DETERMINATION OF CADMIUM / DIFFERENTIAL POLAROGRAPHY / DIFFERENTIAL VOLTAMMETRY / COMPARATIVE VOLTAMME/ 65A-01-03
TIAL VOLTAMMETER (DERIVATIVE COMPUTER / INSTRUMENT DESI/ DIFFERENTIATING CIRCUIT FOR THE CONTROLLED POTEN 65A-01-11
AL POLAROGRAPHY CN AN ELECTRONIC SELF-RECORDING INTEGRC - DIFFERENTIATING POLARCGRAPH DIFFERENTI 65TR-02
NS ELECTRON AND OPTICAL MICROSCOPY AND ELECTRON DIFFRACTION RESEARCH ASSISTANCE TO OTHER DIVISIO 65A-10-04
ABSCRPTICN AND DIFFUSE REFLECTANCE SPECTRA OF SOLID MATERIALS 65A-02-01C
CN OF AIRBORNE RADIO IODINE SPECIES BY MEANS OF COMPOSITE DIFFUSION TUBES INVESTIGATI 65PRR-067
ACID IODINE SPECIES FOUND CN RUBBER SECTIONS OF COMPOSITE DIFFUSION TUBES CHEMICAL BEHAVIOR CF THE R 65PRR-069
UNDER MOIST CONDITIONS IN MAY PACKS BY MEANS OF COMPOSITE DIFFUSION TUBES /ATION OF RADIO IODINE BEHAVICR 65PRR-066
OF NONELEMENTAL RADIO IODINE VAPCR BY MEANS OF COMPOSITE DIFFUSION TUBES /TIGATION OF CHEMICAL REACTIVITY 65PRR-068
HIC ANALYSIS (VISUAL INTERPOLATICN) (LITHIUM CARBCNATE DILUTICN METHOC OF SPECTROGRAPHIC ANALYSIS) /AP 65A-07-01F
PROBLEMS IN DISPCSAL OF RADIOACTIVE LIQUID WASTES IN SALT 65PRR-023
/EDICTION OF THE SOLUBILITY AND TEMPERATURE DEPENDENCE OF DISSOCIATION CONSTANT OF CARBONIC ACID IN CONCE/ 65TR-08
ATERIALS DISSOLUTION OR DECOMPOSITON OF VARIOUS ORGANIC M 65A-06-04
HOD CHLORIDE, DISTILLATION - INDIRECT IODOMETRIC TITRATION MET 65PRR-126
// MONOVALENT THALLIUM TO THALLIUM METAL REDOX REACTICN / DIVALENT LEAD TC LEAD METAL REDOX REACTION / PO/ 65A-05-03A
CID AND OTHER GLASS CORRODING MEDIA - EVALUATICN WITH THE DIVALENT LEAD TC LEAD METAL REDOX REACTION /IC A 65PRR-040
/LENT LEAD TO LEAD METAL REDOX REACTICN / POLAROGRAPHY OF DIVALENT LEAD TC LEAD METAL REDOX REACTION IN A/ 65A-05-03A
/ THE MEASURED VALUES FOR THE HALF-WAVE POTENTIALS OF THE DIVALENT LEAD TO LEAD METAL REDOX REACTION REDU/ 65PRR-010
OPY AND ELECTRON DIFFRACTICN RESEARCH ASSISTANCE TC OTHER DIVISICNS ELECTRON AND OPTICAL MICROSC 65A-10-04
HIGH TEMPERATURE CHEMICAL DOSIMETRY 65A-03-03
/ NEW SQUARE-WAVE POLAROGRAPH WITH SOLID STATE CIRCUITS / DOUBLE LAYER CAPACITANCE MEASUREMENT / TEFLON D/ 65A-01-14
/PERCMETRY / HAMMER-CCNTROL OF DROPPING MERCURY ELECTRCDE DROP TIME / LINEAR TIME BASE / SMOLER DROPPING / 65A-01-04
G MERCURY ELECTRODE DROP TIME / LINEAR TIME BASE / SMOLER DROPPING MERCURY ELECTRODE) /CONTROL OF DROPPIN 65A-01-04
CIRCUITS / DOUBLE LAYER CAPACITANCE MEASUREMENT / TEFLON DROPPING MERCURY ELECTRODE) /H WITH SOLID STATE 65A-01-14
STUDIES WITH THE TEFLON DROPPING MERCURY ELECTRODE (D.M.E.) 65A-05-03
/AQUEOUS HYDROFLUORIC ACID WITH HORIZONTAL ORIFICE TEFLON DROPPING MERCURY ELECTRODE / SIGNIFICANCE OF JU/ 65A-05-03A
SIGNAL TO NOISE RATIOS (S/N) OF GLASS AND TEFLON DROPPING MERCURY ELECTRODE CAPILLARIES 65A-05-03B
/CLIC VOLTAMMETRY / CHRONOAMPEROMETRY / HAMMER-CONTROL OF DROPPING MERCURY ELECTRODE DROP TIME / LINEAR T/ 65A-01-04
/DING MEDIA (EVALUATICN OF THE HORIZONTAL ORIFICE TEFLON DROPPING MERCURY ELECTRODE FOR OBTAINING FUNDAM/ 65A-05-03A
YDROFLUORIC ACID AND OTHER GLASS CORRODING MEDIA / TEFLON DROPPING MERCURY ELECTRODE FOR POLAROGRAPHY IN H 65PRR-039
AD(II) IN AQUEOUS HYDROFLUORIC ACID (1-12-MOLAR) WITH A DROPPING MERCURY ELECTRODE OF TEFLON /APHY OF LE 65PRR-041
RAPHY IN HYDROFLUORIC ACID AND OTHER GLASS CORRODING MED/ DROPPING MERCURY ELECTRODE OF TEFLON FOR POLAROG 65PRR-040
TION OF LEAD, BISMUTH, CADMIUM, TIN, AND ZINC IN AIRBORNE DUST SAMPLES DETERMINA 65A-07-02A
NITRATE IN TRANSURANIUM (TRU) SOLUTIONS, AZO DYE FILTER PHOTOMETRIC METHOD 65PRR-123
AZO DYES (CALCICHROME / CHLORO PHOSPHONAZO-III) 65A-13-01F
NEW RARE EARTH ALPHA EMITTER, EUROPIUM-148 65PRR-056
STUDIES OF RARE EARTH ALPHA EMITTING RADIONUCLIDES 65A-11-01F
METHOD FOR SEPARATING TRANSPLUTONIUM ELEMENTS FROM RARE EARTH FISSION PRODUCTS 65PRR-223
UCLEAR SPECTROSCOPY OF NEUTRON DEFICIENT HAFNIUM AND RARE EARTH RADIONUCLIDES N 65A-11-01E
REVIEW OF SPECTROSCOPIC PROPERTIES OF RARE EARTHS 65PRR-059
QUANTITATIVE ANALYSIS BY THE X-RAY ABSORPTION EDGE TECHNIQUE 65PRR-166
PERSONNEL EDUCATION 65A-10-05
LEAR TECHNOLOGY EIGHTH CONFERENCE ON ANALYTICAL CHEMISTRY IN NUC 65PRR-053
LEAR TECHNOLOGY A RESUME OF THE EIGHTH CONFERENCE ON ANALYTICAL CHEMISTRY IN NUC 65PRR-054
C POTENTIAL DC POLAROGRAPHY / POTENTIOSTATIC COULOMETRY / ELECTROCHEMISTRY) / DC POLAROGRAPHY / CONTROLLE 65A-01-07
L VOLTAMMETER (DERIVATIVE COMPUTER / INSTRUMENT DESIGN / ELECTROCHEMISTRY / ANALYSIS OF MOLTEN SALTS) /A 65A-01-11
/GRAPHY - A NEW HIGH PRECISION AND HIGH ACCURACY METHOD (ELECTROCHEMISTRY / MODEL-Q-1988-FES CONTROLLED / 65A-01-03
/L DETERMINATICN OF URANIUM / 0.5-ML POLAROGRAPHIC CELL / ELECTROCHEMISTRY / MODEL-Q-1988-FES POLAROGRAPH/ 65A-01-06
CTRODE ELECTROCHEMISTRY WITH THE PYROLYTIC GRAPHITE ELE 65PRR-194
ELECTROCHEMISTRY WITH THE PYROLYTIC GRAPHITE ELECTRODE 65PRR-194
IODINE, AND IODATE WITH THE PYROLYTIC GRAPHITE INDICATING ELECTRODE VOLTAMMETRY OF IODIDE, 65PRR-193
DE DROP TIME / LINEAR TIME BASE / SMOLER DROPPING MERCURY ELECTRODE) /CONTROL OF DROPPING MERCURY ELECTRO 65A-01-04
E LAYER CAPACITANCE MEASUREMENT / TEFLON DROPPING MERCURY ELECTRODE) /H WITH SOLID STATE CIRCUITS / DOUBL 65A-01-14
OTASSIUM FLUORIDE / PYROLYTIC GRAPHITE ELECTRODE / SILVER ELECTRODE) /HIUM FLUORIDE - SODIUM FLUORIDE - P 65A-02-02
STUDIES WITH THE TEFLON DROPPING MERCURY ELECTRODE (D.M.E.) 65A-05-03
GLASSY CARBON INDICATOR ELECTRODE (G.C.E.) 65A-05-02A
LYTIC GRAPHITE ELECTRODE (P.G.E.) AND THE GLASSY CARBON ELECTRODE (G.C.E.) /L TITRIMETRY WITH THE PYRO 65A-05-02C
OXIDE FILM FORMATION ON THE PYROLYTIC GRAPHITE ELECTRODE (P.G.E.) 65A-05-02B
RODE/ DIFFERENTIAL TITRIMETRY WITH THE PYROLYTIC GRAPHITE ELECTRODE (P.G.E.) AND THE GLASSY CARBON ELECT 65A-05-02C
/CE ANALYSIS FOR URANIUM / APPLICATION OF QUASI REFERENCE ELECTRODE / ELIMINATION OF IR LOSS IN POLAROGRA/ 65A-01-06
/RIC ACID WITH HORIZONTAL ORIFICE TEFLON DROPPING MERCURY ELECTRODE / SIGNIFICANCE OF JUNCTION POTENTIALS/ 65A-05-03A
SODIUM FLUORIDE - POTASSIUM FLUORIDE / PYROLYTIC GRAPHITE ELECTRODE / SILVER ELECTRODE) /HIUM FLUORIDE - 65A-02-02
NOISE RATIOS (S/N) OF GLASS AND TEFLON DROPPING MERCURY ELECTRODE CAPILLARIES SIGNAL TO 65A-05-03B
// CHRONOAMPEROMETRY / HAMMER-CONTROL OF DROPPING MERCURY ELECTRODE DROP TIME / LINEAR TIME BASE / SMOLER/ 65A-01-04
/UATION OF THE HORIZONTAL ORIFICE TEFLON DROPPING MERCURY ELECTRODE FOR OBTAINING FUNDAMENTAL POLAROGRAPH/ 65A-05-03A
AND OTHER GLASS CORRODING MEDIA / TEFLON DROPPING MERCURY ELECTRODE FOR POLAROGRAPHY IN HYDROFLUORIC ACID 65PRR-039
A GLASSY CARBON ELECTRODE FOR VOLTAMMETRY 65PRR-061
HYDROFLUORIC ACID (1-12-MOLAR) WITH A DROPPING MERCURY ELECTRODE OF TEFLON /APHY OF LEAD(II) IN AQUEOUS 65PRR-041

ORIC ACID AND OTHER GLASS CORRODING MED/ DROPPING MERCURY ELECTRODE OF TEFLON FOR POLAROGRAPHY IN HYDROFLU 65PRR-040
APPLICATION OF DERIVATIVE TECHNIQUES TO STATIONARY ELECTRODE POLAROGRAPHY 65PRR-038
C POTENTIOSTAT AND ITS USE FOR THE INVESTIGATION OF RAPID ELECTRODE REACTIONS THE ELECTRONI 65TR-06
NG PREPARATION BY ELECTRODEPOSITION OF MOUNTS FOR 4 PI BETA COUNTI 65A-11-03A
STUDIES WITH SOLID INDICATOR ELECTRODES 65A-05-02
FILM FORMATION ON PYROLYTIC GRAPHITE ELECTRODES 65PRR-029
ROUS AMMONIUM SULFATE / CHRONOPOTENTIOMETRY WITH PLATINUM ELECTRODES) /CHOPOTENTIOMETRIC OXIDATION OF FER 65A-01-02
POTENTIAL REVERSIBLE DEPOSITION OF METALS ON SOLID ELECTRODES BY VOLTAMMETRY WITH LINEARLY VARYING 65A-02-02A
POTENTIAL REVERSIBLE DEPOSITION OF METALS ON SOLID ELECTRODES BY VOLTAMMETRY WITH LINEARLY VARYING 65PRR-030
POTENTIAL REVERSIBLE DEPOSITION OF METALS ON SOLID ELECTRODES BY VOLTAMMETRY WITH LINEARLY VARYING 65PRR-191
FRACTION RESEARCH ASSISTANCE TO OTHER DIVISIONS ELECTRON AND OPTICAL MICROSCOPY AND ELECTRON DIF 65A-10-04
R DIVISIONS ELECTRON AND OPTICAL MICROSCOPY AND ELECTRON DIFFRACTION RESEARCH ASSISTANCE TO OTHE 65A-10-04
CTIVE MATERIALS ELECTRON MICROSCOPE FACILITY FOR STUDYING RADIOA 65A-10-01A
OPTICAL AND ELECTRON MICROSCOPY 65A-10
METHODS DEVELOPMENT FOR ELECTRON MICROSCOPY 65A-10-03
ELECTRON MICROSCOPY OF RADIOACTIVE MATERIALS 65A-10-01
CARBIDES IN A NIOBIUM - TUNGSTEN - ZIRCONIUM ALLOY ELECTRON OPTICAL STUDY OF THE EFFECT OF AGING ON 65PRR-210
/AY ANALYSIS (ANALYSIS OF SOLIDS BY X-RAY FLUORESCENCE / ELECTRON PROBE ANALYZER / DETERMINATION OF AREA/ 65A-07-01A
ELECTRONIC GAMMA-RAY SPECTRAL RESOLUTION 65A-11-06A
STIGATION OF RAPID ELECTRODE REACTIONS THE ELECTRONIC POTENTIOSTAT AND ITS USE FOR THE INVE 65TR-06
ELECTRONIC RESOLUTION OF GAMMA-RAY SPECTRA 65PRR-168
ING POLAROGRAPH DIFFERENTIAL POLAROGRAPHY ON AN ELECTRONIC SELF-RECORDING INTEGRO - DIFFERENTIAT 65TR-02
AUTOMATIC COULOMETRIC TITRATOR, ORNL MODEL-C-2005, ELECTRONIC, CONTROLLED POTENTIAL 65PRR-146
HIGH VOLTAGE PAPER ELECTROPHORESIS 65A-11-04D
/FOR URANIUM / APPLICATION OF QUASI REFERENCE ELECTRODE / ELIMINATION OF IR LOSS IN POLAROGRAPHY / POLARO/ 65A-01-06
IRECT IODOMETRIC TITRATION METHOD OXALATE IN ELUATES FROM IRRADIATED ION EXCHANGE RESINS, IND 65PRR-158
RECENT ADVANCES IN ANALYTICAL EMISSION SPECTROMETRY 65PRR-212
NEW RARE EARTH ALPHA EMITTER, EUROPIUM-148 65PRR-056
ABSOLUTE LIQUID SCINTILLATION COUNTING OF BETA EMITTERS 65PRR-014
HALF-LIVES OF LONG-LIVED BETA EMITTERS (CHLORINE-36 BERYLLIUM-10) 65A-11-01D
STUDIES OF RARE EARTH ALPHA EMITTING RADIONUCLIDES 65A-11-01F
HEMISTRY AND / EXPERIMENTAL STUDY OF ARTIFACTS AND ERRORS ENCOUNTERED IN GAMMA-RAY SPECTROMETRY FOR RADIOC 65A-11-03E
HEMISTRY AND / EXPERIMENTAL STUDY OF ARTIFACTS AND ERRORS ENCOUNTERED IN GAMMA-RAY SPECTROMETRY FOR RADIOC 65PRR-003
TRACE HIGH ENERGY GAMMA RADIOACTIVITY IN ISOTOPE PRODUCTS 65A-11-02E
ANALYTICAL SERVICES FOR CHEMICAL ENGINEERS 65PRR-182
-55 MEASUREMENT OF WEAR RATES IN AUTOMOTIVE ENGINES BY LIQUID SCINTILLATION COUNTING OF IRON 65A-11-07C
-55 WEAR RATES IN AUTOMOTIVE ENGINES BY LIQUID SCINTILLATION COUNTING OF IRON 65PRR-048
TION OF THE OVERLAPPING BAND STRUCTURE AND STUDIES OF THE ENVIRONMENTAL EFFECTS /A I. MATHEMATICAL RESOLU 65PRR-161
SKY-ILKOVIC EQUATION / POLARGRM / CHRNOGRM / KARAOGLANOFF EQUATION) /AMS AND CHRONOPOTENTIOGRAMS (HEYROV 65A-01-13
/ POLAROGRAMS AND CHRONOPOTENTIOGRAMS (HEYROVSKY-ILKOVIC EQUATION / POLARGRM / CHRNOGRM / KARAOGLANOFF E/ 65A-01-13
S EQUILIBRIUM DISTRIBUTION OF METAL - ION COMPLEXE 65PRR-074
HE MOLTEN SALT REACTOR EX/ ANALYTICAL LABORATORY HOT CELL EQUIPMENT I. SAMPLE PREPARATION APPARATUS FOR T 65PRR-027
ESISTANT PIPETTER ANALYTICAL LABORATORY HOT CELL EQUIPMENT II. A REMOTELY CONTROLLED CORROSION R 65PRR-028
METER ANALYTICAL LABORATORY HOT CELL EQUIPMENT III. A REMOTELY OPERATED FILTER PHOTO 65PRR-026
/ / POTASSIUM HEXA CHLORO RHENATE(IV) / HOLMIUM SULFIDE / ERBIUM SULFIDE / THULIUM PHOSPHIDE / THULIUM NI/ 65A-12
RADIOCHEMISTRY AND / EXPERIMENTAL STUDY OF ARTIFACTS AND ERRORS ENCOUNTERED IN GAMMA-RAY SPECTROMETRY FOR 65A-11-03E
RADIOCHEMISTRY AND / EXPERIMENTAL STUDY OF ARTIFACTS AND ERRORS ENCOUNTERED IN GAMMA-RAY SPECTROMETRY FOR 65PRR-003
/N OF URANIUM, AND THE PURIFICATION OF PROMETHIUM FOR THE ESTABLISHMENT OF A PRIMARY SPECTROGRAPHIC STAND/ 65PRR-089
OUTPUT SPECTRA OF 14-MEV NEUTRON GENERATORS - RAPID ESTIMATION AND INFLUENCE IN ACTIVATION ANALYSIS 65PRR-044
E/ OUTPUT SPECTRUM FROM 14-MEV NEUTRON GENERATORS - RAPID ESTIMATION AND INFLUENCE IN CROSS SECTION MEASUR 65PRR-043
CNATED STRAIGHT CHAIN ALKYL PHENOXY POLY (ETHYLENE OXY) ETHANOL) /IPAL-LO-436 / AMMONIUM SALT OF A SULF 65A-05-04M
/UTYL PHENYL PHOSPHONATE IN DI SEC BUTYL PHOSPHONATE - DI ETHYL BENZENE SOLUTIONS, DIELECTRIC CONSTANT ME/ 65PRR-156
SALT OF A SULFONATED STRAIGHT CHAIN ALKYL PHENOXY POLY (ETHYLENE OXY) ETHANOL) /IPAL-LO-436 / AMMONIUM 65A-05-04M
NEW RARE EARTH ALPHA EMITTER, EUROPIUM-148 65PRR-056
/REACTIONS (HELIUM-3 ACTIVATION ANALYSIS / SENSITIVITY / EXCITATION FUNCTIONS FOR HELIUM-3 INDUCED NUCLE/ 65A-11-08A
LEAD-210 IN AUTO EXHAUST FUMES AND IN NATURAL MATERIALS 65A-11-02H
/IES OF URANIUM IN A TRI N OCTYL PHOSPHINE OXIDE (TOPO) EXTRACT (MICROANALYTICAL DETERMINATION OF URAN/ 65A-01-06
POTENTIAL DC POLAROGRAPHY IN A TRI OCTYL PHOSPHINE OXIDE EXTRACT /ACE QUANTITIES OF URANIUM BY CONTROLLED 65PRR-160
N,N,N TRI HEXYL PHOSPHORIC TRI AMIDE (THPA) AS AN EXTRACTANT FOR METAL IONS 65A-11-04A
/ TRI ALKYL AND HEXA ALKYL PHOSPHORO THIOIC TRI AMIDES AS EXTRACTANTS FOR METAL IONS - SELECTIVE EXTRACTIO 65PRR-015
USE OF METAL DI N BUTYL PHOSPHORO DI THIOATES AS EXTRACTANTS OF METALS 65PRR-016
AND ZINC AS DI BENZOYL METHANE COMPLEXES SOLVENT EXTRACTION OF BERYLLIUM, COPPER, NICKEL, COBALT, 65TR-10
/OIC TRI AMIDES AS EXTRACTANTS FOR METAL IONS - SELECTIVE EXTRACTION OF MERCURY(II), SILVER(I), AND COPPE/ 65PRR-015
REVIEW OF THE SOLVENT EXTRACTION OF METAL CHELATES 65PRR-058
OF ORGANIC ACIDS WITH HIGH MOLECULAR WEIGH/ LIQUID-LIQUID EXTRACTION OF METAL IONS FROM AQUEOUS SOLUTIONS 65PRR-031
/2 POLAROGRAPH / POTENTIOSTATIC DC POLAROGRAPHY / SOLVENT EXTRACTION PRECEDING POLAROGRAPHIC DETERMINATI/ 65A-01-06
CHRONOPOTENTIOMETER WITH COMPENSATION FOR EXTRANEOUS CURRENTS 65PRR-051
CURRENT) CHRONOPOTENTIOMETER WITH COMPENSATION FOR EXTRANEOUS CURRENTS (VOLTAMMETRY AT CONTROLLED 65A-01-01
/F TECHNIQUE OF CHRONOPOTENTIOMETRY WITH COMPENSATION FOR EXTRANEOUS CURRENTS (VOLTAMMETRY WITH CONTROLL/ 65A-01-02
/UP MONTHLY SUMMARY - NOV.,1964 / DEC.,1964 / JAN.,1965 / FEB.,1965 / MAR.,1965 / APR.,1965 / MAY,1965 / / 65PRR-104
/ED CURRENT / CHRONOPOTENTIOMETRIC REDUCTION OF POTASSIUM FERRI CYANIDE / CHRONOPOTENTIOMETRIC REDUCTION / 65A-01-02
/ASSIUM FERRI CYANIDE / CHRONOPOTENTIOMETRIC REDUCTION OF FERRIC PER CHLORATE / CHRONOPOTENTIOMETRIC OXID/ 65A-01-02
/ATION OF OXALIC ACID / CHRONOPOTENTIOMETRIC OXIDATION OF FERROUS AMMONIUM SULFATE / CHRONOPOTENTIOMETRY / 65A-01-02
CHROMIUM IN MSRE FUEL, AMPEROMETRIC FERROUS SULFATE TITRATION METHOD 65PRR-136
GAS CHROMATOGRAPHIC DETERMINATION OF STYROFOAM DIELECTRIC FILM PYROLYSIS AND 65A-05-01B
FILM FORMATION ON PYROLYTIC GRAPHITE ELECTRODES 65PRR-029
DE (P.G.E.) OXIDE FILM FORMATION ON THE PYROLYTIC GRAPHITE ELECTRO 65A-05-02B
REMOTELY OPERATED FILTER PHOTOMETER 65A-04-01C
L LABORATORY HOT CELL EQUIPMENT III. A REMOTELY OPERATED FILTER PHOTOMETER ANALYTICA 65PRR-026
NITRATE IN TRANSURANIUM (TRU) SOLUTIONS, AZO DYE FILTER PHOTOMETRIC METHOD 65PRR-123
RECENT FINDINGS IN RADIOCHEMISTRY 65PRR-196
UTIONS, MEASUREMENT OF PROMPT GAMMA-RAYS FROM SPONTANEOUS FISSION /252 IN TRANSURANIUM (TRU) PROCESS SOL 65PRR-130
/OF THE ABSENCE OF LONG-LIVED ISOTOPES OF PROMETHIUM FROM FISSION OF URANIUM, AND THE PURIFICATION OF PRO/ 65PRR-089
RAPID SEPARATION OF TECHNETIUM FROM FISSION PRODUCTS 65A-11-04C
RAPID SEPARATION OF TECHNETIUM FROM FISSION PRODUCTS 65PRR-076
OD FOR SEPARATING TRANSPLUTONIUM ELEMENTS FROM RARE EARTH FISSION PRODUCTS METH 65PRR-223
SOLUTIONS PRIOR TO THE DETERMINATION OF TOTAL LANTHANIDE FISSION PRODUCTS /SURANIUM ELEMENTS FROM AQUEOUS 65PRR-032
MASS FISSION YIELD CURVE FOR AMERICIUM-241 65PRR-046
MASS FISSION YIELD CURVE FOR NEPTUNIUM-237 65PRR-094
WATER) FLAME PHOTOMETRY (COLLECTION OF BARIUM FROM TAP 65A-07-01D
LD COATING ON SILICON RADIATION DETECTORS BY USE OF X-RAY FLUORESCENCE) /RMINATION OF AREAL DENSITY OF GO 65A-07-01A
NATION OF A/ X-RAY ANALYSIS (ANALYSIS OF SOLIDS BY X-RAY FLUORESCENCE / ELECTRON PROBE ANALYZER / DETERMI 65A-07-01A
OF OXIDIZING POWER OF SOLUTIONS OF HYDROLYZED XENON HEXA FLUORIDE DETERMINATION 65A-05-04L

C THIOCYANATE DETERMINATION OF NIOBIUM IN THE PRESENCE OF FLUORIDE SPECTROPHOTOMETRI 65A-05-04H
II) ION IN LITHIUM FLUORIDE - SODIUM FLUORIDE - POTASSIUM FLUORIDE ABSORPTION SPECTRUM OF COBALTI 65A-02-01B
C TITRATION OF URANIUM(VI) IN AQUEOUS SOLUTIONS OF SODIUM FLUORIDE CONTROLLED POTENTIAL COULOMETRI 65PRR-034
ABSORPTION AND REFLECTANCE SPECTRA OF IRRADIATED LITHIUM FLUORIDE - BERYLLIUM FLUORIDE - ZIRCONIUM FLUORI 65-02-01A
/FLUORIDE SALT SYSTEMS (SILVER / IRON / NICKEL / LITHIUM FLUORIDE - BERYLLIUM FLUORIDE / LITHIUM FLUORID/ 65A-02-02
N SPECTRUM OF COBALT(II) ION IN LITHIUM FLUORIDE - SODIUM FLUORIDE - POTASSIUM FLUORIDE ABSORPTIO 65A-02-01B
/LUORIDE - BERYLLIUM FLUORIDE / LITHIUM FLUORIDE - SODIUM FLUORIDE - POTASSIUM FLUORIDE / PYROLYTIC GRAPH/ 65A-02-02
ABSORPTION SPECTRUM OF COBALT(II) ION IN LITHIUM FLUORIDE - SODIUM FLUORIDE - POTASSIUM FLUORIDE 65A-02-01B
/NICKEL / LITHIUM FLUORIDE - BERYLLIUM FLUORIDE / LITHIUM FLUORIDE - SODIUM FLUORIDE - POTASSIUM FLUORIDE/ 65A-02-02
/DIATED LITHIUM FLUORIDE - BERYLLIUM FLUORIDE - ZIRCONIUM FLUORIDE - THORIUM FLUORIDE - URANIUM(IV) FLUOR/ 65-02-01A
ORIDE - BERYLLIUM FLUORIDE - ZIRCONIUM FLUORIDE - THORIUM FLUORIDE - URANIUM(IV) FLUORIDE /TED LITHIUM FLU 65-02-01A
/TANCE SPECTRA OF IRRADIATED LITHIUM FLUORIDE - BERYLLIUM FLUORIDE - ZIRCONIUM FLUORIDE - THORIUM FLUORID/ 65-02-01A
/ (SILVER / IRON / NICKEL / LITHIUM FLUORIDE - BERYLLIUM FLUORIDE / LITHIUM FLUORIDE - SODIUM FLUORIDE -/ 65A-02-02
/LUORIDE / LITHIUM FLUORIDE - SODIUM FLUORIDE - POTASSIUM FLUORIDE / PYROLYTIC GRAPHITE ELECTRODE / SILVE/ 65A-02-02
IDE - ZIRCONIUM FLUORIDE - THORIUM FLUORIDE - URANIUM(IV) FLUORIDE /TED LITHIUM FLUORIDE - BERYLLIUM FLUOR 65-02-01A
FLUORIDE IN MSRE FUEL, PYROLYSIS METHOD 65PRR-137
LITHIUM FLUORIDE - BERYLLIUM FLUO/ VOLTAMMETRY OF MOLTEN FLUORIDE SALT SYSTEMS (SILVER / IRON / NICKEL / 65A-02-02
DETERMINATION OF IODINE AND FLUORINE IN ,KIO2F2 65A-05-04F
COMPUTER PROGRAM FOR CALCULATING NEUTRON FLUX 65A-11-06F
STANDARDIZATION OF NEUTRON FLUX MEASUREMENTS 65A-11-09C
ACTIVATION ANALYSIS - REACTOR NEUTRON FLUXES AND INTERFERENCES 65PRR-197
ALKYL BENZENE SULFONATE (ABS) CONTROL FOR THE FOAM SEPARATION PROCESS 65PRR-183
GOLD ATOMS SPUTTERED FROM NEUTRON IRRADIATED GOLD FOILS 65A-11-08F
REPORT ON FOREIGN TRAVEL JAN.16-FEB.20,1965 65PRR-101
REPORT ON FOREIGN TRAVEL JUNE-25-JULY-30,1965 65PRR-072
REPORT ON FOREIGN TRAVEL JUNE-30-JULY-10,1965 65PRR-070
REPORT ON FOREIGN TRAVEL MAY-15-JUNE-9,1965 65PRR-095
REPORT OF FOREIGN TRAVEL OCT.10-28,1964 65PRR-087
REPORT ON FOREIGN TRAVEL OCT.19-30,1964 65PRR-099
REPORT ON FOREIGN TRAVEL SEPT.2-OCT.6,1965 65PRR-085
NEUTRON ACTIVATION ANALYSIS OF HUMAN HAIR - FORENSIC APPLICATIONS 65A-11-08D
CAUTION AND PRECAUTION IN FORENSIC APPLICATIONS 65PRR-186
APPLICATION OF NEUTRON ACTIVATION ANALYSIS TO FORENSIC SCIENCE 65PRR-185
INFLUENCE OF THE RECORDING ARRANGEMENT ON THE FORM OF POLAROGRAPHIC CURVES 65TR-01
IDENTITY, CHARACTER AND CHEMICAL BEHAVIOR OF VAPOR FORMS OF RADIO IODINE 65PRR-162
CONCENTRATED LITHIUM CHLORIDE SOLUTION - DETERMINATION OF FREE ACID AND HYDROLYZABLE CATION REACTIONS IN 65PRR-022
POLAROGRAPHIC DETERMINATION OF NICKEL IN MSRE FUEL 65A-04-01E
COULOMETRIC TITRATION OF URANIUM(VI) IN A SYNTHETIC MSRE FUEL CONTROLLED POTENTIAL 65A-04-01D
MONITORING OF LEACHED FUEL ELEMENTS WITH A NEUTRON GENERATOR 65A-11-09F
OF THE COATING DENSITY OF PYROLYTIC CARBON COATED NUCLEAR FUEL PARTICLES DETERMINATION 65PRR-164
ZIRCONIUM IN MSRE FUEL, AMPEROMETRIC CUPFERRON TITRATION METHOD 65PRR-135
HOD CHROMIUM IN MSRE FUEL, AMPEROMETRIC FERROUS SULFATE TITRATION MET 65PRR-136
IC TI/ URANIUM IN MOLTEN SALT REACTOR EXPERIMENT (MSRE) FUEL, AUTOMATIC, CONTROLLED POTENTIAL, COULOMETR 65PRR-121
T/ URANIUM IN THE MOLTEN SALT REACTOR EXPERIMENT (MSRE) FUEL, HIGH SENSITIVITY, AUTOMATIC, CONTROLLED PO 65PRR-122
URANIUM IN MSRE FUEL, POLAROGRAPHIC METHOD 65PRR-138
FLUORIDE IN MSRE FUEL, PYROLYSIS METHOD 65PRR-137
R SUL/ NICKEL, IN MOLTEN SALT REACTOR EXPERIMENT (MSRE) FUEL, SPECTROPHOTOMETRIC DI METHYL GLYOXIME (PE 65PRR-134
RMINATION OF URANIUM(VI) IN URANIUM OXIDE - THORIUM OXIDE FUELS DETE 65A-05-04G
LEAD-210 IN AUTO EXHAUST FUMES AND IN NATURAL MATERIALS 65A-11-02H
/ HELIUM-3 ACTIVATION ANALYSIS / SENSITIVITY / EXCITATION FUNCTIONS FOR HELIUM-3 INDUCED NUCLEAR REACTION/ 65A-11-08A
/ ORIFICE TEFLON DROPPING MERCURY ELECTRODE FOR OBTAINING FUNDAMENTAL POLAROGRAPHIC DATA / MONOVALENT THA/ 65A-05-03A
GLASSY CARBON INDICATOR ELECTRODE (G.C.E.) 65A-05-02A
TE ELECTRODE (P.G.E.) AND THE GLASSY CARBON ELECTRODE (G.C.E.) /L TITRIMETRY WITH THE PYROLYTIC GRAPHI 65A-05-02C
ABSOLUTE COUNTING OF GAMMA ACTIVITY 65PRR-188
HLORIC ACID GAS CHROMATOGRAPHIC ANALYSIS OF GAMMA IRRADIATED MIXTURES OF METHANOL AND HYDROC 65A-03-02
VI) IN PHOSPHORIC ACID SOLUTIONS OF / EFFECT OF COBALT-60 GAMMA RADIATION ON THE DETERMINATION OF URANIUM(65PRR-021
TRACE HIGH ENERGY GAMMA RADIOACTIVITY IN ISOTOPE PRODUCTS 65A-11-02E
GAMMA RADIOLYSIS OF ACID CHLORIDE SOLUTIONS 65A-03-01
CERIUM-141, AND NEODYMIUM-147 GAMMA-RAY BRANCHINGS IN THE DECAY OF INDIUM-116, 65PRR-169
GAMMA-RAY BRANCHINGS IN URANIUM-237 65A-11-01C
PROGRAM NETAREA (GAMMA-RAY PHOTOPEAK AREA) 65A-11-06E
ELECTRONIC RESOLUTION OF GAMMA-RAY SPECTRA 65PRR-168
ELECTRONIC GAMMA-RAY SPECTRAL RESOLUTION 65A-11-06A
ARTIFACTS IN GAMMA-RAY SPECTROMETRY 65A-11-03F
/XPERIMENTAL STUDY OF ARTIFACTS AND ERRORS ENCOUNTERED IN GAMMA-RAY SPECTROMETRY FOR RADIOCHEMISTRY AND A/ 65A-11-03E
/XPERIMENTAL STUDY OF ARTIFACTS AND ERRORS ENCOUNTERED IN GAMMA-RAY SPECTROMETRY FOR RADIOCHEMISTRY AND A/ 65PRR-003
EVALUATION OF COMPUTER PROGRAMS FOR GAMMA-RAY SPECTROMETRY IN ACTIVATION ANALYSIS 65A-11-06B
THE EVALUATION OF COMPUTER PROGRAMS FOR GAMMA-RAY SPECTROMETRY IN ACTIVATION ANALYSIS 65PRR-171
SURANIUM (TRU) PROCESS SOLUTIONS, MEASUREMENT OF PROMPT GAMMA-RAYS FROM SPONTANEOUS FISSION /252 IN TRAN 65PRR-130
MIXTURES OF METHANOL AND HYDROCHLORIC ACID GAS CHROMATOGRAPHIC ANALYSIS OF GAMMA IRRADIATED 65A-03-02
THE REACTION OF IODINE WITH CYCLOHEXANE AND WITH BENZENE/ GAS CHROMATOGRAPHIC ANALYSIS OF THE PRODUCTS OF 65A-04-04C
ASES IN HELIUM AT REDUCED SAMPLE PRESSURES GAS CHROMATOGRAPHIC DETERMINATION OF PERMANENT G 65PRR-007
IELECTRIC FILM PYROLYSIS AND GAS CHROMATOGRAPHIC DETERMINATION OF STYROFOAM D 65A-05-01B
NE VAPORS GAS CHROMATOGRAPHIC IDENTIFICATION OF RADIO IODI 65PRR-062
NE VAPORS GAS CHROMATOGRAPHIC IDENTIFICATION OF RADIO IODI 65PRR-064
GAS CHROMATOGRAPHY 65A-05-01
GAS CHROMATOGRAPHY 65PRR-200
NUCLEAR SAFETY PROGRAM (IODINE / METHYL IODIDE / GAS CHROMATOGRAPHY) 65A-04-04A
APPLICATIONS OF GAS CHROMATOGRAPHY IN NUCLEAR TECHNOLOGY 65PRR-180
NES AND PYRIMIDINES GAS CHROMATOGRAPHY OF PYROLYTIC PRODUCTS OF PURI 65A-06-03
ANE SYSTEM RADIO GAS CHROMATOGRAPHY OF THE TRITIUM - WATER - METH 65A-05-01A
GAS CHROMATOGRAPHIC DETERMINATION OF PERMANENT GASES IN HELIUM AT REDUCED SAMPLE PRESSURES 65PRR-007
ROMIUM(II) SOLUTIONS INTEGRATED ASSEMBLY FOR GENERATION, STORAGE, AND DELIVERY OF STANDARD CH 65PRR-055
14-MEV NEUTRON GENERATOR 65A-11-09
MONITORING OF LEACHED FUEL ELEMENTS WITH A NEUTRON GENERATOR 65A-11-09F
CTIVATION ANALYSIS OUTPUT SPECTRA OF 14-MEV NEUTRON GENERATORS - RAPID ESTIMATION AND INFLUENCE IN A 65PRR-044
ROSS SECTION MEASURE/ OUTPUT SPECTRUM FROM 14-MEV NEUTRON GENERATORS - RAPID ESTIMATION AND INFLUENCE IN C 65PRR-043
MENT GEOLOGICAL AGE DETERMINATION BY ISOTOPIC MEASURE 65PRR-215
LLARIES SIGNAL TO NOISE RATIOS (S/N) OF GLASS AND TEFLON DROPPING MERCURY ELECTRODE CAPI 65A-05-03B
INSTRUMENTATION FOR POLAROGRAPHY OF GLASS CORRODING MEDIA 65PRR-195
/LECTRODE FOR POLAROGRAPHY IN HYDROFLUORIC ACID AND OTHER GLASS CORRODING MEDIA - EVALUATION WITH MONOVAL/ 65PRR-039
/F TEFLON FOR POLAROGRAPHY IN HYDROFLUORIC ACID AND OTHER GLASS CORRODING MEDIA - EVALUATION WITH THE DIV/ 65PRR-040
NTAL ORIFICE/ POLAROGRAPHY IN HYDROFLUORIC ACID AND OTHER GLASS CORRODING MEDIA (EVALUATION OF THE HORIZO 65A-05-03A

WITH THE PYROLYTIC GRAPHITE ELECTRODE (P.G.E.) AND THE GLASSY CARBON ELECTRODE (G.C.E.) /L TITRIMETRY 65A-05-02C
A GLASSY CARBON ELECTRODE FOR VOLTAMMETRY 65PRR-061
GLASSY CARBON INDICATOR ELECTRODE (G.C.E.) 65A-05-02A
/TURE, OXYGEN, HYDROGEN, NITROGEN, AND CARBON DI OXIDE IN GLOVE BOX ATMOSPHERES ON THE ANALYSIS OF THE AL/ 65PRR-175
OR EXPERIMENT (MSRE) FUEL, SPECTROPHOTOMETRIC DI METHYL GLYOXIME (PER SULFATE OXIDATION) METHOD /REACT 65PRR-134
C FOILS GOLD ATOMS SPUTTERED FROM NEUTRON IRRADIATED GOL 65A-11-08F
/CTRON PROBE ANALYZER / DETERMINATION OF AREAL DENSITY OF GOLD COATING ON SILICON RADIATION DETECTORS BY / 65A-07-01A
GOLD ATOMS SPUTTERED FROM NEUTRON IRRADIATED GOLD FOILS 65A-11-08F
ATOMIC ABSORPTION (DETERMINATION OF GOLD SILVER AND COPPER) 65A-07-01B
E MEASUREMENTS (CESIUM-137 / SAMARIUM-153 / ARSENIC-77 / GOLD-198 / IODINE-133 / PHOSPHORUS-33) HALF-LIF 65A-11-030
PREPARATION AND RECOVERY OF CESIUM COMPOUNDS FOR DENSITY GRADIENT SOLUTIONS 65PRR-116
ELECTROCHEMISTRY WITH THE PYROLYTIC GRAPHITE ELECTRODE 65PRR-194
OXIDE FILM FORMATION ON THE PYROLYTIC GRAPHITE ELECTRODE (P.G.E.) 65A-05-02B
BON ELECTRODE/ DIFFERENTIAL TITRIMETRY WITH THE PYROLYTIC GRAPHITE ELECTRODE (P.G.E.) AND THE GLASSY CAR 65A-05-02C
UORIDE - SODIUM FLUORIDE - POTASSIUM FLUORIDE / PYROLYTIC GRAPHITE ELECTRODE / SILVER ELECTRODE) /HIUM FL 65A-02-02
FILM FORMATION ON PYROLYTIC GRAPHITE ELECTRODES 65PRR-029
TAMMETRY OF IODIDE, IODINE, AND IODATE WITH THE PYROLYTIC GRAPHITE INDICATING ELECTRODE VOL 65PRR-193
ACERS GRAVIMETRIC DETERMINATION OF SODIUM WITH RADIOTR 65PRR-050
GROSS ALPHA 65PRR-150
NUCLEAR SPECTROSCOPY OF NEUTRON DEFICIENT HAFNIUM AND RARE EARTH RADIONUCLIDES 65A-11-01E
TRACE ELEMENTS IN HUMAN HAIR 65PRR-009
NEUTRON ACTIVATION ANALYSIS OF HUMAN HAIR - FORENSIC APPLICATIONS 65A-11-08D
MOVAL AND CHARACTERIZATION OF THE SURFACE CONTAMINANTS OF HAIR /TIVATION ANALYSIS IN PROCEDURES FOR THE RE 65PRR-008
DETERMINATION OF MOLYBDENUM IN HAIR AND WOOL BY NEUTRON ACTIVATION ANALYSIS 65PRR-018
53 / ARSENIC-77 / GOLD-198 / IODINE-133 / PHOSPHORUS-33) HALF-LIFE MEASUREMENTS (CESIUM-137 / SAMARIUM-1 65A-11-03D
ICNUCLIDES HALF-LIVES AND SPECIFIC ACTIVITIES OF COMMON RAD 65PRR-078
E-36 BERYLLIUM-10) HALF-LIVES OF LONG-LIVED BETA EMITTERS (CHLORIN 65A-11-010
/IGNIFICANCE OF JUNCTION POTENTIALS IN THE MEASUREMENT OF HALF-WAVE POTENTIAL VALUES / SENSITIVITY LIMIT / 65A-05-03A
/NTIALS AT THE SALT BRIDGE ON THE MEASURED VALUES FOR THE HALF-WAVE POTENTIALS OF THE DIVALENT LEAD TO LE/ 65PRR-010
BENZYL TRI BUTYL PHOSPHONIUM CHL/ QUATERNARY PHOSPHONIUM HALIDES (BENZYL TRI BUTYL PHOSPHONIUM BROMIDE / 65A-13-01A
/ MODEL-Q-2752 (CYCLIC VOLTAMMETRY / CHRONOAMPEROMETRY / HAMMER-CONTROL OF DROPPING MERCURY ELECTRODE DR/ 65A-01-04
HE NATURE OF THE POLAROGRAPHIC CURRENT IN RELATION TO THE HEIGHT OF A MERCURY COLUMN /HOD OF DETERMINING T 65TR-11
GAS CHROMATOGRAPHIC DETERMINATION OF PERMANENT GASES IN HELIUM AT REDUCED SAMPLE PRESSURES 65PRR-007
HELIUM-3 ACTIVATION ANALYSIS 65PRR-198
S AS NEUTRON S/ THEORY AND EXPERIMENT IN RAPID, SENSITIVE HELIUM-3 ACTIVATION ANALYSIS - HELIUM-3 REACTION 65PRR-045
ITATION FUNCTION/ STUDIES OF HELIUM-3 NUCLEAR REACTIONS (HELIUM-3 ACTIVATION ANALYSIS / SENSITIVITY / EXC 65A-11-08A
/VATION ANALYSIS / SENSITIVITY / EXCITATION FUNCTIONS FOR HELIUM-3 INDUCED NUCLEAR REACTIONS / CROSS SECT/ 65A-11-08A
ACTIVATION ANALYSIS WITH 14-MEV NEUTRONS AND WITH HELIUM-3 IONS 65PRR-176
ANALYSIS / SENSITIVITY / EXCITATION FUNCTION/ STUDIES OF HELIUM-3 NUCLEAR REACTIONS (HELIUM-3 ACTIVATION 65A-11-08A
RIMENT IN RAPID, SENSITIVE HELIUM-3 ACTIVATION ANALYSIS - HELIUM-3 REACTIONS AS NEUTRON SOURCES / AND EXPE 65PRR-045
HEPTA DECYL AMINE 65A-13-01E
SPECTROPHOTOMETRIC HETEROPOLY-BLUE DETERMINATION OF SILICON 65A-05-04I
TANTS FOR METAL IONS - SELECTIVE EXTRACTIO/ TRI ALKYL AND HEXA ALKYL PHOSPHORO THIOIC TRI AMIDES AS EXTRAC 65PRR-015
/XIDE / POTASSIUM OXIDE / LITHIUM HYPOBROMITE / POTASSIUM HEXA CHLORO RHENATE(IV) / HOLMIUM SULFIDE / ERB/ 65A-12
/SODIUM SUPEROXIDE / BARIUM BROMITE MONO HYDRATE / SODIUM HEXA CHLORO RHENATE(IV) / URANIUM-235 TRI CHLOR/ 65A-12
ATION OF OXIDIZING POWER OF SOLUTIONS OF HYDROLYZED XENON HEXA FLUORIDE DETERMIN 65A-05-04L
TANT FOR METAL IONS N,N,N TRI HEXYL PHOSPHORIC TRI AMIDE (THPA) AS AN EXTRAC 65A-11-04A
/MS FOR ANALYSIS OF POLAROGRAMS AND CHRONOPOTENTIOGRAMS (HEYROVSKY-ILKOVIC EQUATION / POLARGRM / CHRNOGR/ 65A-01-13
/ AMPEROMETRIC DC POLAROGRAPHY - A NEW HIGH PRECISION AND HIGH ACCURACY METHOD (ELECTROCHEMISTRY / MODEL/ 65A-01-03
CTS TRACE HIGH ENERGY GAMMA RADIOACTIVITY IN ISOTOPE PRODU 65A-11-02E
HIGH LEVEL ALPHA RADIATION LABORATORY 65B-17-01
/ METAL IONS FROM AQUEOUS SOLUTIONS OF ORGANIC ACIDS WITH HIGH MOLECULAR WEIGHT AMINES - THE TRIVALENT AC/ 65PRR-031
DCHEMIS/ COMPARATIVE AMPEROMETRIC DC POLAROGRAPHY - A NEW HIGH PRECISION AND HIGH ACCURACY METHOD (ELECTR 65A-01-03
APPLICATIONS OF ORNL SPARK SOURCE MASS SPECTROGRAPH TO HIGH PURITY MATERIALS ANALYSIS 65PRR-201
HIGH PURITY POTASSIUM CHLORIDE 65PRR-090
) HIGH RADIATION LEVEL ANALYTICAL FACILITY (HRLAF 65B-17-05
AL) HIGH RADIATION LEVEL ANALYTICAL LABORATORY (HRL 65B-17-03
C TITRATOR - CONTROLLED POTENTIAL COULOMETRIC DETERMINAT/ HIGH SENSITIVITY CONTROLLED POTENTIAL COULOMETRI 65PRR-020
64 HIGH SENSITIVITY COULOMETRIC TITRATOR MODEL-Q-25 65PRR-118
L-Q-2564 (POTENTIOSTAT / CONTROLLED POTENTIAL / BLANK C/ HIGH SENSITIVITY COULOMETRIC TITRATOR, ORNL MODE 65A-01-05
/IUM IN THE MOLTEN SALT REACTOR EXPERIMENT (MSRE) FUEL, HIGH SENSITIVITY, AUTOMATIC, CONTROLLED POTENTI/ 65PRR-122
ING CONDUCTOMETRIC TITRATOR WITH AUTOMATIC TEMPERATURE C/ HIGH SENSITIVITY, DIRECT READING, LINEAR, RECORD 65PRR-036
COULOMETRIC TITRATOR, CONTROLLED POTENTIAL, HIGH SENSITIVITY, ORNL MODEL-Q-2564 65PRR-125
TROMETRIC APPLICATION A PROGRAMMABLE HIGH SPEED DATA ACQUISITION SYSTEM FOR MASS SPEC 65PRR-192
HIGH TEMPERATURE CHEMICAL DOSIMETRY 65A-03-03
HIGH VOLTAGE PAPER ELECTROPHORESIS 65A-11-040
// ERBIUM SULFIDE / THULIUM PHOSPHIDE / THULIUM NITRIDE / HOLMIUM NITRIDE / POTASSIUM SUPEROXIDE / RUBIDI/ 65A-12
/ITHIUM HYPOBROMITE / POTASSIUM HEXA CHLORO RHENATE(IV) / HOLMIUM SULFIDE / ERBIUM SULFIDE / THULIUM PHOS/ 65A-12
TISSUE ANALYSIS (TISSUE HOMOGENIZATION) 65A-07-01E
/ACID AND OTHER GLASS CORRODING MEDIA (EVALUATION OF THE HORIZONTAL ORIFICE TEFLON DROPPING MERCURY ELEC/ 65A-05-03A
/D METAL REDOX REACTION IN AQUEOUS HYDROFLUORIC ACID WITH HORIZONTAL ORIFICE TEFLON DROPPING MERCURY ELEC/ 65A-05-03A
TUS FOR THE MOLTEN SALT REACTOR EX/ ANALYTICAL LABORATORY HOT CELL EQUIPMENT I. SAMPLE PREPARATION APPARA 65PRR-027
RROSION RESISTANT PIPETTER ANALYTICAL LABORATORY HOT CELL EQUIPMENT II. A REMOTELY CONTROLLED CO 65PRR-028
TER PHOTOMETER ANALYTICAL LABORATORY HOT CELL EQUIPMENT III. A REMOTELY OPERATED FIL 65PRR-026
HIGH RADIATION LEVEL ANALYTICAL FACILITY (HRLAF) 65B-17-05
HIGH RADIATION LEVEL ANALYTICAL LABORATORY (HRLAL) 65B-17-03
TRACE ELEMENTS IN HUMAN HAIR 65PRR-009
NEUTRON ACTIVATION ANALYSIS OF HUMAN HAIR - FORENSIC APPLICATIONS 65A-11-080
/HANUM CHLORIDE / SODIUM SUPEROXIDE / BARIUM BROMITE MONO HYDRATE / SODIUM HEXA CHLORO RHENATE(IV) / URAN/ 65A-12
HIC ANALYSIS OF GAMMA IRRADIATED MIXTURES OF METHANOL AND HYDROCHLORIC ACID GAS CHROMATOGRAP 65A-03-02
ON CONCENTRATED SOLUTIONS OF LITHIUM CHLORIDE CONTAINING HYDROCHLORIC ACID ALPHA RADIATION EFFECTS 65PRR-165
AD METAL REDOX REACTION REDUCTION IN AQUEOUS SOLUTIONS OF HYDROCHLORIC ACID AND POTASSIUM CHLORIDE / TO LE 65PRR-010
MERCURY ELECTRODE O/ POLAROGRAPHY OF LEAD(II) IN AQUEOUS HYDROFLUORIC ACID (1-12-MOLAR) WITH A DROPPING 65PRR-041
A (EVALUATION OF THE HORIZONTAL ORIFICE/ POLAROGRAPHY IN HYDROFLUORIC ACID AND OTHER GLASS CORRODING MEDI 65A-05-03A
A / TEFLON DROPPING MERCURY ELECTRODE FOR POLAROGRAPHY IN HYDROFLUORIC ACID AND OTHER GLASS CORRODING MEDI 65PRR-039
DROPPING MERCURY ELECTRODE OF TEFLON FOR POLAROGRAPHY IN HYDROFLUORIC ACID AND OTHER GLASS CORRODING MEDI 65PRR-040
/OF DIVALENT LEAD TO LEAD METAL REDOX REACTION IN AQUEOUS HYDROFLUORIC ACID WITH HORIZONTAL ORIFICE TEFLO/ 65A-05-03A
ALKALI METAL/ ANALYSIS OF THE ALKALI METALS (FOR CARBON HYDROGEN AND OXYGEN / DETERMINATION OF OXYGEN IN 65A-04-03A
/ RAPID DETERMINATION OF LOW CONCENTRATIONS OF OXYGEN AND HYDROGEN IN ALKALI MATERIALS BY A MODIFIED AMAL/ 65PRR-073
BO/ THE EFFECTS OF TRACE IMPURITIES OF MOISTURE, OXYGEN, HYDROGEN, NITROGEN, AND CARBON DI OXIDE IN GLOVE 65PRR-175
ITHIUM CHLORIDE SOLUTION - DETERMINATION OF FREE ACID AND HYDROLYZABLE CATION REACTIONS IN CONCENTRATED L 65PRR-022
DETERMINATION OF OXIDIZING POWER OF SOLUTIONS OF HYDROLYZED XENON HEXA FLUORIDE 65A-05-04L
G AND WEAK ACIDS IN THEIR MIXTURES, POTENTIOMETRIC SODIUM HYDROXIDE TITRATION METHOD STRON 65PRR-127

/DIOACTIVITY IN TRANSURANIUM (TRU) PROCESS SOLUTIONS, 8
/SIUM CHLORIDE / SODIUM OXIDE / POTASSIUM OXIDE / LITHIUM
/TENTIAL COULOMETRY / MODEL-Q-2564 COULOMETRIC TITRATOR /
TRY (COULOMETRIC DETERMINATION OF PLUTONIUM) UTILITY OF
OR FORMS OF RADIO IODINE
THE NEW
OPIC COMPARISON OF LEAD FROM TEKTITES AND CONTEMPORANEOUS
REACTION OF METHYL IODIDE WITH CHEMICALLY
POLAROGRAPHY WITH A TRIANGULAR POTENTIAL
EN, AND CARBON DI OXIDE IN GLOVE BO/ THE EFFECTS OF TRACE
CUMULATIVE
953-1964)
GLASSY CARBON
STUDIES WITH SOLID
CHLORIDE, DISTILLATION -
OXALATE IN ELUATES FROM IRRADIATED ION EXCHANGE RESINS,
GAMMA-RAY BRANCHINGS IN THE DECAY OF
N-10
SYNTHESIS AND
ETRY
OF L LEUCINE, L ISOLEUCINE, AND L VALINE IN A MIXTURE BY
RCUITS AND EXAMPLES OF THE USE OF A POTENTIOSTAT
THE
THERMAL NEUTRON ACTIVATION ANALYSIS OF TECHNETIUM
NC CHLORIDE / CESIUM CHLORIDE / SODIUM OXIDE / POTASSIUM/
OF RADIATION ON ANALYTICAL METHODS / REMOTELY CONTROLLED
/CONTROLLED POTENTIAL VOLTAMMETER (DERIVATIVE COMPUTER /
ANALYTICAL
A SURVEY OF ANALYTICAL
ING MEDIA
DELIVERY OF STANDARD CHROMIUM(II) SOLUTIONS
DIFFERENTIAL POLAROGRAPHY ON AN ELECTRONIC SELF-RECORDING
ACTIVATION ANALYSIS - REACTOR NEUTRON FLUXES AND
RRECTION IN LIQUID SCINTILLATOR SYSTEMS USING AN ISOLATED
TION IN LIQUID SCINTILLATOR SYSTEMS BY USE OF AN ISOLATED
HOD OF/ SEMIQUANTITATIVE SPECTROGRAPHIC ANALYSIS (VISUAL
ECTRODE
VOLTAMMETRY OF IODIDE, IODINE, AND
ITE IN A NEUTRAL NITRATE CONTAINING SOLUTION OF POTASSIUM
NUCLEAR SAFETY PROGRAM (IODINE / METHYL
/BICIUM SUPEROXIDE / URANIUM(III) CHLORIDE / URANIUM(III)
REACTION OF METHYL
APHITE INDICATING ELECTRODE
VOLTAMMETRY OF
, CHARACTER AND CHEMICAL BEHAVIOR OF VAPOR FORMS OF RADIO
NUCLEAR SAFETY PROGRAM (
DETERMINATION OF
CKS BY MEANS OF COMPOSITE DIFFUSI/ INVESTIGATION OF RADIO
SPECTROPHOTOMETRIC STUDY OF THE STABILITY OF
ICNS
UBES
INVESTIGATION OF AIRBORNE RADIO
SITE DIFFUSION TUBES
CHEMICAL BEHAVIOR OF THE RADIO
VOLTAMMETRY OF THE
CHRONOPOTENTIOMETRY OF THE
/VESTIGATION OF CHEMICAL REACTIVITY OF NONELEMENTAL RADIO
GAS CHROMATOGRAPHIC IDENTIFICATION OF RADIO
EFFECTS OF SURFACE MATERIALS ON RADIO
GAS CHROMATOGRAPHIC IDENTIFICATION OF RADIO
/OMATOGRAPHIC ANALYSIS OF THE PRODUCTS OF THE REACTION OF
NDICATING ELECTRODE
VOLTAMMETRY OF IODIDE,
NTS (CESIUM-137 / SAMARIUM-153 / ARSENIC-77 / GOLD-198 /
CHLORIDE, DISTILLATION - INDIRECT
IN ELUATES FROM IRRADIATED ION EXCHANGE RESINS, INDIRECT
EQUILIBRIUM DISTRIBUTION OF METAL -
ON METHOD
OXALATE IN ELUATES FROM IRRADIATED
SSIUM FLUORIDE
ABSORPTION SPECTRUM OF COBALT(II)
UTION OF THE OVERL/ THE ABSORPTION SPECTRUM OF THE URANYL
SURFACE
CTIVATION ANALYSIS WITH 14-MEV NEUTRONS AND WITH HELIUM-3
PHOSPHORIC TRI AMIDE (THPA) AS AN EXTRACTANT FOR METAL
/KYL PHOSPHORO THIOIC TRI AMIDES AS EXTRACTANTS FOR METAL
H HIGH MOLECULAR WEIGH/ LIQUID-LIQUID EXTRACTION OF METAL
/PPLICATION OF QUASI REFERENCE ELECTRODE / ELIMINATION OF
O/ VOLTAMMETRY OF MOLTEN FLUORIDE SALT SYSTEMS (SILVER /
TION OF MILLIGRAM AND MICROGRAM QUANTITIES OF URANIUM AND
DETERMINATION OF TRACE METALS IN NIOBIUM -
/UM BY CONTROLLED POTENTIAL COULOMETRY / DETERMINATION OF
HOD
IN AUTOMOTIVE ENGINES BY LIQUID SCINTILLATION COUNTING OF
IN AUTOMOTIVE ENGINES BY LIQUID SCINTILLATION COUNTING OF
GOLD ATOMS SPUTTERED FROM NEUTRON
RIC TITRATION METHOD
OXALATE IN ELUATES FROM
- ZIRCONIUM FLUOR/ ABSORPTION AND REFLECTANCE SPECTRA OF
ACID
GAS CHROMATOGRAPHIC ANALYSIS OF GAMMA
CTS OF RADIATION ON ANALYTICAL METHODS / REMOTELY/ SAMPLE
C SPECTROMETRY
THE RAPID DETERMINATION OF L LEUCINE, L
A NEW
TRACE HIGH ENERGY GAMMA RADIOACTIVITY IN
AND THE PURIFICATI/ EVIDENCE OF THE ABSENCE OF LONG-LIVED
COOPERATIVE
HYDROXY QUINOLINE PRECIPITATION - BETA COUNTING/ 65PRR-129
HYPOBROMITE / POTASSIUM HEXA CHLORO RHENATE(IV)/ 65A-12
I-VERSUS-Q COULOMETRIC METHOD / CONTROLLED POTE/ 65A-01-07
I-VERSUS-Q PLOTS IN CONTROLLED POTENTIAL COULOME 65A-01-08
IDENTITY, CHARACTER AND CHEMICAL BEHAVIOR OF VAP 65PRR-162
IMAGE OF ANALYTICAL CHEMISTRY IN SCIENCE 65PRR-221
IMPACT CRATERS ISOT 65A-08-04
IMPREGNATED CHARCOAL 65PRR-065
IMPULSE 65TR-05
IMPURITIES OF MOISTURE, OXYGEN, HYDROGEN, NITROG 65PRR-175
INDEXES TO THE ORNL MASTER ANALYTICAL MANUAL 65C-18-01
INDEXES TO THE ORNL MASTER ANALYTICAL MANUAL (I 65PRR-093
INDICATOR ELECTRODE (G.C.E.) 65A-05-02A
INDICATOR ELECTRODES 65A-05-02
INDIRECT IODOMETRIC TITRATION METHOD 65PRR-126
INDIRECT IODOMETRIC TITRATION METHOD 65PRR-158
INDIUM-116, CERIUM-141, AND NEODYMIUM-147 65PRR-169
INFRARED ABSORPTION SPECTRUM OF DI BORANE - BORO 65PRR-079
INFRARED AND NUCLEAR MAGNETIC RESONANCE SPECTROM 65A-09
INFRARED SPECTROMETRY 65A-09-01
INFRARED SPECTROMETRY THE RAPID DETERMINATION 65TR-09
INNER RESISTANCE OF POTENTIOSTATIC REGULATING CI 65TR-07
INORGANIC AND NUCLEAR CHEMISTRY OF TECHNETIUM - 65PRR-077
INORGANIC PREPARATIONS (MAGNESIUM CHLORIDE / ZI 65A-12
INSTRUMENT) /BLY FOR COBALT-60 SOURCE (EFFECTS 65A-01-12
INSTRUMENT DESIGN / ELECTROCHEMISTRY / ANALYSIS/ 65A-01-11
INSTRUMENTATION 65A-01
INSTRUMENTATION DEVELOPMENT AT ORNL 65PRR-203
INSTRUMENTATION FOR POLAROGRAPHY OF GLASS CORROD 65PRR-195
INTEGRATED ASSEMBLY FOR GENERATION, STORAGE, AND 65PRR-055
INTEGRO - DIFFERENTIATING POLAROGRAPH 65TR-02
INTERACTIONS OF CADMIUM-106 WITH ALPHA PARTICLES 65PRR-179
INTERFERENCES 65PRR-197
INTERNAL STANDARD COLOR QUENCH CO 65PRR-047
INTERNAL STANDARD COLOR QUENCH CORREC 65A-11-05B
INTERPOLATION) (LITHIUM CARBONATE DILUTION MET 65A-07-01F
IODATE WITH THE PYROLYTIC GRAPHITE INDICATING EL 65PRR-193
IODIDE DETERMINATION OF NITR 65A-05-04D
IODIDE / GAS CHROMATOGRAPHY) 65A-04-04A
IODIDE / SOLID SOLUTIONS OF URANIUM(III) CHLORI/ 65A-12
IODIDE WITH CHEMICALLY IMPREGNATED CHARCOAL 65PRR-065
IODIDE, IODINE, AND IODATE WITH THE PYROLYTIC GR 65PRR-193
IODINE IDENTITY 65PRR-162
IODINE / METHYL IODIDE / GAS CHROMATOGRAPHY) 65A-04-04A
IODINE AND FLUORINE IN ,KIO2F2 65A-05-04F
IODINE BEHAVIOR UNDER MOIST CONDITIONS IN MAY PA 65PRR-066
IODINE IN CYCLOHEXANE AND IN BENZENE 65A-04-04B
IODINE RADIOACTIVITY IN AQUEOUS OR ORGANIC SOLUT 65PRR-149
IODINE SPECIES BY MEANS OF COMPOSITE DIFFUSION T 65PRR-067
IODINE SPECIES FOUND ON RUBBER SECTIONS OF COMPO 65PRR-069
IODINE SYSTEM 65A-05-02D
IODINE SYSTEM 65A-05-02E
IODINE VAPOR BY MEANS OF COMPOSITE DIFFUSION TU/ 65PRR-068
IODINE VAPORS 65PRR-062
IODINE VAPORS 65PRR-063
IODINE VAPORS 65PRR-064
IODINE WITH CYCLOHEXANE AND WITH BENZENE AT 300/ 65A-04-04C
IODINE, AND IODATE WITH THE PYROLYTIC GRAPHITE I 65PRR-193
IODINE-123 65A-11-01A
IODINE-131 PRODUCTS 65A-11-02B
IODINE-131, PRODUCT ANALYSIS GUIDE 65PRR-153
IODINE-133 / PHOSPHORUS-33) HALF-LIFE MEASUREME 65A-11-03D
IODOMETRIC TITRATION METHOD 65PRR-126
IODOMETRIC TITRATION METHOD OXALATE 65PRR-158
ION COMPLEXES 65PRR-074
ION EXCHANGE RESINS, INDIRECT IODOMETRIC TITRATI 65PRR-158
ION IN LITHIUM FLUORIDE - SODIUM FLUORIDE - POTA 65A-02-01B
ION IN PER CHLORATE MEDIA I. MATHEMATICAL RESOL 65PRR-161
IONIZATION STUDIES 65A-08-03
IONS A 65PRR-176
IONS N,N,N TRI HEXYL 65A-11-04A
IONS - SELECTIVE EXTRACTION OF MERCURY(II), SIL/ 65PRR-015
IONS FROM AQUEOUS SOLUTIONS OF ORGANIC ACIDS WIT 65PRR-031
IR LOSS IN POLAROGRAPHY / POLAROGRAPHIC DETECTI/ 65A-01-06
IRON / NICKEL / LITHIUM FLUORIDE - BERYLLIUM FLU 65A-02-02
IRON /CONTROLLED POTENTIAL COULOMETRIC DETERMINA 65PRR-020
IRON ALLOY 65A-05-04B
IRON BY CONTROLLED POTENTIAL COULOMETRY / MODEL/ 65A-01-07
IRON, 1,10 PHENANTHROLINE SPECTROPHOTOMETRIC MET 65PRR-133
IRON-55 WEAR RATES 65PRR-048
IRON-55 MEASUREMENT OF WEAR RATES 65A-11-07C
IRRADIATED GOLD FOILS 65A-11-08F
IRRADIATED ION EXCHANGE RESINS, INDIRECT IODOMET 65PRR-158
IRRADIATED LITHIUM FLUORIDE - BERYLLIUM FLUORIDE 65-02-01A
IRRADIATED MIXTURES OF METHANOL AND HYDROCHLORIC 65A-03-02
IRRADIATION ASSEMBLY FOR COBALT-60 SOURCE (EFFE 65A-01-12
ISOLEUCINE, AND L VALINE IN A MIXTURE BY INFRARE 65TR-09
ISOMER OF BROMINE - BROMINE-82M 65PRR-013
ISOTOPE PRODUCTS 65A-11-02E
ISOTOPES OF PROMETHIUM FROM FISSION OF URANIUM, 65PRR-089
ISOTOPES PROGRAM 65A-11-02

SS SPECTROMETRY ISCTCPIC ANALYSIS OF TRANSURANIUM ELEMENTS BY MA 65PRR-167
NTEMPORANEOUS IMPACT CRATERS ISCTCPIC COMPARISON OF LEAC FROM TEKTITES AND CO 65A-08-04
THE ISCTCPIC COMPOSITICN OF ATMOSPHERIC NECN 65PRR-071
THE ISCTCPIC COMPOSITION OF ATMOSPHERIC NECN 65PRR-208
GEOLOGICAL AGE OETERMINATICN BY ISCTCPIC MEASUREMENT 65PRR-215
THE USE OF ISCTCPIC NEUTRCN SOURCES FOR CHEMICAL ANALYSIS 65PRR-006
/ELOPMENT GROUP MCNTHLY SUMMARY - NOV.,1964 / DEC.,1964 / JAN.,1965 / FEB.,1965 / MAR.,1965 / APR.,1965 // 65PRR-104
AL QUALITY CCNTRCL REPORT, ANALYTICAL CHEMISTRY OIVISICN, JAN.-DEC.,1964 STATISTIC 65PRR-083
STATISTICAL QUALITY CCNTROL REPORT, JAN.-MAR.,1965, ANALYTICAL CHEMISTRY DIVISION 65PRR-082
STATISTICAL QUALITY CCNTROL REPORT, JAN.-SEPT.,1964, ANALYTICAL CHEMISTRY DIVISION 65PRR-084
REPORT CN FOREIGN TRAVEL JAN.16-FEB.20,1965 65PRR-101
EB.,1965 / MAR.,1965 / APR.,1965 / MAY,1965 / JUNE,1965 / JULY,1965 / AUG.,1965 / SEPT.,1965 / OCT.,1965 / 65PRR-104
ASURED VALUES FOR THE HALF-WAVE POTENTIALS OF / EFFECT OF JUNCTION POTENTIALS AT THE SALT BRIDGE ON THE ME 65PRR-010
/FICE TEFLON DROPPING MERCURY ELECTRODE / SIGNIFICANCE OF JUNCTION POTENTIALS IN THE MEASUREMENT OF HALF-/ 65A-05-03A
/N.,1965 / FEB.,1965 / MAR.,1965 / APR.,1965 / MAY,1965 / JUNE,1965 / JULY,1965 / AUG.,1965 / SEPT.,1965 / 65PRR-104
REPORT CN FOREIGN TRAVEL JUNE-25-JULY-30,1965 65PRR-072
REPORT CN FOREIGN TRAVEL JUNE-30-JULY-10,1965 65PRR-070
RAMS (HEYROVSKY-ILKOVIC EQUATION / POLARGRM / CHRNOGRM / KARACGLANOFF EQUATION) /AMS AND CHRONOPOTENTIOG 65A-01-13
RED SPECTROMETRY THE RAPID DETERMINATION OF L LEUCINE, L ISCLEUCINE, ANC L VALINE IN A MIXTURE BY INFRA 65TR-09
RE BY INFRARED SPECTROMETRY THE RAPID DETERMINATICN OF L LEUCINE, L ISCLEUCINE, AND L VALINE IN A MIXTU 65TR-09
THE RAPID DETERMINATICN CF L LEUCINE, L ISOLEUCINE, AND L VALINE IN A MIXTURE BY INFRARED SPECTROMETRY 65TR-09
H HIGH MOLECULAR WEIGHT AMINES - THE TRIVALENT ACTINIDE - LANTHANIDE ELEMENTS /UTIONS OF ORGANIC ACIDS WIT 65PRR-031
RCM AQUEOUS SOLUTICNS PRIOR TO THE DETERMINATICN OF TOTAL LANTHANIDE FISSICN PRODUCTS /SURANIUM ELEMENTS F 65PRR-032
/II) IODIDE / SOLID SOLLTICNS OF LRANIUM(III) CHLORIDE IN LANTHANUM CHLORIDE / SODIUM SUPEROXIDE / BARIUM/ 65A-12
THE LASER IN METALLURGICAL SPECTROSCOPY 65PRR-174
NS OF URANIUM - A COMPARISCN OF/ COMMENTS CN THE PAPER BY LAUER, BRUCK, ANC LEDUIGOU, PRECISE DETERMINATIO 65PRR-098
N OF URANIUM - A COMPARISCN OF / COMMENTS CN THE PAPER BY LAUER, BRUCK, ANC LEDUIGOU, PRECISE DETERMINATIO 65PRR-202
/UARE-WAVE POLARCGRAPH WITH SOLID STATE CIRCUITS / DOUBLE LAYER CAPACITANCE MEASUREMENT / TEFLON DROPPING/ 65A-01-14
MCNITORING OF LEACHED FUEL ELEMENTS WITH A NEUTRON GENERATOR 65A-11-09F
RADIOTRACER METHOD FOR THE DETERMINATICN OF LEAD 65A-11-07D
ATERS ISOTOPIC COMPARISCN OF LEAD FROM TEKTITES ANC CONTEMPORANEOUS IMPACT CR 65A-08-04
/LIUM TO THALLIUM METAL REDOX REACTION / DIVALENT LEAD TO LEAD METAL REDCX REACTION / POLAROGRAPHY OF DIV/ 65A-05-03A
SS CORRODING MEDIA - EVALUATION WITH THE DIVALENT LEAD TO LEAD METAL REOCX REACTION /IC ACID AND OTHER GLA 65PRR-040
/ METAL REDOX REACTION / POLAROGRAPHY OF DIVALENT LEAD TO LEAD METAL REDCX REACTION IN AQUEOUS HYDROFLUOR/ 65A-05-03A
/UES FOR THE HALF-WAVE POTENTIALS OF THE DIVALENT LEAD TO LEAD METAL REDCX REACTION REDUCTION IN AQUEOUS / 65PRR-010
/ENT THALLIUM TO THALLIUM METAL REDOX REACTION / DIVALENT LEAD TO LEAC METAL REDCX REACTION / POLAROGRAPH/ 65A-05-03A
THER GLASS CORRODING MEDIA - EVALUATION WITH THE DIVALENT LEAD TO LEAC METAL REDCX REACTION /IC ACID AND O 65PRR-040
/ TO LEAC METAL REDCX REACTICN / POLAROGRAPHY CF DIVALENT LEAD TC LEAC METAL REDCX REACTION IN AQUEOUS HY/ 65A-05-03A
/URED VALUES FOR THE HALF-WAVE POTENTIALS OF THE DIVALENT LEAD TC LEAC METAL REDCX REACTION REDUCTION IN / 65PRR-010
E DUST SAMPLES DETERMINATICN OF LEAD, BISMUTH, CADMIUM, TIN, AND ZINC IN AIRBORN 65A-07-02A
TERIALS LEAD-210 IN AUTC EXHAUST FUMES AND IN NATURAL MA 65A-11-02H
AR) WITH A DROPPING MERCURY ELECTRODE O/ POLAROGRAPHY OF LEAD(II) IN AQUEOUS HYDROFLUORIC ACID (1-12-MOL 65PRR-041
CMPARISON OF / COMMENTS CN THE PAPER BY LAUER, BRUCK, AND LECUIGOU, PRECISE DETERMINATION OF URANIUM - A C 65PRR-202
CCMPARISON OF/ CCMMENTS ON THE PAPER BY LAUER, BRUCK, ANC LECUIGOU, PRECISE DETERMINATIONS OF URANIUM - A 65PRR-098
BY INFRARED SPECTROMETRY THE RAPID DETERMINATICN OF L LEUCINE, L ISOLEUCINE, ANC L VALINE IN A MIXTURE 65TR-09
N PRECISION PHOTOMETRIC ANALYSIS - USE OF A RADIOISOTOPIC LIGHT SOURCE A NEW CCNCEPT I 65A-11-07A
/ MEASUREMENT OF HALF-WAVE POTENTIAL VALUES / SENSITIVITY LIMIT OF POLAROGRAPHY / PRECISICN OF DC POLAROG/ 65A-05-03A
/ION OF IR LOSS IN PCLAROGRAPHY / POLAROGRAPHIC DETECTION LIMITS FOR URANIUM / CONCENTRATION ADVANTAGE / / 65A-01-06
/HAMMER-CONTROL CF DROPPING MERCURY ELECTRCDE DROP TIME / LINEAR TIME BASE / SMOLER DROPPING MERCURY ELEC/ 65A-01-04
UTOMATIC TEMPERATURE C/ HIGH SENSITIVITY, DIRECT READING, LINEAR, RECORDING CONDUCTOMETRIC TITRATOR WITH A 65PRR-036
OSITICN OF METALS ON SOLID ELECTRODES BY VOLTAMMETRY WITH LINEARLY VARYING POTENTIAL REVERSIBLE DEP 65A-02-02A
CSITICN OF METALS CN SOLID ELECTRODES BY VOLTAMMETRY WITH LINEARLY VARYING POTENTIAL REVERSIBLE DEP 65PRR-030
CSITION OF METALS ON SOLID ELECTRCDES BY VOLTAMMETRY WITH LINEARLY VARYING POTENTIAL REVERSIBLE DEP 65PRR-191
ABSOLUTE LIQUID SCINTILLATION COUNTING OF BETA EMITTERS 65PRR-014
MEASUREMENT OF WEAR RATES IN AUTOMOTIVE ENGINES BY LIQUID SCINTILLATICN COUNTING OF IRON-55 65A-11-07C
WEAR RATES IN AUTOMOTIVE ENGINES BY LIQUID SCINTILLATICN COUNTING OF IRON-55 65PRR-048
DETECTION N,N,N TRI METHYL BORAZINE AS A LIQUID SCINTILLATOR SOLVENT FOR THERMAL NEUTRON 65A-11-05A
DETECTION N,N,N, TRI METHYL BORAZINE AS A LIQUID SCINTILLATOR SOLVENT FOR THERMAL NEUTRCN 65PRR-049
ASPECTS OF QUENCHING VARIABLES FROM BIOMEDICAL SAMPLES IN LIQUID SCINTILLATOR SYSTEM /AL AND EXPERIMENTAL 65PRR-199
C INTERNAL STANDARD COLOR QUENCH CORRECTICN IN LIQUID SCINTILLATOR SYSTEMS BY USE OF AN ISOLATE 65A-11-05B
TERNAL STANDARD COLOR QUENCH CORRECTICN IN LIQUID SCINTILLATOR SYSTEMS USING AN ISOLATED IN 65PRR-047
LIQUID SCINTILLATORS 65A-11-05
PROBLEMS IN DISPOSAL OF RADIOACTIVE LIQUID WASTES IN SALT 65PRR-023
OUS SOLUTIONS OF ORGANIC ACIDS WITH HIGH MOLECULAR WEIGH/ LIQUID-LIQUID EXTRACTION OF METAL IONS FROM AQUE 65PRR-031
/ATIVE SPECTROGRAPHIC ANALYSIS (VISUAL INTERPOLATION) (LITHIUM CARBONATE DILUTION METHOD OF SPECTROGRA/ 65A-07-01F
ALPHA RADIATION EFFECTS CN CONCENTRATED SOLUTICNS OF LITHIUM CHLORIDE CONTAINING HYDROCHLORIC ACID 65PRR-165
E ACID AND HYDROLYZABLE CATION REACTIONS IN CONCENTRATED LITHIUM CHLORIDE SOLUTION - DETERMINATICN OF FRE 65PRR-022
M FLUOR/ ABSORPTICN AND REFLECTANCE SPECTRA OF IRRADIATED LITHIUM FLUORIDE - BERYLLIUM FLUORIDE - ZIRCONIU 65-02-01A
/ MOLTEN FLUORIDE SALT SYSTEMS (SILVER / IRON / NICKEL / LITHIUM FLUORICE - BERYLLIUM FLUORIDE / LITHIUM/ 65A-02-02
/ IRCN / NICKEL / LITHIUM FLUORICE - BERYLLIUM FLUORIDE / LITHIUM FLUORICE - SODIUM FLUORIDE - POTASSIUM / 65A-02-02
LUORICE ABSORPTICN SPECTRUM OF COBALT(II) ICN IN LITHIUM FLUORICE - SODIUM FLUORIDE - POTASSIUM F 65A-02-01B
/IDE / CESIUM CHLORICE / SODIUM OXIDE / POTASSIUM OXIDE / LITHIUM HYPOBROMITE / POTASSIUM HEXA CHLORO RHE/ 65A-12
-10) HALF-LIVES OF LONG-LIVED BETA EMITTERS (CHLORINE-36 BERYLLIUM 65A-11-01D
F URANIUM, AND THE PURIFICATI/ EVIDENCE OF THE ABSENCE OF LONG-LIVED ISOTOPES OF PROMETHIUM FROM FISSION O 65PRR-089
RATORY MANAGEMENT LOOKS AT ANALYTICAL CHEMISTRY IN A NATIONAL LABO 65PRR-204
/ICATION OF QUASI REFERENCE ELECTRODE / ELIMINATICN OF IR LOSS IN POLAROGRAPHY / POLAROGRAPHIC DETECTION / 65A-01-06
ALI MATERIALS BY A MODIFIED A/ THE RAPID DETERMINATICN OF LOW CONCENTRATIONS OF OXYGEN AND HYDROGEN IN ALK 65PRR-073
CARBON-14, LIQUID SCINTILLATION COUNTING METHOD 65PRR-131
RIDE / SODIUM OXIDE / POTASSIUM/ INORGANIC PREPARATICNS (MAGNESIUM CHLORICE / ZINC CHLORIDE / CESIUM CHLO 65A-12
AGON METHOD MAGNESIUM IN PROCESS WATER, SPECTROPHOTOMETRIC M 65PRR-159
INFRARED AND NUCLEAR MAGNETIC RESONANCE SPECTROMETRY 65A-09
NUCLEAR MAGNETIC RESONANCE SPECTROMETRY 65A-09-02
MAGNESIUM IN PROCESS WATER, SPECTROPHOTOMETRIC MAGCN METHOD 65PRR-159
MAINTENANCE OF THE ORNL MASTER ANALYTICAL MANUAL 65C-18-02
TIONAL LABORATORY MANAGEMENT LOOKS AT ANALYTICAL CHEMISTRY IN A NA 65PRR-204
ORNL MASTER ANALYTICAL MANUAL 65C-18
CUMULATIVE INDEXES TO THE ORNL MASTER ANALYTICAL MANUAL 65C-18-01
MAINTENANCE OF THE ORNL MASTER ANALYTICAL MANUAL 65C-18-02
ORNL MASTER ANALYTICAL MANUAL 65C
INDEXES TO THE ORNL MASTER ANALYTICAL MANUAL (1953-1964) 65PRR-093
) PROCESS OTHER ORNL MASTER ANALYTICAL MANUAL METHODS NEEDED IN THE TRANSURANIUM (TRU 65PRR-128
ORNL MASTER ANALYTICAL MANUAL, SUPPLEMENT 7 65PRR-092

/UMMARY - NOV.,1964 / DEC.,1964 / JAN.,1965 / FEB.,1965 / MAR.,1965 / APR.,1965 / MAY,1965 / JUNE,1965 / / 65PRR-104
MASS FISSION YIELD CURVE FOR AMERICIUM-241 65PRR-046
MASS FISSION YIELD CURVE FOR NEPTUNIUM-237 65PRR-094
SIS APPLICATIONS OF ORNL SPARK SOURCE MASS SPECTROGRAPH TO HIGH PURITY MATERIALS ANALY 65PRR-201
MASS SPECTROMETRIC ANALYSIS 65B-15
A PROGRAMMABLE HIGH SPEED DATA ACQUISITION SYSTEM FOR MASS SPECTROMETRIC APPLICATION 65PRR-192
MASS SPECTROMETRY 65A-08
SPARK SOURCE MASS SPECTROMETRY 65A-08-01
TRANSURANIUM ELEMENT (TRU) MASS SPECTROMETRY 65A-08-02
MASS SPECTROMETRY 65PRR-001
ISOTOPIC ANALYSIS OF TRANSURANIUM ELEMENTS BY MASS SPECTROMETRY 65PRR-167
ORNL MASTER ANALYTICAL MANUAL 65C-18
CUMULATIVE INDEXES TO THE ORNL MASTER ANALYTICAL MANUAL 65C-18-01
MAINTENANCE OF THE ORNL MASTER ANALYTICAL MANUAL 65C-18-02
ORNL MASTER ANALYTICAL MANUAL 65C
INDEXES TO THE ORNL MASTER ANALYTICAL MANUAL (1953-1964) 65PRR-093
RANSURANIUM (TRU) PROCESS OTHER ORNL MASTER ANALYTICAL MANUAL METHODS NEEDED IN THE T 65PRR-128
ORNL MASTER ANALYTICAL MANUAL, SUPPLEMENT 7 65PRR-092
/ION SPECTRUM OF THE URANYL ION IN PER CHLORATE MEDIA I. MATHEMATICAL RESOLUTION OF THE OVERLAPPING BAND/ 65PRR-161
/.,1964 / JAN.,1965 / FEB.,1965 / MAR.,1965 / APR.,1965 / MAY,1965 / JUNE,1965 / JULY,1965 / AUG.,1965 / / 65PRR-104
REPORT ON FOREIGN TRAVEL MAY-15-JUNE-9,1965 65PRR-095
/ EFFECT OF JUNCTION POTENTIALS AT THE SALT BRIDGE ON THE MEASURED VALUES FOR THE HALF-WAVE POTENTIALS OF 65PRR-010
INSTRUMENTATION FOR POLAROGRAPHY OF GLASS CORRODING MEDIA 65PRR-195
/HE ABSORPTION SPECTRUM OF THE URANYL ION IN PER CHLORATE MEDIA I. MATHEMATICAL RESOLUTION OF THE OVERLA/ 65PRR-161
/AROGRAPHY IN HYDROFLUORIC ACID AND OTHER GLASS CORRODING MEDIA - EVALUATION WITH MONOVALENT THALLIUM TO / 65PRR-039
/AROGRAPHY IN HYDROFLUORIC ACID AND OTHER GLASS CORRODING MEDIA - EVALUATION WITH THE DIVALENT LEAD TO LE/ 65PRR-040
/AROGRAPHY IN HYDROFLUORIC ACID AND OTHER GLASS CORRODING MEDIA (EVALUATION OF THE HORIZONTAL ORIFICE TE/ 65A-05-03A
THE POLAROGRAPHIC CURRENT IN RELATION TO THE HEIGHT OF A MERCURY COLUMN /HOD OF DETERMINING THE NATURE OF 65TR-11
ELECTRODE DROP TIME / LINEAR TIME BASE / SMOLER DROPPING MERCURY ELECTRODE) /CONTROL OF DROPPING MERCURY 65A-01-04
/ DOUBLE LAYER CAPACITANCE MEASUREMENT / TEFLON DROPPING MERCURY ELECTRODE) /H WITH SOLID STATE CIRCUITS 65A-01-14
STUDIES WITH THE TEFLON DROPPING MERCURY ELECTRODE (D.M.E.) 65A-05-03
/YDROFLUORIC ACID WITH HORIZONTAL ORIFICE TEFLON DROPPING MERCURY ELECTRODE / SIGNIFICANCE OF JUNCTION PO/ 65A-05-03A
GNAL TO NOISE RATIOS (S/N) OF GLASS AND TEFLON DROPPING MERCURY ELECTRODE CAPILLARIES SI 65A-05-03B
/AMMETRY / CHRONOAMPEROMETRY / HAMMER-CONTROL OF DROPPING MERCURY ELECTRODE DROP TIME / LINEAR TIME BASE / 65A-01-04
/A (EVALUATION OF THE HORIZONTAL ORIFICE TEFLON DROPPING MERCURY ELECTRODE FOR OBTAINING FUNDAMENTAL POL/ 65A-05-03A
IC ACID AND OTHER GLASS CORRODING MEDIA / TEFLON DROPPING MERCURY ELECTRODE FOR POLAROGRAPHY IN HYDROFLUOR 65PRR-039
AQUEOUS HYDROFLUORIC ACID (1-12-MOLAR) WITH A DROPPING MERCURY ELECTRODE OF TEFLON /APHY OF LEAD(II) IN 65PRR-041
HYDROFLUORIC ACID AND OTHER GLASS CORRODING MED/ DROPPING MERCURY ELECTRODE OF TEFLON FOR POLAROGRAPHY IN 65PRR-040
MERCURY-197 65A-11-01B
MEASUREMENT OF MERCURY-197 65PRR-012
MEASUREMENT OF MERCURY-197 65PRR-170
S AS EXTRACTANTS FOR METAL IONS - SELECTIVE EXTRACTION OF MERCURY(II), SILVER(I), AND COPPER(I) /TRI AMIDE 65PRR-015
EQUILIBRIUM DISTRIBUTION OF METAL - ION COMPLEXES 65PRR-074
REVIEW OF THE SOLVENT EXTRACTION OF METAL CHELATES 65PRR-058
TANTS OF METALS USE OF METAL DI N BUTYL PHOSPHORO DI THIOATES AS EXTRAC 65PRR-016
HEXYL PHOSPHORIC TRI AMIDE (THPA) AS AN EXTRACTANT FOR METAL IONS N,N,N TRI 65A-11-04A
/EXA ALKYL PHOSPHORO THIOIC TRI AMIDES AS EXTRACTANTS FOR METAL IONS - SELECTIVE EXTRACTION OF MERCURY(II/ 65PRR-015
DS WITH HIGH MOLECULAR WEIGH/ LIQUID-LIQUID EXTRACTION OF METAL IONS FROM AQUEOUS SOLUTIONS OF ORGANIC ACI 65PRR-031
/TAL POLAROGRAPHIC DATA / MONOVALENT THALLIUM TO THALLIUM METAL REDOX REACTION / DIVALENT LEAD TO LEAD ME/ 65A-05-03A
/TO THALLIUM METAL REDOX REACTION / DIVALENT LEAD TO LEAD METAL REDOX REACTION / POLAROGRAPHY OF DIVALENT/ 65A-05-03A
RRODING MEDIA - EVALUATION WITH THE DIVALENT LEAD TO LEAD METAL REDOX REACTION /IC ACID AND OTHER GLASS CO 65PRR-040
/L REDOX REACTION / POLAROGRAPHY OF DIVALENT LEAD TO LEAD METAL REDOX REACTION IN AQUEOUS HYDROFLUORIC AC/ 65A-05-03A
/OR THE HALF-WAVE POTENTIALS OF THE DIVALENT LEAD TO LEAD METAL REDOX REACTION REDUCTION IN AQUEOUS SOLUT/ 65PRR-010
THE LASER IN METALLURGICAL SPECTROSCOPY 65PRR-174
METAL DI N BUTYL PHOSPHORO DI THIOATES AS EXTRACTANTS OF METALS USE OF 65PRR-016
ADING SPECTROGRAPHIC DETERMINATION OF THE RATIOS OF THREE METALS DIRECT RE 65PRR-086
CACTIVE MSRE SALT / SEPARATION OF CURIUM FROM CONTAMINANT METALS) /LS (DETERMINATION OF CHROMIUM IN RADI 65A-07-01C
INATION OF OXYGEN IN ALKALI METAL/ ANALYSIS OF THE ALKALI METALS (FOR CARBON HYDROGEN AND OXYGEN / DETERM 65A-04-03A
DE IN GLOVE BOX ATMOSPHERES ON THE ANALYSIS OF THE ALKALI METALS /N, HYDROGEN, NITROGEN, AND CARBON DI OXI 65PRR-175
N HYDROGEN AND OXYGEN / DETERMINATION OF OXYGEN IN ALKALI METALS BY 14-MEV NEUTRON ACTIVATION ANALYSIS) / 65A-04-03A
LOSE COLUMN SEPARATION OF TRACE METALS FROM URANIUM BY CHROMATOGRAPHY ON A CELLU 65A-11-04E
SE COLUMN SEPARATION OF TRACES OF METALS FROM URANIUM BY CHROMATOGRAPHY ON CELLULO 65PRR-037
DETERMINATION OF TRACE METALS IN NIOBIUM - IRON ALLOY 65A-05-04B
INEARLY VARYING POTENTIAL REVERSIBLE DEPOSITION OF METALS ON SOLID ELECTRODES BY VOLTAMMETRY WITH L 65A-02-02A
INEARLY VARYING POTENTIAL REVERSIBLE DEPOSITION OF METALS ON SOLID ELECTRODES BY VOLTAMMETRY WITH L 65PRR-030
INEARLY VARYING POTENTIAL REVERSIBLE DEPOSITION OF METALS ON SOLID ELECTRODES BY VOLTAMMETRY WITH L 65PRR-191
BERYLLIUM, COPPER, NICKEL, COBALT, AND ZINC AS DI BENZOYL METHANE COMPLEXES SOLVENT EXTRACTION OF 65TR-10
METHYL METHANE SULFONATE (CARCINOGEN) 65A-13-01D
RADIO GAS CHROMATOGRAPHY OF THE TRITIUM - WATER - METHANE SYSTEM 65A-05-01A
CHROMATOGRAPHIC ANALYSIS OF GAMMA IRRADIATED MIXTURES OF METHANOL AND HYDROCHLORIC ACID GAS 65A-03-02
OTOMETRIC DETERMINATION OF NITRATED 4 SEC BUTYL 2 (ALPHA METHYL BENZYL) PHENOL (BAMBP) SPECTROPH 65A-05-04E
FOR THERMAL NEUTRON DETECTION N,N,N TRI METHYL BORAZINE AS A LIQUID SCINTILLATOR SOLVENT 65A-11-05A
FOR THERMAL NEUTRON DETECTION N,N,N, TRI METHYL BORAZINE AS A LIQUID SCINTILLATOR SOLVENT 65PRR-049
/ REACTOR EXPERIMENT (MSRE) FUEL, SPECTROPHOTOMETRIC DI METHYL GLYOXIME (PER SULFATE OXIDATION) METHO/ 65PRR-134
NUCLEAR SAFETY PROGRAM (IODINE / METHYL IODIDE / GAS CHROMATOGRAPHY) 65A-04-04A
AL REACTION OF METHYL IODIDE WITH CHEMICALLY IMPREGNATED CHARCO 65PRR-065
METHYL METHANE SULFONATE (CARCINOGEN) 65A-13-01D
CONTROLLED POTENTIAL POLAROGRAPHY AND COULOMETRY AS MICRO ANALYTICAL TECHNIQUES 65PRR-181
/E DC POLAROGRAPHY AND CONTROLLED POTENTIAL COULOMETRY AS MICRO ANALYTICAL TECHNIQUES (DETERMINATION OF / 65A-01-07
RAISED ON TWO SOILS AS DETERMINED BY NEUTRON ACTIVATION / MICRO ELEMENT CONTENT OF WOOL FROM TWIN WETHERS 65PRR-019
/NIUM IN A TRI N OCTYL PHOSPHINE OXIDE (TOPO) EXTRACT (MICROANALYTICAL DETERMINATION OF URANIUM / 0.5-/ 65A-01-06
LLED POTENTIAL COULOMETRIC DETERMINATION OF MILLIGRAM AND MICROGRAM QUANTITIES OF URANIUM AND IRON /CONTRO 65PRR-020
/VATIVE DC POLAROGRAPHY - APPLICATION TO DETERMINATION OF MICROGRAM QUANTITIES OF URANIUM IN A TRI N OCTY/ 65A-01-06
ERIALS ELECTRON MICROSCOPE FACILITY FOR STUDYING RADIOACTIVE MAT 65A-10-01A
OPTICAL AND ELECTRON MICROSCOPY 65A-10
METHODS DEVELOPMENT FOR ELECTRON MICROSCOPY 65A-10-03
ISTANCE TO OTHER DIVISIONS ELECTRON AND OPTICAL MICROSCOPY AND ELECTRON DIFFRACTION RESEARCH ASS 65A-10-04
ELECTRON MICROSCOPY OF RADIOACTIVE MATERIALS 65A-10-01
/ATOR - CONTROLLED POTENTIAL COULOMETRIC DETERMINATION OF MILLIGRAM AND MICROGRAM QUANTITIES OF URANIUM A/ 65PRR-020
TERMINATION OF L LEUCINE, L ISOLEUCINE, AND L VALINE IN A MIXTURE BY INFRARED SPECTROMETRY THE RAPID DE 65TR-09
/OLAROGRAPHY / PRECISION OF DC POLAROGRAPHY / SUBTRACTIVE MODE AMPEROMETRIC POLAROGRAPHY / COMPARATIVE PO/ 65A-05-03A

ERS, REMOTELY SERVO CONTROLLED, CORROSION RESISTANT, ORNL	MODEL-Q-1348A, MODEL-Q-1348B, AND MODEL-Q-1348C	65PRR-124
OTELY SERVO CONTROLLED CORROSION RESISTANT PIPETTER, ORNL	MODEL-Q-1348B REM	65A-04-018
ERVO CONTROLLED, CORROSION RESISTANT, ORNL MODEL-Q-1348A,	MODEL-Q-1348B, AND MODEL-Q-1348C /RS, REMOTELY S	65PRR-124
RROSION RESISTANT, ORNL MODEL-Q-1348A, MODEL-Q-1348B, AND	MODEL-Q-1348C /RS, REMOTELY SERVO CONTROLLED, CO	65PRR-124
/ PRECISION AND HIGH ACCURACY METHOD (ELECTROCHEMISTRY /	MODEL-Q-1988-FES CONTROLLED POTENTIAL DC POLARO/	65A-01-03
/URANIUM / 0.5-ML POLAROGRAPHIC CELL / ELECTROCHEMISTRY /	MODEL-Q-1988-FES POLAROGRAPH / MODEL-Q-2792 POL/	65A-01-06
CONTROLLED POTENTIAL AND DERIVATIVE POLAROGRAPH,	MODEL-Q-1988A	65PRR-117
AUTOMATIC COULOMETRIC TITRATOR, ORNL	MODEL-Q-2005, ELECTRONIC, CONTROLLED POTENTIAL	65PRR-146
HIGH-SENSITIVITY COULOMETRIC TITRATOR	MODEL-Q-2564	65PRR-118
IC TITRATOR, CONTROLLED POTENTIAL, HIGH SENSITIVITY, ORNL	MODEL-Q-2564 COULOMETR	65PRR-125
AL / BLANK C/ HIGH SENSITIVITY COULOMETRIC TITRATOR, ORNL	MODEL-Q-2564 (POTENTIOSTAT / CONTROLLED POTENTI	65A-01-05
/TERMINATION OF IRON BY CONTROLLED POTENTIAL COULOMETRY /	MODEL-Q-2564 COULOMETRIC TITRATOR / I-VERSUS-Q /	65A-01-07
ME/ CONTROLLED POTENTIAL DC POLAROGRAPH VOLTAMMETER, ORNL	MODEL-Q-2792 (CYCLIC VOLTAMMETRY / CHRONOAMPERO	65A-01-04
VOLTAMMETER CHECK OUT AND TEST PROCEDURE FOR	MODEL-Q-2792 CONTROLLED POTENTIAL DC POLAROGRAPH	65PRR-080
/CELL / ELECTROCHEMISTRY / MODEL-Q-1988-FES POLAROGRAPH /	MODEL-Q-2792 POLAROGRAPH / POTENTIOSTATIC DC PO/	65A-01-06
	MODERN ANALYTICAL CHEMISTRY	65PRR-209
ITE DIFFUSI/ INVESTIGATION OF RADIO IODINE BEHAVIOR UNDER	MOIST CONDITIONS IN MAY PACKS BY MEANS OF COMPOS	65PRR-066
DI OXIDE IN GLOVE BO/ THE EFFECTS OF TRACE IMPURITIES OF	MOISTURE, OXYGEN, HYDROGEN, NITROGEN, AND CARBON	65PRR-175
/L IONS FROM AQUEOUS SOLUTIONS OF ORGANIC ACIDS WITH HIGH	MOLECULAR WEIGHT AMINES - THE TRIVALENT ACTINIO/	65PRR-031
ICKEL / LITHIUM FLUORIDE - BERYLLIUM FLUO/ VOLTAMMETRY OF	MOLTEN FLUORIDE SALT SYSTEMS (SILVER / IRON / N	65A-02-02
	MOLTEN SALT REACTOR EXPERIMENT	65A-04-01
TOMATIC, CONTROLLED POTENTIAL, COULOMETRIC TI/ URANIUM IN	MOLTEN SALT REACTOR EXPERIMENT (MSRE) FUEL, AU	65PRR-121
GH SENSITIVITY, AUTOMATIC, CONTROLLED POT/ URANIUM IN THE	MOLTEN SALT REACTOR EXPERIMENT (MSRE) FUEL, HI	65PRR-122
ECTROPHOTOMETRIC DI METHYL GLYOXIME (PER SUL/ NICKEL, IN	MOLTEN SALT REACTOR EXPERIMENT (MSRE) FUEL, SP	65PRR-134
T CELL EQUIPMENT I. SAMPLE PREPARATION APPARATUS FOR THE	MOLTEN SALT REACTOR EXPERIMENT /AL LABORATORY HO	65PRR-027
ANALYTICAL STUDIES OF	MOLTEN SALT SYSTEMS	65A-02
ABSORPTION SPECTROPHOTOMETRIC STUDIES RELATED TO	MOLTEN SALT SYSTEMS	65A-02-01
UTER / INSTRUMENT DESIGN / ELECTROCHEMISTRY / ANALYSIS OF	MOLTEN SALTS) /AL VOLTAMMETER (DERIVATIVE COMP	65A-01-11
N ANALYSIS DETERMINATION OF	MOLYBDENUM IN HAIR AND WOOL BY NEUTRON ACTIVATIO	65PRR-018
PROCESS SOLUTIONS, 8 HYDROXY QUINOLINE PRECIPITATION - /	MOLYBDENUM RADIOACTIVITY IN TRANSURANIUM (TRU)	65PRR-129
DECAY OF	MOLYBDENUM-99 - TECHNETIUM-99M	65PRR-011
CN GENERATOR	MONITORING OF LEACHED FUEL ELEMENTS WITH A NEUTR	65A-11-09F
/ELECTRODE FOR OBTAINING FUNDAMENTAL POLAROGRAPHIC DATA /	MONOVALENT THALLIUM TO THALLIUM METAL REDOX REA/	65A-05-03A
IC ACID AND OTHER GLASS CORRODING MEDIA - EVALUATION WITH	MONOVALENT THALLIUM TO THALLIUM REDOX REACTION /	65PRR-039
965 / ANALYTICAL CHEMISTRY RESEARCH AND DEVELOPMENT GROUP	MONTHLY SUMMARY - NOV.,1964 / DEC.,1964 / JAN.,1	65PRR-104
PREPARATION BY ELECTRODEPOSITION OF	MOUNTS FOR 4 PI BETA COUNTING	65A-11-03A
ULOMETRIC TI/ URANIUM IN MOLTEN SALT REACTOR EXPERIMENT (	MSRE) FUEL, AUTOMATIC, CONTROLLED POTENTIAL, CO	65PRR-121
LLED POT/ URANIUM IN THE MOLTEN SALT REACTOR EXPERIMENT (	MSRE) FUEL, HIGH SENSITIVITY, AUTOMATIC, CONTRO	65PRR-122
ME (PER SUL/ NICKEL, IN MOLTEN SALT REACTOR EXPERIMENT (	MSRE) FUEL, SPECTROPHOTOMETRIC DI METHYL GLYOXI	65PRR-134
POLAROGRAPHIC DETERMINATION OF NICKEL IN	MSRE FUEL	65A-04-01E
NTIAL COULOMETRIC TITRATION OF URANIUM(VI) IN A SYNTHETIC	MSRE FUEL CONTROLLED POTE	65A-04-01D
OD ZIRCONIUM IN	MSRE FUEL, AMPEROMETRIC CUPFERRON TITRATION METH	65PRR-135
N METHOD CHROMIUM IN	MSRE FUEL, AMPEROMETRIC FERROUS SULFATE TITRATIO	65PRR-136
URANIUM IN	MSRE FUEL, POLAROGRAPHIC METHOD /	65PRR-138
FLUORIDE IN	MSRE FUEL, PYROLYSIS METHOD	65PRR-137
/IVE MATERIALS (DETERMINATION OF CHROMIUM IN RADIOACTIVE	MSRE SALT / SEPARATION OF CURIUM FROM CONTAMINA/	65A-07-01C
	MSRE SAMPLE PREPARATION APPARATUS	65A-04-01A
AN EXTRACTANT FOR METAL IONS	N,N,N TRI HEXYL PHOSPHORIC TRI AMIDE (THPA) AS	65A-11-04A
OR SOLVENT FOR THERMAL NEUTRON DETECTION	N,N,N TRI METHYL BORAZINE AS A LIQUID SCINTILLAT	65A-11-05A
TOR SOLVENT FOR THERMAL NEUTRON DETECTION	N,N,N, TRI METHYL BORAZINE AS A LIQUID SCINTILLA	65PRR-049
MANAGEMENT LOOKS AT ANALYTICAL CHEMISTRY IN A	NATIONAL LABORATORY	65PRR-204
LEAD-210 IN AUTO EXHAUST FUMES AND IN	NATURAL MATERIALS	65A-11-02H
COUNTERS DETECTION OF	NATURAL RADIONUCLIDES BY WELL TYPE SCINTILLATION	65A-11-03B
TO THE HEIGHT O/ CONCERNING THE METHOD OF DETERMINING THE	NATURE OF THE POLAROGRAPHIC CURRENT IN RELATION	65TR-11
OTHER ORNL MASTER ANALYTICAL MANUAL METHODS	NEEDED IN THE TRANSURANIUM (TRU) PROCESS	65PRR-128
AY BRANCHINGS IN THE DECAY OF INDIUM-116, CERIUM-141, AND	NEODYMIUM-147 GAMMA-R	65PRR-169
THE ISOTOPIC COMPOSITION OF ATMOSPHERIC	NEON	65PRR-071
THE ISOTOPIC COMPOSITION OF ATMOSPHERIC	NEON	65PRR-208
MASS FISSION YIELD CURVE FOR	NEPTUNIUM-237	65PRR-094
PROGRAM	NETAREA (GAMMA-RAY PHOTOPEAK AREA)	65A-11-06E
IODIDE DETERMINATION OF NITRITE IN A	NEUTRAL NITRATE CONTAINING SOLUTION OF POTASSIUM	65A-05-04D
PARTICLE SIZE DISTRIBUTION IN URANIUM(IV) OXIDE,	NEUTRON ACTIVATION - SEDIMENTATION METHOD	65PRR-154
DETERMINATION OF TECHNETIUM-99 BY	NEUTRON ACTIVATION ANALYSIS	65A-11-08C
APPLIED	NEUTRON ACTIVATION ANALYSIS	65A-11-08E
DETERMINATION OF URANIUM IN SEAWATER BY	NEUTRON ACTIVATION ANALYSIS	65A-11-08G
DETERMINATION OF MOLYBDENUM IN HAIR AND WOOL BY	NEUTRON ACTIVATION ANALYSIS	65PRR-018
YGEN / DETERMINATION OF OXYGEN IN ALKALI METALS BY 14-MEV	NEUTRON ACTIVATION ANALYSIS) /N HYDROGEN AND OX	65A-04-03A
) STUDY OF PRECISION AND ACCURACY IN	NEUTRON ACTIVATION ANALYSIS (DEAD TIME EFFECTS	65A-11-08B
OL FROM TWIN WETHERS RAISED ON TWO SOILS AS DETERMINED BY	NEUTRON ACTIVATION ANALYSIS /EMENT CONTENT OF WO	65PRR-019
NSIC APPLICATIONS	NEUTRON ACTIVATION ANALYSIS OF HUMAN HAIR - FORE	65A-11-08D
	NEUTRON ACTIVATION ANALYSIS OF RIVER WATER	65A-11-08H
INORGANIC AND NUCLEAR CHEMISTRY OF TECHNETIUM - THERMAL	NEUTRON ACTIVATION ANALYSIS OF TECHNETIUM	65PRR-077
APPLICATION OF	NEUTRON ACTIVATION ANALYSIS TO FORENSIC SCIENCE	65PRR-185
CLIDES NUCLEAR SPECTROSCOPY OF	NEUTRON DEFICIENT HAFNIUM AND RARE EARTH RADIONU	65A-11-01E
HYL BORAZINE AS A LIQUID SCINTILLATOR SOLVENT FOR THERMAL	NEUTRON DETECTION N,N,N TRI MET	65A-11-05A
HYL BORAZINE AS A LIQUID SCINTILLATOR SOLVENT FOR THERMAL	NEUTRON DETECTION N,N,N, TRI MET	65PRR-049
COMPUTER PROGRAM FOR CALCULATING	NEUTRON FLUX	65A-11-06F
STANDARDIZATION OF	NEUTRON FLUX MEASUREMENTS	65A-11-09C
ACTIVATION ANALYSIS - REACTOR	NEUTRON FLUXES AND INTERFERENCES	65PRR-197
14-MEV	NEUTRON GENERATOR	65A-11-09
MONITORING OF LEACHED FUEL ELEMENTS WITH A	NEUTRON GENERATOR	65A-11-09F
NCE IN CROSS SECTION MEASURE/ OUTPUT SPECTRUM FROM 14-MEV	NEUTRON GENERATORS - RAPID ESTIMATION AND INFLUE	65PRR-043
NCE IN ACTIVATION ANALYSIS OUTPUT SPECTRA OF 14-MEV	NEUTRON GENERATORS - RAPID ESTIMATION AND INFLUE	65PRR-044
GOLD ATOMS SPUTTERED FROM	NEUTRON IRRADIATED GOLD FOILS	65A-11-08F
STUDIES OF	NEUTRON PRODUCING TARGETS	65A-11-09B
CATALOG OF 14-MEV	NEUTRON REACTIONS	65A-11-09A
14-MEV	NEUTRON REACTIONS	65PRR-100
TIVE HELIUM-3 ACTIVATION ANALYSIS - HELIUM-3 REACTIONS AS	NEUTRON SOURCES / AND EXPERIMENT IN RAPID, SENSI	65PRR-045
THE USE OF ISOTOPIC	NEUTRON SOURCES FOR CHEMICAL ANALYSIS	65PRR-006
ACTIVATION OF NICKEL-64 WITH 14-MEV	NEUTRONS	65A-11-09D

ACTIVATION ANALYSIS WITH PARTICLES OTHER THAN NEUTRONS 65PRR-189
ACTIVATION ANALYSIS WITH 14-MEV NEUTRONS AND WITH HELIUM-3 IONS 65PRR-176
ACTIVATION CROSS SECTIONS FOR THE REACTIONS OF 14-MEV NEUTRONS WITH TECHNETIUM-99 65A-11-09E/
ACTIVATION CROSS SECTIONS FOR THE REACTIONS OF 14.8-MEV NEUTRONS WITH TECHNETIUM-99 65PRR-075
/MMETRY OF MOLTEN FLUORIDE SALT SYSTEMS (SILVER / IRON / NICKEL / LITHIUM FLUORIDE - BERYLLIUM FLUORIDE / 65A-02-02
POLAROGRAPHIC DETERMINATION OF NICKEL IN MSRE FUEL 65A-04-01E
CMPLEXES SOLVENT EXTRACTION OF BERYLLIUM, COPPER, NICKEL, COBALT, AND ZINC AS DI BENZOYL METHANE C 65TR-10
) FUEL, SPECTROPHOTOMETRIC DI METHYL GLYOXIME (PER SUL/ NICKEL, IN MOLTEN SALT REACTOR EXPERIMENT (MSRE 65PRR-134
ACTIVATION OF NICKEL-64 WITH 14-MEV NEUTRONS 65A-11-09D
DETERMINATION OF TRACE METALS IN NIOBIUM - IRON ALLOY 65A-05-04B
RON OPTICAL STUDY OF THE EFFECT OF AGING ON CARBIDES IN A NIOBIUM - TUNGSTEN - ZIRCONIUM ALLOY ELECT 65PRR-210
SPECTROPHOTOMETRIC THIOCYANATE DETERMINATION OF NIOBIUM IN THE PRESENCE OF FLUORIDE 65A-05-04H
TIONS NIOBIUM RADIOACTIVITY IN AQUEOUS OR ORGANIC SOLU 65PRR-142
ATION DETERMINATION OF NITRATE BY CONTROLLED POTENTIAL COULOMETRIC TITR 65A-05-04J
DETERMINATION OF NITRITE IN A NEUTRAL NITRATE CONTAINING SOLUTION OF POTASSIUM IODIDE 65A-05-04D
YE FILTER PHOTOMETRIC METHOD NITRATE IN TRANSURANIUM (TRU) SOLUTIONS, AZO D 65PRR-123
RIC METHOD NITRATE, PHENOL DI SULFONIC ACID SPECTROPHOTOMET 65PRR-132
HENOL (BAMBP) SPECTROPHOTOMETRIC DETERMINATION OF NITRATED 4 SEC BUTYL 2 (ALPHA METHYL BENZYL) P 65A-05-04E
/M SULFIDE / ERBIUM SULFIDE / THULIUM PHOSPHIDE / THULIUM NITRIDE / HOLMIUM NITRIDE / POTASSIUM SUPEROXID/ 65A-12
/ SULFIDE / THULIUM PHOSPHIDE / THULIUM NITRIDE / HOLMIUM NITRIDE / POTASSIUM SUPEROXIDE / RUBIDIUM SUPER/ 65A-12
OF POTASSIUM IODIDE DETERMINATION OF NITRITE IN A NEUTRAL NITRATE CONTAINING SOLUTION 65A-05-04D
/FECTS OF TRACE IMPURITIES OF MOISTURE, OXYGEN, HYDROGEN, NITROGEN, AND CARBON DI OXIDE IN GLOVE BOX ATMO/ 65PRR-175
G MERCURY ELECTRODE CAPILLARIES SIGNAL TO NOISE RATIOS (S/N) OF GLASS AND TEFLON DROPPIN 65A-05-03B
OSITE DIFFUSION / INVESTIGATION OF CHEMICAL REACTIVITY OF NONELEMENTAL RADIO IODINE VAPOR BY MEANS OF COMP 65PRR-068
/EMISTRY RESEARCH AND DEVELOPMENT GROUP MONTHLY SUMMARY - NOV.,1964 / DEC.,1964 / JAN.,1965 / FEB.,1965 // 65PRR-104
ANNUAL PROGRESS REPORT FOR PERIOD ENDING NOV.15,1964, ANALYTICAL CHEMISTRY DIVISION 65PRR-091
NUCLEAR AND RADIOCHEMICAL ANALYSES 65A-11
RADIO AND NUCLEAR CHEMISTRY 65PRR-177
N ACTIVATION ANALYSIS OF TECHNETIUM INORGANIC AND NUCLEAR CHEMISTRY OF TECHNETIUM - THERMAL NEUTRO 65PRR-077
INATION OF THE COATING DENSITY OF PYROLYTIC CARBON COATED NUCLEAR FUEL PARTICLES DETERM 65PRR-164
INFRARED AND NUCLEAR MAGNETIC RESONANCE SPECTROMETRY 65A-09
NUCLEAR MAGNETIC RESONANCE SPECTROMETRY 65A-09-02
RADIOCHEMICAL AND NUCLEAR METHODS 65PRR-004
NUCLEAR PROPERTIES OF RADIONUCLIDES 65A-11-01
/ SENSITIVITY / EXCITATION FUNCTION/ STUDIES OF HELIUM-3 NUCLEAR REACTIONS (HELIUM-3 ACTIVATION ANALYSIS 65A-11-08A
/ SENSITIVITY / EXCITATION FUNCTIONS FOR HELIUM-3 INDUCED NUCLEAR REACTIONS / CROSS SECTIONS) / ANALYSIS 65A-11-08A
NUCLEAR SAFETY 65A-04-04
NUCLEAR SAFETY PROGRAM 65A-10-02
/ GAS CHROMATOGRAPHY) NUCLEAR SAFETY PROGRAM (IODINE / METHYL IODIDE 65A-04-04A
M AND RARE EARTH RADIONUCLIDES NUCLEAR SPECTROSCOPY OF NEUTRON DEFICIENT HAFNIU 65A-11-01E
ANALYTICAL CHEMISTRY IN NUCLEAR TECHNOLOGY 65PRR-052
EIGHTH CONFERENCE ON ANALYTICAL CHEMISTRY IN NUCLEAR TECHNOLOGY 65PRR-053
APPLICATIONS OF GAS CHROMATOGRAPHY IN NUCLEAR TECHNOLOGY 65PRR-180
ESUME OF THE EIGHTH CONFERENCE ON ANALYTICAL CHEMISTRY IN NUCLEAR TECHNOLOGY A R 65PRR-054
/RPTION SPECTRAL MEASUREMENTS (SOLAR CELL WINDOWS / RIBO NUCLEASE / BACILLUS-SUBTILIS / ALIPAL-LO-436 / / 65A-05-04M
TRANSFER RIBO NUCLEIC ACID (TRNA) ANALYTICAL STUDIES 65A-06-01
STUDIES ON THE PHENYL ALANYL TRANSFER RIBO NUCLEIC ACID (TRNA) SYSTEM 65A-06-02
AUTOMATED NUCLECTIDE ANALYZER 65PRR-207
Y,1965 / JUNE,1965 / JULY,1965 / AUG.,1965 / SEPT.,1965 / OCT.,1965 /EB.,1965 / MAR.,1965 / APR.,1965 / MA 65PRR-104
REPORT OF FOREIGN TRAVEL OCT.10-28,1964 65PRR-087
REPORT ON FOREIGN TRAVEL OCT.19-30,1964 65PRR-099
/ERMINATION OF MICROGRAM QUANTITIES OF URANIUM IN A TRI N OCTYL PHOSPHINE OXIDE (TOPO) EXTRACT (MICROA/ 65A-01-06
URANIUM BY CONTROLLED POTENTIAL DC POLAROGRAPHY IN A TRI OCTYL PHOSPHINE OXIDE EXTRACT /ACE QUANTITIES OF 65PRR-160
REMOTELY OPERATED FILTER PHOTOMETER 65A-04-01C
ANALYTICAL LABORATORY HOT CELL EQUIPMENT III. A REMOTELY OPERATED FILTER PHOTOMETER 65PRR-026
OPTICAL AND ELECTRON MICROSCOPY 65A-10
ARCH ASSISTANCE TO OTHER DIVISIONS ELECTRON AND OPTICAL MICROSCOPY AND ELECTRON DIFFRACTION RESE 65A-10-04
IN A NIOBIUM - TUNGSTEN - ZIRCONIUM ALLOY ELECTRON OPTICAL STUDY OF THE EFFECT OF AGING ON CARBIDES 65PRR-210
/IQUID EXTRACTION OF METAL IONS FROM AQUEOUS SOLUTIONS OF ORGANIC ACIDS WITH HIGH MOLECULAR WEIGHT AMINES/ 65PRR-031
,P=S AND PHOSPHORO DI THIOATE, ,P(S)SH GROUP/ A REVIEW OF ORGANIC COMPOUNDS CONTAINING PHOSPHORO THIOATE, 65PRR-017
, ,P=S AND PHOSPHORO DI THIOATE, ,P(S)SH GROUP/ REVIEW OF ORGANIC COMPOUNDS THAT CONTAIN PHOSPHORO THIOATE 65A-11-04B
DISSOLUTION OR DECOMPOSITON OF VARIOUS ORGANIC MATERIALS 65A-06-04
ORGANIC PREPARATIONS 65A-13
BARIUM RADIOACTIVITY IN AQUEOUS OR ORGANIC SOLUTIONS 65PRR-139
CERIUM RADIOACTIVITY IN AQUEOUS OR ORGANIC SOLUTIONS 65PRR-141
NIOBIUM RADIOACTIVITY IN AQUEOUS OR ORGANIC SOLUTIONS 65PRR-142
TELLURIUM RADIOACTIVITY IN AQUEOUS OR ORGANIC SOLUTIONS 65PRR-144
ZIRCONIUM RADIOACTIVITY IN AQUEOUS OR ORGANIC SOLUTIONS 65PRR-145
IODINE RADIOACTIVITY IN AQUEOUS OR ORGANIC SOLUTIONS 65PRR-149
RADIOACTIVITY AND / OR BARIUM RADIOACTIVITY IN AQUEOUS OR ORGANIC SOLUTIONS STRONTIUM 65PRR-140
/OX REACTION IN AQUEOUS HYDROFLUORIC ACID WITH HORIZONTAL ORIFICE TEFLON DROPPING MERCURY ELECTRODE / SIG/ 65A-05-03A
/HER GLASS CORRODING MEDIA (EVALUATION OF THE HORIZONTAL ORIFICE TEFLON DROPPING MERCURY ELECTRODE FOR O/ 65A-05-03A
A SURVEY OF ANALYTICAL INSTRUMENTATION DEVELOPMENT AT ORNL 65PRR-203
METHODS USED AT ORNL FOR THE PRECISE DETERMINATION OF PLUTONIUM 65PRR-103
METHODS USED AT ORNL FOR THE PRECISE DETERMINATION OF PLUTONIUM 65PRR-206
METHODS USED AT ORNL FOR THE PRECISE DETERMINATION OF URANIUM 65PRR-102
METHODS USED AT ORNL FOR THE PRECISE DETERMINATION OF URANIUM 65PRR-205
ORNL MASTER ANALYTICAL MANUAL 65C-18
CUMULATIVE INDEXES TO THE ORNL MASTER ANALYTICAL MANUAL 65C-18-01
MAINTENANCE OF THE ORNL MASTER ANALYTICAL MANUAL 65C-18-02
ORNL MASTER ANALYTICAL MANUAL 65C
INDEXES TO THE ORNL MASTER ANALYTICAL MANUAL (1953-1964) 65PRR-093
THE TRANSURANIUM (TRU) PROCESS OTHER ORNL MASTER ANALYTICAL MANUAL METHODS NEEDED IN 65PRR-128
ORNL MASTER ANALYTICAL MANUAL, SUPPLEMENT 7 65PRR-092
/PETTERS, REMOTELY SERVO CONTROLLED, CORROSION RESISTANT, ORNL MODEL-Q-1348A, MODEL-Q-1348B, AND MODEL-Q-/ 65PRR-124
REMOTELY SERVO CONTROLLED CORROSION RESISTANT PIPETTER, ORNL MODEL-Q-1348B 65A-04-01B
IAL AUTOMATIC COULOMETRIC TITRATOR, ORNL MODEL-Q-2005, ELECTRONIC, CONTROLLED POTENT 65PRR-146
CMETRIC TITRATOR, CONTROLLED POTENTIAL, HIGH SENSITIVITY, ORNL MODEL-Q-2564 COUL 65PRR-125
TENTIAL / BLANK C/ HIGH SENSITIVITY COULOMETRIC TITRATOR, ORNL MODEL-Q-2564 (POTENTIOSTAT / CONTROLLED PO 65A-01-05
MPEROME/ CONTROLLED POTENTIAL DC POLAROGRAPH VOLTAMMETER, ORNL MODEL-Q-2792 (CYCLIC VOLTAMMETRY / CHRONDA 65A-01-04
TY MATERIALS ANALYSIS APPLICATIONS OF ORNL SPARK SOURCE MASS SPECTROGRAPH TO HIGH PURI 65PRR-201
PID ESTIMATION AND INFLUENCE IN ACTIVATION ANALYSIS OUTPUT SPECTRA OF 14-MEV NEUTRON GENERATORS - RA 65PRR-044
RAPID ESTIMATION AND INFLUENCE IN CROSS SECTION MEASURE/ OUTPUT SPECTRUM FROM 14-MEV NEUTRON GENERATORS - 65PRR-043

/IN PER CHLORATE MEDIA I. MATHEMATICAL RESOLUTICN OF THE 65PRR-161
RESINS, INDIRECT ICDCMETRIC TITRATICN METHOD 65PRR-158
/ FERRIC PER CHLCRATE / CHRCNOPDTENTICMETRIC OXIDATICN OF 65A-01-02
FUEL, SPECTROPHOTCMETRIC DI METHYL GLYOXIME (PER SULFATE 65PRR-134
/IOMETRIC OXIDATICN CF OXALIC ACIC / CHRCNCPOTENTICMETRIC 65A-01-02
/ RECUCTION OF FERRIC PER CHLORATE / CHRCNCPOTENTIOMETRIC 65A-01-02
S FOR THE EVALUATION OF ATMOSPHERIC POLLUTICN - SULFUR DI 65PRR-005
FOR THE EVALUATICN OF ATMOSPHERIC PCLLUTICN - SULPHUR DI 65A-11-07B
DETERMINATICN OF URANIUM(VI) IN URANIUM 65A-05-04G
TIAL CCULOME/ URANIUM(IV) TO URANIUM(VI) RATIO IN THORIUM 65PRR-155
/CROGRAM QUANTITIES OF URANIUM IN A TRI N CCTYL PHOSPHINE 65A-01-06
/NC CHLORIDE / CESIUM CHLORIDE / SODIUM OXIDE / POTASSIUM 65A-12
/SIUM CHLORIDE / ZINC CHLORIDE / CESIUM CHLORIDE / SODIUM 65A-12
F URANIUM(VI) IN PHOSPHORIC ACID SOLUTIONS OF URANIUM(IV) 65PRR-021
ROLLED POTENTIAL CC PDLAROGRAPHY IN A TRI CCTYL PHCSPHINE 65PRR-160
LECTRODE (P.G.E.) 65A-05-02B
DETERMINATICN OF URANIUM(VI) IN URANIUM CXIDE - THCRIUM 65A-05-04G
/S CF MCISTURE, OXYGEN, HYCROGEN, NITROGEN, AND CARBCN DI 65PRR-175
/NIUM(IV) TO URANILM(VI) RATIO IN THORIUM CXIDE - URANIUM 65PRR-155
PARTICLE SIZE DISTRIBLTICN IN URANIUM(IV) 65PRR-154
HEXA FLUORIDE DETERMINATICN OF 65A-05-04L
2,6 DI 65A-13-018
A SULFONATED STRAIGHT CHAIN ALKYL PHENOXY POLY (ETHYLENE 65A-05-04M
/ ANALYSIS OF THE ALKALI METALS (FOR CARBCN HYDROGEN AND 65A-04-03A
IFIED A/ THE RAPID OETERMINATION CF LOW CONCENTRATICNS OF 65PRR-073
/TALS (FOR CARBCN HYDROGEN AND CXYGEN / DETERMINATICN OF 65A-04-03A
IN GLOVE BO/ THE EFFECTS OF TRACE IMPURITIES OF MOISTURE, 65PRR-175
XIDE FILM FORMATICN CN THE PYROLYTIC GRAPHITE ELECTRODE (65A-05-02B
/NTIAL TITRIMETRY WITH THE PYROLYTIC GRAPHITE ELECTRODE (65A-05-02C
CN OF RADIO IOCINE BEHAVIOR UNDER MOIST CONDITICNS IN MAY 65PRR-066
RELEASE OF TRITILM BY TRITIATED 65A-11-02G
ERMINATIONS OF URANILM - A COMPARISCN CF/ COMMENTS ON THE 65PRR-098
ERMINATICN OF URANIUM - A COMPARISCN OF / CCMMENTS ON THE 65PRR-202
HIGH VOLTAGE 65A-11-040
NEUTRON ACTIVATICN - SECIMENTATICN METHOD 65PRR-178
INTERACTICNS CF CAOMIUM-106 WITH ALPHA 65PRR-154
E CCATING DENSITY OF PYRCLYTIC CARBCN COATED NUCLEAR FUEL 65PRR-179
ACTIVATION ANALYSIS WITH 65PRR-164
/FERRI CYANIDE / CHRCNOPOTENTIOMETRIC REDUCTION OF FERRIC 65PRR-189
F THE OVERL/ THE ABSCRPTION SPECTRUM OF THE URANYL ICN IN 65A-01-02
NT (MSRE) FUEL, SPECTROPHOTCMETRIC DI METHYL GLYOXIME (65PRR-161
IRCN, 1,10 65PRR-134
INATION OF NITRATEC 4 SEC BUTYL 2 (ALPHA METHYL BENZYL) 65A-10-05
C NITRATE, 65PRR-133
-436 / AMMONIUM SALT OF A SULFONATED STRAIGHT CHAIN ALKYL 65A-05-04E
) SYSTEM STUDIES CN THE 65PRR-132
DI ETHYL BENZENE SOLUTIONS, DIELECTRIC CON/ DI SEC BUTYL 65A-05-04M
/RHENATE(IV) / HOLMIUM SULFIDE / ERBIUM SULFIDE / THULIUM 65A-C6-02
/TICN OF MICROGRAM QUANTITIES OF URANIUM IN A TRI N OCTYL 65PRR-156
UM BY CONTROLLED POTENTIAL OC POLAROGRAPHY IN A TRI OCTYL 65A-12
TRIC CON/ DI SEC BUTYL PHENYL PHCSPHONATE IN DI SEC BUTYL 65PRR-160
YL BENZENE SOLUTICNS, DIELECTRIC CCN/ DI SEC BUTYL PHENYL 65PRR-156
AZO DYES (CALCICHROME / CHLCRO 65PRR-156
UM CHL/ QUATERNARY PHOSPHCNIUM HALIDES (BENZYL TRI BUTYL 65A-13-01F
(BENZYL TRI BUTYL PHOSPHONIUM BRCMIDE / BENZYL TRI BUTYL 65A-13-01A
UM BROMIDE / BENZYL TRI BUTYL PHOSPHONIUM CHL/ QUATERNARY 65A-13-01A
6D GAMMA RADIATICN ON THE DETERMINATICN OF URANIUM(VI) IN 65A-13-01A
OR METAL IONS N,N,N TRI HEXYL 65PRR-021
/GANIC COMPOUNDS THAT CCNTAIN PHCSPHORO THIOATE, ,P=S AND 65A-11-04A
/CRGANIC COMPOUNCS CCNTAINING PHCSPHORO THIDATE, ,P=S AND 65A-11-04B
USE OF METAL OI N BUTYL 65PRR-017
, ,P(S)SH GROUP/ REVIEW OF ORGANIC CCMPOUNDS THAT CONTAIN 65PRR-016
, ,P(S)SH GROUP/ A REVIEW CF ORGANIC COMPOUNDS CONTAINING 65A-11-04B
ETAL IONS - SELECTIVE EXTRACTIO/ TRI ALKYL AND HEXA ALKYL 65PRR-017
137 / SAMARIUM-153 / ARSENIC-77 / GOLD-198 / IODINE-133 / 65PRR-015
REMOTELY OPERATED FILTER 65A-11-030
ATORY HOT CELL EQUIPMENT III. A REMOTELY OPERATED FILTER 65A-04-01C
GHT SOURCE A NEW CONCEPT IN PRECISION 65PRR-026
NITRATE IN TRANSURANIUM (TRU) SOLUTICNS, AZO OYE FILTER 65A-11-07A
) FLAME 65PRR-123
PROGRAM NETAREA (GAMMA-RAY 65A-07-010
PREPARATION BY ELECTRODEPOSITION OF MOUNTS FOR 4 65A-11-06E
EQUIPMENT II. A REMOTELY CONTROLLED CORROSION RESISTANT 65A-11-03A
REMOTELY SERVO CONTROLLED CORROSION RESISTANT 65PRR-028
RESISTANT, ORNL MODEL-Q-1348A, MCCEL-Q-1348B, AND MODEL-/ 65A-04-01B
CN OF FERROUS AMMCNIUM SULFATE / CHRONOPOTENTICMETRY WITH 65PRR-119
METRIC DETERMINATION OF PLUTONIUM) UTILITY OF I-VERSUS-Q 65PRR-124
METHODS USED AT CRNL FOR THE PRECISE DETERMINATICN OF 65A-01-02
METHODS USED AT CRNL FCR THE PRECISE DETERMINATICN OF 65A-01-08
OLLED POTENTIAL COULCMETRY (COULCMETRIC DETERMINATICN OF 65PRR-103
/UM BY CONTROLLED POTENTIAL COULCMETRY / DETERMINATICN OF 65PRR-206
LAROGRAMS 65A-01-08
MS AND CHRONOPOTENTIOGRAMS (HEYRCVSKY-ILKCVIC EQUATION / 65A-01-07
POLARGRM - A COMPUTER PROGRAM FOR ANALYSIS OF 65PRR-096
ILKOVIC EQUATION / POL/ COMPUTER PROGRAMS FOR ANALYSIS OF 65A-01-13
SPEED OF RESPCNSE OF A 65PRR-096
CN AN ELECTRCNIC SELF-RECORDING INTEGRO - DIFFERENTIATING 65A-01-13
APH WITH SOLID STATE CIRCUITS / DOUBLE LAY/ A SQUARE-WAVE 65TR-04
/CTROCHEMISTRY / MODEL-Q-1988-FES CCNTROLLED POTENTIAL DC 65TR-02
/POLAROGRAPHIC CELL / ELECTROCHEMISTRY / MCDEL-Q-1988-FES 65A-01-14
OVERLAPPING BANC STRUCTURE ANC STUDIES OF THE E/ 65A-01-03
OXALATE IN ELUATES FRCM IRRADIATED ION EXCHANGE 65A-01-06
OXALIC ACID / CHRONOPOTENTIOMETRIC OXIDATION CF/ 65PRR-161
OXIDATION) METHOC /REACTOR EXPERIMENT (MSRE) 65PRR-158
OXIDATICN OF FERRCUS AMMONIUM SULFATE / CHRONOP/ 65A-01-02
OXIDATICN OF OXALIC ACID / CHRONOPOTENTICMETRIC/ 65A-01-02
OXIDE RADIO RELEASE METHOD 65PRR-134
OXIDE RACIO RELEASE METHCOS 65PRR-005
OXIDE - THORIUM OXIDE FUELS 65A-11-07B
OXIDE - URANIUM OXIDE MIXTURES, CONTROLLED POTEN 65A-05-04G
OXIDE (TOPO) EXTRACT (MICROANALYTICAL DETERM/ 65PRR-155
OXIDE / LITHIUM HYPOBROMITE / POTASSIUM HEXA CH/ 65A-01-06
OXIDE / POTASSIUM OXIDE / LITHIUM HYPOBRCMITE // 65A-12
OXIDE /6D GAMMA RADIATION ON THE DETERMINATION O 65A-12
OXIDE EXTRACT /ACE QUANTITIES OF URANIUM BY CONT 65PRR-021
OXIDE FILM FORMATION CN THE PYROLYTIC GRAPHITE E 65PRR-160
OXIDE FUELS 65A-05-02B
OXIDE IN GLCVE BOX ATMOSPHERES ON THE ANALYSIS / 65A-05-04G
OXIDE MIXTURES, CONTROLLED POTENTIAL COULOMETRI/ 65PRR-175
OXIDE, NEUTRON ACTIVATION - SEDIMENTATICN METHOD 65PRR-155
OXIDIZING PCWER OF SOLUTICNS OF HYDROLYZED XENON 65PRR-154
OXIMINC CYCLOHEXANONE 65A-05-04L
OXY) ETHANOL) /IPAL-LO-436 / AMMONIUM SALT OF 65A-13-018
OXYGEN / DETERMINATION OF OXYGEN IN ALKALI METAL 65A-05-04M
OXYGEN ANO HYDRCGEN IN ALKALI MATERIALS BY A MOD 65A-04-03A
OXYGEN IN ALKALI METALS BY 14-MEV NEUTRON ACTIV/ 65PRR-073
OXYGEN, HYDROGEN, NITROGEN, ANO CARBON DI OXIDE 65A-04-03A
P.G.E.) 0 65PRR-175
P.G.E.) ANC THE GLASSY CARBON ELECTRODE (G.C./ 65A-05-02B
PACKS BY MEANS CF COMPOSITE DIFFUSION TUBES /ATI 65A-05-02C
PAINT 65PRR-066
PAPER BY LAUER, BRUCK, ANC LECUIGOU, PRECISE DET 65A-11-02G
PAPER BY LAUER, BRUCK, ANC LECUIGOU, PRECISE DET 65PRR-098
PAPER ELECTRCPHCRESIS 65PRR-202
PARTICLE ACCELERATORS IN PURE MATERIALS ANALYSES 65A-11-040
PARTICLE SIZE DISTRIBUTION IN URANIUM(IV) OXIDE, 65PRR-178
PARTICLES 65PRR-154
PARTICLES DETERMINATICN OF TH 65PRR-179
PARTICLES OTHER THAN NEUTRCNS 65PRR-164
PER CHLORATE / CHRCNOPOTENTIOMETRIC OXICATION O/ 65PRR-189
PER CHLORATE MECIA I. MATHEMATICAL RESCLUTION O 65A-01-02
PER SULFATE OXICATION) METHOC /REACTOR EXPERIME 65PRR-161
PERSCNNEL EDUCATION 65PRR-134
PHENANTHROLINE SPECTRCPHOTOMETRIC METHOC 65A-10-05
PHENCL (BAMBP) SPECTROPHOTOMETRIC OETERM 65PRR-133
PHENCL OI SULFCNIC ACID SPECTROPHOTOMETRIC METHO 65A-05-04E
PHENCXY POLY (ETHYLENE OXY) ETHANOL) /IPAL-LO 65PRR-132
PHENYL ALANYL TRANSFER RIBO NUCLEIC ACID (TRNA 65A-05-04M
PHENYL PHOSPHONATE IN OI SEC BUTYL PHOSPHONATE - 65A-C6-02
PHCSPHICE / THULIUM NITRIDE / HOLMIUM NITRIDE // 65PRR-156
PHCSPHINE OXIDE (TOPO) EXTRACT (MICROANALYTI/ 65A-12
PHCSPHINE OXIDE EXTRACT /ACE QUANTITIES OF URANI 65A-01-06
PHCSPHONATE - CI ETHYL BENZENE SOLUTIONS, DIELEC 65PRR-160
PHCSPHONATE IN CI SEC BUTYL PHOSPHONATE - OI ETH 65PRR-156
PHCSPHCNAZO-III) 65PRR-156
PHCSPHONIUM BROMICE / BENZYL TRI BUTYL PHOSPHCNI 65A-13-01F
PHCSPHONIUM CHLCRIDE) /ARY PHOSPHCNIUM HALIDES 65A-13-01A
PHCSPHCNIUM HALIDES (BENZYL TRI BUTYL PHOSPHONI 65A-13-01A
PHCSPHORIC ACID SOLUTIONS OF URANIUM(IV) OXIDE / 65A-13-01A
PHCSPHCRIC TRI AMIDE (THPA) AS AN EXTRACTANT F 65PRR-021
PHCSPHCRO OI THIOATE, ,P(S)SH GROUPS AS SEPARAT/ 65A-11-04A
PHCSPHCRO OI THIOATE, ,P(S)SH GROUPS AS SEPARAT/ 65A-11-04B
PHCSPHCRO DI THIOATES AS EXTRACTANTS OF METALS 65PRR-017
PHCSPHCRO THIOATE, ,P=S ANC PHOSPHORO OI THIOATE 65PRR-016
PHCSPHORO THIOATE, ,P=S ANC PHOSPHORO DI THIOATE 65A-11-04B
PHCSPHORO THIOIC TRI AMIOES AS EXTRACTANTS FOR M 65PRR-017
PHCSPHORUS-33) HALF-LIFE MEASUREMENTS (CESIUM- 65PRR-015
PHCTCMETER 65A-11-030
PHCTCMETER ANALYTICAL LABOR 65A-04-01C
PHCTCMETRIC ANALYSIS - USE OF A RADIOISOTOPIC LI 65PRR-026
PHCTCMETRIC METHOC 65A-11-07A
PHCTCMETRY (COLLECTION OF BARIUM FROM TAP WATER 65PRR-123
PHCTCPEAK AREA) 65A-07-010
PI BETA COUNTING 65A-11-06E
PIPETTER ANALYTICAL LABORATORY HOT CELL 65A-11-03A
PIPETTER, ORNL MCDEL-Q-1348B 65PRR-028
PIPETTERS 65A-04-01B
PIPETTERS, REMCTELY SERVO CONTROLLED, CORROSION 65PRR-119
PLATINUM ELECTRCDES) /ONOPOTENTIOMETRIC OXIDATI 65PRR-124
PLCTS IN CONTROLLED POTENTIAL COULOMETRY (COULO 65A-01-02
PLUTCNIUM 65A-01-08
PLUTCNIUM 65PRR-103
PLUTONIUM) UTILITY OF I-VERSUS-Q PLOTS IN CONTR 65PRR-206
PLUTONIUM BY CONTROLLED POTENTIAL COULOMETRY / / 65A-01-08
POLARGRM - A COMPUTER PROGRAM FOR ANALYSIS OF PO 65A-01-07
POLARGRM / CHRNOGRM / KARAOGLANOFF EQUATION) /A 65PRR-096
POLAROGRAMS 65A-01-13
POLAROGRAMS ANC CHRONCPOTENTIOGRAMS (HEYROVSKY- 65PRR-096
POLAROGRAPH 65A-01-13
POLAROGRAPH DIFFERENTIAL POLAROGRAPHY 65TR-04
POLAROGRAPH (CESIGN OF NEW SQUARE-WAVE POLAROGR 65TR-02
POLAROGRAPH / CONTROLLED POTENTIAL CC PCLAROGRA/ 65A-01-14
POLAROGRAPH / MCCEL-Q-2792 POLAROGRAPH / POTENT/ 65A-01-03
65A-01-06

/OCHEMISTRY / MODEL-Q-1988-FES POLAROGRAPH / MODEL-Q-2792	POLAROGRAPH / POTENTIOSTATIC DC POLAROGRAPHY / /	65A-01-06
C TEST PROCEDURE FOR MODEL-Q-2792 CONTROLLED POTENTIAL DC	POLAROGRAPH VOLTAMMETER CHECK OUT AN	65PRR-080
LIC VOLTAMMETRY / CHRONOAMPEROME/ CONTROLLED POTENTIAL DC	POLAROGRAPH VOLTAMMETER, ORNL MODEL-Q-2792 (CYC	65A-01-04
AY/ A SQUARE-WAVE POLAROGRAPH (DESIGN OF NEW SQUARE-WAVE	POLAROGRAPH WITH SOLID STATE CIRCUITS / DOUBLE L	65A-01-14
CONTROLLED POTENTIAL AND DERIVATIVE	POLAROGRAPH, MODEL-Q-1988A	65PRR-117
/RACT (MICROANALYTICAL DETERMINATION OF URANIUM / 0.5-ML	POLAROGRAPHIC CELL / ELECTROCHEMISTRY / MODEL-Q/	65A-01-06
C/ CONCERNING THE METHOD OF DETERMINING THE NATURE OF THE	POLAROGRAPHIC CURRENT IN RELATION TO THE HEIGHT	65TR-11
INFLUENCE OF THE RECORDING ARRANGEMENT ON THE FORM OF	POLAROGRAPHIC CURVES	65TR-01
/LON DROPPING MERCURY ELECTRODE FOR OBTAINING FUNDAMENTAL	POLAROGRAPHIC DATA / MONOVALENT THALLIUM TO THA/	65A-05-03A
/NCE ELECTRODE / ELIMINATION OF IR LOSS IN POLAROGRAPHY /	POLAROGRAPHIC DETECTION LIMITS FOR URANIUM / CO/	65A-01-06
EL	POLAROGRAPHIC DETERMINATION OF NICKEL IN MSRE FU	65A-04-01E
/IOSTATIC DC POLAROGRAPHY / SOLVENT EXTRACTION PRECEEDING	POLAROGRAPHIC DETERMINATION OF URANIUM / TRACE /	65A-01-06
URANIUM IN MSRE FUEL,	POLAROGRAPHIC METHOD	65PRR-138
LICATION OF DERIVATIVE TECHNIQUES TO STATIONARY ELECTRODE	POLAROGRAPHY APP	65PRR-038
SUBTRACTIVE MODE AMPEROMETRIC POLAROGRAPHY / COMPARATIVE	POLAROGRAPHY) // PRECISION OF DC POLAROGRAPHY /	65A-05-03A
OGRAPHY / DELTA-E-DIFFERENTIAL POLAROGRAPHY / SUBTRACTIVE	POLAROGRAPHY) /POLAROGRAPHY (COMPARATIVE POLAR	65A-01-15
URACY METHOD (ELECTROCHEMIS/ COMPARATIVE AMPEROMETRIC DC	POLAROGRAPHY - A NEW HIGH PRECISION AND HIGH ACC	65A-01-03
ICROGRAM QUANTITIES C/ CONTROLLED POTENTIAL DERIVATIVE DC	POLAROGRAPHY - APPLICATION TO DETERMINATION OF M	65A-01-06
E-DIFFERENTIAL POLA/ CONTROLLED POTENTIAL DIFFERENTIAL DC	POLAROGRAPHY (COMPARATIVE POLAROGRAPHY / DELTA-	65A-01-15
CISION OF DC POLAROGRAPHY / SUBTRACTIVE MODE AMPEROMETRIC	POLAROGRAPHY (COMPARATIVE POLAROGRAPHY) // PRE	65A-05-03A
/CN / BLANK CORRECTIONS IN COULOMETRY / POTENTIOSTATIC DC	POLAROGRAPHY / CONTROLLED POTENTIAL DC POLAROGR/	65A-01-07
/LEC POTENTIAL DIFFERENTIAL DC POLAROGRAPHY (COMPARATIVE	POLAROGRAPHY / DELTA-E-DIFFERENTIAL POLAROGRAPH/	65A-01-15
/CONTROLLED POTENTIAL DC POLAROGRAPHY / POTENTIOSTATIC DC	POLAROGRAPHY / DETERMINATION OF CADMIUM / DIFFE/	65A-01-03
/L TECHNIQUES (DETERMINATION OF URANIUM BY DERIVATIVE DC	POLAROGRAPHY / DETERMINATION OF URANIUM BY CONT/	65A-01-07
/C POLAROGRAPHY / DETERMINATION OF CADMIUM / DIFFERENTIAL	POLAROGRAPHY / DIFFERENTIAL VOLTAMMETRY / COMPA/	65A-01-03
/OF QUASI REFERENCE ELECTRODE / ELIMINATION OF IR LOSS IN	POLAROGRAPHY / POLAROGRAPHIC DETECTION LIMITS F/	65A-01-06
/TIAL VOLTAMMETRY / COMPARATIVE VOLTAMMETRY / SUBTRACTIVE	POLAROGRAPHY / POTENTIAL-SWEEP CHRONOAMPEROMETR/	65A-01-03
/POTENTIOSTATIC DC POLAROGRAPHY / CONTROLLED POTENTIAL DC	POLAROGRAPHY / POTENTIOSTATIC COULOMETRY / ELEC/	65A-01-07
/OLLED POTENTIAL DC POLAROGRAPH / CONTROLLED POTENTIAL DC	POLAROGRAPHY / POTENTIOSTATIC DC POLAROGRAPHY //	65A-01-03
/ENT OF HALF-WAVE POTENTIAL VALUES / SENSITIVITY LIMIT OF	POLAROGRAPHY / PRECISION OF DC POLAROGRAPHY / S/	65A-05-03A
/LAROGRAPH / MODEL-Q-2792 POLAROGRAPH / POTENTIOSTATIC DC	POLAROGRAPHY / SOLVENT EXTRACTION PRECEDING PO/	65A-01-06
/ES / SENSITIVITY LIMIT OF POLAROGRAPHY / PRECISION OF DC	POLAROGRAPHY / SUBTRACTIVE MODE AMPEROMETRIC PO/	65A-05-03A
OGRAPHY (COMPARATIVE POLAROGRAPHY / DELTA-E-DIFFERENTIAL	POLAROGRAPHY / SUBTRACTIVE POLAROGRAPHY) /POLAR	65A-01-15
AS MICRO ANALYTICAL / CONTROLLED POTENTIAL DERIVATIVE DC	POLAROGRAPHY AND CONTROLLED POTENTIAL COULOMETRY	65A-01-07
TECHNIQUES CONTROLLED POTENTIAL	POLAROGRAPHY AND COULOMETRY AS MICRO ANALYTICAL	65PRR-181
/F TRACE QUANTITIES OF URANIUM BY CONTROLLED POTENTIAL DC	POLAROGRAPHY IN A TRI OCTYL PHOSPHINE OXIDE EXT/	65PRR-160
S CORRODING MEDIA (EVALUATION OF THE HORIZONTAL ORIFICE/	POLAROGRAPHY IN HYDROFLUORIC ACID AND OTHER GLAS	65A-05-03A
S CORRODING MEDIA / TEFLON DROPPING MERCURY ELECTRODE FOR	POLAROGRAPHY IN HYDROFLUORIC ACID AND OTHER GLAS	65PRR-039
S CORRODING MED/ DROPPING MERCURY ELECTRODE OF TEFLON FOR	POLAROGRAPHY IN HYDROFLUORIC ACID AND OTHER GLAS	65PRR-040
/ REACTION / DIVALENT LEAD TO LEAD METAL REDOX REACTION /	POLAROGRAPHY OF DIVALENT LEAD TO LEAD METAL RED/	65A-05-03A
INSTRUMENTATION FOR	POLAROGRAPHY OF GLASS CORRODING MEDIA	65PRR-195
ACID (1-12-MOLAR) WITH A DROPPING MERCURY ELECTRODE O/	POLAROGRAPHY OF LEAD(II) IN AQUEOUS HYDROFLUORIC	65PRR-041
EGRO - DIFFERENTIATING POLAROGRAPH DIFFERENTIAL	POLAROGRAPHY ON AN ELECTRONIC SELF-RECORDING INT	65TR-02
	POLAROGRAPHY WITH A TRIANGULAR POTENTIAL IMPULSE	65TR-05
RADIO RELEASE METHODS FOR THE EVALUATION OF ATMOSPHERIC	POLLUTION - SULFUR DI OXIDE	65PRR-005
RADIO RELEASE METHODS FOR THE EVALUATION OF ATMOSPHERIC	POLLUTION - SULPHUR DI OXIDE	65A-11-07B
MMONIUM SALT OF A SULFONATED STRAIGHT CHAIN ALKYL PHENOXY	POLY (ETHYLENE OXY) ETHANOL) /IPAL-LO-436 / A	65A-05-04M
HIGH PURITY	POTASSIUM CHLORIDE	65PRR-090
N REDUCTION IN AQUEOUS SOLUTIONS OF HYDROCHLORIC ACID AND	POTASSIUM CHLORIDE / TO LEAD METAL REDOX REACTIO	65PRR-010
/H CONTROLLED CURRENT / CHRONOPOTENTIOMETRIC REDUCTION OF	POTASSIUM FERRI CYANIDE / CHRONOPOTENTIOMETRIC /	65A-01-02
OF COBALT(II) ION IN LITHIUM FLUORIDE - SODIUM FLUORIDE -	POTASSIUM FLUORIDE ABSORPTION SPECTRUM	65A-02-01B
/ERYLLIUM FLUORIDE / LITHIUM FLUORIDE - SODIUM FLUORIDE -	POTASSIUM FLUORIDE / PYROLYTIC GRAPHITE ELECTRO/	65A-02-02
// SODIUM OXIDE / POTASSIUM OXIDE / LITHIUM HYPOBROMITE /	POTASSIUM HEXA CHLORO RHENATE(IV) / HOLMIUM SUL/	65A-12
CN OF NITRITE IN A NEUTRAL NITRATE CONTAINING SOLUTION OF	POTASSIUM IODIDE DETERMINATI	65A-05-040
/ORIDE / ZINC CHLORIDE / CESIUM CHLORIDE / SODIUM OXIDE /	POTASSIUM OXIDE / LITHIUM HYPOBROMITE / POTASSI/	65A-12
/ THULIUM PHOSPHIDE / THULIUM NITRIDE / HOLMIUM NITRIDE /	POTASSIUM SUPEROXIDE / RUBIDIUM SUPEROXIDE / UR/	65A-12
ETRIC TITRATOR, ORNL MODEL-Q-2005, ELECTRONIC, CONTROLLED	POTENTIAL AUTOMATIC COULOM	65PRR-146
CN SOLID ELECTRODES BY VOLTAMMETRY WITH LINEARLY VARYING	POTENTIAL REVERSIBLE DEPOSITION OF METALS	65A-02-02A
CN SOLID ELECTRODES BY VOLTAMMETRY WITH LINEARLY VARYING	POTENTIAL REVERSIBLE DEPOSITION OF METALS	65PRR-030
CN SOLID ELECTRODES BY VOLTAMMETRY WITH LINEARLY VARYING	POTENTIAL REVERSIBLE DEPOSITION OF METALS	65PRR-191
/ TITRATOR, ORNL MODEL-Q-2564 (POTENTIOSTAT / CONTROLLED	POTENTIAL / BLANK CORRECTIONS IN CONTROLLED POT/	65A-01-05
88A CONTROLLED	POTENTIAL AND DERIVATIVE POLAROGRAPH, MODEL-Q-19	65PRR-117
/Y CONTROLLED POTENTIAL COULOMETRIC TITRATOR - CONTROLLED	POTENTIAL COULOMETRIC DETERMINATION OF MILLIGRA/	65PRR-020
DETERMINATION OF NITRATE BY CONTROLLED	POTENTIAL COULOMETRIC TITRATION	65A-05-04J
/IC TITRATOR / I-VERSUS-Q COULOMETRIC METHOD / CONTROLLED	POTENTIAL COULOMETRIC TITRATION / BLANK CORRECT/	65A-01-07
NT (MSRE) FUEL, HIGH SENSITIVITY, AUTOMATIC, CONTROLLED	POTENTIAL COULOMETRIC TITRATION METHOD /EXPERIME	65PRR-122
TIO IN THORIUM OXIDE - URANIUM OXIDE MIXTURES, CONTROLLED	POTENTIAL COULOMETRIC TITRATION METHOD /M(VI) RA	65PRR-155
N A SYNTHETIC MSRE FUEL CONTROLLED	POTENTIAL COULOMETRIC TITRATION OF URANIUM(VI) I	65A-04-01D
N AQUEOUS SOLUTIONS OF SODIUM FLUORIDE CONTROLLED	POTENTIAL COULOMETRIC TITRATION OF URANIUM(VI) I	65PRR-034
NTIAL COULOMETRIC DETERMINAT/ HIGH SENSITIVITY CONTROLLED	POTENTIAL COULOMETRIC TITRATOR - CONTROLLED POTE	65PRR-020
/ CONTROLLED POTENTIAL / BLANK CORRECTIONS IN CONTROLLED	POTENTIAL COULOMETRY) /EL-Q-2564 (POTENTIOSTAT	65A-01-05
OF PLUTONIUM) LTILITY OF I-VERSUS-Q PLOTS IN CONTROLLED	POTENTIAL COULOMETRY (COULOMETRIC DETERMINATION	65A-01-08
/AL COULOMETRY / DETERMINATION OF PLUTONIUM BY CONTROLLED	POTENTIAL COULOMETRY / DETERMINATION OF IRON BY/	65A-01-07
/DC POLAROGRAPHY / DETERMINATION OF URANIUM BY CONTROLLED	POTENTIAL COULOMETRY / DETERMINATION OF PLUTONI/	65A-01-07
/TENTIAL COULOMETRY / DETERMINATION OF IRON BY CONTROLLED	POTENTIAL COULOMETRY / MODEL-Q-2564 COULOMETRIC/	65A-01-07
/LLED POTENTIAL DERIVATIVE DC POLAROGRAPHY AND CONTROLLED	POTENTIAL COULOMETRY AS MICRO ANALYTICAL TECHNI/	65A-01-07
/ METHOD (ELECTROCHEMISTRY / MODEL-Q-1988-FES CONTROLLED	POTENTIAL DC POLAROGRAPH / CONTROLLED POTENTIAL/	65A-01-03
CHECK OUT AND TEST PROCEDURE FOR MODEL-Q-2792 CONTROLLED	POTENTIAL DC POLAROGRAPH VOLTAMMETER	65PRR-080
-Q-2792 (CYCLIC VOLTAMMETRY / CHRONOAMPEROME/ CONTROLLED	POTENTIAL DC POLAROGRAPH VOLTAMMETER, ORNL MODEL	65A-01-04
/COULOMETRY / POTENTIOSTATIC DC POLAROGRAPHY / CONTROLLED	POTENTIAL DC POLAROGRAPHY / POTENTIOSTATIC COUL/	65A-01-07
/988-FES CONTROLLED POTENTIAL DC POLAROGRAPH / CONTROLLED	POTENTIAL DC POLAROGRAPHY / POTENTIOSTATIC DC P/	65A-01-03
/TERMINATION OF TRACE QUANTITIES OF URANIUM BY CONTROLLED	POTENTIAL DC POLAROGRAPHY IN A TRI OCTYL PHOSPH/	65PRR-160
CN TO DETERMINATION OF MICROGRAM QUANTITIES O/ CONTROLLED	POTENTIAL DERIVATIVE DC POLAROGRAPHY - APPLICATI	65A-01-06
LED POTENTIAL COULOMETRY AS MICRO ANALYTICAL / CONTROLLED	POTENTIAL DERIVATIVE DC POLAROGRAPHY AND CONTROL	65A-01-07
TIVE POLAROGRAPHY / DELTA-E-DIFFERENTIAL POLA/ CONTROLLED	POTENTIAL DIFFERENTIAL DC POLAROGRAPHY (COMPARA	65A-01-15
POLAROGRAPHY WITH A TRIANGULAR	POTENTIAL IMPULSE	65TR-05
NALYTICAL TECHNIQUES CONTROLLED	POTENTIAL POLAROGRAPHY AND COULOMETRY AS MICRO A	65PRR-181
/E OF JUNCTION POTENTIALS IN THE MEASUREMENT OF HALF-WAVE	POTENTIAL VALUES / SENSITIVITY LIMIT OF POLAROG/	65A-05-03A
STRUMENT DESI/ DIFFERENTIATING CIRCUIT FOR THE CONTROLLED	POTENTIAL VOLTAMMETER (DERIVATIVE COMPUTER / IN	65A-01-11
T REACTOR EXPERIMENT (MSRE) FUEL, AUTOMATIC, CONTROLLED	POTENTIAL, COULOMETRIC TITRATION METHOD /TEN SAL	65PRR-121
COULOMETRIC TITRATOR, CONTROLLED	POTENTIAL, HIGH SENSITIVITY, ORNL MODEL-Q-2564	65PRR-125
RY / COMPARATIVE VOLTAMMETRY / SUBTRACTIVE POLAROGRAPHY /	POTENTIAL-SWEEP CHRONOAMPEROMETRY) /L VOLTAMMET	65A-01-03

LUES FOR THE HALF-WAVE POTENTIALS OF / EFFECT OF JUNCTION POTENTIALS AT THE SALT BRIDGE ON THE MEASURED VA 65PRR-010
/CN DROPPING MERCURY ELECTRODE / SIGNIFICANCE OF JUNCTION POTENTIALS IN THE MEASUREMENT OF HALF-WAVE POTE/ 65A-05-03A
/THE SALT BRIDGE ON THE MEASURED VALUES FOR THE HALF-WAVE POTENTIALS OF THE DIVALENT LEAD TO LEAD METAL R/ 65PRR-010
STRONG AND WEAK ACIDS IN THEIR MIXTURES, POTENTIOMETRIC SODIUM HYDROXIDE TITRATION METHOD 65PRR-127
IOSTATIC REGULATING CIRCUITS AND EXAMPLES OF THE USE OF A POTENTIOSTAT THE INNER RESISTANCE OF POTENT 65TR-07
/GH SENSITIVITY COULOMETRIC TITRATOR, ORNL MODEL-Q-2564 (POTENTIOSTAT / CONTROLLED POTENTIAL / BLANK COR/ 65A-01-05
F RAPID ELECTRODE REACTIONS THE ELECTRONIC POTENTIOSTAT AND ITS USE FOR THE INVESTIGATION O 65TR-06
DC POLAROGRAPHY / CONTROLLED POTENTIAL DC POLAROGRAPHY / POTENTIOSTATIC COULOMETRY / ELECTROCHEMISTRY) / 65A-01-07
/OULOMETRIC TITRATION / BLANK CORRECTIONS IN COULOMETRY / POTENTIOSTATIC DC POLAROGRAPHY / CONTROLLED POT/ 65A-01-07
/ DC POLAROGRAPH / CONTROLLED POTENTIAL DC POLAROGRAPHY / POTENTIOSTATIC DC POLAROGRAPHY / DETERMINATION / 65A-01-03
/ODEL-Q-1988-FES POLAROGRAPH / MODEL-Q-2792 POLAROGRAPH / POTENTIOSTATIC DC POLAROGRAPHY / SOLVENT EXTRAC/ 65A-01-06
TRANSIENT PROCESSES WITH POTENTIOSTATIC REGULATING CIRCUITS 65TR-03
OF THE USE OF A POTENTIOSTAT THE INNER RESISTANCE OF POTENTIOSTATIC REGULATING CIRCUITS AND EXAMPLES 65TR-07
RIDE DETERMINATION OF OXIDIZING POWER OF SOLUTIONS OF HYDROLYZED XENON HEXA FLUO 65A-05-04L
MEDIUM POWER REACTOR EXPERIMENT 65A-04-03
REVIEW OF THE THEORY AND PRACTICE OF SCINTILLATION COUNTING 65PRR-025
CAUTION AND PRECAUTION IN FORENSIC APPLICATIONS 65PRR-186
/PH / POTENTIOSTATIC DC POLAROGRAPHY / SOLVENT EXTRACTION PRECEDING POLAROGRAPHIC DETERMINATION OF URANI/ 65A-01-06
ANSURANIUM (TRU) PROCESS SOLUTIONS, 8 HYDROXY QUINOLINE PRECIPITATION - BETA COUNTING METHOD /VITY IN TR 65PRR-129
METHODS USED AT ORNL FOR THE PRECISE DETERMINATION OF PLUTONIUM 65PRR-103
METHODS USED AT ORNL FOR THE PRECISE DETERMINATION OF PLUTONIUM 65PRR-206
METHODS USED AT ORNL FOR THE PRECISE DETERMINATION OF URANIUM 65PRR-102
METHODS USED AT ORNL FOR THE PRECISE DETERMINATION OF URANIUM 65PRR-205
OF / COMMENTS ON THE PAPER BY LAUER, BRUCK, AND LEDUIGOU, PRECISE DETERMINATION OF URANIUM - A COMPARISON 65PRR-202
OF/ COMMENTS ON THE PAPER BY LAUER, BRUCK, AND LEDUIGOU, PRECISE DETERMINATIONS OF URANIUM - A COMPARISON 65PRR-098
LYSIS (DEAD TIME EFFECTS) STUDY OF PRECISION AND ACCURACY IN NEUTRON ACTIVATION ANA 65A-11-08B
IS/ COMPARATIVE AMPEROMETRIC DC POLAROGRAPHY - A NEW HIGH PRECISION AND HIGH ACCURACY METHOD (ELECTROCHEM 65A-01-03
/E POTENTIAL VALUES / SENSITIVITY LIMIT OF POLAROGRAPHY / PRECISION OF DC POLAROGRAPHY / SUBTRACTIVE MODE/ 65A-05-03A
SOTOPIC LIGHT SOURCE A NEW CONCEPT IN PRECISION PHOTOMETRIC ANALYSIS - USE OF A RADIOI 65A-11-07A
ENDENCE OF DISSOCIATION CONSTANT OF CARBONIC ACID IN CON/ PREDICTION OF THE SOLUBILITY AND TEMPERATURE DEP 65TR-08
ORGANIC PREPARATIONS 65A-13
E / CESIUM CHLORIDE / SODIUM OXIDE / POTASSIUM/ INORGANIC PREPARATIONS (MAGNESIUM CHLORIDE / ZINC CHLORID 65A-12
ERMINATION OF PERMANENT GASES IN HELIUM AT REDUCED SAMPLE PRESSURES GAS CHROMATOGRAPHIC DET 65PRR-007
THE PURIFICATION OF PROMETHIUM FOR THE ESTABLISHMENT OF A PRIMARY SPECTROGRAPHIC STANDARD /F URANIUM, AND 65PRR-089
/ REMOVAL OF TRANSURANIUM ELEMENTS FROM AQUEOUS SOLUTIONS PRIOR TO THE DETERMINATION OF TOTAL LANTHANIDE / 65PRR-032
/IS (ANALYSIS OF SOLIDS BY X-RAY FLUORESCENCE / ELECTRON PROBE ANALYZER / DETERMINATION OF AREAL DENSITY/ 65A-07-01A
CERIUM-144 PRODUCT 65A-11-02D
CARBON-14, PRODUCT ANALYSIS GUIDE 65PRR-152
IODINE-131, PRODUCT ANALYSIS GUIDE 65PRR-153
FOR MASS SPECTROMETRIC APPLICATION A PROGRAMMABLE HIGH SPEED DATA ACQUISITION SYSTEM 65PRR-192
OTENTIOGRAMS (HEYROVSKY-ILKOVIC EQUATION / POL/ COMPUTER PROGRAMS FOR ANALYSIS OF POLAROGRAMS AND CHRONOP 65A-01-13
N ANALYSIS EVALUATION OF COMPUTER PROGRAMS FOR GAMMA-RAY SPECTROMETRY IN ACTIVATIO 65A-11-06B
N ANALYSIS THE EVALUATION OF COMPUTER PROGRAMS FOR GAMMA-RAY SPECTROMETRY IN ACTIVATIO 65PRR-171
NALYTICAL CHEMISTRY DIVISION ANNUAL PROGRESS REPORT FOR PERIOD ENDING NOV.15,1964, A 65PRR-091
ANALYTICAL CHEMISTRY FOR REACTOR PROJECTS 65A-04
/METHIUM FROM FISSION OF URANIUM, AND THE PURIFICATION OF PROMETHIUM FOR THE ESTABLISHMENT OF A PRIMARY S/ 65PRR-089
FICATI/ EVIDENCE OF THE ABSENCE OF LONG-LIVED ISOTOPES OF PROMETHIUM FROM FISSION OF URANIUM, AND THE PURI 65PRR-089
THE ANALYSIS OF PROMETHIUM-147 65PRR-088
IN TRANSURANIUM (TRU) PROCESS SOLUTIONS, MEASUREMENT OF PROMPT GAMMA-RAYS FROM SPONTANEOUS FISSION /252 65PRR-130
ALPHA PROPORTIONAL COUNTER 65PRR-147
ETRIC TITRATOR WITH AUTOMATIC TEMPERATURE COMPENSATION OR PROPORTIONAL TEMPERATURE CONTROL /DING CONDUCTOM 65PRR-036
ARBITRARY-SET, PROPORTIONAL TEMPERATURE CONTROLLER 65PRR-035
PARTICLE ACCELERATORS IN PURE MATERIALS ANALYSES 65PRR-178
/RATION OF BERKELIUM FROM OTHER ELEMENTS - APPLICATION TO PURIFICATION AND RADIOCHEMICAL DETERMINATION OF/ 65PRR-033
/ ISOTOPES OF PROMETHIUM FROM FISSION OF URANIUM, AND THE PURIFICATION OF PROMETHIUM FOR THE ESTABLISHMEN/ 65PRR-089
GAS CHROMATOGRAPHY OF PYROLYTIC PRODUCTS OF PURINES AND PYRIMIDINES 65A-06-03
PLICATIONS OF ORNL SPARK SOURCE MASS SPECTROGRAPH TO HIGH PURITY MATERIALS ANALYSIS AP 65PRR-201
HIGH PURITY POTASSIUM CHLORIDE 65PRR-090
GAS CHROMATOGRAPHY OF PYROLYTIC PRODUCTS OF PURINES AND PYRIMIDINES 65A-06-03
OF STYROFOAM DIELECTRIC FILM PYROLYSIS AND GAS CHROMATOGRAPHIC DETERMINATION 65A-05-01B
FLUORIDE IN MSRE FUEL, PYROLYSIS METHOD 65PRR-137
DETERMINATION OF THE COATING DENSITY OF PYROLYTIC CARBON COATED NUCLEAR FUEL PARTICLES 65PRR-164
ELECTROCHEMISTRY WITH THE PYROLYTIC GRAPHITE ELECTRODE 65PRR-194
OXIDE FILM FORMATION ON THE PYROLYTIC GRAPHITE ELECTRODE (P.G.E.) 65A-05-02B
GLASSY CARBON ELECTRODE/ DIFFERENTIAL TITRIMETRY WITH THE PYROLYTIC GRAPHITE ELECTRODE (P.G.E.) AND THE 65A-05-02C
/ITHIUM FLUORIDE - SODIUM FLUORIDE - POTASSIUM FLUORIDE / PYROLYTIC GRAPHITE ELECTRODE / SILVER ELECTRODE/ 65A-02-02
FILM FORMATION ON PYROLYTIC GRAPHITE ELECTRODES 65PRR-029
VOLTAMMETRY OF IODIDE, IODINE, AND IODATE WITH THE PYROLYTIC GRAPHITE INDICATING ELECTRODE 65PRR-193
GAS CHROMATOGRAPHY OF PYROLYTIC PRODUCTS OF PURINES AND PYRIMIDINES 65A-06-03
QUALITY CONTROL 65B-14
ISION, JAN.-DEC.,1964 STATISTICAL QUALITY CONTROL REPORT, ANALYTICAL CHEMISTRY DIV 65PRR-083
AL CHEMISTRY DIVISION STATISTICAL QUALITY CONTROL REPORT, APR.-JUNE,1965, ANALYTIC 65PRR-081
AL CHEMISTRY DIVISION STATISTICAL QUALITY CONTROL REPORT, JAN.-MAR.,1965, ANALYTIC 65PRR-082
CAL CHEMISTRY DIVISION STATISTICAL QUALITY CONTROL REPORT, JAN.-SEPT.,1964, ANALYTI 65PRR-084
GE TECHNIQUE QUANTITATIVE ANALYSIS BY THE X-RAY ABSORPTION ED 65PRR-166
RIALS - SPECTROCHEMICAL AND X-RAY TECHNIQUES THE QUANTITATIVE ANALYSIS OF HIGHLY RADIOACTIVE MATE 65PRR-173
/OF URANIUM / TRACE ANALYSIS FOR URANIUM / APPLICATION OF QUASI REFERENCE ELECTRODE / ELIMINATION OF IR L/ 65A-01-06
L PHOSPHONIUM BROMIDE / BENZYL TRI BUTYL PHOSPHONIUM CHL/ QUATERNARY PHOSPHONIUM HALIDES (BENZYL TRI BUTY 65A-13-01A
BY USE OF AN ISOLATED INTERNAL STANDARD COLOR QUENCH CORRECTION IN LIQUID SCINTILLATOR SYSTEMS 65A-11-05B
USING AN ISOLATED INTERNAL STANDARD COLOR QUENCH CORRECTION IN LIQUID SCINTILLATOR SYSTEMS 65PRR-047
IQUID SCINTILLAT/ THEORETICAL AND EXPERIMENTAL ASPECTS OF QUENCHING VARIABLES FROM BIOMEDICAL SAMPLES IN L 65PRR-199
VITY IN TRANSURANIUM (TRU) PROCESS SOLUTIONS, 8 HYDROXY QUINOLINE PRECIPITATION - BETA COUNTING METHOD / 65PRR-129
/ETERMINATION OF AREAL DENSITY OF GOLD COATING ON SILICON RADIATION DETECTORS BY USE OF X-RAY FLUORESCENC/ 65A-07-01A
ITHIUM CHLORIDE CONTAINING HYDROCHLORIC ACID ALPHA RADIATION EFFECTS ON CONCENTRATED SOLUTIONS OF L 65PRR-165
HIGH LEVEL ALPHA RADIATION LABORATORY 65B-17-01
HIGH RADIATION LEVEL ANALYTICAL FACILITY (HRLAF) 65B-17-05
HIGH RADIATION LEVEL ANALYTICAL LABORATORY (HRLAL) 65B-17-03
EFFECTS OF RADIATION ON ANALYTICAL METHODS 65A-03
/E IRRADIATION ASSEMBLY FOR COBALT-60 SOURCE (EFFECTS OF RADIATION ON ANALYTICAL METHODS / REMOTELY CONT/ 65A-01-12
PHOSPHORIC ACID SOLUTIONS OF / EFFECT OF COBALT-60 GAMMA RADIATION ON THE DETERMINATION OF URANIUM(VI) IN 65PRR-021
RADIO AND NUCLEAR CHEMISTRY 65PRR-177
- METHANE SYSTEM RADIO GAS CHROMATOGRAPHY OF THE TRITIUM - WATER 65A-05-01A

ENTITY, CHARACTER AND CHEMICAL BEHAVIOR OF VAPOR FORMS OF RADIC IODINE ID 65PRR-162
MAY PACKS BY MEANS OF COMPOSITE DIFFUSI/ INVESTIGATION OF RADIC IODINE BEHAVIOR UNDER MOIST CONDITIONS IN 65PRR-066
SION TUBES INVESTIGATION OF AIRBORNE RADIC IODINE SPECIES BY MEANS OF COMPOSITE DIFFU 65PRR-067
COMPOSITE DIFFUSION TUBES CHEMICAL BEHAVIOR OF THE RADIO IODINE SPECIES FOUND ON RUBBER SECTIONS OF 65PRR-069
ON / INVESTIGATION OF CHEMICAL REACTIVITY OF NONELEMENTAL RADIC IODINE VAPOR BY MEANS OF COMPOSITE DIFFUSI 65PRR-068
GAS CHROMATOGRAPHIC IDENTIFICATION OF RADIC IODINE VAPORS 65PRR-062
EFFECTS OF SURFACE MATERIALS ON RADIC IODINE VAPORS 65PRR-063
GAS CHROMATOGRAPHIC IDENTIFICATION OF RADIC IODINE VAPORS 65PRR-064
SPHERIC POLLUTION - SULPHUR DI OXIDE RADIO RELEASE METHODS FOR THE EVALUATION OF ATMO 65A-11-07B
SPHERIC POLLUTION - SULFUR DI OXIDE RADIO RELEASE METHODS FOR THE EVALUATION OF ATMO 65PRR-005
REVIEW OF RADIOACTIVATION ANALYSIS 65PRR-042
RADIOACTIVATION ANALYSIS AS AN ANALYTICAL TOOL 65PRR-187
PROBLEMS IN DISPOSAL OF RADIOACTIVE LIQUID WASTES IN SALT 65PRR-023
ELECTRON MICROSCOPY OF RADIOACTIVE MATERIALS 65A-10-01
ELECTRON MICROSCOPE FACILITY FOR STUDYING RADIOACTIVE MATERIALS 65A-10-01A
Y TECHNIQUES THE QUANTITATIVE ANALYSIS OF HIGHLY RADIOACTIVE MATERIALS - SPECTROCHEMICAL AND X-RA 65PRR-173
M IN RADIOACTIVE MSRE SALT / / SPECTROGRAPHIC ANALYSIS OF RADIOACTIVE MATERIALS (DETERMINATION OF CHROMIU 65A-07-01C
/ OF RADIOACTIVE MATERIALS (DETERMINATION OF CHROMIUM IN RADIOACTIVE MSRE SALT / SEPARATION OF CURIUM FR/ 65A-07-01C
MEASUREMENT OF RADIOACTIVITY 65A-11-03
QUEOUS OR ORGANIC SOLUTIONS STRONTIUM RADIOACTIVITY AND / OR BARIUM RADIOACTIVITY IN A 65PRR-140
BARIUM RADIOACTIVITY IN AQUEOUS OR ORGANIC SOLUTIONS 65PRR-139
STRONTIUM RADIOACTIVITY AND / OR BARIUM RADIOACTIVITY IN AQUEOUS OR ORGANIC SOLUTIONS 65PRR-140
CERIUM RADIOACTIVITY IN AQUEOUS OR ORGANIC SOLUTIONS 65PRR-141
NIOBIUM RADIOACTIVITY IN AQUEOUS OR ORGANIC SOLUTIONS 65PRR-142
TELLURIUM RADIOACTIVITY IN AQUEOUS OR ORGANIC SOLUTIONS 65PRR-144
ZIRCONIUM RADIOACTIVITY IN AQUEOUS OR ORGANIC SOLUTIONS 65PRR-145
IODINE RADIOACTIVITY IN AQUEOUS OR ORGANIC SOLUTIONS 65PRR-149
RUTHENIUM RADIOACTIVITY IN AQUEOUS SOLUTIONS 65PRR-143
ATE METHOD CESIUM RADIOACTIVITY IN AQUEOUS SOLUTIONS, CHLORO STANN 65PRR-151
TRACE HIGH ENERGY GAMMA RADIOACTIVITY IN ISOTOPE PRODUCTS 65A-11-02E
LUTIONS, 8 HYDROXY QUINOLINE PRECIPITATION - / MOLYBDENUM RADIOACTIVITY IN TRANSURANIUM (TRU) PROCESS SO 65PRR-129
AVAILABILITY AND USE OF RADIOACTIVITY STANDARDS 65PRR-024
NUCLEAR AND RADIOCHEMICAL ANALYSES 65A-11
RADIOCHEMICAL AND NUCLEAR METHODS 65PRR-004
IUM FROM OTHER ELEMENTS - APPLICATION TO PURIFICATION AND RADIOCHEMICAL DETERMINATION OF BERKELIUM /BERKEL 65PRR-033
RADIOCHEMICAL STUDIES 65A-11-04
RECENT FINDINGS IN RADIOCHEMISTRY 65PRR-196
ACTS AND ERRORS ENCOUNTERED IN GAMMA-RAY SPECTROMETRY FOR RADIOCHEMISTRY AND ACTIVATION ANALYSIS /OF ARTIF 65A-11-03E
ACTS AND ERRORS ENCOUNTERED IN GAMMA-RAY SPECTROMETRY FOR RADIOCHEMISTRY AND ACTIVATION ANALYSIS /OF ARTIF 65PRR-003
RADIOISOTOPES - RADIOCHEMISTRY LABORATORY 65B-17-04
ANALYTICAL RADIOCHEMISTRY OF TECHNETIUM 65PRR-002
APPLICATIONS OF RADIOISOTOPES 65PRR-190
RADIOISOTOPES - RADIOCHEMISTRY LABORATORY 65B-17-04
APPLICATION OF RADIOISOTOPES TO ANALYTICAL CHEMISTRY 65A-11-07
NEW CONCEPT IN PRECISION PHOTOMETRIC ANALYSIS - USE OF A RADIOISOTOPIC LIGHT SOURCE A 65A-11-07A
GAMMA RADIOLYSIS OF ACID CHLORIDE SOLUTIONS 65A-03-01
NUCLEAR PROPERTIES OF RADIONUCLIDES 65A-11-01
STUDIES OF RARE EARTH ALPHA EMITTING RADIONUCLIDES 65A-11-01F
HALF-LIVES AND SPECIFIC ACTIVITIES OF COMMON RADIONUCLIDES 65PRR-078
SPECTROSCOPY OF NEUTRON DEFICIENT HAFNIUM AND RARE EARTH RADIONUCLIDES NUCLEAR 65A-11-01E
S DETECTION OF NATURAL RADIONUCLIDES BY WELL TYPE SCINTILLATION COUNTER 65A-11-03B
RADIOTRACER METHOD FOR THE DETERMINATION OF LEAD 65A-11-07D
GRAVIMETRIC DETERMINATION OF SODIUM WITH RADIOTRACERS 65PRR-050
IVATION / MICRO ELEMENT CONTENT OF WOOL FROM TWIN WETHERS RAISED ON TWO SOILS AS DETERMINED BY NEUTRON ACT 65PRR-019
AND L VALINE IN A MIXTURE BY INFRARED SPECTROMETRY THE RAPID DETERMINATION OF L LEUCINE, L ISOLEUCINE, 65TR-C9
GEN AND HYDROGEN IN ALKALI MATERIALS BY A MODIFIED A/ THE RAPID DETERMINATION OF LOW CONCENTRATIONS OF OXY 65PRR-073
CTRONIC POTENTIOSTAT AND ITS USE FOR THE INVESTIGATION OF RAPID ELECTRODE REACTIONS THE ELE 65TR-C6
LYSIS OUTPUT SPECTRA OF 14-MEV NEUTRON GENERATORS - RAPID ESTIMATION AND INFLUENCE IN ACTIVATION ANA 65PRR-044
MEASURE/ OUTPUT SPECTRUM FROM 14-MEV NEUTRON GENERATORS - RAPID ESTIMATION AND INFLUENCE IN CROSS SECTION 65PRR-043
OUS SOLUTIONS PRIOR TO THE DETERMINATION OF TOTAL LANTHA/ RAPID REMOVAL OF TRANSURANIUM ELEMENTS FROM AQUE 65PRR-032
UCTS RAPID SEPARATION OF TECHNETIUM FROM FISSION PROD 65A-11-04C
UCTS RAPID SEPARATION OF TECHNETIUM FROM FISSION PROD 65PRR-076
HELIUM-3 REACTIONS AS NEUTRON S/ THEORY AND EXPERIMENT IN RAPID, SENSITIVE HELIUM-3 ACTIVATION ANALYSIS - 65PRR-045
NEW RARE EARTH ALPHA EMITTER, EUROPIUM-148 65PRR-056
STUDIES OF RARE EARTH ALPHA EMITTING RADIONUCLIDES 65A-11-01F
METHOD FOR SEPARATING TRANSPLUTONIUM ELEMENTS FROM RARE EARTH FISSION PRODUCTS 65PRR-223
NUCLEAR SPECTROSCOPY OF NEUTRON DEFICIENT HAFNIUM AND RARE EARTH RADIONUCLIDES 65A-11-01E
REVIEW OF SPECTROSCOPIC PROPERTIES OF RARE EARTHS 65PRR-059
ION COUNTING OF IRON-55 MEASUREMENT OF WEAR RATES IN AUTOMOTIVE ENGINES BY LIQUID SCINTILLAT 65A-11-07C
ION COUNTING OF IRON-55 WEAR RATES IN AUTOMOTIVE ENGINES BY LIQUID SCINTILLAT 65PRR-048
CONTROLLED POTENTIAL COULOME/ URANIUM(IV) TO URANIUM(VI) RATIO IN THORIUM OXIDE - URANIUM OXIDE MIXTURES, 65PRR-155
URY ELECTRODE CAPILLARIES SIGNAL TO NOISE RATIOS (S/N) OF GLASS AND TEFLON DROPPING MERC 65A-05-03B
DIRECT READING SPECTROGRAPHIC DETERMINATION OF THE RATIOS OF THREE METALS 65PRR-086
/APHIC DATA / MONOVALENT THALLIUM TO THALLIUM METAL REDOX REACTION / DIVALENT LEAD TO LEAD METAL REDOX RE/ 65A-05-03A
/METAL REDOX REACTION / DIVALENT LEAD TO LEAD METAL REDOX REACTION / POLAROGRAPHY OF DIVALENT LEAD TO LEA/ 65A-05-03A
A - EVALUATION WITH MONOVALENT THALLIUM TO THALLIUM REDOX REACTION /IC ACID AND OTHER GLASS CORRODING MEDI 65PRR-039
A - EVALUATION WITH THE DIVALENT LEAD TO LEAD METAL REDOX REACTION /IC ACID AND OTHER GLASS CORRODING MEDI 65PRR-040
/TION / POLAROGRAPHY OF DIVALENT LEAD TO LEAD METAL REDOX REACTION IN AQUEOUS HYDROFLUORIC ACID WITH HORI/ 65A-05-03A
ZENE/ GAS CHROMATOGRAPHIC ANALYSIS OF THE PRODUCTS OF THE REACTION OF IODINE WITH CYCLOHEXANE AND WITH BEN 65A-04-04C
NATED CHARCOAL REACTION OF METHYL IODIDE WITH CHEMICALLY IMPREG 65PRR-065
/WAVE POTENTIALS OF THE DIVALENT LEAD TO LEAD METAL REDOX REACTION REDUCTION IN AQUEOUS SOLUTIONS OF HYDR/ 65PRR-010
CATALOG OF 14-MEV NEUTRON REACTIONS 65A-11-09A
14-MEV NEUTRON REACTIONS 65PRR-100
STAT AND ITS USE FOR THE INVESTIGATION OF RAPID ELECTRODE REACTIONS THE ELECTRONIC POTENTIO 65TR-C6
TIVITY / EXCITATION FUNCTION/ STUDIES OF HELIUM-3 NUCLEAR REACTIONS (HELIUM-3 ACTIVATION ANALYSIS / SENSI 65A-11-08A
IVITY / EXCITATION FUNCTIONS FOR HELIUM-3 INDUCED NUCLEAR REACTIONS / CROSS SECTIONS) / ANALYSIS / SENSIT 65A-11-08A
RAPID, SENSITIVE HELIUM-3 ACTIVATION ANALYSIS - HELIUM-3 REACTIONS AS NEUTRON SOURCES / AND EXPERIMENT IN 65PRR-045
ION - DETERMINATION OF FREE ACID AND HYDROLYZABLE CATION REACTIONS IN CONCENTRATED LITHIUM CHLORIDE SOLUT 65PRR-022
9 ACTIVATION CROSS SECTIONS FOR THE REACTIONS OF 14.8-MEV NEUTRONS WITH TECHNETIUM-9 65PRR-075
ACTIVATION CROSS SECTIONS FOR THE REACTIONS OF 14-MEV NEUTRONS WITH TECHNETIUM-99 65A-11-09E
MEANS OF COMPOSITE DIFFUSION / INVESTIGATION OF CHEMICAL REACTIVITY OF NONELEMENTAL RADIO IODINE VAPOR BY 65PRR-068
MOLTEN SALT REACTOR EXPERIMENT 65A-04-01
MEDIUM POWER REACTOR EXPERIMENT 65A-04-03

TROLLED POTENTIAL, COULCMETRIC TI/ URANIUM IN MOLTEN SALT	REACTOR EXPERIMENT (MSRE) FUEL, AUTOMATIC, CON	65PRR-121
TY, AUTCMATIC, CCNTROLLEC POT/ URANIUM IN THE MOLTEN SALT	REACTCR EXPERIMENT (MSRE) FUEL, HIGH SENSITIVI	65PRR-122
TRIC DI METHYL GLYCXIME (PER SUL/ NICKEL, IN MOLTEN SALT	REACTCR EXPERIMENT (MSRE) FUEL, SPECTROPHOTOME	65PRR-134
MENT I. SAMPLE PREPARATICN APPARATUS FOR THE MOLTEN SALT	REACTCR EXPERIMENT /AL LABORATORY HOT CELL EQUIP	65PRR-027
ACTIVATION ANALYSIS -	REACTOR NEUTRON FLUXES ANC INTERFERENCES	65PRR-197
ANALYTICAL CHEMISTRY FOR	REACTCR PROJECTS	65A-04
OS OF THREE METALS DIRECT	READING SPECTRCGRAPHIC DETERMINATICN OF THE RATI	65PRR-086
OR WITH AUTOMATIC TEMPERATURE C/ HIGH SENSITIVITY, DIRECT	READING, LINEAR, RECORDING CONDUCTOMETRIC TITRAT	65PRR-036
O DI THIOATE, ,P(S)SH GROUPS AS SEPARATORY ANO ANALYTICAL	REAGENTS /G PHCSPHORO THIOATE, ,P=S AND PHOSPHOR	65PRR-017
O DI THIOATE, ,P(S)SH GROUPS AS SEPARATICN ANO ANALYTICAL	REAGENTS /N PHCSPHORO THIOATE, ,P=S AND PHOSPHOR	65A-11-04B
IC CURVES INFLUENCE OF THE	RECORDING ARRANGEMENT ON THE FORM OF POLAROGRAPH	65TR-01
TEMPERATURE C/ HIGH SENSITIVITY, DIRECT REAOING, LINEAR,	RECORDING CONDUCTOMETRIC TITRATOR WITH AUTOMATIC	65PRR-036
/LAROGRAPHIC OATA / MCNOVALENT THALLIUM TO THALLIUM METAL	REDOX REACTION / DIVALENT LEAC TO LEAD METAL RE/	65A-05-03A
/LLIUM METAL REOOX REACTICN / DIVALENT LEAC TO LEAC METAL	REDOX REACTICN / POLAROGRAPHY OF DIVALENT LEAD /	65A-05-03A
G MEDIA - EVALUATICN WITH MCNOVALENT THALLIUM TO THALLIUM	REDOX REACTICN /IC ACID ANC OTHER GLASS CORRODIN	65PRR-039
G MECIA - EVALUATION WITH THE DIVALENT LEAC TO LEAC METAL	REDOX REACTICN /IC ACID ANC OTHER GLASS CORRODIN	65PRR-040
/X REACTION / POLAROGRAPHY OF DIVALENT LEAD TO LEAC METAL	REDOX REACTION IN AQUEOUS HYDROFLUORIC ACID WIT/	65A-05-03A
/ HALF-WAVE POTENTIALS OF THE DIVALENT LEAC TO LEAC METAL	REDOX REACTION REDUCTION IN AQUEOUS SOLUTIONS O/	65PRR-010
MATOGRAPHIC DETERMINATICN CF PERMANENT GASES IN HELIUM AT	REDUCEC SAMPLE PRESSURES GAS CHRO	65PRR-007
/NTIALS OF THE DIVALENT LEAD TO LEAD METAL REDCX REACTION	REDUCTION IN AQUEOUS SOLUTIONS OF HYDROCHLORIC /	65PRR-010
/UCTICN OF POTASSIUM FERRI CYANIDE / CHRCNCPOTENTIOMETRIC	REDUCTION OF FERRIC PER CHLORATE / CHRCNOPOTENT/	65A-01-02
/LTAMMETRY WITH CCNTROLLED CURRENT / CHRCNCPOTENTICMETRIC	REDUCTION OF POTASSIUM FERRI CYANIDE / CHRONOPO/	65A-01-02
/NIUM / TRACE ANALYSIS FOR URANIUM / APPLICATICN OF QUASI	REFERENCE ELECTRODE / ELIMINATION OF IR LOSS IN/	65A-01-06
CE - BERYLLIUM FLUORIDE - ZIRCONIUM FLUOR/ ABSCRPTICN AND	REFLECTANCE SPECTRA OF IRRADIATED LITHIUM FLUORI	65-02-01A
ABSORPTION AND DIFFUSE	REFLECTANCE SPECTRA OF SOLID MATERIALS	65A-02-01C
TRANSIENT PROCESSES WITH POTENTICSTATIC	REGULATING CIRCUITS	65TR-03
POTENTIOSTAT THE INNER RESISTANCE OF POTENTICSTATIC	REGULATING CIRCUITS AND EXAMPLES OF THE USE OF A	65TR-07
C POLLUTION - SULPHUR DI OXIDE RADIO	RELEASE METHODS FOR THE EVALUATION OF ATMOSPHERI	65A-11-07B
C POLLUTION - SULFUR DI OXIDE RADIO	RELEASE METHODS FOR THE EVALUATION OF ATMOSPHERI	65PRR-005
	RELEASE OF TRITIUM BY TRITIATED PAINT	65A-11-02G
ANALYTICAL LABCRATORY HCT CELL EQUIPMENT II. A	REMOTELY CONTRCLLEC CCRROSION RESISTANT PIPETTER	65PRR-028
-60 SOURCE (EFFECTS OF RACIATION ON ANALYTICAL METHODS /	REMOTELY CONTROLLED INSTRUMENT) /BLY FOR COBALT	65A-01-12
	REMOTELY OPERATED FILTER PHOTOMETER	65A-04-01C
ANALYTICAL LABORATORY HCT CELL EQUIPMENT III. A	REMOTELY OPERATED FILTER PHOTOMETER	65PRR-026
PETTER, ORNL MODEL-Q-1348B	REMOTELY SERVO CONTROLLED CORROSION RESISTANT PI	65A-04-018
ORNL MOCEL-Q-1348A, MODEL-Q-1348B, AND MODEL-/ PIPETTERS,	REMOTELY SERVO CONTROLLED, CORROSION RESISTANT,	65PRR-124
OXALATE IN ELUATES FROM IRRADIATED ICN EXCHANGE	RESINS, INDIRECT IODOMETRIC TITRATION METHOD	65PRR-158
AND EXAMPLES OF THE USE OF A POTENTIOSTAT THE INNER	RESISTANCE CF POTENTICSTATIC REGULATING CIRCUITS	65TR-07
Y HOT CELL ECUIPMENT II. A REMOTELY CONTRCLLED CORROSION	RESISTANT PIPETTER ANALYTICAL LABORATOR	65PRR-028
REMOTELY SERVO CONTRCLLED CORROSION	RESISTANT PIPETTER, ORNL MODEL-Q-1348B	65A-04-01B
C MODEL-/ PIPETTERS, REMCTELY SERVO CCNTROLLED, CORROSION	RESISTANT, DRNL MODEL-Q-1348A, MODEL-Q-1348B, AN	65PRR-124
ELECTRONIC GAMMA-RAY SPECTRAL	RESOLUTION	65A-11-06A
ELECTRCNIC	RESOLUTION OF GAMMA-RAY SPECTRA	65PRR-168
/OF THE URANYL ICN IN PER CHLORATE MEDIA I. MATHEMATICAL	RESOLUTION OF THE OVERLAPPING BAND STRUCTURE AN/	65PRR-161
INFRARED AND NUCLEAR MAGNETIC	RESCNANCE SPECTROMETRY	65A-09
NUCLEAR MAGNETIC	RESCNANCE SPECTROMETRY	65A-09-02
SPEED OF	RESPCNSE OF A PCLAROGRAPH	65TR-04
EMISTRY IN NUCLEAR TECHNOLOGY A	RESUME CF THE EIGHTH CONFERENCE ON ANALYTICAL CH	65PRR-054
OES BY VOLTAMMETRY WITH LINEARLY VARYING POTENTIAL	REVERSIBLE DEPOSITION OF METALS ON SOLID ELECTRO	65A-02-02A
DES BY VOLTAMMETRY WITH LINEARLY VARYING POTENTIAL	REVERSIBLE DEPCSITION OF METALS ON SOLID ELECTRO	65PRR-030
CES BY VOLTAMMETRY WITH LINEARLY VARYING POTENTIAL	REVERSIBLE DEPCSITICN OF METALS ON SOLID ELECTRO	65PRR-191
	REVIEW OF AMPERCMETRIC TITRATIONS	65PRR-060
	REVIEW OF ANALYTICAL CHEMISTRY OF THALLIUM	65PRR-057
THIOATE, ,P=S AND PHOSPHORO DI THIOATE, ,P(S)SH GROUP/ A	REVIEW OF ORGANIC COMPOUNDS CONTAINING PHOSPHORO	65PRR-017
RO THIOATE, ,P=S AND PHOSPHORO DI THIOATE, ,P(S)SH GROUP/	REVIEW OF ORGANIC COMPOUNDS THAT CONTAIN PHOSPHO	65A-11-04B
	REVIEW OF RACICACTIVATION ANALYSIS	65PRR-042
S	REVIEW OF SPECTROSCOPIC PROPERTIES OF RARE EARTH	65PRR-059
ES	REVIEW OF THE SOLVENT EXTRACTION OF METAL CHELAT	65PRR-058
CN COUNTING	REVIEW OF THE THECRY AND PRACTICE OF SCINTILLATI	65PRR-025
	REVIEWS AND TECHNICAL ASSISTANCE	65A-11-02A
/SIUM OXIDE / LITHIUM HYPOBROMITE / POTASSIUM HEXA CHLORO	RHENATE(IV) / HOLMIUM SULFIDE / ERBIUM SULFIDE /	65A-12
ROXIDE / BARIUM BROMITE MCNO HYDRATE / SODIUM HEXA CHLORO	RHENATE(IV) / URANIUM-235 TRI CHLORIDE) /M SUPE	65A-12
/ ABSORPTION SPECTRAL MEASUREMENTS (SOLAR CELL WINDOWS /	RIBO NUCLEASE / BACILLUS-SUBTILIS / ALIPAL-LO-43	65A-05-04M
TRANSFER	RIBO NUCLEIC ACID (TRNA) ANALYTICAL STUDIES	65A-06-01
STUDIES ON THE PHENYL ALANYL TRANSFER	RIBO NUCLEIC ACID (TRNA) SYSTEM	65A-06-02
NEUTRCN ACTIVATION ANALYSIS OF	RIVER WATER	65A-11-08H
CHEMICAL BEHAVICR OF THE RADIC IODINE SPECIES FOUND ON	RUBBER SECTICNS OF COMPOSITE DIFFUSION TUBES	65PRR-069
/ULIUM NITRIDE / HOLMIUM NITRIDE / POTASSIUM SUPEROXIDE /	RUBICIUM SUPEROXIDE / URANIUM(III) CHLORIDE / U/	65A-12
	RUBICIUM-85 (N,2N) CROSS SECTION	65A-11-02F
	RUTHENIUM RACIOACTIVITY IN AQUEOUS SOLUTIONS	65PRR-143
RODE CAPILLARIES SIGNAL TO NOISE RATIOS (	S/N) OF GLASS AND TEFLON DROPPING MERCURY ELECT	65A-05-03B
NUCLEAR	SAFETY	65A-04-04
NUCLEAR	SAFETY PROGRAM	65A-10-02
RCMATOGRAPHY) NUCLEAR	SAFETY PROGRAM (IODINE / METHYL IODIDE / GAS CH	65A-04-04A
PROBLEMS IN DISPOSAL OF RADICACTIVE LIQUID WASTES IN	SALT	65PRR-023
/ATERIALS (DETERMINATICN CF CHROMIUM IN RADIOACTIVE MSRE	SALT / SEPARATICN OF CURIUM FROM CONTAMINANT ME/	65A-07-01C
WAVE POTENTIALS OF / EFFECT OF JUNCTICN POTENTIALS AT THE	SALT BRIDGE ON THE MEASURED VALUES FOR THE HALF-	65PRR-010
/ NUCLEASE / BACILLUS-SUBTILIS / ALIPAL-LO-436 / AMMCNIUM	SALT OF A SULFCNATED STRAIGHT CHAIN ALKYL PHENO/	65A-05-04M
MOLTEN	SALT REACTOR EXPERIMENT	65A-04-01
, CONTROLLED POTENTIAL, COULOMETRIC TI/ URANIUM IN MOLTEN	SALT REACTOR EXPERIMENT (MSRE) FUEL, AUTOMATIC	65PRR-121
ITIVITY, AUTCMATIC, CCNTROLLED POT/ URANIUM IN THE MOLTEN	SALT REACTOR EXPERIMENT (MSRE) FUEL, HIGH SENS	65PRR-122
OTOMETRIC DI METHYL GLYOXIME (PER SUL/ NICKEL, IN MOLTEN	SALT REACTOR EXPERIMENT (MSRE) FUEL, SPECTROPH	65PRR-134
EQUIPMENT I. SAMPLE PREPARATION APPARATUS FOR THE MOLTEN	SALT REACTOR EXPERIMENT /AL LABORATORY HOT CELL	65PRR-027
ANALYTICAL STUDIES OF MOLTEN	SALT SYSTEMS	65A-02
ABSORPTION SPECTROPHOTCMETRIC STUDIES RELATED TO MOLTEN	SALT SYSTEMS	65A-02-01
FLUORIDE - BERYLLIUM FLUO/ VOLTAMMETRY OF MOLTEN FLUORIDE	SALT SYSTEMS (SILVER / IRON / NICKEL / LITHIUM	65A-02-02
INSTRUMENT DESIGN / ELECTROCHEMISTRY / ANALYSIS OF MOLTEN	SALTS) /AL VOLTAMMETER (DERIVATIVE COMPUTER /	65A-01-11
3 / PHOSPHORUS-32) HALF-LIFE MEASUREMENTS (CESIUM-137 /	SAMARIUM-153 / ARSENIC-77 / GOLD-198 / IODINE-13	65A-11-03D
(EFFECTS OF RACIATICN ON ANALYTICAL METHCDS / REMOTELY/	SAMPLE IRRACIATICN ASSEMBLY FOR COBALT-60 SOURCE	65A-01-12
MSRE	SAMPLE PREPARATION APPARATUS	65A-04-01A
REACTOR EX/ ANALYTICAL LABORATORY HOT CELL EQUIPMENT I.	SAMPLE PREPARATICN APPARATUS FOR THE MOLTEN SALT	65PRR-027
HIC DETERMINATION OF PERMANENT GASES IN HELIUM AT REDUCED	SAMPLE PRESSURES GAS CHRCMATOGRAP	65PRR-007
APPLICATICN OF NEUTRCN ACTIVATION ANALYSIS TC FORENSIC	SCIENCE	65PRR-185

THE NEW IMAGE OF ANALYTICAL CHEMISTRY IN SCIENCE 65PRR-221
DETECTION OF NATURAL RADIONUCLIDES BY WELL TYPE SCINTILLATION COUNTERS 65A-11-03B
REVIEW OF THE THEORY AND PRACTICE OF SCINTILLATION COUNTING 65PRR-025
CARBON-14, LIQUID SCINTILLATION COUNTING METHOD 65PRR-131
ABSOLUTE LIQUID SCINTILLATION COUNTING OF BETA EMITTERS 65PRR-014
MEASUREMENT OF WEAR RATES IN AUTOMOTIVE ENGINES BY LIQUID SCINTILLATION COUNTING OF IRON-55 65A-11-07C
WEAR RATES IN AUTOMOTIVE ENGINES BY LIQUID SCINTILLATION COUNTING OF IRON-55 65PRR-048
CN N,N,N TRI METHYL BORAZINE AS A LIQUID SCINTILLATOR SOLVENT FOR THERMAL NEUTRON DETECTI 65A-11-05A
CN N,N,N, TRI METHYL BORAZINE AS A LIQUID SCINTILLATOR SOLVENT FOR THERMAL NEUTRON DETECTI 65PRR-049
OF QUENCHING VARIABLES FROM BIOMEDICAL SAMPLES IN LIQUID SCINTILLATOR SYSTEM /AL AND EXPERIMENTAL ASPECTS 65PRR-199
NAL STANDARD COLOR QUENCH CORRECTION IN LIQUID SCINTILLATOR SYSTEMS BY USE OF AN ISOLATED INTER 65A-11-05B
STANDARD COLOR QUENCH CORRECTION IN LIQUID SCINTILLATOR SYSTEMS USING AN ISOLATED INTERNAL 65PRR-047
LIQUID SCINTILLATORS 65A-11-05
DETERMINATION OF TITANIUM IN SEAWATER 65A-05-04A
DETECTION LIMITS FOR URANIUM / CONCENTRATION ADVANTAGE / SEAWATER) /LOSS IN POLAROGRAPHY / POLAROGRAPHIC 65A-01-06
DETERMINATION OF URANIUM IN SEAWATER BY NEUTRON ACTIVATION ANALYSIS 65A-11-08G
SPHONATE - DI ETHYL BENZENE SOLUTIONS, DIELECTRIC CON/ DI SEC BUTYL PHENYL PHOSPHONATE IN DI SEC BUTYL PHO 65PRR-156
NS, DIELECTRIC CON/ DI SEC BUTYL PHENYL PHOSPHONATE IN DI SEC BUTYL PHOSPHONATE - DI ETHYL BENZENE SOLUTIO 65PRR-156
EP) SPECTROPHOTOMETRIC DETERMINATION OF NITRATED 4 SEC BUTYL 2 (ALPHA METHYL BENZYL) PHENOL (BAM 65A-05-04E
E DISTRIBUTION IN URANIUM(IV) OXIDE, NEUTRON ACTIVATION - SEDIMENTATION METHOD PARTICLE SIZ 65PRR-154
/SPHORO THIOIC TRI AMIDES AS EXTRACTANTS FOR METAL IONS - SELECTIVE EXTRACTION OF MERCURY(II), SILVER(I),/ 65PRR-015
RAPH DIFFERENTIAL POLAROGRAPHY ON AN ELECTRONIC SELF-RECORDING INTEGRO - DIFFERENTIATING POLAROG 65TR-02
L INTERPOLATION) (LITHIUM CARBONATE DILUTION METHOD OF/ SEMIQUANTITATIVE SPECTROGRAPHIC ANALYSIS (VISUA 65A-07-01F
3 REACTIONS AS NEUTRON S/ THEORY AND EXPERIMENT IN RAPID, SENSITIVE HELIUM-3 ACTIVATION ANALYSIS - HELIUM- 65PRR-045
TH FISSION PRODUCTS METHOD FOR SEPARATING TRANSPLUTONIUM ELEMENTS FROM RARE EAR 65PRR-223
THIOATE, ,P=S AND PHOSPHORO DI THIOATE, ,P(S)SH GROUPS AS SEPARATION AND ANALYTICAL REAGENTS /N PHOSPHORO 65A-11-04B
PLICATION TO PURIFICATION AND RADIOCHEMICAL DETERMINATIO/ SEPARATION OF BERKELIUM FROM OTHER ELEMENTS - AP 65PRR-033
LS (DETERMINATION OF CHROMIUM IN RADIOACTIVE MSRE SALT / SEPARATION OF CURIUM FROM CONTAMINANT METALS) / 65A-07-01C
RAPID SEPARATION OF TECHNETIUM FROM FISSION PRODUCTS 65A-11-04C
RAPID SEPARATION OF TECHNETIUM FROM FISSION PRODUCTS 65PRR-076
ATOGRAPHY ON A CELLULOSE COLUMN SEPARATION OF TRACE METALS FROM URANIUM BY CHROM 65A-11-04E
HROMATOGRAPHY ON CELLULOSE COLUMN SEPARATION OF TRACES OF METALS FROM URANIUM BY C 65PRR-037
ALKYL BENZENE SULFONATE (ABS) CONTROL FOR THE FOAM SEPARATION PROCESS 65PRR-183
THIOATE, ,P=S AND PHOSPHORO DI THIOATE, ,P(S)SH GROUPS AS SEPARATORY AND ANALYTICAL REAGENTS /G PHOSPHORO 65PRR-017
PR.,1965 / MAY,1965 / JUNE,1965 / JULY,1965 / AUG.,1965 / SEPT.,1965 / OCT.,1965 /EB.,1965 / MAR.,1965 / A 65PRR-104
REPORT ON FOREIGN TRAVEL SEPT.2-OCT.6,1965 65PRR-085
SERVICE ANALYSES 65B
ANALYTICAL SERVICES FOR CHEMICAL ENGINEERS 65PRR-182
RNL MODEL-Q-1348B REMOTELY SERVO CONTROLLED CORROSION RESISTANT PIPETTER, O 65A-04-01B
L-Q-1348A, MODEL-Q-1348B, AND MODEL-/ PIPETTERS, REMOTELY SERVO CONTROLLED, CORROSION RESISTANT, ORNL MODE 65PRR-124
CN DROPPING MERCURY ELECTRODE CAPILLARIES SIGNAL TO NOISE RATIOS (S/N) OF GLASS AND TEFL 65A-05-03B
SPECTROPHOTOMETRIC HETEROPOLY-BLUE DETERMINATION OF SILICON 65A-05-04I
/YZER / DETERMINATION OF AREAL DENSITY OF GOLD COATING ON SILICON RADIATION DETECTORS BY USE OF X-RAY FLU/ 65A-07-01A
LLIUM FLUO/ VOLTAMMETRY OF MOLTEN FLUORIDE SALT SYSTEMS (SILVER / IRON / NICKEL / LITHIUM FLUORIDE - BERY 65A-02-02
ATOMIC ABSORPTION (DETERMINATION OF GOLD SILVER AND COPPER) 65A-07-01B
IDE - POTASSIUM FLUORIDE / PYROLYTIC GRAPHITE ELECTRODE / SILVER ELECTRODE) /HIUM FLUORIDE - SODIUM FLUOR 65A-02-02
NTS FOR METAL IONS - SELECTIVE EXTRACTION OF MERCURY(II), SILVER(I), AND COPPER(I) /TRI AMIDES AS EXTRACTA 65PRR-015
ABSORBANCE SPECTRA OF SINGLE CRYSTALS 65PRR-214
DROPPING MERCURY ELECTRODE DROP TIME / LINEAR TIME BASE / SMOLER DROPPING MERCURY ELECTRODE) /CONTROL OF 65A-01-04
OF DISSOCIATION CONSTANT OF CARBONIC ACID IN CONCENTRATED SODIUM CHLORIDE SOLUTION /EMPERATURE DEPENDENCE 65TR-08
LOMETRIC TITRATION OF URANIUM(VI) IN AQUEOUS SOLUTIONS OF SODIUM FLUORIDE CONTROLLED POTENTIAL COU 65PRR-034
SORPTION SPECTRUM OF COBALT(II) ION IN LITHIUM FLUORIDE - SODIUM FLUORIDE - POTASSIUM FLUORIDE AB 65A-02-01B
/THIUM FLUORIDE - BERYLLIUM FLUORIDE / LITHIUM FLUORIDE - SODIUM FLUORIDE - POTASSIUM FLUORIDE / PYROLYTI/ 65A-02-02
/RIDE / SODIUM SUPEROXIDE / BARIUM BROMITE MONO HYDRATE / SODIUM HEXA CHLORO RHENATE(IV) / URANIUM-235 TR/ 65A-12
STRONG AND WEAK ACIDS IN THEIR MIXTURES, POTENTIOMETRIC SODIUM HYDROXIDE TITRATION METHOD 65PRR-127
/(MAGNESIUM CHLORIDE / ZINC CHLORIDE / CESIUM CHLORIDE / SODIUM OXIDE / POTASSIUM OXIDE / LITHIUM HYPOBR/ 65A-12
/LUTIONS OF URANIUM(III) CHLORIDE IN LANTHANUM CHLORIDE / SODIUM SUPEROXIDE / BARIUM BROMITE MONO HYDRATE/ 65A-12
GRAVIMETRIC DETERMINATION OF SODIUM WITH RADIOTRACERS 65PRR-050
/ ELEMENT CONTENT OF WOOL FROM TWIN WETHERS RAISED ON TWO SOILS AS DETERMINED BY NEUTRON ACTIVATION ANALY/ 65PRR-019
BTILIS / ALIPAL-LO-43/ ABSORPTION SPECTRAL MEASUREMENTS (SOLAR CELL WINDOWS / RIBO NUCLEASE / BACILLUS-SU 65A-05-04M
RYING POTENTIAL REVERSIBLE DEPOSITION OF METALS ON SOLID ELECTRODES BY VOLTAMMETRY WITH LINEARLY VA 65A-02-02A
RYING POTENTIAL REVERSIBLE DEPOSITION OF METALS ON SOLID ELECTRODES BY VOLTAMMETRY WITH LINEARLY VA 65PRR-030
RYING POTENTIAL REVERSIBLE DEPOSITION OF METALS ON SOLID ELECTRODES BY VOLTAMMETRY WITH LINEARLY VA 65PRR-191
STUDIES WITH SOLID INDICATOR ELECTRODES 65A-05-02
ABSORPTION AND DIFFUSE REFLECTANCE SPECTRA OF SOLID MATERIALS 65A-02-01C
/PEROXIDE / URANIUM(III) CHLORIDE / URANIUM(III) IODIDE / SOLID SOLUTIONS OF URANIUM(III) CHLORIDE IN LAN/ 65A-12
/POLAROGRAPH (DESIGN OF NEW SQUARE-WAVE POLAROGRAPH WITH SOLID STATE CIRCUITS / DOUBLE LAYER CAPACITANCE/ 65A-01-14
ALYZER / DETERMINATION OF A/ X-RAY ANALYSIS (ANALYSIS OF SOLIDS BY X-RAY FLUORESCENCE / ELECTRON PROBE AN 65A-07-01A
ATION CONSTANT OF CARBONIC ACID IN CON/ PREDICTION OF THE SOLUBILITY AND TEMPERATURE DEPENDENCE OF DISSOCI 65TR-08
COBALT, AND ZINC AS DI BENZOYL METHANE COMPLEXES SOLVENT EXTRACTION OF BERYLLIUM, COPPER, NICKEL, 65TR-10
REVIEW OF THE SOLVENT EXTRACTION OF METAL CHELATES 65PRR-058
/EL-Q-2792 POLAROGRAPH / POTENTIOSTATIC DC POLAROGRAPHY / SOLVENT EXTRACTION PRECEEDING POLAROGRAPHIC DET/ 65A-01-06
N,N,N TRI METHYL BORAZINE AS A LIQUID SCINTILLATOR SOLVENT FOR THERMAL NEUTRON DETECTION 65A-11-05A
N,N,N, TRI METHYL BORAZINE AS A LIQUID SCINTILLATOR SOLVENT FOR THERMAL NEUTRON DETECTION 65PRR-049
ISION PHOTOMETRIC ANALYSIS - USE OF A RADIOISOTOPIC LIGHT SOURCE A NEW CONCEPT IN PREC 65A-11-07A
CDS / REMOTELY/ SAMPLE IRRADIATION ASSEMBLY FOR COBALT-60 SOURCE (EFFECTS OF RADIATION ON ANALYTICAL METH 65A-01-12
MODIFICATIONS TO THE COBALT-60 SOURCE ASSEMBLY 65A-03-04
S ANALYSIS APPLICATIONS OF ORNL SPARK SOURCE MASS SPECTROGRAPH TO HIGH PURITY MATERIAL 65PRR-201
SPARK SOURCE MASS SPECTROMETRY 65A-08-01
IUM-3 ACTIVATION ANALYSIS - HELIUM-3 REACTIONS AS NEUTRON SOURCES / AND EXPERIMENT IN RAPID, SENSITIVE HEL 65PRR-045
THE USE OF ISOTOPIC NEUTRON SOURCES FOR CHEMICAL ANALYSIS 65PRR-006
TERIALS ANALYSIS APPLICATIONS OF ORNL SPARK SOURCE MASS SPECTROGRAPH TO HIGH PURITY MA 65PRR-201
SPARK SOURCE MASS SPECTROMETRY 65A-08-01
INVESTIGATION OF AIRBORNE RADIO IODINE SPECIES BY MEANS OF COMPOSITE DIFFUSION TUBES 65PRR-067
FFUSION TUBES CHEMICAL BEHAVIOR OF THE RADIO IODINE SPECIES FOUND ON RUBBER SECTIONS OF COMPOSITE DI 65PRR-069
HALF-LIVES AND SPECIFIC ACTIVITIES OF COMMON RADIONUCLIDES 65PRR-078
ELECTRONIC RESOLUTION OF GAMMA-RAY SPECTRA 65PRR-168
UM FLUORIDE - ZIRCONIUM FLUOR/ ABSORPTION AND REFLECTANCE SPECTRA OF IRRADIATED LITHIUM FLUORIDE - BERYLLI 65-02-01A
ABSORBANCE SPECTRA OF SINGLE CRYSTALS 65PRR-214
ABSORPTION AND DIFFUSE REFLECTANCE SPECTRA OF SOLID MATERIALS 65A-02-01C
IMATION AND INFLUENCE IN ACTIVATION ANALYSIS OUTPUT SPECTRA OF 14-MEV NEUTRON GENERATORS - RAPID EST 65PRR-044
O NUCLEASE / BACILLUS-SUBTILIS / ALIPAL-LO-43/ ABSORPTION SPECTRAL MEASUREMENTS (SOLAR CELL WINDOWS / RIB 65A-05-04M
ELECTRONIC GAMMA-RAY SPECTRAL RESOLUTION 65A-11-06A

X-RAY AND SPECTROCHEMICAL ANALYSES 65A-07
X-RAY AND SPECTROCHEMICAL ANALYSES (X-10) 65A-07-01
X-RAY AND SPECTROCHEMICAL ANALYSES (Y-12) 65A-07-02
SPECTROCHEMICAL ANALYSIS LABORATORY 65B-16
E QUANTITATIVE ANALYSIS OF HIGHLY RADIOACTIVE MATERIALS - SPECTROCHEMICAL AND X-RAY TECHNIQUES TH 65PRR-173
APPLICATIONS OF ORNL SPARK SOURCE MASS SPECTROGRAPH TO HIGH PURITY MATERIALS ANALYSIS 65PRR-201
AL INTERPOLATION) (LITHIUM CARBONATE DILUTION METHOD OF SPECTROGRAPHIC ANALYSIS) /APHIC ANALYSIS (VISU 65A-07-01F
(LITHIUM CARBONATE DILUTION METHOD OF/ SEMIQUANTITATIVE SPECTROGRAPHIC ANALYSIS (VISUAL INTERPOLATION) 65A-07-01F
(DETERMINATION OF CHROMIUM IN RADIOACTIVE MSRE SALT / / SPECTROGRAPHIC ANALYSIS OF RADIOACTIVE MATERIALS 65A-07-01C
REE METALS DIRECT READING SPECTROGRAPHIC DETERMINATION OF THE RATIOS OF TH 65PRR-086
FICATION OF PROMETHIUM FOR THE ESTABLISHMENT OF A PRIMARY SPECTROGRAPHIC STANDARD /F URANIUM, AND THE PURI 65PRR-089
MASS SPECTROMETRIC ANALYSIS 65B-15
PROGRAMMABLE HIGH SPEED DATA ACQUISITION SYSTEM FOR MASS SPECTROMETRIC APPLICATION A 65PRR-192
MASS SPECTROMETRY 65A-08
SPARK SOURCE MASS SPECTROMETRY 65A-08-01
TRANSURANIUM ELEMENT (TRU) MASS SPECTROMETRY 65A-08-02
INFRARED AND NUCLEAR MAGNETIC RESONANCE SPECTROMETRY 65A-09
INFRARED SPECTROMETRY 65A-09-01
NUCLEAR MAGNETIC RESONANCE SPECTROMETRY 65A-09-02
ARTIFACTS IN GAMMA-RAY SPECTROMETRY 65A-11-03F
MASS SPECTROMETRY 65PRR-001
ISOTOPIC ANALYSIS OF TRANSURANIUM ELEMENTS BY MASS SPECTROMETRY 65PRR-167
RECENT ADVANCES IN ANALYTICAL EMISSION SPECTROMETRY 65PRR-212
CINE, L ISOLEUCINE, AND L VALINE IN A MIXTURE BY INFRARED SPECTROMETRY THE RAPID DETERMINATION OF L LEU 65TR-09
/L STUDY OF ARTIFACTS AND ERRORS ENCOUNTERED IN GAMMA-RAY SPECTROMETRY FOR RADIOCHEMISTRY AND ACTIVATION / 65A-11-03E
/L STUDY OF ARTIFACTS AND ERRORS ENCOUNTERED IN GAMMA-RAY SPECTROMETRY FOR RADIOCHEMISTRY AND ACTIVATION / 65PRR-003
EVALUATION OF COMPUTER PROGRAMS FOR GAMMA-RAY SPECTROMETRY IN ACTIVATION ANALYSIS 65A-11-06B
THE EVALUATION OF COMPUTER PROGRAMS FOR GAMMA-RAY SPECTROMETRY IN ACTIVATION ANALYSIS 65PRR-171
SPECTROMETRY IN ANALYTICAL CHEMISTRY 65PRR-172
BORON, SPECTROPHOTOMETRIC CURCUMIN METHOD 65PRR-157
EC BUTYL 2 (ALPHA METHYL BENZYL) PHENOL (BAMBP) SPECTROPHOTOMETRIC DETERMINATION OF NITRATED 4 S 65A-05-04E
ARSENAZO-III SPECTROPHOTOMETRIC DETERMINATION OF URANIUM WITH 65A-05-04C
NICKEL, IN MOLTEN SALT REACTOR EXPERIMENT (MSRE) FUEL, SPECTROPHOTOMETRIC DI METHYL GLYOXIME (PER SULF 65PRR-134
OF SILICON SPECTROPHOTOMETRIC HETEROPOLY-BLUE DETERMINATION 65A-05-04I
MAGNESIUM IN PROCESS WATER, SPECTROPHOTOMETRIC MAGON METHOD 65PRR-159
NITRATE, PHENOL DI SULFONIC ACID SPECTROPHOTOMETRIC METHOD 65PRR-132
IRON, 1,10 PHENANTHROLINE SPECTROPHOTOMETRIC METHOD 65PRR-133
T SYSTEMS ABSORPTION SPECTROPHOTOMETRIC STUDIES RELATED TO MOLTEN SAL 65A-02-01
INE IN CYCLOHEXANE AND IN BENZENE SPECTROPHOTOMETRIC STUDY OF THE STABILITY OF IOD 65A-04-04B
NIOBIUM IN THE PRESENCE OF FLUORIDE SPECTROPHOTOMETRIC THIOCYANATE DETERMINATION OF 65A-05-04H
REVIEW OF SPECTROSCOPIC PROPERTIES OF RARE EARTHS 65PRR-059
THE LASER IN METALLURGICAL SPECTROSCOPY 65PRR-174
RE EARTH RADIONUCLIDES NUCLEAR SPECTROSCOPY OF NEUTRON DEFICIENT HAFNIUM AND RA 65A-11-01E
ESTIMATION AND INFLUENCE IN CROSS SECTION MEASURE/ OUTPUT SPECTRUM FROM 14-MEV NEUTRON GENERATORS - RAPID 65PRR-043
SODIUM FLUORIDE - POTASSIUM FLUORIDE ABSORPTION SPECTRUM OF COBALT(II) ION IN LITHIUM FLUORIDE - 65A-02-01B
SYNTHESIS AND INFRARED ABSORPTION SPECTRUM OF DI BORANE - BORON-10 65PRR-079
I. MATHEMATICAL RESOLUTION OF THE OVERL/ THE ABSORPTION SPECTRUM OF THE URANYL ION IN PER CHLORATE MEDIA 65PRR-161
TRIC APPLICATION A PROGRAMMABLE HIGH SPEED DATA ACQUISITION SYSTEM FOR MASS SPECTROME 65PRR-192
SPEED OF RESPONSE OF A POLAROGRAPH 65TR-04
PROCESS SOLUTIONS, MEASUREMENT OF PROMPT GAMMA-RAYS FROM SPONTANEOUS FISSION /252 IN TRANSURANIUM (TRU) 65PRR-130
GOLD ATOMS SPUTTERED FROM NEUTRON IRRADIATED GOLD FOILS 65A-11-08F
AVE POLAROGRAPH WITH SOLID STATE CIRCUITS / DOUBLE LAY/ A SQUARE-WAVE POLAROGRAPH (DESIGN OF NEW SQUARE-W 65A-01-14
S / DOUBLE LAY/ A SQUARE-WAVE POLAROGRAPH (DESIGN OF NEW SQUARE-WAVE POLAROGRAPH WITH SOLID STATE CIRCUIT 65A-01-14
E SPECTROPHOTOMETRIC STUDY OF THE STABILITY OF IODINE IN CYCLOHEXANE AND IN BENZEN 65A-04-04B
IN LIQUID SCINTILLATOR SYSTEMS USING AN ISOLATED INTERNAL STANDARD COLOR QUENCH CORRECTION 65PRR-047
IQUID SCINTILLATOR SYSTEMS BY USE OF AN ISOLATED INTERNAL STANDARD COLOR QUENCH CORRECTION IN L 65A-11-05B
METHIUM FOR THE ESTABLISHMENT OF A PRIMARY SPECTROGRAPHIC STANDARD /F URANIUM, AND THE PURIFICATION OF PRO 65PRR-089
EGRATED ASSEMBLY FOR GENERATION, STORAGE, AND DELIVERY OF STANDARD CHROMIUM(II) SOLUTIONS INT 65PRR-055
(THEORY OF COULOME/ PREPARATION OF CHAPTER FOR WELCHERS STANDARD METHODS OF CHEMICAL ANALYSIS, VOL. III. 65A-01-10
STANDARDIZATION OF NEUTRON FLUX MEASUREMENTS 65A-11-09C
AVAILABILITY AND USE OF RADIOACTIVITY STANDARDS 65PRR-024
CESIUM RADIOACTIVITY IN AQUEOUS SOLUTIONS, CHLORO STANNATE METHOD 65PRR-151
APPLICATION OF DERIVATIVE TECHNIQUES TO STATIONARY ELECTRODE POLAROGRAPHY 65PRR-038
HEMISTRY DIVISION, JAN.-DEC.,1964 STATISTICAL QUALITY CONTROL REPORT, ANALYTICAL C 65PRR-083
65, ANALYTICAL CHEMISTRY DIVISION STATISTICAL QUALITY CONTROL REPORT, APR.-JUNE,19 65PRR-081
65, ANALYTICAL CHEMISTRY DIVISION STATISTICAL QUALITY CONTROL REPORT, JAN.-MAR.,19 65PRR-082
964, ANALYTICAL CHEMISTRY DIVISION STATISTICAL QUALITY CONTROL REPORT, JAN.-SEPT.,1 65PRR-084
THE PRESENT STATUS OF THE ATOMIC WEIGHTS 65PRR-163
OLUTIONS INTEGRATED ASSEMBLY FOR GENERATION, STORAGE, AND DELIVERY OF STANDARD CHROMIUM(II) S 65PRR-055
/SUBTILIS / ALIPAL-LO-436 / AMMONIUM SALT OF A SULFONATED STRAIGHT CHAIN ALKYL PHENOXY POLY (ETHYLENE OX/ 65A-05-04M
METRIC SODIUM HYDROXIDE TITRATION METHOD STRONG AND WEAK ACIDS IN THEIR MIXTURES, POTENTI 65PRR-127
IVITY IN AQUEOUS OR ORGANIC SOLUTIONS STRONTIUM RADIOACTIVITY AND / OR BARIUM RADIOACT 65PRR-140
/EDIA I. MATHEMATICAL RESOLUTION OF THE OVERLAPPING BAND STRUCTURE AND STUDIES OF THE ENVIRONMENTAL EFFE/ 65PRR-161
PYROLYSIS AND GAS CHROMATOGRAPHIC DETERMINATION OF STYROFOAM DIELECTRIC FILM 65A-05-01B
/Y LIMIT OF POLAROGRAPHY / PRECISION OF DC POLAROGRAPHY / SUBTRACTIVE MODE AMPEROMETRIC POLAROGRAPHY / CO/ 65A-05-03A
RATIVE POLAROGRAPHY / DELTA-E-DIFFERENTIAL POLAROGRAPHY / SUBTRACTIVE POLAROGRAPHY) /POLAROGRAPHY (COMPA 65A-01-15
/Y / DIFFERENTIAL VOLTAMMETRY / COMPARATIVE VOLTAMMETRY / SUBTRACTIVE POLAROGRAPHY / POTENTIAL-SWEEP CHRO/ 65A-01-03
/CID / CHRONOPOTENTIOMETRIC OXIDATION OF FERROUS AMMONIUM SULFATE / CHRONOPOTENTIOMETRY WITH PLATINUM ELE/ 65A-01-02
MSRE) FUEL, SPECTROPHOTOMETRIC DI METHYL GLYOXIME (PER SULFATE OXIDATION) METHOD /REACTOR EXPERIMENT (65PRR-134
CHROMIUM IN MSRE FUEL, AMPEROMETRIC FERROUS SULFATE TITRATION METHOD 65PRR-136
/YPOBROMITE / POTASSIUM HEXA CHLORO RHENATE(IV) / HOLMIUM SULFIDE / ERBIUM SULFIDE / THULIUM PHOSPHIDE / / 65A-12
/SSIUM HEXA CHLORO RHENATE(IV) / HOLMIUM SULFIDE / ERBIUM SULFIDE / THULIUM PHOSPHIDE / THULIUM NITRIDE // 65A-12
N PROCESS ALKYL BENZENE SULFONATE (ABS) CONTROL FOR THE FOAM SEPARATIO 65PRR-183
METHYL METHANE SULFONATE (CARCINOGEN) 65A-13-01D
ANALYSIS OF ALKYL BENZENE SULFONATE DETERGENTS 65A-05-04K
// BACILLUS-SUBTILIS / ALIPAL-LO-436 / AMMONIUM SALT OF A SULFONATED STRAIGHT CHAIN ALKYL PHENOXY POLY (/ 65A-05-04M
NITRATE, PHENOL DI SULFONIC ACID SPECTROPHOTOMETRIC METHOD 65PRR-132
ASE METHODS FOR THE EVALUATION OF ATMOSPHERIC POLLUTION - SULFUR DI OXIDE RADIO RELE 65PRR-005
SULFUR-33 (N,P) CROSS SECTION 65A-11-03C
ASE METHODS FOR THE EVALUATION OF ATMOSPHERIC POLLUTION - SULPHUR DI OXIDE RADIO RELE 65A-11-07B
/LYTICAL CHEMISTRY RESEARCH AND DEVELOPMENT GROUP MONTHLY SUMMARY - NOV.,1964 / DEC.,1964 / JAN.,1965 / F/ 65PRR-104
/ OF URANIUM(III) CHLORIDE IN LANTHANUM CHLORIDE / SODIUM SUPEROXIDE / BARIUM BROMITE MONO HYDRATE / SODI/ 65A-12

/HOSPHIDE / THULIUM NITRIDE / HOLMIUM NITRIDE / POTASSIUM SUPEROXIDE / RUBIDIUM SUPEROXIDE / URANIUM(III)/ 65A-12
/RIDE / HOLMIUM NITRIDE / POTASSIUM SUPEROXIDE / RUBIDIUM SUPEROXIDE / URANIUM(III) CHLORIDE / URANIUM(II/ 65A-12
IN PROCEDURES FOR THE REMOVAL AND CHARACTERIZATION OF THE SURFACE CONTAMINANTS OF HAIR /TIVATION ANALYSIS 65PRR-008
SURFACE IONIZATION STUDIES 65A-08-03
EFFECTS OF SURFACE MATERIALS ON RADIO IODINE VAPORS 65PRR-063
AT ORNL A SURVEY OF ANALYTICAL INSTRUMENTATION DEVELOPMENT 65PRR-203
BORANE - BORON-10 SYNTHESIS AND INFRARED ABSORPTION SPECTRUM OF DI 65PRR-079
OLLED POTENTIAL COULOMETRIC TITRATION OF URANIUM(VI) IN A SYNTHETIC MSRE FUEL CONTR 65A-04-010
FLAME PHOTOMETRY (COLLECTION OF BARIUM FROM TAP WATER) 65A-07-010
STUDIES OF NEUTRON PRODUCING TARGETS 65A-11-09B
ANALYTICAL RADIOCHEMISTRY OF TECHNETIUM 65PRR-002
RY OF TECHNETIUM - THERMAL NEUTRON ACTIVATION ANALYSIS OF TECHNETIUM INORGANIC AND NUCLEAR CHEMIST 65PRR-077
OF TECHNETIUM INORGANIC AND NUCLEAR CHEMISTRY OF TECHNETIUM - THERMAL NEUTRON ACTIVATION ANALYSIS 65PRR-077
RAPID SEPARATION OF TECHNETIUM FROM FISSION PRODUCTS 65A-11-04C
RAPID SEPARATION OF TECHNETIUM FROM FISSION PRODUCTS 65PRR-076
TECHNETIUM STUDIES 65A-05-04N
CROSS SECTIONS FOR THE REACTIONS OF 14-MEV NEUTRONS WITH TECHNETIUM-99 ACTIVATION 65A-11-09E
ROSS SECTIONS FOR THE REACTIONS OF 14.8-MEV NEUTRONS WITH TECHNETIUM-99 ACTIVATION C 65PRR-075
DETERMINATION OF TECHNETIUM-99 BY NEUTRON ACTIVATION ANALYSIS 65A-11-08C
DECAY OF MOLYBDENUM-99 - TECHNETIUM-99M 65PRR-011
REVIEWS AND TECHNICAL ASSISTANCE 65A-11-02A
ANALYTICAL CHEMISTRY IN NUCLEAR TECHNOLOGY 65PRR-052
EIGHTH CONFERENCE ON ANALYTICAL CHEMISTRY IN NUCLEAR TECHNOLOGY 65PRR-053
APPLICATIONS OF GAS CHROMATOGRAPHY IN NUCLEAR TECHNOLOGY 65PRR-180
THE EIGHTH CONFERENCE ON ANALYTICAL CHEMISTRY IN NUCLEAR TECHNOLOGY A RESUME OF 65PRR-054
ACID (1-12-MOLAR) WITH A DROPPING MERCURY ELECTRODE OF TEFLON /APHY OF LEAD(II) IN AQUEOUS HYDROFLUORIC 65PRR-041
C STATE CIRCUITS / DOUBLE LAYER CAPACITANCE MEASUREMENT / TEFLON DROPPING MERCURY ELECTRODE) /H WITH SOLI 65A-01-14
STUDIES WITH THE TEFLON DROPPING MERCURY ELECTRODE (D.M.E.) 65A-05-03
/ION IN AQUEOUS HYDROFLUORIC ACID WITH HORIZONTAL ORIFICE TEFLON DROPPING MERCURY ELECTRODE / SIGNIFICANC/ 65A-05-03A
SIGNAL TO NOISE RATIOS (S/N) OF GLASS AND TEFLON DROPPING MERCURY ELECTRODE CAPILLARIES 65A-05-03B
/S CORRODING MEDIA (EVALUATION OF THE HORIZONTAL ORIFICE TEFLON DROPPING MERCURY ELECTRODE FOR OBTAINING/ 65A-05-03A
HY IN HYDROFLUORIC ACID AND OTHER GLASS CORRODING MEDIA / TEFLON DROPPING MERCURY ELECTRODE FOR POLAROGRAP 65PRR-039
OTHER GLASS CORRODING MED/ DROPPING MERCURY ELECTRODE OF TEFLON FOR POLAROGRAPHY IN HYDROFLUORIC ACID AND 65PRR-040
ISOTOPIC COMPARISON OF LEAD FROM TEKTITES AND CONTEMPORANEOUS IMPACT CRATERS 65A-08-04
LUTIONS TELLURIUM RADIOACTIVITY IN AQUEOUS OR ORGANIC SO 65PRR-144
HIGH TEMPERATURE CHEMICAL DOSIMETRY 65A-03-03
/LINEAR, RECORDING CONDUCTOMETRIC TITRATOR WITH AUTOMATIC TEMPERATURE COMPENSATION OR PROPORTIONAL TEMPER/ 65PRR-036
R WITH AUTOMATIC TEMPERATURE COMPENSATION OR PROPORTIONAL TEMPERATURE CONTROL /DING CONDUCTOMETRIC TITRATO 65PRR-036
ARBITRARY-SET, PROPORTIONAL TEMPERATURE CONTROLLER 65PRR-035
OF CARBONIC ACID IN CON/ PREDICTION OF THE SOLUBILITY AND TEMPERATURE DEPENDENCE OF DISSOCIATION CONSTANT 65TR-08
REVIEW OF ANALYTICAL CHEMISTRY OF THALLIUM 65PRR-057
/ FUNDAMENTAL POLAROGRAPHIC DATA / MONOVALENT THALLIUM TO THALLIUM METAL REDOX REACTION / DIVALENT LEAD T/ 65A-05-03A
CORRODING MEDIA - EVALUATION WITH MONOVALENT THALLIUM TO THALLIUM REDOX REACTION /IC ACID AND OTHER GLASS 65PRR-039
/OR OBTAINING FUNDAMENTAL POLAROGRAPHIC DATA / MONOVALENT THALLIUM TO THALLIUM METAL REDOX REACTION / DIV/ 65A-05-03A
OTHER GLASS CORRODING MEDIA - EVALUATION WITH MONOVALENT THALLIUM TO THALLIUM REDOX REACTION /IC ACID AND 65PRR-039
G VARIABLES FROM BIOMEDICAL SAMPLES IN LIQUID SCINTILLAT/ THEORETICAL AND EXPERIMENTAL ASPECTS OF QUENCHIN 65PRR-199
-3 ACTIVATION ANALYSIS - HELIUM-3 REACTIONS AS NEUTRON S/ THEORY AND EXPERIMENT IN RAPID, SENSITIVE HELIUM 65PRR-045
REVIEW OF THE THEORY AND PRACTICE OF SCINTILLATION COUNTING 65PRR-025
/CHERS STANDARD METHODS OF CHEMICAL ANALYSIS, VOL. III. (THEORY OF COULOMETRIC METHODS / TECHNIQUE OF CO/ 65A-01-10
M INORGANIC AND NUCLEAR CHEMISTRY OF TECHNETIUM - THERMAL NEUTRON ACTIVATION ANALYSIS OF TECHNETIU 65PRR-077
TRI METHYL BORAZINE AS A LIQUID SCINTILLATOR SOLVENT FOR THERMAL NEUTRON DETECTION N,N,N 65A-11-05A
TRI METHYL BORAZINE AS A LIQUID SCINTILLATOR SOLVENT FOR THERMAL NEUTRON DETECTION N,N,N, 65PRR-049
/DS THAT CONTAIN PHOSPHORO THIOATE, ,P=S AND PHOSPHORO DI THIOATE, ,P(S)SH GROUPS AS SEPARATION AND ANALY/ 65A-11-04B
/UNDS CONTAINING PHOSPHORO THIOATE, ,P=S AND PHOSPHORO DI THIOATE, ,P(S)SH GROUPS AS SEPARATORY AND ANALY/ 65PRR-017
GROUP/ REVIEW OF ORGANIC COMPOUNDS THAT CONTAIN PHOSPHORO THIOATE, ,P=S AND PHOSPHORO DI THIOATE, ,P(S)SH 65A-11-04B
GROUP/ A REVIEW OF ORGANIC COMPOUNDS CONTAINING PHOSPHORO THIOATE, ,P=S AND PHOSPHORO DI THIOATE, ,P(S)SH 65PRR-017
USE OF METAL DI N BUTYL PHOSPHORO DI THIOATES AS EXTRACTANTS OF METALS 65PRR-016
ENCE OF FLUORIDE SPECTROPHOTOMETRIC THIOCYANATE DETERMINATION OF NIOBIUM IN THE PRES 65A-05-04H
- SELECTIVE EXTRACTIO/ TRI ALKYL AND HEXA ALKYL PHOSPHORO THIOIC TRI AMIDES AS EXTRACTANTS FOR METAL IONS 65PRR-015
HIUM FLUORIDE - BERYLLIUM FLUORIDE - ZIRCONIUM FLUORIDE - THORIUM FLUORIDE - URANIUM(IV) FLUORIDE /TED LIT 65-02-01A
ED POTENTIAL COULOME/ URANIUM(IV) TO URANIUM(VI) RATIO IN THORIUM OXIDE - URANIUM OXIDE MIXTURES, CONTROLL 65PRR-155
DETERMINATION OF URANIUM(VI) IN URANIUM OXIDE - THORIUM OXIDE FUELS 65A-05-04G
N,N,N TRI HEXYL PHOSPHORIC TRI AMIDE (THPA) AS AN EXTRACTANT FOR METAL IONS 65A-11-04A
// HOLMIUM SULFIDE / ERBIUM SULFIDE / THULIUM PHOSPHIDE / THULIUM NITRIDE / HOLMIUM NITRIDE / POTASSIUM S/ 65A-12
/ CHLORO RHENATE(IV) / HOLMIUM SULFIDE / ERBIUM SULFIDE / THULIUM PHOSPHIDE / THULIUM NITRIDE / HOLMIUM N/ 65A-12
/ETRY / HAMMER-CONTROL OF DROPPING MERCURY ELECTRODE DROP TIME / LINEAR TIME BASE / SMOLER DROPPING MERCU/ 65A-01-04
-CONTROL OF DROPPING MERCURY ELECTRODE DROP TIME / LINEAR TIME BASE / SMOLER DROPPING MERCURY ELECTRODE) 65A-01-04
CISION AND ACCURACY IN NEUTRON ACTIVATION ANALYSIS (DEAD TIME EFFECTS) STUDY OF PRE 65A-11-08B
DETERMINATION OF LEAD, BISMUTH, CADMIUM, TIN, AND ZINC IN AIRBORNE DUST SAMPLES 65A-07-02A
TISSUE ANALYSIS (TISSUE HOMOGENIZATION) 65A-07-01E
TISSUE ANALYSIS (TISSUE HOMOGENIZATION) 65A-07-01E
DETERMINATION OF TITANIUM IN SEAWATER 65A-05-04A
ERMINATION OF NITRATE BY CONTROLLED POTENTIAL COULOMETRIC TITRATION DET 65A-05-04J
/-Q COULOMETRIC METHOD / CONTROLLED POTENTIAL COULOMETRIC TITRATION / BLANK CORRECTIONS IN COULOMETRY / P/ 65A-01-07
CHLORIDE, DISTILLATION - INDIRECT IODOMETRIC TITRATION METHOD 65PRR-126
ZIRCONIUM IN MSRE FUEL, AMPEROMETRIC CUPFERRON TITRATION METHOD 65PRR-135
CHROMIUM IN MSRE FUEL, AMPEROMETRIC FERROUS SULFATE TITRATION METHOD 65PRR-136
ACIDS IN THEIR MIXTURES, POTENTIOMETRIC SODIUM HYDROXIDE TITRATION METHOD STRONG AND WEAK 65PRR-127
FROM IRRADIATED ION EXCHANGE RESINS, INDIRECT IODOMETRIC TITRATION METHOD OXALATE IN ELUATES 65PRR-158
SENSITIVITY, AUTOMATIC, CONTROLLED POTENTIAL COULOMETRIC TITRATION METHOD /EXPERIMENT (MSRE) FUEL, HIGH 65PRR-122
URANIUM OXIDE MIXTURES, CONTROLLED POTENTIAL COULOMETRIC TITRATION METHOD /M(VI) RATIO IN THORIUM OXIDE - 65PRR-155
MSRE) FUEL, AUTOMATIC, CONTROLLED POTENTIAL, COULOMETRIC TITRATION METHOD /TEN SALT REACTOR EXPERIMENT (65PRR-121
L CONTROLLED POTENTIAL COULOMETRIC TITRATION OF URANIUM(VI) IN A SYNTHETIC MSRE FUE 65A-04-010
SODIUM FLUORIDE CONTROLLED POTENTIAL COULOMETRIC TITRATION OF URANIUM(VI) IN AQUEOUS SOLUTIONS OF 65PRR-034
REVIEW OF AMPEROMETRIC TITRATIONS 65PRR-060
RMINAT/ HIGH SENSITIVITY CONTROLLED POTENTIAL COULOMETRIC TITRATOR - CONTROLLED POTENTIAL COULOMETRIC DETE 65PRR-020
/NTROLLED POTENTIAL COULOMETRY / MODEL-Q-2564 COULOMETRIC TITRATOR / 1-VERSUS-Q COULOMETRIC METHOD / CONT/ 65A-01-07
HIGH-SENSITIVITY COULOMETRIC TITRATOR MODEL-Q-2564 65PRR-118
/TIVITY, DIRECT READING, LINEAR, RECORDING CONDUCTOMETRIC TITRATOR WITH AUTOMATIC TEMPERATURE COMPENSATIO/ 65PRR-036
, ORNL MODEL-Q-2564 COULOMETRIC TITRATOR, CONTROLLED POTENTIAL, HIGH SENSITIVITY 65PRR-125
LED POTENTIAL AUTOMATIC COULOMETRIC TITRATOR, ORNL MODEL-Q-2005, ELECTRONIC, CONTROL 65PRR-146
TROLLED POTENTIAL / BLANK C/ HIGH SENSITIVITY COULOMETRIC TITRATOR, ORNL MODEL-Q-2564 (POTENTIOSTAT / CON 65A-01-05
(P.G.E.) AND THE GLASSY CARBON ELECTRODE/ DIFFERENTIAL TITRIMETRY WITH THE PYROLYTIC GRAPHITE ELECTRODE 65A-05-02C

RADIOACTIVATION ANALYSIS AS AN ANALYTICAL TOCL 65PRR-187
/QUANTITIES OF URANIUM IN A TRI N OCTYL PHOSPHINE OXIDE (TOPO) EXTRACT (MICROANALYTICAL DETERMINATION / 65A-01-06
ENTS FROM AQUEOUS SOLUTIONS PRIOR TO THE DETERMINATION OF TOTAL LANTHANIDE FISSION PRODUCTS /URANIUM ELEM 65PRR-032
/TION PRECEEDING POLAROGRAPHIC DETERMINATION OF URANIUM / TRACE ANALYSIS FOR URANIUM / APPLICATION OF QUA/ 65A-01-06
TRACE ELEMENTS IN HUMAN HAIR 65PRR-009
PRODUCTS TRACE HIGH ENERGY GAMMA RADIOACTIVITY IN ISOTOPE 65A-11-02E
NITROGEN, AND CARBON DI OXIDE IN GLOVE BO/ THE EFFECTS OF TRACE IMPURITIES OF MOISTURE, OXYGEN, HYDROGEN, 65PRR-175
CELLULOSE COLUMN SEPARATION OF TRACE METALS FROM URANIUM BY CHROMATOGRAPHY ON A 65A-11-04E
DETERMINATION OF TRACE METALS IN NIOBIUM - IRON ALLOY 65A-05-04B
IAL DC POLAROGRAPHY IN A TRI OCTYL PHOS/ DETERMINATION OF TRACE QUANTITIES OF URANIUM BY CONTROLLED POTENT 65PRR-160
CN CELLULOSE COLUMN SEPARATION OF TRACES OF METALS FROM URANIUM BY CHROMATOGRAPHY 65PRR-037
TUDIES TRANSFER RIBO NUCLEIC ACID (TRNA) ANALYTICAL S 65A-06-01
STUDIES ON THE PHENYL ALANYL TRANSFER RIBO NUCLEIC ACID (TRNA) SYSTEM 65A-06-02
NG CIRCUITS TRANSIENT PROCESSES WITH POTENTIOSTATIC REGULATI 65TR-03
PRODUCTS METHOD FOR SEPARATING TRANSPLUTONIUM ELEMENTS FROM RARE EARTH FISSION 65PRR-223
OTHER ORNL MASTER ANALYTICAL MANUAL METHODS NEEDED IN THE TRANSURANIUM (TRU) PROCESS 65PRR-128
ENT OF PROMPT GAMMA-RAYS FROM SPONTAN/ CALIFORNIUM-252 IN TRANSURANIUM (TRU) PROCESS SOLUTIONS, MEASUREM 65PRR-130
Y QUINOLINE PRECIPITATION - / MOLYBDENUM RADIOACTIVITY IN TRANSURANIUM (TRU) PROCESS SOLUTIONS, 8 HYDROX 65PRR-129
HOTOMETRIC METHOD NITRATE IN TRANSURANIUM (TRU) SOLUTIONS, AZO DYE FILTER P 65PRR-123
TRANSURANIUM ELEMENT (TRU) MASS SPECTROMETRY 65A-08-02
TRANSURANIUM ELEMENT PROCESSING PROGRAM 65A-04-02
ISOTOPIC ANALYSIS OF TRANSURANIUM ELEMENTS BY MASS SPECTROMETRY 65PRR-167
OR TO THE DETERMINATION OF TOTAL LANTHA/ RAPID REMOVAL OF TRANSURANIUM ELEMENTS FROM AQUEOUS SOLUTIONS PRI 65PRR-032
REPORT ON FOREIGN TRAVEL JAN.16-FEB.20,1965 65PRR-101
REPORT ON FOREIGN TRAVEL JUNE-25-JULY-30,1965 65PRR-072
REPORT ON FOREIGN TRAVEL JUNE-30-JULY-10,1965 65PRR-070
REPORT ON FOREIGN TRAVEL MAY-15-JUNE-9,1965 65PRR-095
REPORT OF FOREIGN TRAVEL OCT.10-28,1964 65PRR-087
REPORT ON FOREIGN TRAVEL OCT.19-30,1964 65PRR-099
REPORT ON FOREIGN TRAVEL SEPT.2-OCT.6,1965 65PRR-085
IDES AS EXTRACTANTS FOR METAL IONS - SELECTIVE EXTRACTIO/ TRI ALKYL AND HEXA ALKYL PHOSPHORO THIOIC TRI AM 65PRR-015
NS N,N,N TRI HEXYL PHOSPHORIC TRI AMIDE (THPA) AS AN EXTRACTANT FOR METAL IO 65A-11-04A
TIVE EXTRACTIO/ TRI ALKYL AND HEXA ALKYL PHOSPHORO THIOIC TRI AMIDES AS EXTRACTANTS FOR METAL IONS - SELEC 65PRR-015
PHOSPHONIUM CHL/ QUATERNARY PHOSPHONIUM HALIDES (BENZYL TRI BUTYL PHOSPHONIUM BROMIDE / BENZYL TRI BUTYL 65A-13-01A
M HALIDES (BENZYL TRI BUTYL PHOSPHONIUM BROMIDE / BENZYL TRI BUTYL PHOSPHONIUM CHLORIDE) /ARY PHOSPHONIU 65A-13-01A
NO HYDRATE / SODIUM HEXA CHLORO RHENATE(IV) / URANIUM-235 TRI CHLORIDE) /M SUPEROXIDE / BARIUM BROMITE MO 65A-12
TRACTANT FOR METAL IONS N,N,N TRI HEXYL PHOSPHORIC TRI AMIDE (THPA) AS AN EX 65A-11-04A
VENT FOR THERMAL NEUTRON DETECTION N,N,N TRI METHYL BORAZINE AS A LIQUID SCINTILLATOR SOL 65A-11-05A
VENT FOR THERMAL NEUTRON DETECTION N,N,N, TRI METHYL BORAZINE AS A LIQUID SCINTILLATOR SOL 65PRR-049
/TO DETERMINATION OF MICROGRAM QUANTITIES OF URANIUM IN A TRI N OCTYL PHOSPHINE OXIDE (TOPO) EXTRACT (/ 65A-01-06
S OF URANIUM BY CONTROLLED POTENTIAL DC POLAROGRAPHY IN A TRI OCTYL PHOSPHINE OXIDE EXTRACT /ACE QUANTITIE 65PRR-160
POLAROGRAPHY WITH A TRIANGULAR POTENTIAL IMPULSE 65TR-05
RELEASE OF TRITIUM BY TRITIATED PAINT 65A-11-02G
RADIO GAS CHROMATOGRAPHY OF THE TRITIUM - WATER - METHANE SYSTEM 65A-05-01A
RELEASE OF TRITIUM BY TRITIATED PAINT 65A-11-02G
OF ORGANIC ACIDS WITH HIGH MOLECULAR WEIGHT AMINES - THE TRIVALENT ACTINIDE - LANTHANIDE ELEMENTS /UTIONS 65PRR-031
TRANSFER RIBO NUCLEIC ACID (TRNA) ANALYTICAL STUDIES 65A-06-01
STUDIES ON THE PHENYL ALANYL TRANSFER RIBO NUCLEIC ACID (TRNA) SYSTEM 65A-06-02
TRANSURANIUM ELEMENT (TRU) MASS SPECTROMETRY 65A-08-02
ER ANALYTICAL MANUAL METHODS NEEDED IN THE TRANSURANIUM (TRU) PROCESS OTHER ORNL MAST 65PRR-128
AMMA-RAYS FROM SPONTAN/ CALIFORNIUM-252 IN TRANSURANIUM (TRU) PROCESS SOLUTIONS, MEASUREMENT OF PROMPT G 65PRR-130
CIPITATION - / MOLYBDENUM RADIOACTIVITY IN TRANSURANIUM (TRU) PROCESS SOLUTIONS, 8 HYDROXY QUINOLINE PRE 65PRR-129
OD NITRATE IN TRANSURANIUM (TRU) SOLUTIONS, AZO DYE FILTER PHOTOMETRIC METH 65PRR-123
ORNE RADIO IODINE SPECIES BY MEANS OF COMPOSITE DIFFUSION TUBES INVESTIGATION OF AIRB 65PRR-067
E SPECIES FOUND ON RUBBER SECTIONS OF COMPOSITE DIFFUSION TUBES CHEMICAL BEHAVIOR OF THE RADIO IODIN 65PRR-069
T CONDITIONS IN MAY PACKS BY MEANS OF COMPOSITE DIFFUSION TUBES /ATION OF RADIO IODINE BEHAVIOR UNDER MCIS 65PRR-066
MENTAL RADIO IODINE VAPOR BY MEANS OF COMPOSITE DIFFUSION TUBES /TIGATION OF CHEMICAL REACTIVITY OF NONELE 65PRR-068
L STUDY OF THE EFFECT OF AGING ON CARBIDES IN A NIOBIUM - TUNGSTEN - ZIRCONIUM ALLOY ELECTRON OPTICA 65PRR-210
Y NEUTRON ACTIVATION / MICRO ELEMENT CONTENT OF WOCL FROM TWIN WETHERS RAISED ON TWO SOILS AS DETERMINED B 65PRR-019
METHODS USED AT ORNL FOR THE PRECISE DETERMINATION OF URANIUM 65PRR-102
METHODS USED AT ORNL FOR THE PRECISE DETERMINATION OF URANIUM 65PRR-205
R BY LAUER, BRUCK, AND LEDUIGOU, PRECISE DETERMINATION OF URANIUM - A COMPARISON OF DIFFERENT METHODS /APE 65PRR-202
BY LAUER, BRUCK, AND LEDUIGOU, PRECISE DETERMINATIONS OF URANIUM - A COMPARISON OF DIFFERENT METHODS /PER 65PRR-098
/AROGRAPHIC DETERMINATION OF URANIUM / TRACE ANALYSIS FOR URANIUM / APPLICATION OF QUASI REFERENCE ELECTR/ 65A-01-06
LOSS IN POLAROGRAPHY / POLAROGRAPHIC DETECTION LIMITS FOR URANIUM / CONCENTRATION ADVANTAGE / SEAWATER) / 65A-01-06
/ENT EXTRACTION PRECEDING POLAROGRAPHIC DETERMINATION OF URANIUM / TRACE ANALYSIS FOR URANIUM / APPLICAT/ 65A-01-06
/XIDE (TOPO) EXTRACT (MICROANALYTICAL DETERMINATION OF URANIUM / 0.5-ML POLAROGRAPHIC CELL / ELECTROCH/ 65A-01-06
IC DETERMINATION OF MILLIGRAM AND MICROGRAM QUANTITIES OF URANIUM AND IRON /CONTROLLED POTENTIAL COULOMETR 65PRR-020
SEPARATION OF TRACE METALS FROM URANIUM BY CHROMATOGRAPHY ON A CELLULOSE COLUMN 65A-11-04E
SEPARATION OF TRACES OF METALS FROM URANIUM BY CHROMATOGRAPHY ON CELLULOSE COLUMN 65PRR-037
/URANIUM BY DERIVATIVE DC POLAROGRAPHY / DETERMINATION OF URANIUM BY CONTROLLED POTENTIAL COULOMETRY / DE/ 65A-01-07
IN A TRI OCTYL PHOS/ DETERMINATION OF TRACE QUANTITIES OF URANIUM BY CONTROLLED POTENTIAL DC POLAROGRAPHY 65PRR-160
/OMETRY AS MICRO ANALYTICAL TECHNIQUES (DETERMINATION OF URANIUM BY DERIVATIVE DC POLAROGRAPHY / DETERMI/ 65A-01-07
/ APPLICATION TO DETERMINATION OF MICROGRAM QUANTITIES OF URANIUM IN A TRI N OCTYL PHOSPHINE OXIDE (TOPO/ 65A-01-06
) FUEL, AUTOMATIC, CONTROLLED POTENTIAL, COULOMETRIC TI/ URANIUM IN MOLTEN SALT REACTOR EXPERIMENT (MSRE 65PRR-121
URANIUM IN MSRE FUEL, POLAROGRAPHIC METHOD 65PRR-138
IS DETERMINATION OF URANIUM IN SEAWATER BY NEUTRON ACTIVATION ANALYS 65A-11-08G
MSRE) FUEL, HIGH SENSITIVITY, AUTOMATIC, CONTROLLED POT/ URANIUM IN THE MOLTEN SALT REACTOR EXPERIMENT (65PRR-122
DETERMINATION OF URANIUM(VI) IN URANIUM OXIDE - THORIUM OXIDE FUELS 65A-05-04G
LOME/ URANIUM(IV) TO URANIUM(VI) RATIO IN THORIUM OXIDE - URANIUM OXIDE MIXTURES, CONTROLLED POTENTIAL COU 65PRR-155
SPECTROPHOTOMETRIC DETERMINATION OF URANIUM WITH ARSENAZO-III 65A-05-04C
/NCE OF LONG-LIVED ISOTOPES OF PROMETHIUM FROM FISSION OF URANIUM, AND THE PURIFICATION OF PROMETHIUM FOR/ 65PRR-089
M BROMITE MONO HYDRATE / SODIUM HEXA CHLORO RHENATE(IV) / URANIUM-235 TRI CHLORIDE) /M SUPEROXIDE / BARIU 65A-12
GAMMA-RAY BRANCHINGS IN URANIUM-237 65A-11-01C
/M NITRIDE / POTASSIUM SUPEROXIDE / RUBIDIUM SUPEROXIDE / URANIUM(III) CHLORIDE / URANIUM(III) IODIDE / S/ 65A-12
/III) CHLORIDE / URANIUM(III) IODIDE / SOLID SOLUTIONS OF URANIUM(III) CHLORIDE IN LANTHANUM CHLORIDE / S/ 65A-12
/PEROXIDE / RUBIDIUM SUPEROXIDE / URANIUM(III) CHLORIDE / URANIUM(III) IODIDE / SOLID SOLUTIONS OF URANIU/ 65A-12
YLLIUM FLUORIDE - ZIRCONIUM FLUORIDE - THORIUM FLUORIDE - URANIUM(IV) FLUORIDE /TED LITHIUM FLUORIDE - BER 65-02-01A
ERMINATION OF URANIUM(VI) IN PHOSPHORIC ACID SOLUTIONS OF URANIUM(IV) OXIDE /60 GAMMA RADIATION ON THE DET 65PRR-021
ATION METHOD PARTICLE SIZE DISTRIBUTION IN URANIUM(IV) OXIDE, NEUTRON ACTIVATION - SEDIMENT 65PRR-154
E - URANIUM OXIDE MIXTURES, CONTROLLED POTENTIAL COULOME/ URANIUM(IV) TO URANIUM(VI) RATIO IN THORIUM OXID 65PRR-155
CONTROLLED POTENTIAL COULOMETRIC TITRATION OF URANIUM(VI) IN A SYNTHETIC MSRE FUEL 65A-04-01D
IDE CONTROLLED POTENTIAL COULOMETRIC TITRATION OF URANIUM(VI) IN AQUEOUS SOLUTIONS OF SODIUM FLUOR 65PRR-034

/ECT OF COBALT-60 GAMMA RADIATION ON THE DETERMINATION OF URANIUM(VI) IN PHOSPHORIC ACID SOLUTIONS OF URA/ 65PRR-021
LS DETERMINATION OF URANIUM(VI) IN URANIUM OXIDE - THORIUM OXIDE FUE 65A-05-04G
DE MIXTURES, CONTROLLED POTENTIAL COULOME/ URANIUM(IV) TO URANIUM(VI) RATIO IN THORIUM OXIDE - URANIUM OXI 65PRR-155
L RESOLUTION OF THE OVERL/ THE ABSORPTION SPECTRUM OF THE URANYL ION IN PER CHLORATE MEDIA I. MATHEMATICA 65PRR-161
IAL COULOMETRY (COULOMETRIC DETERMINATION OF PLUTONIUM) UTILITY OF I-VERSUS-Q PLOTS IN CONTROLLED POTENT 65A-01-08
THE RAPID DETERMINATION OF L LEUCINE, L ISOLEUCINE, AND L VALINE IN A MIXTURE BY INFRARED SPECTROMETRY 65TR-09
/ION POTENTIALS IN THE MEASUREMENT OF HALF-WAVE POTENTIAL VALUES / SENSITIVITY LIMIT OF POLAROGRAPHY / PR/ 65A-05-03A
/F JUNCTION POTENTIALS AT THE SALT BRIDGE ON THE MEASURED VALUES FOR THE HALF-WAVE POTENTIALS OF THE DIVA/ 65PRR-010
ATION OF CHEMICAL REACTIVITY OF NONELEMENTAL RADIO IODINE VAPOR BY MEANS OF COMPOSITE DIFFUSION TUBES /TIG 65PRR-068
IDENTITY, CHARACTER AND CHEMICAL BEHAVIOR OF VAPOR FORMS OF RADIO IODINE 65PRR-162
GAS CHROMATOGRAPHIC IDENTIFICATION OF RADIO IODINE VAPORS 65PRR-062
EFFECTS OF SURFACE MATERIALS ON RADIO IODINE VAPORS 65PRR-063
GAS CHROMATOGRAPHIC IDENTIFICATION OF RADIO IODINE VAPORS 65PRR-064
TILLAT/ THEORETICAL AND EXPERIMENTAL ASPECTS OF QUENCHING VARIABLES FROM BIOMEDICAL SAMPLES IN LIQUID SCIN 65PRR-199
ION METHOD OF/ SEMIQUANTITATIVE SPECTROGRAPHIC ANALYSIS (VISUAL INTERPOLATION) (LITHIUM CARBONATE DILUT 65A-07-01F
/PTER FOR WELCHERS STANDARD METHODS OF CHEMICAL ANALYSIS, VOL. III. (THEORY OF COULOMETRIC METHODS / TEC/ 65A-01-10
HIGH VOLTAGE PAPER ELECTROPHORESIS 65A-11-04D
CURE FOR MODEL-Q-2792 CONTROLLED POTENTIAL DC POLAROGRAPH VOLTAMMETER CHECK OUT AND TEST PROCE 65PRR-080
ESI/ DIFFERENTIATING CIRCUIT FOR THE CONTROLLED POTENTIAL VOLTAMMETER (DERIVATIVE COMPUTER / INSTRUMENT D 65A-01-11
TRY / CHRONOAMPEROME/ CONTROLLED POTENTIAL DC POLAROGRAPH VOLTAMMETER, ORNL MODEL-Q-2792 (CYCLIC VOLTAMME 65A-01-04
A GLASSY CARBON ELECTRODE FOR VOLTAMMETRY 65PRR-061
/L DC POLAROGRAPH VOLTAMMETER, ORNL MODEL-Q-2792 (CYCLIC VOLTAMMETRY / CHRONOAMPEROMETRY / HAMMER-CONTRO/ 65A-01-04
/ON OF CADMIUM / DIFFERENTIAL POLAROGRAPHY / DIFFERENTIAL VOLTAMMETRY / COMPARATIVE VOLTAMMETRY / SUBTRAC/ 65A-01-03
/AL POLAROGRAPHY / DIFFERENTIAL VOLTAMMETRY / COMPARATIVE VOLTAMMETRY / SUBTRACTIVE POLAROGRAPHY / POTENT/ 65A-01-03
POTENTIOMETER WITH COMPENSATION FOR EXTRANEOUS CURRENTS (VOLTAMMETRY AT CONTROLLED CURRENT) CHRONO 65A-01-01
HE PYROLYTIC GRAPHITE INDICATING ELECTRODE VOLTAMMETRY OF IODIDE, IODINE, AND IODATE WITH T 65PRR-193
LVER / IRON / NICKEL / LITHIUM FLUORIDE - BERYLLIUM FLUO/ VOLTAMMETRY OF MOLTEN FLUORIDE SALT SYSTEMS (SI 65A-02-02
VOLTAMMETRY OF THE IODINE SYSTEM 65A-05-02D
OMETRY (VOLTAMMETRY WITH CONTROLLED CURRENT / DERIVATIVE VOLTAMMETRY WITH CONTROLLED CURRENT) /NOPOTENTI 65A-01-09
/OTENTIOMETRY WITH COMPENSATION FOR EXTRANEOUS CURRENTS (VOLTAMMETRY WITH CONTROLLED CURRENT / CHRONOPOT/ 65A-01-02
VOLTAMMETRY WITH CONTR/ DERIVATIVE CHRONOPOTENTIOMETRY (VOLTAMMETRY WITH CONTROLLED CURRENT / DERIVATIVE 65A-01-09
REVERSIBLE DEPOSITION OF METALS ON SOLID ELECTRODES BY VOLTAMMETRY WITH LINEARLY VARYING POTENTIAL 65A-02-02A
REVERSIBLE DEPOSITION OF METALS ON SOLID ELECTRODES BY VOLTAMMETRY WITH LINEARLY VARYING POTENTIAL 65PRR-030
REVERSIBLE DEPOSITION OF METALS ON SOLID ELECTRODES BY VOLTAMMETRY WITH LINEARLY VARYING POTENTIAL 65PRR-191
PROBLEMS IN DISPOSAL OF RADIOACTIVE LIQUID WASTES IN SALT 65PRR-023
NEUTRON ACTIVATION ANALYSIS OF RIVER WATER 65A-11-08H
FLAME PHOTOMETRY (COLLECTION OF BARIUM FROM TAP WATER) 65A-07-01D
RADIO GAS CHROMATOGRAPHY OF THE TRITIUM - WATER - METHANE SYSTEM 65A-05-01A
MAGNESIUM IN PROCESS WATER, SPECTROPHOTOMETRIC MAGON METHOD 65PRR-159
IUM HYDROXIDE TITRATION METHOD STRONG AND WEAK ACIDS IN THEIR MIXTURES, POTENTIOMETRIC SOD 65PRR-127
ILLATION COUNTING OF IRON-55 MEASUREMENT OF WEAR RATES IN AUTOMOTIVE ENGINES BY LIQUID SCINT 65A-11-07C
ILLATION COUNTING OF IRON-55 WEAR RATES IN AUTOMOTIVE ENGINES BY LIQUID SCINT 65PRR-048
/M AQUEOUS SOLUTIONS OF ORGANIC ACIDS WITH HIGH MOLECULAR WEIGHT AMINES - THE TRIVALENT ACTINIDE - LANTHA/ 65PRR-031
THE PRESENT STATUS OF THE ATOMIC WEIGHTS 65PRR-163
VOL. III. (THEORY OF COULOME/ PREPARATION OF CHAPTER FOR WELCHERS STANDARD METHODS OF CHEMICAL ANALYSIS, 65A-01-10
TRON ACTIVATION / MICRO ELEMENT CONTENT OF WOOL FROM TWIN WETHERS RAISED ON TWO SOILS AS DETERMINED BY NEU 65PRR-019
IPAL-LO-43/ ABSORPTION SPECTRAL MEASUREMENTS (SOLAR CELL WINDOWS / RIBO NUCLEASE / BACILLUS-SUBTILIS / AL 65A-05-04M
WOOL ANALYSIS 65A-11-08I
DETERMINATION OF MOLYBDENUM IN HAIR AND WOOL BY NEUTRON ACTIVATION ANALYSIS 65PRR-018
TERMINED BY NEUTRON ACTIVATION / MICRO ELEMENT CONTENT OF WOOL FROM TWIN WETHERS RAISED ON TWO SOILS AS DE 65PRR-019
QUANTITATIVE ANALYSIS BY THE X-RAY ABSORPTION EDGE TECHNIQUE 65PRR-166
ORESCENCE / ELECTRON PROBE ANALYZER / DETERMINATION OF A/ X-RAY ANALYSIS (ANALYSIS OF SOLIDS BY X-RAY FLU 65A-07-01A
X-RAY AND SPECTROCHEMICAL ANALYSES 65A-07
X-RAY AND SPECTROCHEMICAL ANALYSES (X-10) 65A-07-01
X-RAY AND SPECTROCHEMICAL ANALYSES (Y-12) 65A-07-02
OF GOLD COATING ON SILICON RADIATION DETECTORS BY USE OF X-RAY FLUORESCENCE) /RMINATION OF AREAL DENSITY 65A-07-01A
ETERMINATION OF A/ X-RAY ANALYSIS (ANALYSIS OF SOLIDS BY X-RAY FLUORESCENCE / ELECTRON PROBE ANALYZER / D 65A-07-01A
SIS OF HIGHLY RADIOACTIVE MATERIALS - SPECTROCHEMICAL AND X-RAY TECHNIQUES THE QUANTITATIVE ANALY 65PRR-173
X-RAY AND SPECTROCHEMICAL ANALYSES (X-10) 65A-07-01
TERMINATION OF OXIDIZING POWER OF SOLUTIONS OF HYDROLYZED XENON HEXA FLUORIDE DE 65A-05-04L
X-RAY AND SPECTROCHEMICAL ANALYSES (Y-12) 65A-07-02
MASS FISSION YIELD CURVE FOR AMERICIUM-241 65PRR-046
MASS FISSION YIELD CURVE FOR NEPTUNIUM-237 65PRR-094
VENT EXTRACTION OF BERYLLIUM, COPPER, NICKEL, COBALT, AND ZINC AS DI BENZOYL METHANE COMPLEXES SOL 65TR-10
POTASSIUM/ INORGANIC PREPARATIONS (MAGNESIUM CHLORIDE / ZINC CHLORIDE / CESIUM CHLORIDE / SODIUM OXIDE / 65A-12
DETERMINATION OF LEAD, BISMUTH, CADMIUM, TIN, AND ZINC IN AIRBORNE DUST SAMPLES 65A-07-02A
THE EFFECT OF AGING ON CARBIDES IN A NIOBIUM - TUNGSTEN - ZIRCONIUM ALLOY ELECTRON OPTICAL STUDY OF 65PRR-210
/RA OF IRRADIATED LITHIUM FLUORIDE - BERYLLIUM FLUORIDE - ZIRCONIUM FLUORIDE - THORIUM FLUORIDE - URANIUM/ 65-02-01A
ITRATION METHOD ZIRCONIUM IN MSRE FUEL, AMPEROMETRIC CUPFERRON T 65PRR-135
LUTIONS / ZIRCONIUM RADIOACTIVITY IN AQUEOUS OR ORGANIC SO 65PRR-145
/O) EXTRACT (MICROANALYTICAL DETERMINATION OF URANIUM / 0.5-ML POLAROGRAPHIC CELL / ELECTROCHEMISTRY / / 65A-01-06
IRON, 1,10 PHENANTHROLINE SPECTROPHOTOMETRIC METHOD 65PRR-133
/ POLAROGRAPHY OF LEAD(II) IN AQUEOUS HYDROFLUORIC ACID (1-12-MOLAR) WITH A DROPPING MERCURY ELECTRODE O 65PRR-041
ACTIVATION CROSS SECTIONS FOR THE REACTIONS OF 14.8-MEV NEUTRONS WITH TECHNETIUM-99 65PRR-075
AND OXYGEN / DETERMINATION OF OXYGEN IN ALKALI METALS BY 14-MEV NEUTRON ACTIVATION ANALYSIS) /N HYDROGEN 65A-04-03A
14-MEV NEUTRON GENERATOR 65A-11-09
INFLUENCE IN CROSS SECTION MEASURE/ OUTPUT SPECTRUM FROM 14-MEV NEUTRON GENERATORS - RAPID ESTIMATION AND 65PRR-043
INFLUENCE IN ACTIVATION ANALYSIS OUTPUT SPECTRA OF 14-MEV NEUTRON GENERATORS - RAPID ESTIMATION AND 65PRR-044
CATALOG OF 14-MEV NEUTRON REACTIONS 65A-11-09A
14-MEV NEUTRON REACTIONS 65PRR-100
ACTIVATION OF NICKEL-64 WITH 14-MEV NEUTRONS 65A-11-09D
ACTIVATION ANALYSIS WITH 14-MEV NEUTRONS AND WITH HELIUM-3 IONS 65PRR-176
ACTIVATION CROSS SECTIONS FOR THE REACTIONS OF 14-MEV NEUTRONS WITH TECHNETIUM-99 65A-11-09E
INDEXES TO THE ORNL MASTER ANALYTICAL MANUAL (1953-1964) 65PRR-093
SPECTROPHOTOMETRIC DETERMINATION OF NITRATED 4 SEC BUTYL 2 (ALPHA METHYL BENZYL) PHENOL (BAMBP) 65A-05-04E
2,6 DI OXIMINO CYCLOHEXANONE 65A-13-01B
N OF IODINE WITH CYCLOHEXANE AND WITH BENZENE AT 300C AND 20 ATMOSPHERES /S OF THE PRODUCTS OF THE REACTIO 65A-04-04C
E REACTION OF IODINE WITH CYCLOHEXANE AND WITH BENZENE AT 300C AND 20 ATMOSPHERES /S OF THE PRODUCTS OF TH 65A-04-04C
PREPARATION BY ELECTRODEPOSITION OF MOUNTS FOR 4 PI BETA COUNTING 65A-11-03A
AMBP) SPECTROPHOTOMETRIC DETERMINATION OF NITRATED 4 SEC BUTYL 2 (ALPHA METHYL BENZYL) PHENOL (B 65A-05-04E
ORNL MASTER ANALYTICAL MANUAL, SUPPLEMENT 7 65PRR-092
/RADIOACTIVITY IN TRANSURANIUM (TRU) PROCESS SOLUTIONS, 8 HYDROXY QUINOLINE PRECIPITATION - BETA COUNTI/ 65PRR-129

Author Index

A

*ACKLEY, R. D. 65PRR-066
*ACKLEY, R. D. 65PRR-067
*ACKLEY, R. D. 65PRR-068
*ACKLEY, R. D. 65PRR-069
*ACKLEY, R. D. 65PRR-162

*ADAMS, R. E. 65PRR-062
*ADAMS, R. E. 65PRR-063
*ADAMS, R. E. 65PRR-064
*ADAMS, R. E. 65PRR-065
*ADAMS, R. E. 65PRR-162

=ALEXANDER, T. D. 65A-11-06B
=ALEXANDER, T. D. 65PRR-171

*ANDERSON, N. G. 65PRR-207

APPLE, R. F. 65A-04-01
APPLE, R. F. 65PRR-121
APPLE, R. F. 65PRR-122
APPLE, R. F. 65PRR-135
APPLE, R. F. 65PRR-136
APPLE, R. F. 65PRR-137
APPLE, R. F. 65PRR-138

ATTRILL, J. E. 65A-04-04A
ATTRILL, J. E. 65PRR-007
ATTRILL, J. E. 65PRR-062
ATTRILL, J. E. 65PRR-063
ATTRILL, J. E. 65PRR-064
ATTRILL, J. E. 65PRR-065
ATTRILL, J. E. 65PRR-162
ATTRILL, J. E. 65PRR-207

B

=BARKER, G. C. 65A-01-14
BATE, L. C. 65A-11-04E
BATE, L. C. 65A-11-08D
BATE, L. C. 65A-11-08I
BATE, L. C. 65PRR-008
BATE, L. C. 65PRR-009
BATE, L. C. 65PRR-018
BATE, L. C. 65PRR-019
BATE, L. C. 65PRR-037
BATE, L. C. 65PRR-154

*BAYBARZ, R. D. 65PRR-165

*BEATTY, R. L. 65PRR-164

BELEW, W. L. 65A-01-03
BELEW, W. L. 65A-01-04
BELEW, W. L. 65A-01-06
BELEW, W. L. 65A-01-07
BELEW, W. L. 65A-05-03B
BELEW, W. L. 65PRR-010
BELEW, W. L. 65PRR-160
BELEW, W. L. 65PRR-181

*BELL, J. T. 65PRR-161

*BIGELOW, J. E. 65PRR-165

BIGGERS, R. E. 65PRR-161

BOYD, C. M. 65PRR-007
BOYD, C. M. 65PRR-155

BRADY, L. J. 65PRR-052
BRADY, L. J. 65PRR-053
BRADY, L. J. 65PRR-054

*BROOKSBANK, R. E. 65PRR-139

*BROWNING, W. E., JR. 65PRR-062
*BROWNING, W. E., JR. 65PRR-063
*BROWNING, W. E., JR. 65PRR-064
*BROWNING, W. E., JR. 65PRR-065
*BROWNING, W. E., JR. 65PRR-066
*BROWNING, W. E., JR. 65PRR-067
*BROWNING, W. E., JR. 65PRR-068
*BROWNING, W. E., JR. 65PRR-069
*BROWNING, W. E., JR. 65PRR-162

BURROS, C. L. 65PRR-139

C

CAMERON, A. E. 65A-07-02
CAMERON, A. E. 65A-08
CAMERON, A. E. 65B-15
CAMERON, A. E. 65B-16
CAMERON, A. E. 65PRR-001
CAMERON, A. E. 65PRR-070
CAMERON, A. E. 65PRR-071
CAMERON, A. E. 65PRR-163
CAMERON, A. E. 65PRR-192
CAMERON, A. E. 65PRR-208
CAMERON, A. E. 65PRR-215 - 220

CANADA, D. C. 65A-05-04H
CANADA, D. C. 65A-05-04I
CANADA, D. C. 65A-05-04J
CANADA, D. C. 65A-05-04K

CARTER, J. A. 65A-07-02
CARTER, J. A. 65A-07-02A
CARTER, J. A. 65A-08-01
CARTER, J. A. 65B-16
CARTER, J. A. 65PRR-086
CARTER, J. A. 65PRR-116

*CASE, F. N. 65A-11-02B

CHRISTIE, W. H. 65PRR-192

*CLARK, G. W. 65PRR-214

COMBS, Z. 65A-07-01F

*COOK, J. L. 65PRR-164

COOPER, J. H. 65A-04-02
COOPER, J. H. 65B-17-01

CORBIN, L. T. 65A-04
CORBIN, L. T. 65B-14
CORBIN, L. T. 65B-17

COSTANZO, D. A. 65A-04-02
COSTANZO, D. A. 65PRR-022
COSTANZO, D. A. 65PRR-123
COSTANZO, D. A. 65PRR-165

=CROWTHER, P. 65A-11-03E
=CROWTHER, P. 65PRR-003
=CROWTHER, P. 65PRR-011
=CROWTHER, P. 65PRR-169

D

DALE, J. M. 65A-02-02
DALE, J. M. 65A-04-01
DALE, J. M. 65PRR-020
DALE, J. M. 65PRR-030
DALE, J. M. 65PRR-191

*DAVIS, M. E. 65PRR-162

=DEAN, J. A. 65A-01-06
=DEAN, J. A. 65PRR-076
=DEAN, J. A. 65PRR-160

*DRUSCHEL, R. E. 65PRR-140
*DRUSCHEL, R. E. 65PRR-141
*DRUSCHEL, R. E. 65PRR-142
*DRUSCHEL, R. E. 65PRR-143
*DRUSCHEL, R. E. 65PRR-144
*DRUSCHEL, R. E. 65PRR-145

=DUNLAP, L. B. 65PRR-034

DUNN, H. W. 65A-07-01A
DUNN, H. W. 65PRR-166

=DUNN, J. W., III 65A-11-07C
=DUNN, J. W., III 65PRR-048

DYER, F. F. 65A-11-06B
DYER, F. F. 65A-11-06C
DYER, F. F. 65A-11-06F
DYER, F. F. 65A-11-08B
DYER, F. F. 65A-11-08D
DYER, F. F. 65PRR-009
DYER, F. F. 65PRR-171
DYER, F. F. 65PRR-198

E

EBY, R. E. 65A-08-02
EBY, R. E. 65PRR-167

ELDRIDGE, J. S. 65A-11-01A
ELDRIDGE, J. S. 65A-11-01B
ELDRIDGE, J. S. 65A-11-01C
ELDRIDGE, J. S. 65A-11-02C
ELDRIDGE, J. S. 65A-11-03E
ELDRIDGE, J. S. 65A-11-06A
ELDRIDGE, J. S. 65PRR-003
ELDRIDGE, J. S. 65PRR-011
ELDRIDGE, J. S. 65PRR-012
ELDRIDGE, J. S. 65PRR-168
ELDRIDGE, J. S. 65PRR-169
ELDRIDGE, J. S. 65PRR-170

EMERY, J. F. 65A-11-06B
EMERY, J. F. 65A-11-06D
EMERY, J. F. 65A-11-08B
EMERY, J. F. 65A-11-08C
EMERY, J. F. 65A-11-08E
EMERY, J. F. 65A-11-08F
EMERY, J. F. 65PRR-013
EMERY, J. F. 65PRR-077
EMERY, J. F. 65PRR-171

*EMPSON, F. M. 65PRR-023

F

FARRAR, L. G. 65B-17-05

FELDMAN, C. 65A-07-01
FELDMAN, C. 65A-07-01B
FELDMAN, C. 65A-07-01C
FELDMAN, C. 65A-07-01D
FELDMAN, C. 65PRR-072
FELDMAN, C. 65PRR-172
FELDMAN, C. 65PRR-173
FELDMAN, C. 65PRR-174

FERGUSON, N. M. 65A-07-01B
FERGUSON, N. M. 65A-07-01D

FISHER, D. J. 65A-01
FISHER, D. J. 65A-01-03
FISHER, D. J. 65A-01-06
FISHER, D. J. 65A-01-07
FISHER, D. J. 65A-01-08
FISHER, D. J. 65A-01-11
FISHER, D. J. 65A-01-15
FISHER, D. J. 65A-04-01
FISHER, D. J. 65A-04-02
FISHER, D. J. 65A-05-03B
FISHER, D. J. 65PRR-028
FISHER, D. J. 65PRR-036
FISHER, D. J. 65PRR-124
FISHER, D. J. 65PRR-160
FISHER, D. J. 65PRR-181

=FLORENCE, T. M. 65A-05-04A
=FLORENCE, T. M. 65A-05-04B
=FLORENCE, T. M. 65A-05-04C

=FREEMAN, D. B. 65PRR-029

G

=GARDNER, R. P. 65A-11-07C
=GARDNER, R. P. 65PRR-048

GHANN, C. L. 65PRR-151

GOEKING, C. F. 65PRR-046

GOLDBERG, G. 65A-04-03A
GOLDBERG, G. 65PRR-052
GOLDBERG, G. 65PRR-053
GOLDBERG, G. 65PRR-054
GOLDBERG, G. 65PRR-073
GOLDBERG, G. 65PRR-175

GOLDSTEIN, G. 65PRR-077
GOLDSTEIN, G. 65PRR-078
GOLDSTEIN, G. 65A-11-01D
GOLDSTEIN, G. 65A-11-03A
GOLDSTEIN, G. 65A-11-04C
GOLDSTEIN, G. 65A-11-08C
GOLDSTEIN, G. 65A-11-09E
GOLDSTEIN, G. 65PRR-002
GOLDSTEIN, G. 65PRR-014
GOLDSTEIN, G. 65PRR-074
GOLDSTEIN, G. 65PRR-075
GOLDSTEIN, G. 65PRR-076

=GREEN, J. G. 65PRR-207

H

=HAGA, F. E. 65A-01-01
=HAGA, F. E. 65A-04-01
=HAGA, F. E. 65A-04-02
=HAGA, F. E. 65PRR-027
=HAGA, F. E. 65PRR-028
=HAGA, F. E. 65PRR-051
=HAGA, F. E. 65PRR-124

HAHN, R. L. 65A-11-06E
HAHN, R. L. 65A-11-08A
HAHN, R. L. 65PRR-045
HAHN, R. L. 65PRR-176
HAHN, R. L. 65PRR-177
HAHN, R. L. 65PRR-178
HAHN, R. L. 65PRR-179
HAHN, R. L. 65PRR-198

HANDLEY, T. H. 65A-11-01E
HANDLEY, T. H. 65A-11-01F
HANDLEY, T. H. 65A-11-02G
HANDLEY, T. H. 65A-11-04A
HANDLEY, T. H. 65A-11-04B
HANDLEY, T. H. 65PRR-015
HANDLEY, T. H. 65PRR-016
HANDLEY, T. H. 65PRR-017
HANDLEY, T. H. 65PRR-056

HARMON, T. G. 65A-10

HEACKER, F. K. 65A-04-01

=HEALY, W. B. 65A-11-08I
=HEALY, W. B. 65PRR-018
=HEALY, W. B. 65PRR-019

=HERTEL, G. R. 65A-08-03

HILL, F. M. 65PRR-156

HOLSOPPLE, H. L. 65A-11-05A
HOLSOPPLE, H. L. 65A-13
HOLSOPPLE, H. L. 65PRR-049
HOLSOPPLE, H. L. 65PRR-079

=HONAKER, C. B. 65A-06-03

HORTON, A. D. 65A-03-02
HORTON, A. D. 65A-04-04C
HORTON, A. D. 65A-05-01A
HORTON, A. D. 65A-05-01B
HORTON, A. D. 65PRR-180

HORTON, C. A. 65A-06-01

J

=JOHNSON, B. C. 65A-05-03B

JONES, F. S. 65A-07-01E

JONES, H. C. 65A-01-01
JONES, H. C. 65A-01-04
JONES, H. C. 65A-01-05
JONES, H. C. 65A-01-07
JONES, H. C. 65A-01-08
JONES, H. C. 65A-01-15
JONES, H. C. 65PRR-020
JONES, H. C. 65PRR-036
JONES, H. C. 65PRR-051
JONES, H. C. 65PRR-080
JONES, H. C. 65PRR-125
JONES, H. C. 65PRR-146
JONES, H. C. 65PRR-181

K

KELLEY, M. T. 65A-01
KELLEY, M. T. 65A-01-06

KELLEY, M. T. 65A-01-07
KELLEY, M. T. 65A-01-08
KELLEY, M. T. 65A-04
KELLEY, M. T. 65A-04-01
KELLEY, M. T. 65A-07-01
KELLEY, M. T. 65A-10
KELLEY, M. T. 65A-12
KELLEY, M. T. 65C-18
KELLEY, M. T. 65PRR-081
KELLEY, M. T. 65PRR-082
KELLEY, M. T. 65PRR-083
KELLEY, M. T. 65PRR-084
KELLEY, M. T. 65PRR-085
KELLEY, M. T. 65PRR-160
KELLEY, M. T. 65PRR-180
KELLEY, M. T. 65PRR-181
KELLEY, M. T. 65TR-01
KELLEY, M. T. 65TR-02
KELLEY, M. T. 65TR-03
KELLEY, M. T. 65TR-04
KELLEY, M. T. 65TR-05
KELLEY, M. T. 65TR-06
KELLEY, M. T. 65TR-07

*KLEIN, A. S. 65PRR-093

=KOCHANNY, G. L. 65PRR-021

KOSKELA, U. 65A-04-01

KUBOTA, H. 65A-03-01
KUBOTA, H. 65A-03-03
KUBOTA, H. 65A-03-04
KUBOTA, H. 65PRR-022
KUBOTA, H. 65PRR-023
KUBOTA, H. 65PRR-126
KUBOTA, H. 65PRR-127
KUBOTA, H. 65PRR-157
KUBOTA, H. 65PRR-158
KUBOTA, H. 65PRR-159
KUBOTA, H. 65TR-08
KUBOTA, H. 65TR-09
KUBOTA, H. 65TR-10

L

LAING, W. R. 65B-17-02
LAING, W. R. 65PRR-052
LAING, W. R. 65PRR-053
LAING, W. R. 65PRR-054
LAING, W. R. 65PRR-164
LAING, W. R. 65PRR-182
LAING, W. R. 65PRR-183 - 184

LAMB, C. E. 65A-04-01
LAMB, C. E. 65B-17-03

*LANDRY, J. W. 65A-11-09F

LAVALLE, D. E. 65A-12
LAVALLE, D. E. 65PRR-090

=LEE, T. 65PRR-086

*LEWIS, R. E. 65A-11-03C

=LUDWIG, T. G. 65PRR-019

LUND, J. R. 65A-04-01
LUND, J. R. 65A-05-04L
LUND, J. R. 65A-05-04M
LUND, J. R. 65A-06-04
LUND, J. R. 65A-09-01

LUPICA, S. B. 65A-07-01A

LYNN, E. C. 65PRR-183 - 184

LYON, W. S. 65A-11
LYON, W. S. 65A-11-01B
LYON, W. S. 65A-11-03E
LYON, W. S. 65PRR-003
LYON, W. S. 65PRR-005
LYON, W. S. 65PRR-006
LYON, W. S. 65PRR-012
LYON, W. S. 65PRR-024
LYON, W. S. 65PRR-025
LYON, W. S. 65PRR-052
LYON, W. S. 65PRR-053
LYON, W. S. 65PRR-054
LYON, W. S. 65PRR-087
LYON, W. S. 65PRR-169
LYON, W. S. 65PRR-170
LYON, W. S. 65PRR-185
LYON, W. S. 65PRR-186

LYON, W. S. 65PRR-187
LYON, W. S. 65PRR-188
LYON, W. S. 65PRR-189
LYON, W. S. 65PRR-190

M

MADDOX, W. L. 65A-01-11
MADDOX, W. L. 65A-01-12
MADDOX, W. L. 65A-04-01
MADDOX, W. L. 65A-04-02
MADDOX, W. L. 65PRR-026
MADDOX, W. L. 65PRR-027
MADDOX, W. L. 65PRR-028

=MAMANTOV, G. 65A-02-02
=MAMANTOV, G. 65PRR-029
=MAMANTOV, G. 65PRR-030
=MAMANTOV, G. 65PRR-191

MANNING, D. L. 65A-02-02
MANNING, D. L. 65A-04-01
MANNING, D. L. 65PRR-030
MANNING, D. L. 65PRR-191

=MATTISON, L. E. 65A-11-07D

MCKOWN, H. S. 65PRR-192

MEYER, A. S. 65A-04-01
MEYER, A. S. 65A-04-04C
MEYER, A. S. 65A-05-01
MEYER, A. S. 65PRR-007
MEYER, A. S. 65PRR-180

MEYER, H. C. 65A-04-02
MEYER, H. C. 65PRR-128
MEYER, H. C. 65PRR-129

MILLER, F. J. 65A-05-02
MILLER, F. J. 65A-05-04N
MILLER, F. J. 65PRR-029
MILLER, F. J. 65PRR-061
MILLER, F. J. 65PRR-193
MILLER, F. J. 65PRR-194
MILLER, F. J. 65TR-11

MOORE, F. L. 65A-04-02
MOORE, F. L. 65PRR-031
MOORE, F. L. 65PRR-032
MOORE, F. L. 65PRR-033
MOORE, F. L. 65PRR-130
MOORE, F. L. 65PRR-223

MORTON, J. E. 65PRR-131

=MOUNTCASTLE, W. R. 65PRR-034

MUELLER, T. R. 65A-01-01
MUELLER, T. R. 65A-01-02
MUELLER, T. R. 65A-01-04
MUELLER, T. R. 65A-01-09
MUELLER, T. R. 65A-04-01
MUELLER, T. R. 65A-04-02
MUELLER, T. R. 65PRR-035
MUELLER, T. R. 65PRR-036
MUELLER, T. R. 65PRR-038
MUELLER, T. R. 65PRR-051

MULLINS, W. T. 65PRR-032
MULLINS, W. T. 65PRR-033

MURRAY, M. M. 65A-09-01

MUSICK, W. R. 65A-07-01C

=MUZZARELLI, R. A. A. 65A-11-04E
=MUZZARELLI, R. A. A. 65PRR-037

N

*NEWMAN, E. 65PRR-056

=NORRIS, J. A. 65PRR-088

O

*ORR, P. B. 65PRR-089

P

=PAPPAS, W. S. 65PRR-116

PARKER, H. A. 65PRR-094

=PERONE, S. P. 65PRR-038

=PETERS, D. G. 65A-01-02

PIERCE, G. V. 65A-01-07
PIERCE, G. V. 65A-01-08
PIERCE, G. V. 65PRR-181

*PRESSLY, R. S. 65PRR-089

PRITCHARD, C. A. 65A-07-01F

Q

QUINCY, R. B. 65A-12
QUINCY, R. B. 65PRR-090

R

RAAEN, H. P. 65A-05-03A
RAAEN, H. P. 65A-05-03B
RAAEN, H. P. 65C-18
RAAEN, H. P. 65PRR-010
RAAEN, H. P. 65PRR-039
RAAEN, H. P. 65PRR-040
RAAEN, H. P. 65PRR-041
RAAEN, H. P. 65PRR-091
RAAEN, H. P. 65PRR-092
RAAEN, H. P. 65PRR-093
RAAEN, H. P. 65PRR-195

=RAINS, T. C. 65A-07-01C
=RAINS, T. C. 65A-07-01D

REYNOLDS, S. A. 65A-11-02A
REYNOLDS, S. A. 65A-11-02B
REYNOLDS, S. A. 65A-11-02C
REYNOLDS, S. A. 65A-11-02D
REYNOLDS, S. A. 65A-11-02E
REYNOLDS, S. A. 65A-11-02F
REYNOLDS, S. A. 65A-11-02H
REYNOLDS, S. A. 65A-11-03B
REYNOLDS, S. A. 65A-11-03C
REYNOLDS, S. A. 65A-11-03D
REYNOLDS, S. A. 65A-11-03F
REYNOLDS, S. A. 65PRR-004
REYNOLDS, S. A. 65PRR-042
REYNOLDS, S. A. 65PRR-078
REYNOLDS, S. A. 65PRR-147
REYNOLDS, S. A. 65PRR-148
REYNOLDS, S. A. 65PRR-196

RICCI, E. 65A-11-06E
RICCI, E. 65A-11-08A
RICCI, E. 65A-11-09C
RICCI, E. 65PRR-043
RICCI, E. 65PRR-044
RICCI, E. 65PRR-045
RICCI, E. 65PRR-197
RICCI, E. 65PRR-198

RICKARD, R. R. 65PRR-046
RICKARD, R. R. 65PRR-094
RICKARD, R. R. 65PRR-149

ROEMER, A. F. 65PRR-066
ROEMER, A. F. 65PRR-067
ROEMER, A. F. 65PRR-068
ROEMER, A. F. 65PRR-069

ROSS, H. H. 65A-11-05A
ROSS, H. H. 65A-11-05B
ROSS, H. H. 65A-11-07A
ROSS, H. H. 65A-11-07B
ROSS, H. H. 65A-11-07C
ROSS, H. H. 65PRR-005
ROSS, H. H. 65PRR-047
ROSS, H. H. 65PRR-048
ROSS, H. H. 65PRR-049
ROSS, H. H. 65PRR-095
ROSS, H. H. 65PRR-199
ROSS, H. H. 65PRR-200

ROSS, W. J. 65A-04-03A
ROSS, W. J. 65A-11-08G
ROSS, W. J. 65A-11-08H
ROSS, W. J. 65A-11-09A
ROSS, W. J. 65A-11-09B
ROSS, W. J. 65A-11-09D
ROSS, W. J. 65A-11-09F
ROSS, W. J. 65PRR-050
ROSS, W. J. 65PRR-100

RUBIN, I. B. 65A-06-02

S

=SAVAGE, S. 65A-01-13
=SAVAGE, S. 65PRR-096
=SAVAGE, S. 65PRR-097

=SCHAAP, W. B. 65A-01-15

*SCHMITT, J. M. 65A-05-01A

*SCHONFELD, E. 65A-11-06B
*SCHONFELD, E. 65PRR-171

SCROGGIE, L. E. 65A-09-01
SCROGGIE, L. E. 65A-09-02
SCROGGIE, L. E. 65PRR-079

SHULTS, W. D. 65A-01-01
SHULTS, W. D. 65A-01-02
SHULTS, W. D. 65A-01-03
SHULTS, W. D. 65A-01-05
SHULTS, W. D. 65A-01-07
SHULTS, W. D. 65A-01-08
SHULTS, W. D. 65A-01-09
SHULTS, W. D. 65A-01-10
SHULTS, W. D. 65A-01-13
SHULTS, W. D. 65A-01-15
SHULTS, W. D. 65PRR-020
SHULTS, W. D. 65PRR-051
SHULTS, W. D. 65PRR-096
SHULTS, W. D. 65PRR-097
SHULTS, W. D. 65PRR-181

SITES, J. R. 65A-08-01
SITES, J. R. 65B-15
SITES, J. R. 65PRR-201

SMITH, D. H. 65A-08-04

SPITZER, E. J. 65PRR-089

STELZNER, R. W. 65A-01-14
STELZNER, R. W. 65A-04-02
STELZNER, R. W. 65PRR-036
STELZNER, R. W. 65PRR-098
STELZNER, R. W. 65PRR-202
STELZNER, R. W. 65PRR-203

=STEVENS, C. M. 65PRR-001

STRAIN, J. E. 65A-04-03A
STRAIN, J. E. 65A-11-09A
STRAIN, J. E. 65A-11-09B
STRAIN, J. E. 65A-11-09D
STRAIN, J. E. 65A-11-09F
STRAIN, J. E. 65PRR-006
STRAIN, J. E. 65PRR-099
STRAIN, J. E. 65PRR-100
STRAIN, J. E. 65PRR-198

STRINGER, C. D. 65PRR-132

SUSANO, C. D. 65PRR-052
SUSANO, C. D. 65PRR-053
SUSANO, C. D. 65PRR-054
SUSANO, C. D. 65PRR-204

T

TALBOTT, C. K. 65A-04-01

=TERRY, A. A. 65PRR-055

=THIELE, D. 65A-01-03

THOMASON, P. F. 65A-04-01
THOMASON, P. F. 65A-04-02
THOMASON, P. F. 65A-05-02
THOMASON, P. F. 65A-05-03
THOMASON, P. F. 65A-05-04
THOMASON, P. F. 65A-09
THOMASON, P. F. 65A-13
THOMASON, P. F. 65PRR-034
THOMASON, P. F. 65PRR-101
THOMASON, P. F. 65PRR-102
THOMASON, P. F. 65PRR-103
THOMASON, P. F. 65PRR-205
THOMASON, P. F. 65PRR-206

*TOTH, K. S. 65PRR-056

TRAVAGLINI, A. L. 65A-05-04D

TRAVAGLINI, A. L. 65A-05-04E
TRAVAGLINI, A. L. 65A-05-04F
TRAVAGLINI, A. L. 65A-05-04G
TRAVAGLINI, A. L. 65A-05-04L

=TRUNDLE, A. S., III 65PRR-192

V

VAUGHAN, W. F. 65PRR-133
VAUGHAN, W. F. 65PRR-134

=VISSER, D. R. 65PRR-207

W

WALKER, R. L. 65PRR-150

WALTON, J. R. 65PRR-071
WALTON, J. R. 65PRR-208

=WAMPLER, J. M. 65A-08-04

=WEBER, C. W. 65PRR-116

WHITE, J. C. 65A-02
WHITE, J. C. 65A-03
WHITE, J. C. 65A-04
WHITE, J. C. 65A-05
WHITE, J. C. 65A-06
WHITE, J. C. 65A-09
WHITE, J. C. 65A-11
WHITE, J. C. 65A-13
WHITE, J. C. 65PRR-057
WHITE, J. C. 65PRR-058
WHITE, J. C. 65PRR-104 - 115
WHITE, J. C. 65PRR-209
WHITE, J. C. 65PRR-221 - 222

=WILD, J. F. 65A-11-04D

*WILLIAMS, I. R. 65PRR-056

WILLMARTH, T. E. 65A-10
WILLMARTH, T. E. 65PRR-210 - 211

WILSON, G. R. 65B-14

WRIGHT, H. W. 65A-10

=WRIGHT, R. R. 65PRR-116

WYATT, E. I. 65B-17-04
WYATT, E. I. 65PRR-046
WYATT, E. I. 65PRR-094
WYATT, E. I. 65PRR-139
WYATT, E. I. 65PRR-140
WYATT, E. I. 65PRR-141
WYATT, E. I. 65PRR-142
WYATT, E. I. 65PRR-143
WYATT, E. I. 65PRR-144
WYATT, E. I. 65PRR-145
WYATT, E. I. 65PRR-147
WYATT, E. I. 65PRR-148
WYATT, E. I. 65PRR-149
WYATT, E. I. 65PRR-150
WYATT, E. I. 65PRR-151
WYATT, E. I. 65PRR-152
WYATT, E. I. 65PRR-153

Y

YOAKUM, A. M. 65A-07-01F
YOAKUM, A. M. 65PRR-212 - 213

YOUNG, J. P. 65A-02-01
YOUNG, J. P. 65A-04-04B
YOUNG, J. P. 65PRR-059
YOUNG, J. P. 65PRR-214

Z

ZITTEL, H. E. 65A-03-04
ZITTEL, H. E. 65A-05-02
ZITTEL, H. E. 65PRR-029
ZITTEL, H. E. 65PRR-060
ZITTEL, H. E. 65PRR-061

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	MONTHLY	WEEKLY	TOTAL
ORNL EMPLOYEES			
ADMINISTRATIVE AND CLERICAL	5	11	16
SCIENTISTS	109		109
ANALYSTS		86	86
SUBTOTAL	114	97	211
OTHERS			
ORINS FELLOW	3		3
NONCITIZEN GUEST	1		1
STUDENT GUEST	1		1
SUBTOTAL	5	—	5
GRAND TOTAL	119	97	216

[1]DUAL CAPACITY
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