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ANALYTICAL CHEMISTRY DIVISION
ANNUAL PROGRESS REPORT
FOR PERIOD ENDING NOVEMBER 15, 1964

OAK RIDGE NATIONAL LABORATORY
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ANNUAL PROGRESS REPORT
For Period Ending November 15, 1964

M. T. Kelley, Director
C. D. Susano, Associate Director

JANUARY 1965

OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee
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Summary

PART A. ANALYTICAL RESEARCH

1. Analytical Instrumentation

Two papers were presented at the 3rd International Congress of Polarography. The first summarized the controlled-potential dc polarography done at ORNL since mid-1959; the second described the quasi-reference electrode (Q.R.E.) and research results obtained with it in controlled-potential polarography, controlled-potential voltammetry, and chronopotentiometry.

A prototype controlled-potential dc polarograph-voltammeter was designed and constructed; solid-state instead of vacuum-tube operational amplifiers and power supplies are used. The instrument is to be used with the short-drop-time Smoler vertical-orifice dropping-mercury electrodes (D.M.E.) and with solid working electrodes. The prototype will be used for continued research in polarography and voltammetry and as the basis for design of several controlled-potential dc polarograph-voltammeters for work in the Analytical Chemistry Division.

To facilitate the evaluation of dc polarographic instrumentation, computer programs were written. These programs solve the Koutecký equation for a value of the diffusion coefficient and provide computer-plotted, normalized, theoretical curves (simple, reversible, diffusion-controlled process) for the regular polarographic wave and for the first-, second-, and third-derivative waves.

Two ORNL model Q-2564 high-sensitivity coulometric titrators were fabricated and installed, one in the High-Radiation-Level Analytical Laboratory (HRLAL) and the other in the High-Level Alpha Radiation Laboratory. A check-out and test procedure and a troubleshooting guide were prepared for this instrument. About 25 complete sets of engineering drawings for the titrator were sent to interested installations in the United States and abroad.

A paper that described the application of the derivative method to stationary-electrode voltammetry with linearly varying potential was presented at the 148th American Chemical Society Meeting. The accuracy and reproducibility of first-derivative linear-sweep voltammetry are about the same as those of regular linear-sweep voltammetry; the sensitivity is at least an order of magnitude greater.

A safety circuit for the expensive ($633) multiplier phototube of the ORNL model VII flame spectrophotometer was designed and installed. Already, the circuit has twice provided automatic shutdown that saved the phototube.

Study of the significance of the placement of the reference electrode in controlled-potential coulometry was continued with the Bi^{3+}-UO_2^{2+} system. There was no chemical evidence that variation in placement of the reference electrode, even under drastic cell conditions, causes the voltage that drives the electrochemical reaction to exceed the command voltage set into the potentiostat.

The development of analytical instruments necessary to the MSRE and TRU programs was continued (see Sect. 4). Some of the results may be applied in the future to in-line monitoring problems. Several remotely operated instruments for use in hot cells were redesigned to simplify manipulation and maintenance.

For the MSRE program, the following instrument development was done or is in progress. Applications of the Q.R.E. to the analysis of molten salts by electrochemical methods were continued. The ORNL model Q-1734 remotely operated filter photometer was redesigned to make greater use of commercially available components and of better materials of construction and to simplify operation of the instrument in hot cells. Two of these units were fabricated for use in the HRLAL. An assembly drawing of the redesigned photometer is

given in ORNL-Dwg. 64-3171. The ORNL corrosion-resistant servopipetter was redesigned to permit easier manipulations in hot cells. The new design features are a single cable connection (removable by the master-slave manipulators) to the pipetter and a new stand and mount that replace the ORNL screw-operated elevator. The relative standard deviation of 100-μl deliveries is 0.2%. Five of the new units (designated Q-1348B) were fabricated at ORNL, tested with satisfactory results, and delivered to the HRLAL. Terminal engineering drawings for the Q-1348B are being prepared. The remotely operated decapper was redesigned to accommodate sample bottles of various heights and diameters. Two of the new decappers were fabricated for use in the HRLAL. Application of the ORNL model Q-2564 high-sensitivity coulometric titrators to analyses for the MSRE program was continued. Another of these titrators that was constructed recently was checked out against the design specifications. For the following equipment that is essential to the remotely operated analysis of MSRE samples, design work was completed, engineering drawings were prepared, and a spare set of the instruments is being constructed: one pyrohydrolysis unit, one transfer-tube decoupling device, one modified Spex Industries Inc. Mixer/Mill, two powder-transfer devices, one redesigned ladle-cutting device, five electrochemical apparatuses, and 250 copper pulverizer-mixers (copper ladles).

For the TRU program, the following instrument development work has been done or is in progress. Drawings for an alternative pipet delivery unit and an alternative stand-jack for the ORNL model Q-1728 velocity-servo, automatic, recording potentiometric titrator were prepared and were added to the Q-1728 series of drawings. Two of these titrators, at least one of which is to be used with these alternative units, are to be fabricated and will be checked out after construction to ensure that the design specifications have been met by the fabricator. The following remotely operated apparatus for use in the TRU analytical hot cells is under development. The ORNL corrosion-resistant servopipetter is being redesigned to deliver a full 1-ml sample and to be capable of passage through an 8 × 8 in. cylindrical access area; it is to be capable of assembly and disassembly within the hot-cell enclosure. Engineering drawings of this device will be prepared. A remotely operated analytical balance for weighing TRU samples prior to their analysis is being designed. Remotely operated solvent-extraction apparatus for use within the TRU analytical hot cells is being designed, and engineering drawings will be prepared after the prototype unit has been proved satisfactory.

2. Analytical Studies of Molten-Salt Systems

Absorption spectrophotometric studies of various molten-salt systems were continued. The atmosphere of the nickel high-temperature spectrophotometric cell assembly was freed from water, oxygen, and HF by placing chips of titanium sponge in the sample compartment. This procedure resulted in the stability of the absorption spectrum of U^{4+} in LiF-NaF-KF for 15 hr.

The absorption spectrum of U^{3+} at 500°C in $LiF\text{-}BeF_2\text{-}ZrF_4$ was obtained by reduction of U^{4+} by either Zr or U metal. The molar absorptivity of the strongest U^{3+} peak at 875 mμ in the near-infrared region was estimated to be 40 liters $mole^{-1}$ cm^{-1}. In the ultraviolet region, at 360 mμ, the molar absorptivity is about 500 liters $mole^{-1}$ cm^{-1}. The spectrum of U^{3+} in $LiF\text{-}BeF_2$ appears to be identical with that in $LiF\text{-}BeF_2\text{-}ZrF_4$. The intensity of the 360-mμ peak in these melts provides a sensitive means of observing reactions that generate or consume U^{3+}.

A special holder for very small crystals was devised to obtain spectra from 280 to 750 mμ of crystals whose diameter is of the order of 0.1 ma.

The absorption spectrum of UF_6 was obtained using a vacuum-tight fluorinated-nickel gas cell equipped with crystalline Al_2O_3 windows and Teflon gaskets. The spectrum is complex in the region 340 to 420 mμ with a maximum at 368 mμ, at which wavelength the molar absorptivity of UF_6 is 5.5 liters $mole^{-1}$ cm^{-1}.

The experimental equipment used to investigate the electroanalytical characteristics of molten-fluoride salt systems was modified to eliminate possible contamination from moisture. With this equipment the decomposition potentials of such metals as Zr, Cr, Fe, Ni, and Mo in molten fluorides were obtained.

Rapid-scan and anodic-stripping voltammetry of nickel in molten LiF-NaF-KF were studied. By means of the anodic-stripping technique, as

little as 1 ppm of nickel can be measured in the melt. Voltammetry was also applied to the MSR coolant in fuel salts. The presence of hafnium was indicated by these studies. Further studies were also carried out in an effort to determine chromium in the fuel salt.

Work was continued on the development of a reference electrode that would be compatible with molten-fluoride salts. The platinum quasi-reference electrode is not particularly stable. The Ni-NiO electrode appears to be a useful reference with the three-electrode controlled-potential circuit; however, it is not satisfactory in the two-electrode circuit because the cell current that flows through the reference electrode seems to produce polarization and subsequent erratic potential measurements.

Several instrumental approaches to the on-stream analysis of radioactive molten-salt systems were investigated, including electron-spin-resonance measurements and x-ray absorption or emission measurements. None of these techniques seemingly offer much promise for this application.

3. Effects of Radiation on Analytical Methods

The study of the effect of ^{60}Co gamma radiation on the reaction of nickel with dimethylglyoxime (*I*) was completed. A postirradiation effect on the Ni-*I* complex was noted in that the color of the complex returns after it has been removed from the source of radiation. The radiation damage to the colored complex is apparently due to reduction of the Ni(III) in the complex. A detailed study of the effect of gamma radiation on the determination of uranium(VI) in phosphoric acid solutions of uranium(IV) oxide was completed. Errors due to radiation can be minimized by excluding oxygen from solutions of UO_2 and by maintaining the UO_2 concentration as low as possible. Studies of the effect of gamma radiation on hydrochloric, sulfuric, phosphoric, and acetic acid solutions of arsenazo III were completed. Similar studies of 2-thenoyltrifluoroacetone (TTA) were initiated.

Studies were also initiated on the radiolysis of solutions of high chloride concentration. Free chlorine is not produced at chloride concentrations below 1 *M*. Organic solvents such as methanol are effective in eliminating chlorine produced by radiolysis in high chloride concentrations.

4. Analytical Chemistry for Reactor Projects

Analytical research and development is being carried out for the MSRE, GCR, and TRU Programs. Since details of the progress of these programs are included in the respective periodical reactor-project progress reports, only a brief summary is included in this annual report. For the MSRE, development and evaluation of methods required for the analysis of the fuel and related problems and investigation of the feasibility of various techniques for in-line monitoring are being studied. For the GCR, evaluation of various types of detectors in the gas chromatographic determination of impurities in helium was continued. For TRU, the effort is to provide methods of analysis needed to control the process being carried out at ORNL. Radiochemical, instrumental, and ionic methods are under investigation.

5. Chemical Analysis of Advanced Reactor Fuels

A gas chromatograph was designed for the analysis of corrosive gases such as nitrogen dioxide, dinitrogen trioxide, chlorine, and nitrosyl chloride. The use of tantalum appears to provide the necessary corrosion-resistant material. Gas chromatography was used to analyze aliphatic diluents used in solvent-extraction processes. The argon-beta ionization detector was successfully applied to this problem.

The combustion–gas chromatographic determination of organic carbon in aqueous and nitric acid solutions was studied further in an effort to increase the recovery of known amounts of organic carbon.

The sealed-tube dissolution technique was applied with limited success to a variety of difficultly soluble compounds such as graphite-coated UO_2, carbides, and several organic compounds. In general, the perchloric acid ("liquid fire") technique is more successful for the dissolution of fluidized coke or graphite-coated materials.

The horizontal-orifice Teflon dropping-mercury electrode (D.M.E.) was used to obtain fundamental polarographic data for the $Tl^+ \rightleftharpoons Tl^0$ reaction in 0.1 *M* KCl–1 m*M* HCl and the $Pb^{2+} \rightleftharpoons Pb^0$ reaction in 1 *M* HCl. The data obtained are interchangeable with those taken with glass D.M.E.'s. The horizontal-orifice Teflon D.M.E. was also

used to determine for the first time the polarographic characteristics of the $Pb^{2+} \rightleftharpoons Pb^0$ reaction in hydrofluoric acid solutions of concentrations from 1 to 12 *M*. A procedure was developed for fabricating a vertical-orifice Teflon D.M.E.

A study of the chronopotentiometry of plutonium in mineral acid media was completed. Diffusion coefficients of the different plutonium species were measured, and the possible analytical usefulness of chronopotentiometry for the determination of plutonium was evaluated.

6. Special Research and Development Problems

Studies with 10 *M* LiCl solutions have shown that the free-acid concentration of such solutions can be determined in the presence of hydrolyzable cations by direct titration without the addition of complexing agents. When several hydrolyzable cations are present, these cations can be differentiated by titration provided there is sufficient difference in their solubility products. Although not particularly precise, the titration is useful as a rapid semiquantitative method.

The investigation of solid indicator electrodes made of pyrolytic graphite and "glassy" carbon was continued to determine their applications and limitations. These studies included differential potentiometry, particularly for hot-cell operation, voltammetry, amperometry, and chronopotentiometry.

An apparatus using flame ionization detection was developed to measure total concentration of organic contaminants in the atmosphere of reactor-fuel-burnup test assemblies. Hydrocarbon concentrations equivalent to 5 ppb of methane in air can be detected. An instrument was assembled in a portable cabinet for making measurements at various sources. Subsequent investigations revealed that aerosols generated by dispersed oxides of uranium interfered with the hydrocarbon determination.

Modifications were made to the apparatus for the determination of oxygen, carbon, and hydrogen in alkali metals. Some of these modifications were required to determine both oxygen and hydrogen in the same sample.

A microtitration cell that does not require an internal electrode was fabricated of pyrolytic graphite for potentiometric titrations of highly radioactive solutions. Both redox and acid-base potentiometric titrations are possible with this cell for microliter volumes of solutions at a relative standard deviation of 0.3%.

7. Analytical Biochemistry

A new program was initiated to establish a biochemistry group in the Analytical Chemistry Division. Assistance is being provided to the Biology and Chemical Technology Divisions in their program to recover and separate the transfer ribonucleic acids (t-RNA's) from *Escherichia coli* bacteria. Since an immense number of assays is required to determine the efficiency of the solvent-extraction process used to separate the t-RNA's, initial efforts are concerned with modifying procedures to reduce the overall time required for analysis. The precision of alternative methods of rapidly adding reagents by means of various automatic repetitive pipetting is being studied.

8. X-Ray and Spectrochemical Analyses

The analysis of powdered solids was investigated by the x-ray absorption-edge method with a rotating sample holder. Disposable one-piece polystyrene cells were fabricated for use in the analysis of radioactive solutions by the same method. Computer codes were written for determining the height of an x-ray absorption edge by extrapolation and for analyzing multicomponent alloys by x-ray fluorescence. Ruthenium and selenium were determined at the 0.5-μg/ml level by atomic absorption with an air-hydrogen flame and an end-fed absorption tube. A procedure was developed for separating traces of barium from the alkali metals, alkaline-earth elements, and other elements by coprecipitation on lead chromate. The barium is subsequently determined by flame photometry.

9. Mass Spectrometry

The new double-magnetic-deflection mass spectrometer was put in service in a laboratory arranged for containment of alpha radioactivity. Ion detection is by secondary electron multiplier, and pulse counting is used instead of current measurement. Only 1 to 2 ng of sample is required; rare

isotopes in concentration as low as 1 ppm can be measured.

The isotopic composition of atmospheric neon was measured by use of ^{20}Ne and ^{22}Ne standards of high isotopic and chemical purity; the composition (atom percent) is indicated to be: ^{20}Ne, 90.514 ± 0.031; ^{21}Ne, 0.266 ± 0.005; ^{22}Ne, 9.220 ± 0.029.

10. Infrared Spectroscopy

Infrared spectroscopy was used to determine the effect of gamma radiation on such compounds as butyl-α-methylbenzylphenol, 2-thenoyltrifluoroacetone, dimethylglyoxime, sodium dimethylglyoxime, *o*-benzylphenol, di-*sec*-butylphenylphosphonate, ion-exchange resins, and fractions of the products from the nitric acid degradation of diethylbenzene. Infrared absorption spectra were measured for the water-soluble acidic solids and ether-soluble waxes that are the products of the hydrolysis of uranium carbides and thorium carbides with water or nitric acid. Other studies involved the detection and/or identification of impurities in commercial solvents, process streams, or compounds (inorganic and organic) prepared at ORNL; the identification of commercial products; or the characterization of organic compounds. Semiquantitative or quantitative determinations were made of a number of compounds.

11. Optical and Electron Microscopy

The electron microscope facility for the examination of ultrastructures of radioactive materials was established in Building 3019 and will be in operation about January 1965. Studies were made of particulates from simulated fallout experiments in connection with the Nuclear Safety Program. The Philips EM 200 electron microscope was installed; resolution of the order of 6 A or better has been achieved with it. Research assistance was given in electron microscopy and diffraction and in optical microscopy to other divisions in studies of the surface and internal structure of thin films and solids, particle sizes and shapes, and radiation-induced physical and chemical changes in a wide range of materials. An electron microscopical study was made of the effect of cationic surfactants on the structure of thoria sol-gel beads. The examination of the ultrastructure of certain exchange resins was begun. Vapor-deposited thin metallic films were made for the Physics Division to be used in their semiconductor experiments.

12. Nuclear and Radiochemical Analyses

Branching-ratio measurements and decay-scheme studies were made on ^{141}Ce, ^{79}Kr, ^{99}Mo-^{99m}Tc, and a new isomer, ^{82m}Br. A study of successive neutron-capture reactions on La and Ce was begun. A search for new alpha emitters resulted in the discovery of alpha emission in ^{148}Eu. Nuclear levels were studied in a number of even-*A* nuclei, and neutron-deficient hafnium radionuclides produced in ORIC by (α,*xn*) reactions were investigated. The use of lithium-drifted germanium diodes for such decay-scheme studies and the effect of spectrometer live time on precision were evaluated. An IAEA ^{137}Cs standard intercalibration was done, and the absolute determination of ^{125}I by the sum-coincidence method was made. A gamma-spectrometric determination of ^{232}U was developed and applied. New half-life values for ^{76}As, ^{137}Cs, ^{140}La, and ^{232}U were measured. Trialkyl- and hexaalkylphosphorothioic triamides and metal di-*n*-butyl phosphorodithioates were studied as extractants for metals. Trivalent actinides were separated from lanthanides by liquid-liquid extraction with tricaprylmethylammonium thiocyanate. Technetium was separated and determined by cyclohexanone extraction. Cellulose column chromatography was used to separate trace quantities of 13 metal ions from uranium. Separation of tellurium isomers in different chemical media was studied; work continued on the determination of Kr and Xe fission yields from uranium. Studies relative to ore selection for Project Coach were made. A search for a naturally occurring transuranic radionuclide was unsuccessful. Cooperative work in low-level analysis continued. Two new liquid scintillators, 1-methylnaphthalene and N,N′,N″-trimethylborazine, were studied. A liquid-scintillation method for absolute counting of beta emitters was developed. Several different computer techniques were applied to the resolution of gamma-ray spectra.

A radio-release method was developed for the measurement of sulfur dioxide in the atmosphere. Radiotracers were used to study and to improve

chemical procedures for sodium, lithium, and phosphorus. The problem of separation of sodium from activated samples received continuing attention.

A study of precision in activation analysis was begun. In applications of activation analysis to forensic problems, ammunition primer and gunshot residues, as well as hair and paper, were studied. Trace-element nutritional investigations in humans and animals were continued, and a number of unusual samples, both biological and chemical, were analyzed. Analyses for trace elements in uranyl nitrate solutions and in high-purity Be, Al, and Fe were made by neutron activation. In addition, isotopic neutron sources were used for the analysis of organo-boron compounds and high-cross-section materials in reactor fuel elements.

A rapid method to calculate the output spectra from the 14-Mev neutron generator was formulated; a catalog of 14-Mev neutron reactions for elements of $Z = 3$ to 30 was prepared. Work on fast-neutron cross sections for titanium continued. An improved sample-irradiation and sample-handling system (primarily for oxygen determination was designed. The neutron generator was used for the analysis of a number of materials and for the evaluation of neutron yields from experimental targets. Excitation functions and other nuclear data were gathered from experimental runs made at the 5.5-Mev Van de Graaff accelerator with ^3He particles; the potential usefulness of such procedures for activation analysis is being investigated.

13. Inorganic Preparations

The preparation of fused salts for the Metals and Ceramics Division and for the Chemistry Division continued on a reduced scale. For the latter division other preparations were several 30-g quantities of K_2S in fairly high purity and pound quantities of high-purity KCl and LiCl.

The Reactor Chemistry Division was provided with one or more preparations each of anhydrous $AlCl_3$, $ZrCl_4$, $ThCl_4$, and $MgCl_2$ and a preparation of UCl_3 of fairly high purity.

For the Solid State Division dysprosium phosphide, DyP, was prepared in practically 100% purity, and a study of the holmium-sulfur system is being made. High-purity KCl is being produced for this division on a routine basis.

The Metals and Ceramics Division was provided with further preparations of K_2O and some alkali-metal niobates. The preparation of alloys that contain rare-earth metals was continued for the Physics Division. Other miscellaneous isolated preparations on a small scale were made for several other groups.

14. Organic Preparations

A number of special organic compounds that are not commercially available were synthesized on request from other research divisions. These compounds were: methyl isocyanide, N,N′,N″-trimethylborazine, 6-amino-3,9-diethyltridecane, two new monoacid esters, and α,α'-dipiperidyl.

PART B. SERVICE ANALYSES

15. Quality Control

The Statistical Quality Control Program increased during the year. Some 5200 control tests were made. A significant improvement occurred in the quality of work.

16. Mass Spectrometric Analysis

The Mass Spectrometry Laboratory analyzed 950 samples of 34 elements for the Stable Isotope Separation program. In addition, a wide variety of special gas samples were analyzed. Relative isotopic abundances were determined, and a number of radioactive samples were analyzed.

17. Spectrochemical Analysis Laboratory

In the Spectrochemical Analysis Laboratory, over 18,000 results were reported on some 1200 samples; the stable isotopes of about 34 elements were determined. A third of the analyses were for the Isotopes Division. The Wadsworth and Ebert spectrographs were relocated; one arc-spark stand now serves both instruments. With one exposure, 60 to 65 trace elements can be determined.

18. Process Analyses

The Process Analyses Laboratories performed 146,500 analyses. The Low-Level Radiochemical Laboratory was combined with the Radioisotopes-Radiochemistry Laboratory. A new glove-box line was installed in Building 3019 and will soon be in operation. A brief statement of the new developments in each laboratory follows.

The High-Level Alpha Radiation Laboratory reported over 10,000 results. Low levels of plutonium were determined for the Savannah River Laboratory. Plutonium sols were analyzed for nitrogen. Protactinium was determined in samples that contained various interferences.

The General Analyses Laboratory continued work on the determination of free HF and on measurements of coating density. A Technicon AutoAnalyzer was used to determine uranium and the detergent, ABS. Computer programs were coded for pycnometer density, total anion, permeability, and porosity measurements.

The General Hot Analyses Laboratory continued to work on the preparation for handling, treating, and analyzing radioactive materials anticipated from proposed programs. Methods and equipment were established for the MSRE and Curium Programs, and the training of the personnel was initiated. Special hot-cell work included electrolytic disintegration of graphite spheres, dissolution of irradiated graphite, and carbon determination.

The Materials Testing Laboratory continued the analysis of various fluoride salts, rocks and ores, alloys, and aqueous and organic solutions derived from fuel-reprocessing and waste-disposal studies. Specification tests were made on salts to be used in the MSRE.

The Radioisotopes-Radiochemistry Laboratory has had an increase in personnel and in the volume and variety of work performed. The low-level work and the intermediate-level work were consolidated and will be located at Y-12. Hot-cell gamma spectrometry is now being done in Building 3525. Many of the gamma spectra are now resolved by computers. The mass distribution of fission products formed by epicadmium-induced fission of ^{237}Np was measured.

The High-Radiation-Level Analytical Facility recently experienced a wide diversification of analytical work. The samples received varied from graphite-coated uranium particles to large pieces of irradiated tubing. Procedures for the analysis of high-level alpha-emitting materials were adapted for use in glove-box-type shielded work cells.

The new High-Level Analytical Laboratory is completed. Minor changes and installation of equipment are now in progress. This laboratory should be ready to receive samples by November 1964.

A mockup of a cell in the Transuranium Facility is being constructed for use in the development and evaluation of methods.

19. Reactor Engineering Service Laboratory

The Reactor Engineering Service Analytical Chemistry Laboratory made ~5700 analyses on some 1160 samples, about half of which were from the Reactor Division. Both the samples and the methods of analysis varied widely.

PART C. *ORNL MASTER ANALYTICAL MANUAL*

20. *ORNL Master Analytical Manual*

The first revision to the cumulative indexes for the *ORNL Master Analytical Manual* was prepared by means of the same IBM 7090 computer program used last year. Information about the methods written in 1963 was added, thus making the indexes cumulative for the years 1953 through 1963. The updated indexes are available from the U.S. Department of Commerce; they are designated TID-7015 (Indexes), Revision 1.

A new Section 4, "Mass Spectrometric Methods," and also the Subsections 9 02, "Molten-Salt Reactor Experiment (MSRE) Methods," and 9 06, "Transuranium (TRU) Process Methods," were added to the *Manual*. Altogether, 106 new methods and revisions to 46 methods were issued, and the Table of Contents was revised. The sixth supplement to the reprinted form of the *Manual* (TID-7015, Suppl. 6) was issued.

The forthcoming need for new methods for the Division was determined, and methods-writing work was planned accordingly.

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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.

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1. Analytical Instrumentation

M. T. Kelley
D. J. Fisher

1.1 RECENT DEVELOPMENTS IN DC POLAROGRAPHY

D. J. Fisher W. L. Belew
M. T. Kelley

A paper was presented[1] that summarized the work done at ORNL in controlled-potential dc polarography since mid-1959. The viewpoints and conclusions reached in the course of this work were discussed. The topics included were:

1. Potentiostatic compensation for polarographic cell resistance; principles of potentiostatic (controlled-potential) electrolysis
2. Time-derivative dc polarography
   (*a*) Requirements for quantitative time-derivative dc polarography
   (*b*) Additional requirements for optimum resolution and sensitivity
3. Signal-to-noise ratio (S/N)
   (*a*) Definition of S/N
   (*b*) Measurement of low-concentration polarograms
   (*c*) Relative S/N of various kinds of polarized mercury electrodes
   (*d*) Reproducibility of regular wave height and of first-derivative peak height
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   (*f*) Relative signal levels in regular and in first-, second-, and third-derivative dc polarography
4. Resolution
   (*a*) Criterion for relative resolving power of various methods of polarography
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   (*e*) Mathematical resolution of overlapped first-derivative dc polarograms
   (*f*) Mathematical resolution of regular polarography compared with first-derivative dc polarography

[1] D. J. Fisher, W. L. Belew, and M. T. Kelley, "Recent Developments in DC Polarography," presented at the 3rd International Congress of Polarography, Southampton, England, July 19–25, 1964 (to be published in the proceedings of the Congress).

5. DC polarography in nonaqueous solvents and, particularly, in solvent extracts
   (*a*) Electrochemical requirements for polarography in nonaqueous solvents, especially in solvent extracts
   (*b*) Controlled-potential dc polarography of metal complexes in solvent extracts

1.2 THE QUASI-REFERENCE ELECTRODE (Q.R.E.), A SIMPLE REFERENCE ELECTRODE. APPLICATIONS IN CONTROLLED-POTENTIAL POLAROGRAPHY, CONTROLLED-POTENTIAL VOLTAMMETRY, AND CHRONOPOTENTIOMETRY

D. J. Fisher W. L. Belew
M. T. Kelley

The most essential requirement of any simple electrode that is to be used instead of a true reference electrode is constancy of potential during a particular experiment. The quasi-reference electrode (Q.R.E.) is a bright-platinum wire, or other inert conductor, immersed directly in the test solution; the immersed area may be very small. This electrode is used instead of a true reference electrode, but it must be used with potentiostatic or other circuits that prevent the drawing of the cell current through the Q.R.E. With this arrangement, the results are like those obtained with a true reference electrode, except that the Q.R.E. potential lacks thermodynamic meaning; hence, this simple electrode is called a quasi-reference electrode.

For a true reference electrode, the reaction that permits the passage of current through the electrode is reversible and is known; the electrode will not be polarized significantly if the current is substantially lower than the exchange current of the reaction. However, the resistance of the associated salt bridge and interface junctions may be high enough to cause significant *iR* error and current phase shift. Also, junction potentials may be of significant and variable magnitude. Nevertheless, when properly applied, the potential of a true reference electrode relative to that of a standard hydrogen electrode is predictable and reproducible. If electrolysis is performed with a potentiostat, only the potentiostat input current, which is very low, must be sustained by the electrode used as reference. Two extreme cases are represented by a true reference electrode and by the Q.R.E., since in the latter case the reaction or reactions that permit passage of potentiostat input current through the Q.R.E. cannot be predicted. Sometimes it is inconvenient to use a true reference electrode having an exchange current high enough to permit the passage of cell current with negligible polarization. Also, such an electrode may not be available. It is important that a reference electrode having only a moderate exchange current (i.e., less than the cell current) can be used and will function in a potentiostatic circuit as a true reference electrode.

In many practical situations, the lack of thermodynamic significance of the potential assumed by the Q.R.E. is tolerable. The Q.R.E. has been used successfully for controlled-potential polarography, controlled-potential voltammetry, and chronopotentiometry in aqueous, organic, and molten-salt solvents of unspecified composition but constant overall composition. The form of the polarographic waves is conventional, but the observed $E_{1/2}$ is meaningless; a slow drift in Q.R.E. potential does not change the heights of regular polarograms or, at fast scan rates, of derivative polarograms.

The Q.R.E. is rugged, small, convenient to use, and simple to construct. It does not introduce impurities such as water or Hg^{2+}, Ag^{+}, K^{+}, or Cl^{-} ions. It can be used in corrosive solutions, will operate at high temperatures, and has a very low resistance. The Q.R.E. may be used instead of a true reference electrode when the quantity being measured as a function of concentration or of some other chosen variable is either the change in emf between the working and reference electrodes or the change in current at a constant or scanned applied cell voltage. The predominant consideration leading to the choice of any simple rather than true reference electrode is the relative convenience of use of these electrodes. The Q.R.E., like any other simple electrode, is not meant to compete with true reference electrodes if they are necessary or available and are equally convenient to use.

The Q.R.E. was discussed at the 3rd International Congress of Polarography.[2]

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[2] 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," presented at the 3rd International Congress of Polarography, Southampton, England, July 19–25, 1964 (to be published in the proceedings of the Congress).

1.3 CONTROLLED-POTENTIAL DC POLAROGRAPH-VOLTAMMETER. DESIGN AND EVALUATION

D. J. Fisher H. C. Jones
M. T. Kelley R. W. Stelzner
W. L. Belew

A new controlled-potential dc polarograph-voltammeter was designed in which solid-state instead of vacuum-tube operational amplifiers and power supplies are used. A prototype unit was constructed. This compact instrument is intended for use with solid working electrodes and with short-drop-time Smoler vertical-orifice D.M.E.'s at which the current is diffusion controlled. Its features are

1. a fast potentiostatic circuit whose input voltages are referenced to circuit ground potential,
2. scan rates of 0.1, 0.2, 0.3, 0.5, 1.0, 2.0, 3.0, 5.0, 10.0, and 20.0 v/min,
3. current ranges (full-scale) from 0.02 μa to 10 ma based on the 1, 2, 5 repetitive system,
4. X-Y recorder presentation of instantaneous or average current values as a function of the potential difference between the working and reference electrodes,
5. X-Y recorder presentation of the first, second, and third derivatives of the average current, each with calibrated scales, as a function of the potential difference between the working and reference electrodes, the recorded derivative waves and the recorded regular polarographic wave all having about the same height for a one-electron-change redox reaction ($n = 1$), thus eliminating scale-hunting,
6. prior-wave compensation by injection of a constant current of suitable magnitude into the input of the current amplifier.

The use of solid-state amplifiers and power supplies has reduced the amount of heat generated within the instrument. The use of solid-state amplifiers should also result in better control of the potential in the electrochemical cell because of the greater tolerance of these amplifiers to capacitive loading.

The polarograph-voltammeter is being evaluated with the Smoler vertical-orifice D.M.E.; it will also be evaluated with solid working electrodes. To facilitate evaluation of the instrument in its polarographic function, normalized theoretical waves were calculated and plotted at 1-mv increments by a computer (Control Data Corporation model 1604-A). Regular and first-, second-, and third-derivative dc polarographic waves were plotted for a simple, reversible, diffusion-controlled process. The plots were computed for $n = 1$, 2, and 3. The amplitude and form of recorded polarographic waves will be compared with those of the theoretical waves. Similarly, the instrument will be evaluated in its voltammetric function.

The prototype will be used for continued research in polarography and voltammetry and as the basis of design of several controlled-potential dc polarograph-voltammeters for use in the Analytical Chemistry Division.

1.4 CALCULATION OF DIFFUSION COEFFICIENT FROM THE KOUTECKÝ EQUATION

R. W. Stelzner W. L. Belew

The Koutecký equation,[3]

$$\bar{i}_d = 607nmC\left(\frac{D^{1/2}t^{1/6}}{m^{1/3}}\right)\left[1 + 34\left(\frac{D^{1/2}t^{1/6}}{m^{1/3}}\right) + 100\left(\frac{Dt^{1/3}}{m^{2/3}}\right)\right],$$

is regarded as the best mathematical description of the diffusion current in simple, reversible, diffusion-controlled, dc polarographic phenomena. By a trigonometric transformation, this cubic equation was solved for D; this solution is suitable for use in making desk calculations of D. Also, a computer program, DIFFUSE, that solves the Koutecký equation for D was written.[4] This program uses the subroutine VIETA to calculate explicitly the physically realizable root.

[3] J. Koutecký, "Correction for Spherical Diffusion to the Il'kovič Equation," *Czech. J. Phys.* 2, 50 (1953).

[4] R. P. Rannie, Mathematics Division.

1.5 ORNL MODEL Q-2564 HIGH-SENSITIVITY COULOMETRIC TITRATOR

H. C. Jones

Two ORNL model Q-2564 titrators[5,6] were fabricated by the Instrumentation and Controls Division and are now in use, one in the High-Radiation-Level Analytical Laboratory (HRLAL) and the other in the High-Level Alpha Radiation Laboratory. The Instrumentation and Controls Division completed the Q-2564 series of drawings. A check-out and test procedure (ORNL Test Specification ST-260) was written for use in ensuring that each newly constructed titrator meets the performance specifications. A set of simplified operating instructions was also prepared, and a troubleshooting guide was written for use with ST-260 in maintaining the titrators.

A paper describing this instrument and giving procedures for titrating micro- and milligram amounts of uranium and iron was written for publication in the open literature. About 25 complete sets of engineering drawings of the titrator were sent to interested installations in the United States and abroad.

1.6 THEORY AND APPLICATION OF THE DERIVATIVE METHOD TO STATIONARY-ELECTRODE VOLTAMMETRY WITH LINEARLY VARYING POTENTIAL

T. R. Mueller S. P. Perone[7]

The theoretical shapes of the first, second, and third time derivatives of linear-sweep voltammetric curves were calculated for plane and spherical stationary electrodes for the case of simple reversible ion-ion or ion-amalgam electrode processes. Derivative-measurement techniques were tested experimentally with reversible electrodepositions at the hanging-mercury-drop electrode by electrolysis with linearly varying potential. Analytical characteristics of first-, second-, and third-derivative measurements were studied, the latter two in a cursory way only. The accuracy and reproducibility of first-derivative linear-sweep voltammetry are about the same as those of regular linear-sweep voltammetry, but the sensitivity is at least an order of magnitude greater.

This work was reported in a paper given at the 148th American Chemical Society Meeting[8] and is tentatively scheduled for publication in the January issue of *Analytical Chemistry*.

1.7 SAFETY CIRCUIT FOR THE MULTIPLIER PHOTOTUBE OF THE ORNL MODEL VII FLAME SPECTROPHOTOMETER

R. W. Stelzner

The EMI/US[9] 9558QA multiplier phototube, which costs $633, is used in the ORNL model VII flame spectrophometer[10] to span the wavelength range from 2000 to 8700 A. The phototube is powered from a John Fluke Manufacturing Company model 412A voltage-regulated power supply. When certain circuit failures occur in this power supply, voltages greater than the tube manufacturer's recommended maximum are applied to the phototube, possibly resulting in its loss.

A safety circuit has been developed and installed that senses the onset of an excessive applied voltage of either polarity and automatically shuts down the power supply, even when the instrument is unattended. A 1-ma relay and a suitable resistor string, plus the existing switching circuitry of the model 412A power supply, are used to accomplish rapid shutdown and manual start-up. In two such power-supply failures, the safety circuit operated successfully to save the multiplier phototube.

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[5]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, p. 3.

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

[7]Assistant Professor, Department of Chemistry, Purdue University, Lafayette, Ind.

[8]S. P. Perone and T. R. Mueller, "The Application of Derivative Techniques to Stationary Electrode Polarography," presented at the 148th Meeting of the American Chemical Society, Chicago, Illinois, Aug. 30–Sept. 4, 1964.

[9]Electra Megadyne, Inc. (EMI/US) has been purchased by Whittaker Corporation, 9229 Sunset Blvd., Los Angeles, Calif., and has been renamed GENCOM Division, Whittaker Corporation, 151 West 46th Street, New York 36, N.Y.

[10]H. C. Jones, D. J. Fisher, and M. T. Kelley, "Model VII Flame Spectrophotometer," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1963*, ORNL-3537, pp. 7–8.

1.8 SIGNIFICANCE OF PLACEMENT OF THE REFERENCE ELECTRODE IN CONTROLLED-POTENTIAL COULOMETRY

T. R. Mueller

The Cu^{2+}-UO_2^{2+} experiment,[11] made to detect possible error in the controlled potential (driving voltage of electrochemical reaction at the controlled working electrode exceeding command voltage set into the potentiostat) arising from nonuniform current distribution in an electrochemical cell, was repeated with the Bi^{3+}-UO_2^{2+} system in 1 *M* $HClO_4$. Since the exchange current for the reduction of Bi^{3+} is small with respect to that of Cu^{2+}, the probability of loss of uranium should be greater in the Bi^{3+}-UO_2^{2+} system than in the Cu^{2+}-UO_2^{2+} system. As for the Cu^{2+}-UO_2^{2+} system, so for the Bi^{3+}-UO_2^{2+} system, there was no chemical evidence (loss of uranium) that variation in placement of the reference electrode, even under drastic cell conditions, causes measurable error in the controlled potential.

[11] R. W. Stelzner, D. J. Fisher, and M. T. Kelley, "Reference Electrode Placement in Controlled-Potential Coulometry," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1963*, ORNL-3537, pp. 9–12.

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 of various molten-salt systems were continued. The primary objective is to evaluate the applicability of absorption spectrophotometry to the determination of ionic species in molten-fluoride-salt systems of interest to the Reactor Chemistry Division. To do this, it is necessary to investigate the nature of the dissolved species as well as their stability and compatibility with respect to other dissolved species and solids that are in contact with the melt.

2.1.*a* Nickel High-Temperature Spectrophotometric Cell Assembly

Hydrolytic contamination of melts occurred in the operation of the nickel high-temperature cell assembly described previously.[1] It was assumed that small amounts of HF gas, liberated from the molten-fluoride samples, reacted with a NiO coating on the interior surface of the nickel cell and that the resultant water reacted with the melt to liberate HF. Because of the relatively large area of nickel metal compared with the small volume of sample, this process could go on indefinitely. The hydrolytic contamination was eliminated by cleaning the interior of the cell assembly with nickel wool and placing chips of sponge titanium in the sample compartment to react preferentially with H_2O, O_2, and HF gas. The stability of the absorption spectrum of U^{4+} in LiF-NaF-KF over a period of about 15 hr indicated that contamination had been eliminated.

Installation of limit-control switches in the power supply of the high-temperature cell assembly has made possible the safe operation of the assembly overnight, if necessary.

2.1.*b* Containers for Molten-Fluoride Salts

The formation and stability of U^{3+} in LiF-BeF_2 and in LiF-BeF_2-ZrF_4, which are the coolant and fuel solvent respectively of the MSR, were studied by allowing various metal reductants to react with the molten salt that contained U^{4+}. The procedure often consisted in placing a piece of the metal

[1] J. P. Young, "Spectrophotometric Studies of Molten-Salt Systems," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1963*, ORNL-3537, pp. 25–30.

reductant of interest in a windowless container of the pendent-drop type[2] and filling the container with the molten salt by immersion. The filled container was brought immediately into the optical path for observation. All the containers for molten salts were made of copper, which is reasonably compatible with reactants and possible products. Studies by the Reactor Chemistry Division have shown that BeF_2-containing melts do not wet a clean copper surface. It is thought that the original captive-liquid cell[3] functions because liquids wet the surface of the container. It was assumed that the small "keeper" holes of the captive-liquid cell might not be needed in a cell designed to hold nonwetting liquids. Recent experimental results confirm this assumption. The path length and constancy of path length of liquids contained in such a modified cell are not known. These studies are being continued.

[2] J. P. Young, "Spectrophotometric Studies of Molten Salts," *Anal. Chem. Div. Ann. Progr. Rept. Dec. 31, 1960*, ORNL-3060, pp. 67–70.

[3] J. P. Young, "Windowless Spectrophotometric Cell for Use with Corrosive Liquids," *Anal. Chem.* **36**, 390 (1964).

2.1.c Absorption Spectra of U^{3+} in $LiF-BeF_2-ZrF_4$ and in $LiF-BeF_2$

The absorption spectrum of U^{3+} at 500°C in $LiF-BeF_2-ZrF_4$ compared with its previously reported[1] spectrum in LiF-NaF-KF is shown in Fig. 2.1. A solution of U^{3+} in $LiF-BeF_2-ZrF_4$ was prepared by reduction of U^{4+} with either Zr^0 or U^0. For this system, the molar absorptivity of the strongest U^{3+} peak in the near-infrared region (875 mμ) is estimated to be 40 liters $mole^{-1}$ cm^{-1}. In the visible-ultraviolet region, the spectrum of U^{3+} is very intense, and the position of the main peak is in the ultraviolet rather than the visible, as might be expected. Indeed, dilute solutions of U^{3+} in BeF_2 melts are yellow, whereas those of U^{3+} in LiF-NaF-KF are red. The molar absorptivity at the wavelength of maximum absorbance, 360 mμ, is estimated to be 500 liters $mole^{-1}$ cm^{-1}. The spectrum of U^{3+} in $LiF-BeF_2$ appears to be identical with that in $LiF-BeF_2-ZrF_4$.

The intensity of the 360-mμ peak of U^{3+} in these melts provides a sensitive means of observing reactions that either generate or consume U^{3+}. An example of this use is the kinetic study made of

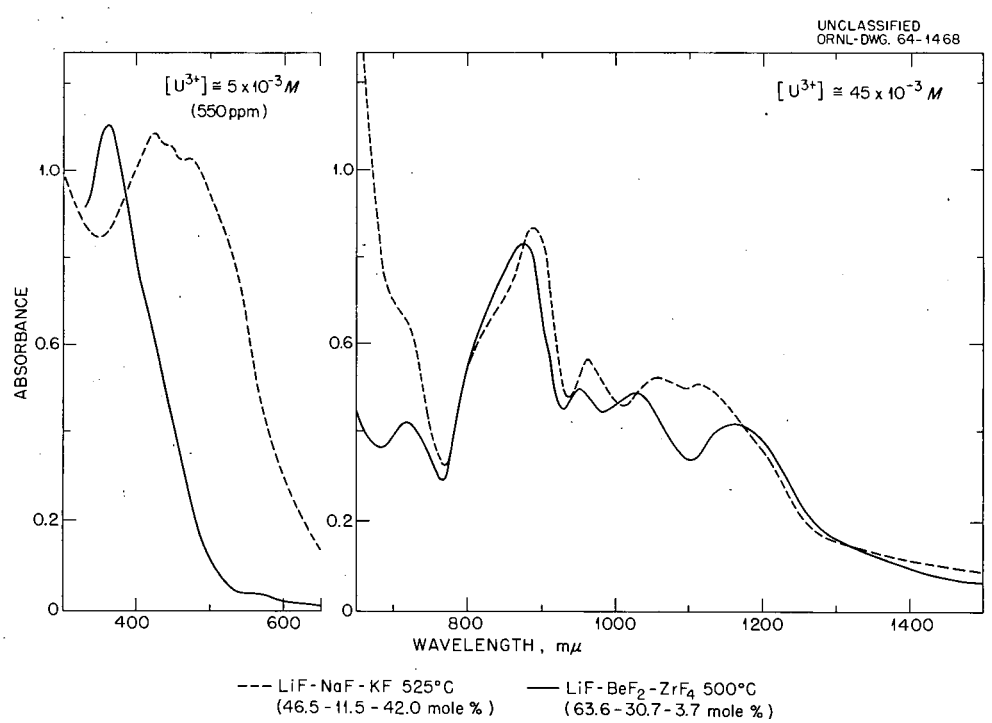

Fig. 2.1. Spectra of U^{3+} in Molten-Fluoride Media.

the reduction of U^{4+} (~1000 ppm) with Zr^0 in LiF-BeF_2-ZrF_4. It was assumed that the reduction of U^{4+}[2] is essentially quantitative and that it is therefore possible to measure the loss of U^{4+} by formation of U^{3+}, observed spectrophotometrically. Such spectrophotometric absorption measurements indicate that the reduction of small amounts of U^{4+} by Zr^0 is first order with respect to U^{4+} concentration over the temperature range studied (480 to 540°C); the specific rate constants are 0.042, 0.065, and 0.10 per minute at 480, 510, and 540°C respectively.

In the molten solvents LiF-BeF_2 and LiF-BeF-ZrF_4 at 500 to 550°C, U^0 is partly oxidized to U^{3+}[4]. The mechanism of this reaction is not understood. Possibly, contaminant ions in the melt, such as Fe^{2+}, Cr^{2+}, and Ni^{2+}, are being reduced. No spectrophotometric evidence for the existence of Zr^{3+} has been observed under any experimental situation where it might be expected to occur, for example, the reaction of Zr^0 with Zr^{4+}. Likewise, in the reaction of U^{4+} with Zr^0, no peaks attributable to any species other than U^{3+} are observed.

A paper in which the results of these studies were discussed was presented.[4]

2.1.*d* Studies of the Absorption Spectra of Molten Alkali-Metal–Alkali-Metal-Halide Systems

The vacuum-tight crystalline Al_2O_3 cell mentioned previously[1] was constructed and used. This work is being done in cooperation with H. R. Bronstein.[5] Attempts were made to observe the absorption spectra of the systems K-KBr and Na-NaI, both above and below their melting points. However, in the molten state both systems are opaque. A vapor bubble was seen in the melt K-KBr; the spectrum of this vapor was that of a mixture of K atoms and K_2. The melt solidified to a violet crystalline mass, the spectrum of which appears to be that of an R_2 band (derived from *F* bands) in KBr. The solidified Na-NaI melt is a blue crystalline mass, the spectrum of which can be ascribed to *F* bands.

[4] J. P. Young, "Direct Spectrophotometric Studies of Redox Reactions in Molten Fluoride Salts," paper presented at 15th Meeting of the Comite International de Thermodynamique et de Cinetique Electrochimiques, London, England, Sept. 21, 1964.

[5] Chemistry Division.

2.1.*e* Holder for Use in Obtaining Absorption Spectra of Small Crystals

In cooperation with G. W. Clark,[6] a special holder for very small crystals (Fig. 2.2) was devised for use in obtaining their spectra. With this holder and the Cary model 14 M recording spectrophotometer, provided with the model 1471200 high-intensity light source, it is possible to obtain spectra of crystals of diameter slightly larger than 0.1 mm in the wavelength range from about 280 to 750 mμ; for crystals of 0.3 mm diameter or larger, spectra can be observed over most of the wavelength range of the Cary spectrophotometer. When the apparatus is properly aligned in the sample compartment, its base-line response is linear and is independent of slit width at slit widths of <0.3 mm. The holder should be generally useful in spectral studies of small crystals, such as those of interest to ceramists, those sometimes obtained from molten salts, and those that are solutes to be dissolved in molten salts.

A detailed description of the holder is being prepared for publication in *Analytical Chemistry*.

2.1.*f* Absorption Spectrum of UF_6 Gas

Although not directly related to molten-fluoride salts, UF_6 is an important chemical entity and

[6] Metals and Ceramics Division.

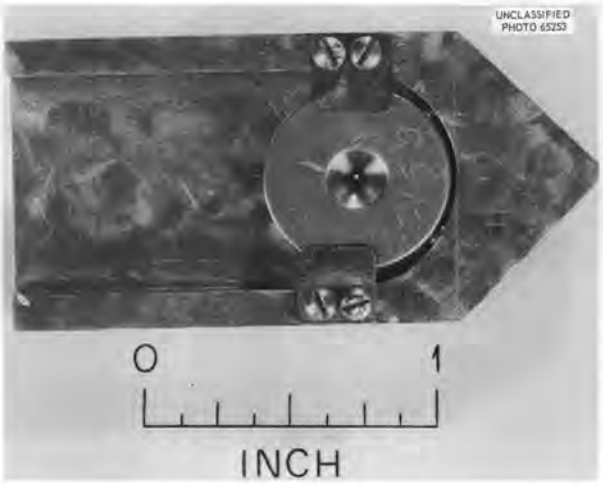

Fig. 2.2. Holder for Use in Obtaining Absorption Spectra of Small Crystals.

one that can form if solutions of U^{4+} in molten-fluoride salts are subjected to oxidizing conditions such as those in the Fluoride Volatility Process. Technique and experience in spectral determinations of such gaseous materials as UF_6 and F_2 are therefore of value in spectrophotometric studies of fluoride systems. J. Winnick[7] was interested in the use of ultraviolet spectrophotometry for in-line determination of UF_6 in gas streams of the Fluoride Volatility Process. Such a gas stream would contain UF_6, MoF_6, and F_2 in varying concentrations. A fluorinated-nickel gas cell of 10 cm path length was constructed; it has crystalline Al_2O_3 windows and Teflon gaskets and is vacuum-tight. This cell was used to determine the absorption spectrum of UF_6. The results show that UF_6 has a complex absorption band in the region from 340 to 420 mμ with a maximum at 368 mμ, at which wavelength the molar absorptivity of UF_6 is 5.5 liters $mole^{-1}$ cm^{-1} $\pm$ 2% over a pressure range from 20 to 100 mm at room temperature. The results from a limited number of spectrophotometric determinations of UF_6 in MoF_6 and F_2 compare favorably with mass spectrometric results. A preliminary report of this work has been written;[8] the study is being continued.

2.2 VOLTAMMETRY OF MOLTEN-FLUORIDE SALT SYSTEMS

J. M. Dale D. L. Manning

2.2.*a* New and Modified Equipment

A vacuum dry box (S. Blickman, Inc., Weehawken, New Jersey) was installed. Leak-rate tests showed that the box conforms to the manufacturer's specification (not >1 μ/hr at 10 μ pressure). Argon is used inside the box as the inert-gas cover. Traces of moisture and oxygen are removed from the argon by passing it through a trap that contains hot titanium sponge metal. The box is used in the handling and transfer of the various fluoride salts being studied by electroanalytical and spectrophotometric techniques.

The apparatus, described earlier,[9] for containing molten fluorides was modified to provide better control of vacuum or inert-gas cover over the melt. Additional graphite electrolysis cells were fabricated. They were cleaned ultrasonically to minimize the amount of graphite dust that floats on top of the melt. Abnormal amounts possibly could cause an electrical short circuit within the cell.

2.2.*b* Decomposition Potentials of Pure Metals in Molten-Fluoride Salts

Before the current-voltage characteristics of various electroactive species in melts are investigated, it is desirable to determine the approximate potential at which a particular species undergoes oxidation or reduction. Table 2.1 gives the approximate decomposition potentials of several metals in molten fluorides. If a reasonable degree of reversibility of the $M \rightleftharpoons M^{n+} + ne$ electrode reaction is assumed, the potentials of the various oxidation-reduction waves can be approximated from such values. For example, in molten LiF-NaF-KF the decomposition potentials of iron and nickel are about the same as the half-wave potentials for the reductions of Fe^{2+} and Ni^{2+} at the pyrolytic-graphite indicator electrode. This relationship between potentials also exists for chromium in molten $LiF\text{-}BeF_2\text{-}ZrF_4$.

For any two particular metals, the difference between decomposition potentials is the only characteristic that should be compared from melt to melt. This limitation is due to the arbitrary zero potential of the platinum quasi-reference electrode. The difference between the decomposition potentials for chromium and iron appears to be larger in $LiF\text{-}BeF_2\text{-}ZrF_4$ than in the other two melts. In both LiF-NaF-KF and $LiF\text{-}BeF_2$ (see Table 2.1) the decomposition potentials of chromium and iron are almost the same; this lack of difference makes their separation difficult, if not impossible.

[7] Summer research participant (Chemical Technology Division), Department of Chemical Engineering, University of Missouri, Columbia.

[8] J. Winnick, *A Preliminary Investigation of the Use of Ultraviolet Spectrometry for the Analysis of UF_6-F_2-MoF_6 Mixtures*, Sept. 14, 1964 (unpublished report).

[9] D. L. Manning, "Voltammetry of Iron in Molten Lithium Fluoride–Potassium Fluoride–Sodium Fluoride," *J. Electroanal. Chem.* 6, 227 (1963).

Table 2.1. Approximate Decomposition Potentials of Pure Metals in Molten Fluorides at 500°C

Metal	Decomposition Potential (v vs platinum quasi-reference electrode)		
	In LiF-NaF-KF (46.5-11.5-42.0 mole %)	In $LiF-BeF_2$[a] (66.0-34.0 mole %)	In $LiF-BeF_2-ZrF_4$ (65.6-30.7-3.7 mole %)
Zr	−1.3	−1.5	−1.5
Cr	−0.5	−0.6	−0.8
Fe	−0.6	−0.5	−0.3
Ni	−0.2	−0.2	+0.1
Mo	+0.3		
W	+0.5		
Cu			+0.5
Ag	+0.8		
Pt	+1.5	+1.6	

[a]G. Mamantov, Reactor Chemistry Division and University of Tennessee, private communication to J. M. Dale and D. L. Manning, July 1964.

2.2.c Voltammetry of Ni^{2+} in Molten LiF-NaF-KF

Rapid-scan and anodic-stripping voltammetry of nickel in molten LiF-NaF-KF (46.5-11.5-42.0 mole %) were studied. The half-wave potential for the reduction $Ni^{2+} \longrightarrow Ni^0$ occurs at about −0.2 v vs the platinum quasi-reference electrode. Current-voltage curves were recorded with a pyrolytic-graphite indicator electrode at voltage scan rates from 50 mv/min to 10 v/min. At the faster scan rates, peak-shaped curves resulted which indicate that the transport process is mainly diffusion controlled. The diffusion coefficient for Ni^{2+} was calculated to be $\sim 1 \times 10^{-6}$ cm^2/sec at 500°C. Anodic stripping shows promise as a way to determine directly trace concentrations of nickel in the melt; as little as 1 ppm can be detected. An article on this work has been published.[10]

2.2.*d* Voltammetry of the MSR Coolant Salt and MSR Fuel Salt

MSR Coolant Salt. – A sample of $LiF-BeF_2$ (66.0-34.0 mole %), which is the coolant salt for the MSR, was received from J. H. Shaffer of the Reactor Chemistry Division. The batch of salt from which the sample was taken had been treated with beryllium metal and then filtered to remove traces of reducible impurities. Current-voltage curves recorded subsequently at a pyrolytic-graphite indicator electrode showed no reduction waves that could be attributed to electroactive impurities such as Fe^{2+} or Ni^{2+}. A small rather ill-defined wave is observed at about −1.1 v vs the platinum quasi-reference electrode. This wave is believed to be due to the reduction of Zr^{4+}, since zirconium in small concentration (700 ppm) was found in the sample by chemical analysis. This wave will be studied further after standard additions of ZrF_4 are made to the melt.

The potential at which the cell current becomes too large to measure (potential limit of the melt) is about −1.7 v vs the platinum quasi-reference electrode. It is believed that at this potential, Be^{2+} is being reduced at the pyrolytic-graphite indicator electrode.

MSR Fuel Salt. – A separate experimental system was set up to permit the study of $LiF-BeF_2-ZrF_4$ (65.6-30.7-3.7 mole %), which is the fuel salt for the MSR. Cathodic scans of the melt have consistently shown a cathodic peak at about −1.3 v vs the platinum quasi-reference electrode. The peak is too large to be accounted for by the impurities known from initial wet analyses to be present in

[10]D. L. Manning, "Voltammetry of Nickel in Molten Lithium Fluoride–Sodium Fluoride–Potassium Fluoride," *J. Electroanal. Chem.* 7, 302 (1964).

the salt. Subsequent spectrographic analysis of the melt indicated the presence of ~1800 ppm hafnium. The current-voltage curves were analyzed on the assumption that the peak was due to hafnium. From a plot of peak current vs square root of voltage scan rate, a value for the electron change (n) of 4.2 was obtained as compared with an expected value of 4. Although this result does not prove that the peak is due to hafnium, it does indicate that further experimental work should be done to determine whether or not hafnium is present.

The cathodic limit of the melt occurs at about −1.5 v vs the platinum quasi-reference electrode. It is believed that Zr^{4+} is being reduced at the pyrolytic-graphite indicator electrode at this potential.

The fuel salt was made 2.5 mM in CrF_2, after which a cathodic wave appeared at about −0.8 v vs the platinum quasi-reference electrode; this is the approximate potential for the oxidation of chromium metal (see Table 2.1). Further study of the chromium system was hindered by difficulties encountered with the pyrolytic-graphite indicating electrode. New methods for fabricating this electrode are being investigated.

2.2.e Reference Electrodes Compatible with Molten-Fluoride Salts

Platinum Quasi-Reference Electrode. – It appears that the platinum quasi-reference electrode is not as well poised in $LiF-BeF_2-ZrF_4$ and $LiF-BeF_2$ as it is in LiF-NaF-KF. This poor behavior was first observed in $LiF-BeF_2-ZrF_4$ when the cathodic limit shifted from about −1.2 to −1.5 v vs the platinum quasi-reference electrode after controlled-potential electrolysis was performed in an attempt to remove reducible impurities from the melt. The negative shift also occurred when the system was evacuated. When the cell was allowed to stand under a confined inert-gas cover, the limit drifted in the positive direction. However, the more negative limit could be maintained after evacuation if the cover gas was allowed to flow through the system. Similar results were found subsequently for $LiF-BeF_2$.

Ni-NiO Reference Electrode. – Reference electrodes similar to the one used by Steinmetz[11] were prepared. These electrodes consist of an intimate mixture of powdered nickel metal and NiO contained in a ZrO_2 tube. Contact to the Ni-NiO mixture is made with a nickel wire. When the electrode is immersed in the melt, the ZrO_2 becomes a solid ionic conductor (oxide ion membrane) and thus serves as a "salt bridge." The electrode supposedly is a second-order electrode that is stabilized by the oxide ion activity in the solid Ni-NiO mixture at 500°C.

Current-voltage curves from the $LiF-BeF_2-ZrF_4$ melt recorded by use of the Ni-NiO electrode did not compare favorably with those obtained with the platinum quasi-reference electrode. The high resistance of the ZrO_2 tube was not the problem, since satisfactory curves were obtained when a 20-kilohm resistor was placed in series with the platinum quasi-reference electrode. The potential of the Ni-NiO reference electrode vs the platinum quasi-reference electrode was followed during a normal voltage scan with the Ni-NiO electrode in the cell circuit. When current started to flow between the working electrode and the counter electrode, the difference in potential between the Ni-NiO and the platinum quasi-reference electrodes varied. Another test was made by using the platinum quasi-reference electrode in the cell circuit and measuring the potential difference between the Ni-NiO electrode and ground during a normal voltage scan. The results showed that the Ni-NiO electrode did not follow the potential of the solution but, in fact, deviated by 0.5 v over a 1.5-v scan. Tests were also performed in which current-voltage curves from the $LiF-BeF_2$ melt were recorded with the Ni-NiO electrode in the circuit while the potential of the Ni-NiO electrode vs the platinum quasi-reference electrode was monitored. In this case the difference in potential between the Ni-NiO reference and platinum quasi-reference electrodes did not deviate by more than ±10 mv throughout the recording of the current-voltage curve.

In an effort to improve the performance of the Ni-NiO reference electrode, the dry Ni-NiO mixture inside the ZrO_2 tube was replaced with $LiF-BeF_2$ melt that contained an excess of NiO (5 wt %) so as to form a saturated solution of NiO. The resistance of the electrode decreased from ~18 to 4-to-6 kilohms, and the general operating characteristics of the electrode improved when it was used in the three-electrode controlled-potential circuit. Over a two-week period the cathodic limit of the $LiF-BeF_2-ZrF_4$ melt remained within

[11] H. Steinmetz, *Development of a Continuous Meter for Oxygen in Sodium*, UNC-5032 (August 1962).

a 0.04-v span with respect to the Ni-NiO electrode, as compared with a 0.3-v span with respect to the platinum quasi-reference electrode. The cathodic limit of the LiF-BeF_2 melt remained constant at -1.85 ± 0.02 v vs the Ni-NiO electrode for a three-week test period. The limits were not affected by static or flowing conditions of the inert-gas cover, as was observed previously for the platinum quasi-reference electrode. It now appears that this type of electrode is a useful reference for molten-salt voltammetry with the three-electrode controlled-potential circuit. However, it could not be used with confidence in a two-electrode circuit, because the cell current that flows through the reference electrode under these conditions could possibly cause polarization and subsequent erratic potential values.

2.3 INSTRUMENTAL APPROACHES TO ANALYSIS OF FUSED SALTS

C. A. Horton

The ability to analyze radioactive molten-fluoride eutectics, either on or off stream, would be helpful in the operation of molten-salt reactors. In addition to absorption spectrophotometry and electroanalytical methods, which are now under study, several other instrumental techniques are being considered on a theoretical basis; some preliminary experimental work was done on a few of these. The determination of minor components of fused alkali-metal fluoride, beryllium fluoride, and uranium(IV) fluoride eutectics has been stressed, prime consideration being given to the effects of high radioactivity. The minor components of importance include oxide, Cr^{2+}, Ni^{2+}, Fe^{2+}, Fe^{3+}, and U^{3+}.

Electron spin resonance was indicated by Llewellyn[12] to be a possible method for determining Cr^{2+}, Ni^{2+}, Cu^{2+}, Fe^{2+}, Fe^{3+}, U^{3+}, and U^{4+} in solid or liquid fluorides. Others[13,14] suggest that in liquid samples the possibility exists for iron only but that electron-spin-resonance measurements might be made at liquid-nitrogen or liquid-helium temperatures. Electron-spin-resonance spectra were obtained at 77 and 295°K for the fluorides of U^{3+}, U^{4+}, and both; these fluorides were present in several concentrations in powdered alkaline-earth-metal fluorides that contained impurity ions of various concentrations and in powdered uranium-doped calcium fluoride. The spectra indicate that, at temperatures of 77°K and lower, U^{3+} ($\sim$250 ppm) can be determined in the presence of 1% of U^{4+} in the molten-fluoride eutectic and very low concentrations of other impurities. At these temperatures, Cr^{2+}, Ni^{2+}, Fe^{2+}, and Fe^{3+} in concentrations from 20 to 50 ppm produce disturbing resonance effects that have not been studied carefully.

Estimation of iron, zirconium, and total uranium in MSRE-type fuels should be possible by x-ray absorption or emission spectroscopy. A special x-ray spectrometer would be necessary. Determination of U^{3+}:U^{4+} ratios by fine-line effects on absorption or emission spectra would probably fail for melts. Radioactivity effects could be minimized in x-ray methods by the combined use of collimation, shielding, and pulsed-source ac detectors, together with a thin, curved analyzing crystal maintained at a high temperature to avoid radiation damage.

Russian workers[15,16] indicate that U^{3+}-doped crystals of alkaline-earth-metal fluorides fluoresce at four wavelengths: 2151, 2210, 2420, and 2490 mμ; of these the first two are also absorption bands. Likewise, similar crystals have a laser emission in the region from 2.2 to 2.8 μ; such laser emission has not been observed for U^{4+}. An unsuccessful attempt was made to duplicate the Russian work using fluoride powders. It was found that fluorescence decreases in intensity at higher temperatures; thus a laser operation would be needed for the estimation of U^{3+}. The high temperature and the corrosive and radioactive

[12] P. M. Llewellyn, "Some Applications of Paramagnetic Resonance," Ph.D. thesis, St. John's College, Oxford University, Oxford, England, 1956; personal communication, 1963–64.

[13] J. Brown, "Electron Spin Resonance in Solid and Liquid Salt Mixtures," *J. Phys. Chem.* **67**, 2524 (1963).

[14] L. Yarnes, M. Kukk, and B. R. Sundheim, "Paramagnetic Resonance Spectrum of Manganese(II) Chloride in Molten Alkali Halides," *J. Chem. Phys.* **40**, 33 (1964).

[15] L. N. Galkin and P. P. Feofilov, "Luminescence of Trivalent Uranium," *Soviet Phys. "Doklady" (English Transl.)* **2**, 255 (1957); *Doklady Akad. Nauk SSSR* **114**, 745.

[16] P. P. Sorokin and M. J. Stevenson, *Advances in Quantum Electronics* (ed. by J. R. Linger), p. 65, Columbia University Press, New York, 1961.

nature of these samples would make experimental production of simulated emission very difficult.

Infrared and Raman spectroscopy, emission and mass spectrometry, and nuclear magnetic resonance[17] seem less applicable for the determination of minor components in fused salts than the methods just discussed. However, infrared and Raman spectroscopy have been used for melts.[18–21]

[17] S. Hafner and N. H. Nachtrieb, "Nuclear Magnetic Resonance in Molten Salts. I. Chemical Shift of Crystalline and Molten Thallium Salts," *J. Chem. Phys.* 40, 2891 (1964).

[18] G. J. Janz, C. Baddiel, and T. R. Kozlowski, "Vibrational Spectra of Molten Mercuric Halides. IV. $HgCl_2KCl$-KNO_3," *J. Chem. Phys.* 40, 2055 (1964).

[19] G. J. Pontrelli, "The Raman Spectra of Selected Molecules at Elevated Temperatures," Ph.D. thesis, University of Wisconsin, 1963.

[20] J. Greenberg, "Near-Ultraviolet, Visible, and Infrared Spectra of Molten KSCN and NaSCN," *J. Chem. Phys.* 39, 3158 (1963).

[21] J. K. Wilmshurst, "Infrared Spectra of Highly Associated Liquids and the Question of Complex Ions in Fused Salts," *J. Chem. Phys.* 39, 1779 (1963).

3. Effects of Radiation on Analytical Methods

J. C. White

3.1 EFFECT OF ^{60}Co GAMMA RADIATION ON THE REACTION OF NICKEL WITH DIMETHYLGLYOXIME

H. E. Zittel

Study of the effect of ^{60}Co gamma radiation on the reaction of nickel with dimethylglyoxime (*I*) was completed. The significance of ethyl alcohol concentration, *I* concentration, radiation-dose level, and accumulated radiation was determined. A very important discrepancy exists between the behavior of *I* and the Ni-*I* complex toward radiation. Spectrophotometric studies of the Ni-*I* complex made immediately after irradiation showed that a postirradiation effect exists. Data taken earlier indicated that the Ni-*I* complex is more stable toward radiation than is *I* itself. The new data indicate that radiation has considerable effect on the color of the Ni-*I* complex but that the color "grows back" after the complex is removed from the radiation source. Extrapolation of the data to zero time (i.e., exact time of removal from the radiation source) shows that the radiation damage to the Ni-*I* complex is a linear function of radiation dose. This postirradiation effect is not a function of the amount of Ni or *I* present. Possibly the radiation damage to the Ni-*I* complex is due to reduction of the Ni(III) in the colored complex.

The results of this study are being prepared for publication.

3.2 EFFECT OF ^{60}Co GAMMA RADIATION ON THE DETERMINATION OF URANIUM(VI) IN PHOSPHORIC ACID SOLUTIONS OF URANIUM(IV) OXIDE

G. L. Kochanny

The effect of ^{60}Co gamma radiation on aerated and deaerated H_3PO_4 solutions of uranium(IV) oxide (UO_2) was studied as a function of temperature, concentration of UO_2, and radiation dose rate. The effect was measured in terms of the radiolytic yield of U(VI), $G_{U(VI)}$. For solutions of high initial UO_2 concentration, $G_{U(VI)}$ is largest for the aerated solutions at 25°C; it is lowest for the deaerated solutions at 140°C. The $G_{U(VI)}$ is lower for solutions of low initial UO_2 concentration than for any of the solutions of high initial UO_2 concentration. At the high starting UO_2 concentration, the initial $G_{U(VI)}$ values are always higher than the succeeding values; this effect is attributed to the depletion of oxygen

originally present in the solution. Gamma radiation causes an error in the determination of the stoichiometry of UO_2; the error is a function of the radiation dose. This error can be minimized by excluding oxygen from solutions of UO_2 and by keeping the initial UO_2 concentration as low as the method of analysis permits. A paper that describes this work is to be published.[1]

3.3 EFFECT OF ^{60}Co GAMMA RADIATION ON ARSENAZO III SOLUTIONS

G. L. Kochanny

Studies on the effect of ^{60}Co gamma radiation on solutions of the disodium salt of arsenazo III (*I*) in various aqueous solutions were completed. The rates of radiolytic decomposition of *I* were determined in hydrochloric, sulfuric, phosphoric, and acetic acid solutions as well as in distilled water. For 2.5×10^{-5} and 1×10^{-4} *M* solutions of *I* in 9 *N* HCl, the average rates of decomposition of *I* are 4×10^{-6} and 2×10^{-6} mole/liter per kilorad of absorbed gamma radiation respectively. For a 1×10^{-4} *M* solution of *I* in 1 *N* HCl, the average rate of decomposition of *I* is 9×10^{-7} mole liter^{-1} kilorad^{-1}. In 9 *N* H_2SO_4 and in 9 *N* H_3PO_4 the initial rate of decomposition is the same as that in 9 *N* HCl, but the rate decreases as the reaction proceeds. In distilled water the average rate of decomposition is about 5×10^{-7} mole liter^{-1} kilorad^{-1}. The initial and final rates of decomposition of *I* in acetic acid solution are fairly constant at 9×10^{-8} and 2×10^{-6} mole liter^{-1} kilorad^{-1} respectively. At a specific absorbed dose, the initial rate of decomposition changes sharply to the final rate of decomposition. The absorbed dose at which this change in rate occurs depends on the acetic acid concentration: for 1 *N* CH_3COOH, 70 kilorads; 2 *N*, 73 kilorads; 4 *N*, 72 kilorads; 9 *N*, 100 kilorads; and 6 *N* CH_3COOH, >180 kilorads. Attempts to identify the products of the radiolytic decomposition of *I* by infrared spectra of solids obtained on evaporation of the solutions were unsuccessful.

[1]G. L. Kochanny, Jr., "Effect of Cobalt-60 Gamma Radiation on the Determination of Uranium(VI) in Phosphoric Acid Solutions of Uranium(IV) Oxide," *Anal. Chim. Acta*, in press.

Study of the effects of gamma radiation on the thorium-*I* complex was begun; initial results show that the complex is decomposed by gamma radiation.

3.4 EFFECT OF ^{60}Co GAMMA RADIATION ON 2-THENOYLTRIFLUOROACETONE (TTA) SOLUTIONS

G. L. Kochanny

A study of the effect of gamma radiation on solutions of 2-thenoyltrifluoroacetone (TTA) was initiated. Xylene was used as the solvent. Gamma ray doses as large as 300 kilorads had no effect on the absorption-spectrum maximum of xylene or on xylene solutions of TTA in the region from 320 to 330 mμ; doses as large as 1800 kilorads did not affect the infrared spectrum. Some variation in the infrared spectrum between 700 and 800 mμ did occur, but the absorption in this region is attributed to the ortho and meta substitution of methyl groups on the benzene ring of xylene.

3.5 RADIOLYSIS OF CHLORIDE SOLUTIONS

Hisashi Kubota

Solutions of high chloride concentration are used in many radiochemical procedures; it is therefore desirable to elucidate the radiolytic behavior of such solutions. Sworski reports[2] that acid chloride solutions react with hydroxyl free radicals in the manner

$$Cl^- + OH + H^+ = H_2O + Cl^{\cdot}.$$

This reaction indicates that free chlorine is produced at the expense of the hydronium ion. No such oxidation is indicated to occur in neutral or basic solutions. Actual determinations of chlorine were not made by Sworski; the maximum chloride concentration studied was 1 *M*. The 10 *M* lithium chloride solutions used in transuranic separation procedures are known to suffer alpha radiolysis and to give up chlorine while losing acidity.

[2]T. J. Sworski, "Yields of Hydrogen Peroxide in the Decomposition of Water by Cobalt γ-Radiation. II. Effect of Chloride Ion," *Radiation Res.* 2, 26 (1955).

Studies were made of the radiolysis of chloride solutions prepared without the special precautions required for fundamental radiation chemistry. The results indicate that chlorine is produced at a rate proportional to both chloride and acid concentrations. At fixed acidity, this production varies linearly with total chloride content. The radiolysis of hydrochloric acid solutions gives an exponential response in which both acid and chloride concentrations come into play. The limit of acid and chloride concentrations below which no chlorine is produced is slightly above 1 *M*.

This radiolytic reaction to produce chlorine can be eliminated effectively by the addition of methanol; other organic solvents produce the same effect. Scavenging of hydroxyl radicals seems to be the principle involved.

The effect of the products of acid chloride radiolysis on analytical procedures is now under study.

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4. Analytical Chemistry for Reactor Projects

J. C. White L. T. Corbin

The Analytical Chemistry Division continued its programs on analytical chemistry research and development for the Molten-Salt Reactor Experiment (MSRE), Gas-Cooled Reactor (GCR) Program, and Transuranium Element Processing (TRU) Program. Details of these investigations were reported regularly in the following progress reports:

MSRE Semiannual Progress Reports:
ORNL-3626 for Period Ending January 31, 1964
ORNL-3708 for Period Ending July 31, 1964

GCR Semiannual Progress Report:
ORNL-3619 for Period Ending March 31, 1964

TRU Quarterly Progress Reports:
ORNL-3597 for Period Ending November 30, 1963
ORNL-3651 for Period Ending February 29, 1964
ORNL-3739 for Period Ending August 31, 1964

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

4.1 MOLTEN-SALT REACTOR EXPERIMENT

A. S. Meyer F. E. Haga
R. F. Apple F. K. Heacker
D. J. Fisher J. M. Dale
M. J. Gaitanis W. L. Maddox
R. W. Stelzner

The program being carried out by the Analytical Chemistry Division for the MSRE can be classified into two categories: (1) development and evaluation of the methods required for the analysis of the MSR fuel and related problems and (2) investigation of the feasibility of various techniques for in-line monitoring of the fuel.

The first category has occupied the major portion of the program and should be essentially complete by the end of this year. In-line analysis is in the initial stages of investigation. Among the analytical methods being considered for in-line monitoring are: electrochemical analyses of molten materials, electron spin resonance, and x-ray measurements. It is not contemplated to use these in-line measurements on the MSR but to evaluate them for possible future use on molten-salt reactors.

Specially designed equipment was devised and tested for sampling and handling the highly radioactive fluoride-salt fuel mixtures ($LiF-BeF_2-ZrF_4-UF_4$). The maximum expected radioactivity of a sample taken for analysis (~10 g) will be ~15 curies; thus, cell operations will be performed in the High-Radiation-Level Analytical Laboratory (HRLAL). Methods were established for determining U, Be, Li, Zr, F^-, Cr, Fe, Ni, Mo, and fission products in MSR fuel. The precision to be expected for these methods under remotely controlled operations is currently being established. Methods for determining oxide and the reducing power of the fuel are still being developed. Special equipment that was designed includes a remotely operated filter photometer, a corrosion-resistant servopipetter, and a bottle decapper to accommodate sample bottles of various heights and diameters.

A gas chromatograph equipped with a highly sensitive helium-breakdown detector is being constructed for analysis of the helium carrier gas to be used in the MSR. This chromatograph has the following stable operation detection limits: for permanent gases (H_2, O_2, N_2, CH_4), 5 ppb; for Kr, 3 ppb; for Xe, 5 ppb; and for CF_4, 30 ppb. The postulated detection limit for H_2O is 10 to 20 ppb.

4.2 GAS-COOLED REACTOR

A. S. Meyer

C. M. Boyd

The Analytical Chemistry Division's effort for the GCR Program has been primarily to evaluate various types of detectors for use in the gas chromatographic determination of impurities in helium. The most promising is the helium-breakdown detector by which voltage changes between electrodes carrying a constant current in the microampere range are measured as a function of changes in the composition of the carrier gas. The detector is sensitive to parts-per-billion concentrations of H_2, O_2, N_2, CH_4, and CO. The other detector studied was the Lipsky beta ionization detector, which features a 180-mc beta source of titanium tritide. Technetium-99 was also considered as an alternative source but it is too difficult to plate.

4.3 TRANSURANIUM ELEMENT PROCESSING

P. F. Thomason
F. L. Moore
D. A. Costanzo
F. E. Haga
W. L. Maddox
T. R. Mueller
H. C. Meyer
R. W. Stelzner
W. T. Mullins
A. L. Travaglini

The Analytical Chemistry Division's program for TRU is basically to provide the methods of analysis needed to control the process being carried out at ORNL. In the overall production scheme for the transplutonium elements, ^{239}Pu is irradiated to >99.9% burnup and is then processed to separate the ^{242}Pu from Am, Cm, and rare-earth fission products. The Am-Cm actinide fraction is next separated from the rare-earth elements by solvent extraction (Tramex process) with a long-chain tertiary amine. The actinide fraction is formed into oxide targets, which are irradiated at a high neutron flux ($\sim 3 \times 10^{15}$ neutrons cm^{-2} sec^{-1}) in the High Flux Isotope Reactor (HFIR) at ORNL. The actinides are recovered from the dissolved fuel element by the Tramex process. The Bk, Cf, Es, and Fm produced will be separated by first isolating Bk with 2-ethylhexylphenylphosphonic acid and then using cation exchange resins to collect Fm, Es, and Cf. As a different portion of the overall program, $^{242-244}Cm$ will be separated from other actinide elements.

Both radiochemical and ionic methods are required; all the operations of these methods must be done in both hot cells and glove boxes (to provide protection against alpha radiation). Radiochemical methods are being evaluated and modified when necessary. Heavy reliance is being placed on instrumental techniques and on alpha and gamma spectrometry in an effort to eliminate chemical operations or reduce them to a minimum. Ionic methods for free acid, lithium, total rare-earth elements, individual actinides, and nitrate are under study. Current work includes the use of a cation-sensitive glass electrode for the determination of lithium in 11 *M* LiCl, a direct ultraviolet spectrophotometric measurement of

traces of nitrate in 11 *M* LiCl, potentiometric titration of free acid in Tramex solutions, development of methods for the separation of actinides from lanthanides, and evaluation of methods for determining fission products under operating conditions that simulate actual conditions. In addition, much effort is being given to develop improved remotely operated apparatus such as servopipetters, analytical balances, and solvent-extraction equipment.

5. Chemical Analysis of Advanced Reactor Fuels

J. C. White

A. S. Meyer P. F. Thomason

5.1 GAS CHROMATOGRAPH FOR THE ANALYSIS OF CORROSIVE GASES

A. D. Horton A. S. Meyer

A gas chromatograph was designed to minimize the corrosive effects of such gases as nitrogen dioxide, dinitrogen trioxide, chlorine, and nitrosyl chloride. The measuring-and-control system for the carrier gas is conventional. All of the system that comes in contact with the sample, except the supporting stirrups of the thermistors, is constructed of materials highly resistant to corrosion.

The gas-sampling manifold is constructed of glass tubing and Delmar-Urry greaseless needle valves. It contains a Dynisco pressure transducer, which has an absolute pressure range from 0 to 1000 mm Hg. The tantalum diaphragm is the only part of the transducer that comes in contact with the sample.

The detector "block" consists of a cylindrical glass bulb (~2-in. diam, $2\frac{1}{2}$ in. high) that contains two wells for the sample and the reference thermistors. These wells are connected to the appropriate inlet and outlet tubes. The 8000-ohm thermistors are sealed in place with Teflon O-rings and threaded Teflon caps. The detector block is filled with mercury to provide thermal inertia, and a thermocouple well is provided for measuring the temperature of the block. The block is heated with a glass electric heating tape and is insulated with Styrofoam block and quartz wool. The entire system is built into a Micro-Tek model 2500 instrument base; the Micro-Tek column-heating system is used. A Gow-Mac model 9999 D-1 power supply is used to operate the detector. The chromatograph has been leak- and vacuum-tested, but it has not been tested otherwise.

5.2 QUALITATIVE AND QUANTITATIVE ANALYSIS OF ALIPHATIC DILUENTS BY GAS CHROMATOGRAPHY

A. D. Horton

Components of diluents used by the Chemical Technology Division in research on solvent extraction were identified by gas chromatography on

Table 5.1. Results of Qualitative and Quantitative Analyses of Experimental Aliphatic Diluents

Component	Amount of Component Present (vol %)	
	In P-170	In P-185
n-Octane	0.01	0.1
n-Nonane	0.2	0.1
n-Decane	5.0	0.2
n-Undecane	51	9.0
n-Dodecane	29	43
n-Tridecane	12	36
n-Tetradecane	3	10
n-Pentadecane		1.0
Isomeric paraffins	0.3	0.2

a Barber-Colman model 20 gas chromatograph with an argon-beta ionization detector.

Two aliphatic diluents, identified only as P-170 and P-185, were resolved on a 10-ft-long by $\frac{1}{8}$-in.-OD SE-30 silicone column at 125°C. The argon pressure on the column inlet was 10 psig. Seven normal paraffins were identified in P-170 and eight in P-185 by comparison of the retention times with those of known mixtures of normal paraffins obtained from Micro-Tek Instrument Company and from Phillips Petroleum Company. The above identifications were confirmed by use of a 6-ft-long by $\frac{1}{4}$-in.-OD Reoplex 400 column. Table 5.1 gives the results of the qualitative and quantitative analyses of the diluent samples.

5.3 COMBUSTION–GAS CHROMATOGRAPHIC DETERMINATION OF CARBON IN AQUEOUS AND NITRIC ACID SOLUTIONS

I. B. Rubin

The combustion–gas chromatographic determination of carbon in aqueous and nitric acid solutions was studied further.[1] Modifications made in the apparatus and procedure gave increased efficiency in the conversion of soluble carbon compounds to carbon dioxide and in the subsequent recovery of this gas. Passage of the combustion gases over hot copper did not affect the quantity of carbon dioxide found. The amount of carbon in distilled concentrated nitric acid was determined, and tests were performed with synthetic samples of sucrose and with nitric acid solutions of various compositions to establish the accuracy and precision of the method.

A slight modification in the combustion procedure for the determination of carbon resulted in increased conversion of carbon to carbon dioxide. By raising the oxygen pressure inside the combustion tube and by decreasing the oxygen flow through the cold trap, the recovery of carbon, as carbon dioxide, was increased from an average of 72.4 to 89.0% over the range from 50 to 400 μg of carbon. When the combustion gases were passed over copper needles heated to 1025°C, the average recovery was 88.5%. The overall relative standard deviation in each of these two recent tests was 4%. The copper reduction tube, which was heated by means of a platinum wire wound around the tube itself, was replaced by a larger tube heated by a separate combustion furnace. The new tube was designed to be more easily detached for inspection and for replacement of the copper. A batch of freshly prepared copper was not effective in reducing nitrogen oxides until at least several portions of nitric acid had been processed. Therefore, fresh charges of copper were pretreated as follows: the copper oxide needles were reduced with hydrogen at ~450°C, ~5 ml of concentrated nitric acid was distilled through the copper at 1025°C under vacuum, the oxidation was completed with air at about 450°C, and the copper oxide was re-reduced with hydrogen.

A number of tests were made to determine a nitric acid blank. Concentrated nitric acid was distilled for use in all the tests. When an apparatus blank equivalent to 4.7 μg of carbon was subtracted, the nitric acid blanks ranged from the equivalent of 7.5 to 25.5 μg of carbon over a sample range from 0.05 to 0.20 ml of acid. Synthetic samples that contained from 50 to 400 μg of carbon as sucrose (0.025 to 0.20 ml of solution) were mixed with 0.05- to 0.20-ml portions of concentrated nitric acid. For samples that contained 50 and 400 μg of carbon, the average net recoveries of the carbon were 79.0 and 81.5% respectively. When the results for all samples were averaged, the relative standard deviation was 17%. Thus, it appears that from 50 to 400 μg of carbon can be determined in the presence of from 0.05 to 0.20 ml of concentrated nitric acid (0.8 to 3.2 millimoles). The accuracy is about 80% on an absolute basis and about 90% when compared with standards; the relative standard deviation is about 15%.

No future work on this project is contemplated.

5.4 DISSOLUTION OR DECOMPOSITION OF VARIOUS MATERIALS

J. R. Lund Hisashi Kubota P. F. Thomason

Wichers, Schlect, and Gordon[2] dissolved a variety of oxides, silicates, and ceramic materials in hydrochloric acid by heating the sample,

[1] I. B. Rubin, "Determination of Carbon in Aqueous and Nitric Acid Solutions," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1963*, ORNL-3537, p. 18.

[2] E. Wichers, W. G. Schlect, and C. L. Gordon, "Preparing Refractory Oxides, Silicates, and Ceramic Materials for Analysis, by Heating with Acids in Sealed Tubes at Elevated Temperatures," Research Paper RP-1621, *J. Res. Natl. Bur. Std.* 33, 451 (1944).

Table 5.2. Results of Attempted Dissolutions and Decompositions by Heating with Acids in Sealed Tubes

Material	Analysis Desired	Acid		Result[a]
		Solvent	Oxidant	
HfO_2	None	HCl	$HClO_4$	S
Fluidized coke	Trace elements	HCl	$HClO_4$	F
UO_2 (graphite-coated)	Trace elements	HCl	$HClO_4$	S
ThC_2-UC_2 (graphite-coated)	Trace elements	HCl	$HClO_4$	F
HgS	Cl^-	HNO_3	HNO_3	S
LiF (irradiated)	F	HCl	$(KI + AlCl_3)$	F
$[(C_4H_9O)_2PSS]_2$	P, S	HNO_3	HNO_3	S
$Hg[(C_4H_9O)_2PSS]_2$	Hg, P, S	HNO_3	HNO_3	S
N-Trimethylborazine	B	HNO_3	HNO_3	S
Organic chloride[b]	Cl^-	HNO_3	$(AgNO_3)$	S

[a]F = failure, did not dissolve; *S* = success, sufficient dissolution and/or reaction to complete analysis.
[b]3,9-Diethyltridecylamine-6-amino-3,9-diethyltridecane hydrochloride.

solvent acid, and an oxidant such as nitric or perchloric acid in a sealed tube at temperatures between 100 and 350°C. The scope of this technique was extended to the use of acids other than hydrochloric acid and is now being modified for hot-cell operation. Table 5.2 summarizes the results obtained to date.

In connection with dissolution problems in general, Smith's "liquid fire" treatments[3] have proved more successful for the destruction and dissolution of fluidized coke and graphite-coated materials than has the sealed-tube technique or fusion with sodium peroxide that contains a little potassium nitrate.

Table 5.2 indicates that the dissolution of high-purity refractory hafnium oxide is successful. Although this is true for limited quantities (~0.1 g), the dissolution of as much as 5 g of the material required a preliminary heating with ammonium bifluoride and then a fuming with sulfuric acid.

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

5.5 POLAROGRAPHY IN HYDROFLUORIC ACID AND OTHER GLASS-CORRODING MEDIA

Helen P. Raaen

Polarography with the Teflon dropping-mercury electrode (D.M.E.)[4] was continued. Results which show that the horizontal-orifice Teflon D.M.E. is satisfactory for obtaining fundamental polarographic data were given in two articles accepted for open-literature publication. With this D.M.E., polarography of the $Pb^{2+} \rightleftharpoons Pb^0$ reaction in 1, 4, 8, and 12 *N* HF was studied in detail, and certain fundamental polarographic data were determined for the first time. This work is being described in an article for open-literature publication. During the measurement of half-wave potentials ($E_{1/2}$) with the horizontal-orifice Teflon D.M.E., the magnitude of junction potentials at the salt bridge was found to be significant. Data that show the effect of such potentials were given

[4]Helen P. Raaen, "Polarography in Hydrofluoric Acid and Other Glass-Corroding Media," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1963*, ORNL-3537, pp. 35–38.

in a communication accepted for journal publication. Further work was done to improve the procedure for fabricating Smoler-type vertical-orifice Teflon capillaries; the improved procedure is given in a topical report.

This polarographic work with the Teflon D.M.E. is discussed below.

5.5.*a* Evaluation of the Horizontal-Orifice Teflon D.M.E. for Use in Obtaining Fundamental Polarographic Data

A means of translating from work with the glass D.M.E. in media that do not corrode glass to work with the Teflon D.M.E. in glass-corroding media was provided by taking fundamental polarographic data with the Teflon D.M.E. for two reference redox reactions in noncorroding media.

The $Tl^+ \rightleftharpoons Tl^0$ Reaction. – The polarographic characteristics of the $Tl^+ \rightleftharpoons Tl^0$ reaction in 0.1 *M* KCl–1 m*M* HCl were determined with the horizontal-orifice Teflon D.M.E. They are in agreement with those established from studies done with glass D.M.E.'s. The agreement indicates that polarograms obtained with Teflon D.M.E.'s have the same meaning as those taken with glass D.M.E.'s and can be analyzed in the same way. The results show that data taken with the two types of electrodes are, indeed, interchangeable. The results also ensure confidence in the certainty of polarographic measurements made in glass-corroding media with the Teflon D.M.E.; this ensurance is needed because reference data for such measurements do not exist. An article[5] that discusses this work with the $Tl^+ \rightleftharpoons Tl^0$ reaction is tentatively scheduled for publication in December.

The $Pb^{2+} \rightleftharpoons Pb^0$ Reaction. – In a similar way, the $Pb^{2+} \rightleftharpoons Pb^0$ reaction in 1 *M* HCl was used to evaluate the horizontal-orifice Teflon D.M.E. The results were of the same nature as those obtained in the evaluation with the $Tl^+ \rightleftharpoons Tl^0$ reaction. An article[6] submitted for publication describes this work.

[5]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, in press.

5.5.*b* Significance of Junction Potentials in the Measurement of $E_{1/2}$ Values

In the first work on the measurement of $E_{1/2}$ values of the reference redox reactions $Tl^+ \rightleftharpoons Tl^0$ and $Pb^{2+} \rightleftharpoons Pb^0$ in HCl media with the Teflon D.M.E., the test solution was bridged to a saturated calomel electrode (S.C.E.) with a NaF–agar-agar gel, which was thought to be suitable as a bridge to HF media. It was found that for certain media, especially HCl solutions, the magnitude of junction potentials for this bridge, as well as for other types of bridges, can be unexpectedly large and can have a significant effect on the measured value for $E_{1/2}$. From experimental data, it was concluded that the test solution should be bridged to the S.C.E. through a saturated solution of KCl only, the junction potential thereby being only that of the (test solution)/(saturated KCl solution). This work was done with W. L. Belew;[7] a Short Communication about it was accepted for publication.[8]

For measurement of $E_{1/2}$ values of redox reactions in aqueous hydrofluoric acid media, a bridge was made from Kel-F tubing and disks of sintered Teflon. This bridge is filled with the supporting medium and is connected to the S.C.E. through a solution of saturated KCl. The small differences among the $E_{1/2}$ values for the $Pb^{2+} \rightleftharpoons Pb^0$ reaction in 1, 4, 8, and 12 *N* HF indicate that the use of this bridge arrangement minimizes junction potentials (see Table 5.3).

5.5.*c* Polarography of the $Pb^{2+} \rightleftharpoons Pb^0$ Reaction in Aqueous Hydrofluoric Acid with the Horizontal-Orifice Teflon D.M.E.

The broad program of detailed study of individual redox reactions in aqueous hydrofluoric acid media was begun. The polarographic characteristics of

[6]Helen P. Raaen, "Teflon Dropping-Mercury Electrode for Polarography in Hydrofluoric Acid and Other Glass-Corroding Media. Evaluation with the Lead(II) $\rightleftharpoons$ Lead Reaction," submitted for publication in *Analytical Chemistry*.

[7]Analytical Instrumentation Group.

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

Table 5.3. Polarographic Characteristics of the $Pb^{2+} \rightleftharpoons Pb^0$ Reaction in Aqueous Hydrofluoric Acid Media

Polarographic Characteristic	HF Concentration, N			
	1.00	4.00	8.00	12.00
Approximate usable range of supporting medium, v vs S.C.E.[a]	+0.4 to −1.25	+0.4 to −1.20	+0.4 to −1.15	+0.4 to −1.10
Number of waves	One	One	One	One
$E_{1/2}$, v vs S.C.E.[b]	−0.380	−0.380	−0.375	−0.370
Reciprocal slope of $\log [\bar{i}_d/(\bar{i}_d - \bar{i})]$ vs $E_{d.e.}$[c]	0.036	0.035	0.036	0.033
$90.6/W_{1/2}$[d]	1.90	1.92	1.92	1.94
n	2	2	2	2
Reversibility	Reversible	Reversible	Reversible	Reversible
$[Pb^{2+}]$ vs $\bar{i}_d$[e]	Linear	Linear	Linear	Linear
$[Pb^{2+}]$ vs $(d\bar{i}/dt)_{max}$	Linear	Linear	Linear	Linear

[a]From dissolution of mercury to evolution of H_2.
[b]Determined from both average-currents regular and derivative polarograms; the value includes junction potentials.
[c]Theoretical value for a reversible 2-electron-change redox reaction = 0.030.
[d]$W_{1/2}$ = width of first-derivative peak at half-peak height.
[e]Over the range of $[Pb^{2+}]$ from about 0.03 to 0.8 mM.

the $Pb^{2+} \rightleftharpoons Pb^0$ reaction in 1, 4, 8, and 12 N HF were determined with the horizontal-orifice Teflon D.M.E. and a bridge of the supporting electrolyte (see Sect. 5.5.*b*). Table 5.3 summarizes these characteristics, which were determined for the first time.

The Teflon D.M.E. behaved excellently during the study. Undamped regular, average-currents regular, and derivative polarograms were recorded. All these are of the form usual for a glass D.M.E. The undamped polarograms indicate precise drop formation. The shapes of the regular and derivative polarographic waves are excellent; residual-current and limiting-current regions are parallel, and there is no evidence of maxima although maximum suppressors were not used. Diffusion currents were reproducible.

The electrocapillary curve of mercury was determined in each of the HF media. The curves have the usual parabolic shape; the maxima are in the region between −0.40 and −0.55 v vs the S.C.E.

The effect of increase in HF concentration on $E_{1/2}$ is not significant; the use of the bridge of supporting medium probably accounts for this small effect, since junction potentials between the test solution and S.C.E. are thus minimized. The slopes of the plots of $[Pb^{2+}]$ vs $\bar{i}_d$ and of $[Pb^{2+}]$ vs $(d\bar{i}/dt)_{max}$ decrease with increase in HF concentration.

A paper that describes this complete study is being prepared for open-literature publication.

5.5.*d* Extension of the Use of the Horizontal-Orifice Teflon D.M.E. by Others

The recent work of Headridge and his associates[9] indicates that the information given in

[9]J. B. Headridge *et al.*, "Polarographic Investigations in Acidic Fluoride Solutions," Program of the 3rd International Congress of Polarography, The University, Southampton, England, July 19–25, 1964.

ORNL-3344[10] is a satisfactory guide for others who wish to make horizontal-orifice Teflon D.M.E.'s and that the Teflon D.M.E.'s are generally useful for polarographic studies of acid fluoride media. Following the instructions given in ORNL-3344, Headridge and co-workers made Teflon D.M.E.'s and used them to study the polarographic behavior of the ions of 26 elements in HF-NH_4F, HF-HCl, and HF-H_2SO_4 supporting electrolytes. To study complexing effects of fluoride ion, they measured half-wave potentials and other characteristics of the polarographic waves of the ions of 15 metals in 0.1 *M* HF–0.1 *M* NH_4F and compared the characteristics with those of the ions in perchlorate media. They state that their results indicate the possibility of determining molybdenum and tungsten polarographically in the presence of Ti, Zr, Nb, and Ta; of determining several common elements in Ti-, Zr-, Nb-, Ta-, and W-base alloys; and of determining tin and lead in alloys by a new polarographic method.

The satisfactory fabrication and use of Teflon D.M.E.'s by Headridge and his associates indicate that this type of electrode is the means of extending the application of both fundamental and applied polarography to glass-corroding media. The potential amount of such polarography is tremendous; it is gratifying to know that others are having success with the fabrication and use of Teflon D.M.E.'s.

5.5.e Vertical-Orifice Rapid Teflon D.M.E.

In work with Ralph L. Clark,[11] an improved procedure was developed for fabricating the vertical-orifice Teflon capillary of a rapid Teflon D.M.E. Special tools were designed and built for use in the fabrication. With a micro drill press and a micro drill, the vertical orifice is drilled in a lapped vertical face of the capillary blank. The blank is held in a special jig mounted on the table of the drill press. To form orifices of about 100-μ diameter or larger, a micro twist drill is used; for smaller diameters, a micro pivot drill is used. Several capillaries that have orifice diameters of about 70 and 100 μ were made. Although these capillaries have not yet been evaluated thoroughly, photomicrographs indicate that they have better geometry at the orifice than has a vertical-orifice glass D.M.E. which has performed satisfactorily. The improved fabrication procedure is given in ORNL-3654.[12]

5.6 CHRONOPOTENTIOMETRY OF PLUTONIUM

D. G. Peters[13] W. D. Shults

A general study of the chronopotentiometric behavior of plutonium in mineral acid media was conducted. The results are presented in a published paper.[14]

The chronopotentiometry of Pu^{3+}, Pu^{4+}, and Pu^{6+} at a platinum electrode in various media was studied to determine the diffusion coefficients of the different plutonium species, the effects of surface oxidation of the platinum electrode, and the possible analytical usefulness of chronopotentiometry for the determination of plutonium.

The reduction of $Pu^{4+} \longrightarrow Pu^{3+}$ at a platinum cathode in perchloric, sulfuric, nitric, and hydrochloric acid solutions was investigated. The relatively small diffusion coefficient of Pu^{4+} in each of these media plus the spontaneous chemical oxidation of the platinum electrode by Pu^{4+} result in a significant positive error in chronopotentiometry with concentrations of Pu^{4+} below 0.01 *M*.

Chronopotentiograms for the $Pu^{3+} \longrightarrow Pu^{4+}$ oxidation in sulfuric acid show one wave due to oxidation of Pu^{3+} followed by a second wave attributable to the formation of a platinum oxide film. Plutonium(III) in sulfuric acid medium chemically reduces the oxide film. The electrooxidation of Pu^{3+} in perchloric acid medium gives a well-defined chronopotentiogram at either prereduced or preoxidized electrodes.

The chronopotentiometric reduction of Pu^{6+} in 1 *f* perchloric acid proceeds stepwise first to Pu^{5+} and then to Pu^{3+}. No evidence that Pu^{5+} undergoes significant disproportionation in the perchloric acid medium was observed under the experimental conditions.

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

[11]Plant and Equipment Division.

[12]Helen P. Raaen and Ralph L. Clark, *Fabrication of the Vertical-Orifice Teflon Capillary of a Teflon Dropping-Mercury Electrode*, ORNL-3654 (August 1964).

[13]Assistant Professor, Department of Chemistry, Indiana University, Bloomington.

[14]D. G. Peters and W. D. Shults, "Chronopotentiometry of Plutonium in Mineral Acid Media," *J. Electroanal. Chem.* 8, 200 (1964).

6. Special Research and Development Problems

J. C. White

P. F. Thomason

6.1 REACTIONS IN CONCENTRATED LITHIUM CHLORIDE SOLUTIONS

Hisashi Kubota

Concentrated solutions of strong acid–strong base salts behave similarly to nonaqueous solvents and can be used as media for titrations of very weak bases.[1] This behavior is especially pronounced in concentrated solutions of lithium chloride and of lithium bromide because of their high solubilities (~14 *M*) and the high affinity of lithium ion for water.

Studies with 10 *M* LiCl solutions have shown that many unique applications are possible with this system. In this medium free acid can be determined in the presence of hydrolyzable cations by direct titration without the need to add a separate complexing agent. This determination is especially useful in the presence of a cation that forms a strong chloro complex. Titration beyond the free-acid end point results in the formation of the hydroxide of the cation, and a sharp end point corresponding to the completion of hydroxide formation is seen.

When a mixture of several hydrolyzable cations is titrated, the cations can be differentiated provided there is a difference of at least seven units between respective *pK*'s (*K* = solubility-product constant), the most insoluble hydroxide being titrated first. This is not a precise analytical procedure, but is useful as a rapid semiquantitative method. The results are described in a paper accepted for publication.[2]

When Al^{3+} or UO_2^{2+} is titrated past the hydroxide end point, another break that corresponds roughly to the formation of aluminate or diuranate is seen. The appearance and stoichiometry of this break seem to be concentration dependent; further work is necessary to establish conditions for more exact response.

Since lithium chloride is a neutral salt, the titration of weak acids as well as weak bases in this medium seems feasible. Rosenthal and Dwyer[3] claim that titrations of weak acids are difficult to carry out with a glass electrode because of alkali errors. Present work using special glass electrodes for highly alkaline solutions and with pyrolytic-graphite electrodes indicates that interaction between lithium and organic acids interferes with titration of weak organic acids; however, weak inorganic acids like boric acid can be readily titrated.

Titration of phosphoric acid in this medium gives rise to one or two breaks, depending on total acid concentration. The breaks correspond to the neutralization of the first and the sum of the second and third hydrogens. These breaks result from precipitation of lithium phosphate. Lithium phosphate is very slightly soluble in 10 *M* LiCl; the solubility is a function of acid concentration. When there is sufficient initial total acidity, the most active acid is titrated before precipitation begins. When the total acidity is small, precipitation sets in before any acid differentiation is possible.

6.2 DISPOSAL OF RADIOACTIVE WASTES IN NATURAL SALT FORMATIONS

Hisashi Kubota

Laboratory studies associated with the disposal of high-level radioactive wastes in natural salt formations are essentially complete. Work of the preceding year established that no detectable chlorine is released from rock salt by ^{60}Co gamma radiation. The field demonstration now under way is being rigged to allow periodic sampling of

[1] F. E. Critchfield and J. B. Johnson, "Titration of Weak Bases in Strong Salt Solutions," *Anal. Chem.* **30**, 1247 (1958).

[2] H. Kubota and D. A. Costanzo, "Reactions in Concentrated Lithium Chloride Solutions. Determination of Free Acid and Hydrolyzable Metal," *Anal. Chem.*, in press.

[3] D. Rosenthal and J. S. Dwyer, "Acid-Base Equilibria in Concentrated Salt Solutions. Acid-Base Titrations and Measurements of pH and H_0," *Anal. Chem.* **35**, 161 (1963).

entrapped air next to the disposal can so that any possible release of chlorine by long-term irradiation can be detected.

One remaining problem is *in situ* dosimetry at various distances in the salt away from the hot can. Glass dosimeters are to be used for this purpose, but it is also desired to use chemical dosimetry as a reference method. Chemical dosimetry is complicated by the expected high temperatures (100 to 250°C) in the surrounding salt.

6.3 SOLID INDICATOR ELECTRODES

F. J. Miller H. E. Zittel

Evaluation of solid indicator electrodes made of pyrolytic graphite and "glassy" carbon was continued to determine their possible applications and limitations.

6.3.*a* Glassy-Carbon Indicator Electrode

Glassy carbon is a gas-impermeable electrically conductive material which is highly resistant to chemical attack. From it, an indicator electrode was made in two ways: by molding a piece of glassy carbon into an epoxy rod with a central tube that allows mercury contact and by brazing a platinum wire to the glassy carbon and fitting the assembly into a leak-tight Kel-F stem. The electrode is suitable for use over the potential range from about +1.2 to −0.8 v vs the S.C.E. in acid medium. The voltammetry of solutions of the following was studied: Ce^{3+}, Ce^{4+}, Cr^{6+}, Fe^{2+}, Hg^{+}, $Fe(CN)_6^{4-}$, Ag^{+}, Cu^{2+}, and UO_2^{2+}. The E_p and i_p values for these ions are, for the most part, reproducible under the conditions studied and appear to be analytically useful. The results of this study were submitted to *Analytical Chemistry*.

6.3.*b* Oxide Film Formation on the Pyrolytic-Graphite Electrode

In collaboration with D. E. Freeman[4] a study was made of the possible existence of an oxide film on the pyrolytic-graphite electrode under oxidizing conditions. It was shown that an oxide film forms if the electrode is exposed for a long time to a strong oxidant such as Ce^{4+} or is anodized at high positive potentials. The film appears to be temporary; the characteristics of the electrode are unaffected by its formation and removal. The results of this work are being presented by D. E. Freeman as a thesis for the master's degree.

6.3.c Differential Potentiometry

The characteristics of the pyrolytic-graphite and the glassy-carbon electrodes are sufficiently dissimilar that coupling these electrodes for differential titrimetry is possible. The feasibility of doing this was demonstrated by carrying out acid-base and redox titrations. In both cases the differential curve obtained is suitable for end-point detection. The influence of chemical parameters of the system on the quality of the titration is being studied in detail. Such an electrode system would be very useful, especially in hot-cell operations where it could replace electrodes that are fragile, subject to radiation damage, and require frequent maintenance.

6.3.*d* Voltammetry

The pyrolytic-graphite indicating electrode was used to study the voltammetry of I^-, I_2, and IO_3^- in H_2SO_4 solution. Change in H_2SO_4 concentration shifts both the E_p and the i_p of the I^-, I_2, and IO_3^- voltammetric curves. The i_p/C ratio is sufficiently constant for quantitative purposes over the concentration range from 0.005 to 1.000 m*M* for I^- and 0.060 to 1.200 m*M* for I_2. The IO_3^- wave is not reproducible. Absorption of an iodine species on the electrode was demonstrated by tracer experiments.

6.3.e Amperometry

A rapid and precise method for the amperometric titration of Cr^{6+} in mixtures of fluoride salts was developed. The Cr^{6+} is titrated with a standard solution of ferrous sulfate; the cell current is followed with a pyrolytic-graphite electrode. In the range from 1 to 50 μg of Cr^{6+} titrated, the relative standard deviation is less than 2%. This

[4]Graduate student, Department of Chemistry, University of Tennessee, Knoxville.

work, which was done jointly with R. F. Apple, has been published.[5]

6.3.*f* Chronopotentiometry

The glassy-carbon electrode exhibits characteristics which indicate that it should be extremely useful as an indicator electrode in chronopotentiometry. The chronopotentiometry of U^{6+} and its complexes is being studied with this electrode.

6.3.*g* Instrumentation

The polarographs now available are not designed for voltammetry; they lack many features that are needed in the voltammetric evaluation of solid electrodes. Accordingly, the Analytical Instrumentation Group was asked to design and construct a prototype voltammeter specifically for use in studying solid electrodes.

6.4 FLAME IONIZATION DETERMINATION OF PARTS-PER-BILLION CONCENTRATIONS OF ORGANIC CONTAMINANTS IN AIR

A. D. Horton A. S. Meyer

An apparatus utilizing flame ionization detection was developed to measure the total concentration of organic contaminants in the atmosphere of reactor-fuel-burnup test assemblies. Several commercial instruments employing flame ionization detection were tested; the model 109 hydrocarbon analyzer was found to offer the best potential sensitivity. Although the most sensitive range of the Beckman instrument is 3 ppm full scale, the sensitivity was increased about 30-fold by using pure hydrogen as the fuel, by increasing the sample flow to the detector, and by reading the signal of a 1-mv recorder. To exploit the inherent sensitivity, it was necessary to limit the background and noise level by eliminating contamination of the sample and by purifying the burner air.

In the conventional mode of operation recommended by the manufacturer, the sample is drawn into the instrument and is pressurized for injection into the flame detector by a Dynavac pump. Since the rubber diaphragm in the pump contaminated the sample slightly, an alternative (intermittent) mode of sampling was developed. In this mode, the sample is drawn through an 80-cc loop on a linear gas chromatographic sample valve. The contents of the loop are then injected into a stream of purified air that flows through the detector. The passage of this large sample through the detector is recorded as a plateau of height proportional to the hydrocarbon concentration. By the intermittent-sampling technique, hydrocarbon concentrations equivalent to 5 volumes of methane per billion volumes of air can be detected over a noise level corresponding to 2.5 ppb. Thus, moderate and high concentrations can be monitored continuously in the conventional mode, and more sensitive measurements can be made in the intermittent mode.

The entire apparatus, which includes the hydrocarbon analyzer, the recorder, the air purifier (a $\frac{1}{2}$-in.-OD by 12-in.-long bed of platinum catalyst heated to 400°C), and the necessary stainless steel valves and connectors to provide the two sampling modes, was assembled in a movable instrument cabinet for making measurements at various sample sources.

Although the instrument is satisfactory for the initial determination of the hydrocarbon concentration in the fuel-burnup test cells, its use during the course of the tests would be complicated by the presence of dispersed oxides of uranium and smaller quantities of the oxides of structural metals. During controlled burnup tests conducted by the Reactor Safety Group, concentrations of oxide aerosols as high as 0.003 mole per cubic meter of air were found. To test for interference by dispersed oxides, aerosols were generated by aspirating measured quantities of solutions of various metallic salts through the hydrogen flame of the burner of a Beckman flame spectrophotometer. The resulting aerosols were mixed with purified air and "analyzed" by sampling them in the intermittent mode. Significantly large and varying amounts of interferences were observed. The signal generated by the iron oxide aerosol from the aspiration of a solution that contained one mole of ferric nitrate was the smallest of those for all the metal oxides tested and was equivalent to that of 0.05 mole of methane. When a solution of uranyl ammonium carbonate was aspirated, the signal due

[5] R. F. Apple and H. E. Zittel, "Amperometric Titration of Chromium in Mixtures of Fluoride Salts," *Anal. Chem.* **36**, 983 (1964).

to the aerosol was the largest obtained and was equivalent to 2 moles of methane per mole of uranium. The aspiration of anions in the form of the corresponding mineral acids did not produce significant detector responses.

These interference data are not suitable for quantitative interpretation, since the efficiency of aerosol formation and the fraction of metal oxide reaching the flame detector could not be determined easily. However, the results indicate that metal oxides must be removed from the samples if hydrocarbon is to be determined accurately. Techniques applicable to the removal of oxides have not been investigated.

6.5 DETERMINATION OF OXYGEN IN ALKALI METALS BY A MODIFICATION OF THE AMALGAMATION METHOD

Gerald Goldberg

To further evaluate the reliability of the amalgamation method for the determination of oxygen in alkali metals,[6] an experiment was conducted to determine whether a portion of the insoluble oxide is entrained in the amalgam. The first amalgam from a sample of potassium to which ~1000 ppm of oxygen had been added as K_2O was drained under vacuum into a molybdenum capsule that contained a strip of high-purity zirconium metal. This capsule was sealed within a dry box. Samples of triple-distilled mercury and of mercury that had been cleaned with the Bethlehem Oxifier were also sealed in separate molybdenum capsules, which contained similar strips of zirconium metal. After equilibration at an elevated temperature, the zirconium strips were analyzed by vacuum fusion for oxygen pickup. The oxygen contents (ppm) were: amalgam, 34; oxified mercury, 24; triple-distilled mercury, 38.

As has been the case with almost all oxide additions made to potassium for equilibrium studies, the recovery (by amalgamation) of oxygen from the potassium sample described above was low (420 ppm). However, the result of this gettering experiment, in addition to the good precision obtained between replicate samples of alkali metals that contained less than 100 ppm of oxygen, indicates that this method is applicable to the determination of oxygen in sodium, potassium, and NaK. Since increase in oxygen concentration enhances corrosion of the containment material, it is possible that part of the oxygen is converted to the metallic oxide of the containment material. The oxygen recovered by amalgamation is that oxygen which remains dissolved in the alkali metal and which is still available to continue to oxidize the containment material.

The calculation of the oxygen content of the alkali metal is based on either the titration of the solution of the residue after amalgamation or the flame photometric analysis of the solution. For each microgram of hydrogen present as hydride in the residue, the result is in error by 8 μg of oxygen. To eliminate this error, any sodium hydride or potassium hydride present is decomposed by refluxing the first amalgam under vacuum for several minutes (see Sect. 6.7). When the hydride concentration is high, a second refluxing may be necessary for complete removal of the hydrogen. This method was proved to be satisfactory by the addition of water in vacuum to the residue after amalgamation of samples to which hydrogen had been added. Less than 2 ppm of hydrogen was recovered from the water addition after refluxing.

Recently, almost continuous discrepancy has existed between the oxygen contents of circulating NaK loops determined by amalgamation and by a plugging meter. In addition, prematurely high plugging temperatures have been recorded. An experiment was conducted to determine whether some compound other than the alkali-metal oxide was causing the erratic behavior of the plugging meter. A valved, crimped, bypass tube was added to the loop, and the crimped area was maintained at a low temperature to form a plug with a large amount of solid. Several such plugs were obtained. The plugged crimped sections were cut from the tubes, and the samples were analyzed by amalgamation.

The first amalgamation did not give rise to any off-gas (hydrogen) after refluxing. The residues were clean white. The oxygen results obtained for those residues that were dissolved and titrated for total alkalinity compared favorably with the results obtained by flame photometric analysis of the solutions after the titrations. There was no evidence of either corrosion products or carbon in the solutions. Carbon dioxide was not evolved

[6] 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.

from those residues to which dilute acid was added under vacuum, nor was acetylene detected. A small amount of HCN was liberated during the acid addition. These results indicate that a cyanide was formed during the operation of the loop, even though nitrogen is not used as the blanket gas.

The almost pure oxide residues (5 to 6 mg) contained more K_2O than Na_2O. This relationship had been noted also during the analysis of NaK removed from the high-temperature loops by a bucket sampler. Large amounts of K_2O were found, along with larger amounts of Na_2O, in cold traps that were a part of these loops. The residues after amalgamation of samples of NaK from low-temperature systems usually contain 90% or more of Na_2O. At this time it can only be postulated that the erratic behavior of the plugging meter is due to the presence of the mixed oxides of sodium and potassium in the loops, since the solubility curves were based on pure systems.

6.6 DETERMINATION OF CARBON IN ALKALI METALS BY HIGH-TEMPERATURE OXIDATION

Gerald Goldberg

Several modifications were made to the apparatus for the determination of carbon in alkali metals by high-temperature oxidation.[7] They were necessary because the procedure was changed from ignition in oxygen at reduced pressure to ignition in a mixture of about 10% oxygen in helium at a pressure slightly above atmospheric and at a flow rate of ~100 cc/min. It should now be possible to analyze larger samples with less vaporization during oxidation of the alkali metal. Recovery of carbon will not be affected by this reduction in the amount of oxygen present, since the method depends on the reaction of the carbon impurity with the alkali-metal silicate formed when the metal is converted to the oxide within the quartz boat.

Vacuum-tight low-flow meters were constructed, and the oxygen was metered directly from the gas cylinder. The gases are purified before being added to the ignition tube. The trap that was packed with glass beads was replaced with an Anhydrone-filled trap. The flowing gases are passed from the apparatus through a mercury bubbler. The U-tube is evacuated just prior to vaporization of the collected CO_2 for subsequent manometric measurement. The blank is as low as the blank obtained by the reduced pressure method.[7] The apparatus is now similar to those used at other installations involved in the determination of carbon in alkali metals.

[7]G. Goldberg, "Determination of Carbon in Sodium," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1963*, ORNL-3537, p. 53.

6.7 DETERMINATION OF HYDROGEN IN ALKALI METALS BY AMALGAMATION AND VACUUM EXTRACTION

Gerald Goldberg

The procedure for the determination of hydrogen in alkali metals by amalgamation and vacuum extraction[8] was modified to compensate for the portion of hydrogen that is liberated as a result of the heat of reaction of the first amalgamation of the sample. Additional heating and refluxing of the first amalgam liberates the hydrogen that is present as the hydrides, and possibly the hyoxides, of sodium and potassium. The Anhydrone and liquid-nitrogen-cooled moisture traps were eliminated, and the hydrogen is now pumped directly into an evacuated sample bulb. In this manner the method is greatly simplified, and it is still possible to determine both oxygen and hydrogen impurities in the same sample.

The recovery of hydrogen from samples of NaK that contained 20 to 30 cc of hydrogen was better than 95%. Lithium added to one sample was converted to the hydride, which remained stable during the refluxing. Controlled addition of hydrogen at low temperature and subsequent filtration of the lithium hydride may be a means to reduce the lithium impurity that sometimes exists in the alkali metals. A paper describing this modified method was presented.[9]

[8]G. Goldberg, "Determination of Hydrogen in the Alkali Metals," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1963*, ORNL-3537, p. 49.

[9]G. Goldberg, "The Rapid Determination of Low Concentrations of Oxygen and Hydrogen in Alkali Metals by a Modified Amalgamation Technique," presented at the Eighth Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Oct. 6–8, 1964.

6.8 NONAQUEOUS TITRIMETRY OF PHENOLS

J. R. Lund

Members of the Chemical Technology Division are investigating the properties of 4-sec-butyl-2(α-methylbenzyl)phenol, referred to as BAMBP. The phenol, dissolved in such diluents as Amsco 125-82, kerosene, or *n*-octane, is used as an extractant for removing cesium from waste water-processing solutions.

A method was developed whereby the concentration of phenol is determined by neutralization of phenol, which is weakly acidic, with a standard solution of tetrabutylammonium hydroxide in a nonaqueous medium. The course of the neutralization is followed with a glass electrode–S.C.E. pair. The nonaqueous pyridine phase, in which the glass electrode is immersed, is separated from the aqueous phase and S.C.E. by an agar-agar plug. Recently, plugs of ~3% agar-agar in 6% lithium chloride solution were substituted for the agar-agar–saturated potassium chloride plugs as salt bridges. Advantages of the new plugs have not been established.

6.9 TITRIMETRY OF ACIDS IN IRRADIATED LIQUIDS

J. R. Lund Hisashi Kubota

Radiation experiments made in the Chemical Technology Division indicate that certain organic liquids undergo radiolytic decomposition. Free acid and/or carbonyl groups were determined in these irradiated liquids. The free acid was titrated in 10 *M* LiCl with a standard solution of sodium hydroxide. Attempts to determine the carbonyl groups by a similar titration of pyridinium hydrochloride, liberated by the reaction of the carbonyl group with hydroxylamine hydrochloride and pyridine, were unsuccessful.

6.10 DETERMINATION OF SOLIDS IN RADIOACTIVE WASTE-DISPOSAL SLURRIES

J. R. Lund Hisashi Kubota

A method suitable for field use by the Health Physics Division was developed for determining solids in cement slurries. These slurries are used in the hydrofracture experiment being made in conjunction with a study of the disposal of intermediate-level radioactive wastes. The method is empirical; the percent of solids is related to the "as is" weight of residue obtained from centrifugation of a weighed test portion of the slurry.

6.11 DETERMINATION OF CHLORIDE WITH A PYROLYTIC-GRAPHITE INDICATOR ELECTRODE

J. R. Lund F. J. Miller H. E. Zittel

Dilute solutions of sodium chloride ($\sim 10^{-4}$ *M*) resulted from experiments made by members of the Chemistry Division to determine the amount of sodium chloride held within the lattice of certain ion exchange resins. The sensitivity of the amperometric method of determining the end point of the titration of chloride with a standard solution of Ag^+ was improved by substituting the pyrolytic-graphite indicator electrode for the rotating platinum electrode normally used.

6.12 PYROLYTIC-GRAPHITE CUP AS BOTH VESSEL AND INDICATOR ELECTRODE FOR MICRO REDOX AND ACID-BASE POTENTIOMETRIC TITRATIONS

P. F. Thomason

A microtitration cell that does not require an internal electrode was needed for potentiometric titrations of highly radioactive materials. Such a cell was fabricated and tested for the titration of microliter volumes of acid-base and of Fe^{2+}-Ce^{4+} solutions. These titrations carried out both manually and automatically gave a relative standard deviation of 0.3%. The *Microchemical Journal* has in press a paper that describes this work.[10]

[10] P. F. Thomason, "Pyrolytic Graphite Cup as Both Vessel and Electrode for Micro Redox and Acid-Base Potentiometric Titrations," *Microchem. J.* 8(3), in press.

6.13 COULOMETRIC DETERMINATION OF ^{99}Tc

A. L. Travaglini

The concentrations of several ^{99}Tc solutions standardized by a radioactivity counting method were checked by coulometric reduction of the ^{99}Tc in a solution of tripolyphosphate as the supporting electrolyte. The method used in this work is that described by Terry and Zittel.[11] Results obtained with the two methods were within the limits of experimental error.

6.14 COLORIMETRIC DETERMINATION OF SAMARIUM WITH ARSENAZO III

A. L. Travaglini

A series of samples that contained small concentrations of samarium (1 to 5 μg/ml) were analyzed colorimetrically with arsenazo III as the chromogenic reagent. The color developed rapidly in solutions whose pH was adjusted to 2.0 to 2.5 with glacial acetic acid. Sulfate prevented or limited color formation. All solutions were measured at 655 mμ on a Beckman model DU spectrophotometer.

6.15 ANALYSIS OF ARC-FUSED EUROPIUM MOLYBDATE

A. L. Travaglini

A sample presumed to be europium molybdate was analyzed for europium by precipitation with 8-hydroxyquinoline, ignition of the precipitate at 1000°C for several hours, and weighing the residue as Eu_2O_3. Molybdenum was determined by precipitation with benzoin α-oxime, ignition of the precipitate at 525°C, and weighing the residue as MoO_3. The results were 81.5% Eu and 9.8% Mo. The exact composition of the sample was not determined; it appears to be a mixture of Eu_2O_3 and $EuMoO_3$.

6.16 COULOMETRIC DETERMINATION OF NICKEL

A. L. Travaglini

Accurate determinations of the amount of radioactive nickel in a large number of solutions were required. A coulometric method was used to minimize handling. To determine the chemical factor for nickel, a standard solution was made by dissolving a weighed piece of zone-refined nickel in a minimum of concentrated HNO_3 and diluting the solution to volume in a calibrated 50-ml flask. A calibrated 500-μl pipet was used to withdraw each test portion of standard and unknown. The test portions were titrated in a supporting electrolyte of 1 *M* NH_4OH–1 *M* NH_4Cl. The supporting electrolyte was prereduced at –1.20 v vs the S.C.E., the sample was added, and the solution was prereduced at –0.825 v. The final reduction was done at –1.20 v.

Good precision was obtained on duplicate samples.

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[11] A. A. Terry and H. E. Zittel, "Determination of Technetium by Controlled-Potential Coulometric Titration in Buffered Sodium Tripolyphosphate Medium," *Anal. Chem.* **35**, 614 (1963).

7. Analytical Biochemistry

J. C. White

C. A. Horton I. B. Rubin

A program was initiated in July 1964 with the ultimate objective to establish a biochemistry analytical group in the Analytical Chemistry Division. This effort is being jointly sponsored by the Biology and Chemical Technology Divisions. Two analytical chemists were assigned initially to assist these research divisions in their program to recover and separate the transfer ribonucleic acids (t-RNA's) from *Escherichia coli* bacteria (*E. coli*). The t-RNA's are intermediates in the synthesis of proteins from α-amino acids. The primary objective of this program is to develop methods for obtaining a purified mixture of t-RNA's from *E. coli* and for subsequently fractionating this mixture into the individual t-RNA species. Reverse-phase chromatography is used to fractionate the mixture of t-RNA's into its components. The organic extractant is a quaternary ammonium compound in an organic diluent (0.1 *M* dimethyldilaurylammonium chloride in amyl acetate) supported on specially treated diatomaceous earth. A sodium chloride solution of increasing concentration is used to elute different t-RNA's. These fractions from the column are assayed by spectral absorbance measurements at 260 mμ and by reacting the fraction with ^{14}C-labeled amino acids. Analytical Chemistry Division personnel are doing these assays. An immense number of assays is required to elucidate the efficiency of the column separation. Initial efforts are thus concerned with modifying the assay procedures to reduce the overall time required for analysis by improving individual steps in the procedure and ultimately to automate the assay insofar as is possible.

Since many pipet operations on small volumes of reagents are required, alternative methods of adding reagents and performing the time reaction are being investigated.[1] Laing-Levy glass pipets are normally used to take the final aliquot of the mixture to a paper disk, which is washed, dried, and counted for ^{14}C in scintillation fluid. Among a number of others being considered is the Cooke-Takatsy[2,3] loop, which is a coil of fairly closely wound stainless steel wire with small loops at the sides of the main coil. These loops are sturdy, fairly inexpensive, and easily cleaned; they have adequate precision and accuracy if used properly and permit aliquots to be taken and delivered rapidly. Tests are under way to determine their adequacy as a substitute for the glass pipets in the normal procedure.

The possibility is being considered of depositing final aliquots along a paper strip, which could be washed automatically, dried, and counted by a ^{14}C counter, such as a chromatogram radioactivity scanner. Preliminary test results are encouraging. The possibility of conducting the reaction directly on a paper disk or strip is also under study. Whatman No. 1 and No. 17 and Millipore HAWP papers will not be satisfactory, whereas Whatman 3MM may be. Elevated room temperature ($\sim$37°C) and high humidity accelerate the reaction. More studies are needed to determine whether reproducible and complete reaction can be achieved.

Various automatic diluters and pipetters are also being studied to locate one that will increase the number of determinations per day.

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[1] 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).

[2] J. L. Sever, "Application of a Microtechnique to Viral Serological Investigations," *J. Immunol.* **88**, 320 (1962).

[3] G. Takatsy, "A New Method for Preparation of Serial Dilutions in a Quick and Accurate Way," *Acta Microbiol. Acad. Sci. Hung.* **3**, 191 (1955).

8. X-Ray and Spectrochemical Analyses

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

M. T. Kelley

Cyrus Feldman

8.1.*a* X-Ray Analysis

H. W. Dunn

X-Ray Absorption-Edge Analysis of Solids. – Preliminary work was done toward determining the absolute quantity of an element present in a powdered sample by rotating the sample in an x-ray beam and measuring the intensity of the transmitted x ray on each side of the x-ray absorption edge of the element determined. Tests were conducted with mixtures of ZrO_2 and MnO_2 by use of the K_α edge of Zr. Table 8.1 gives the results of these tests.

It is believed that the random nature of the errors is due to imperfect homogenization of the sample. If the homogeneity can be improved, this sample form should be as satisfactory as the liquid form.

Table 8.1. Accuracy of Determination of Zirconium in Powdered Samples by the X-Ray Absorption-Edge Method

Zr (mg)		Error
Present	Found	(%)
0.10	0.072	–28
0.50	0.600	+20
1.0	0.978	–2.2
2.0	2.026	+1.3
5.0	5.24	+4.8
25.0	24.7	–1.4

X-Ray Absorption-Edge Analysis of Radioactive Solutions. – Disposable one-piece polystyrene cells were fabricated for use in the analysis of radioactive solutions by the x-ray absorption-edge method (Fig. 8.1). Both 1.0- and 0.5-cm-thick cells were made.

The cell is filled in a shielded area by remote-handling equipment; the opening is then sealed with tape. The cell is placed in a shielded container and is transferred to the x-ray laboratory for analysis.

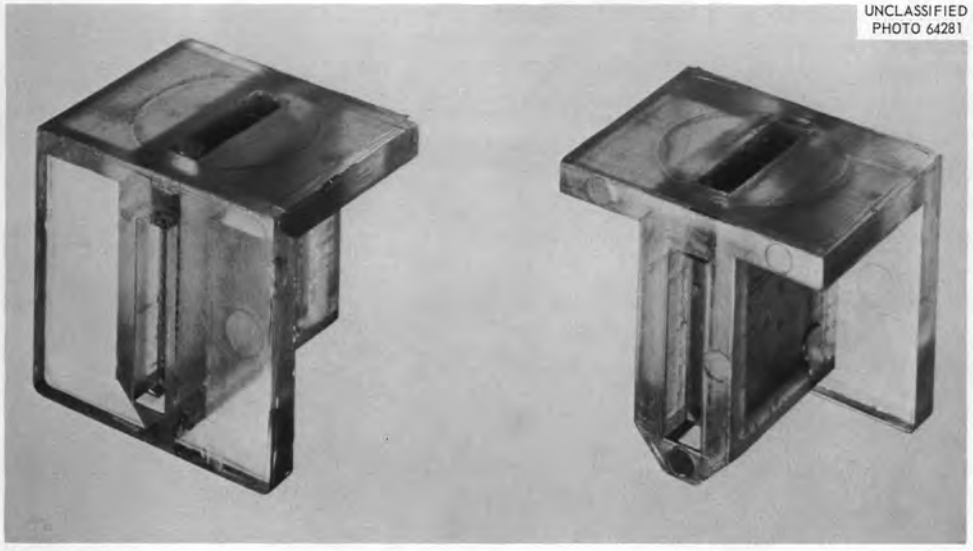

Fig. 8.1. Disposable Polystyrene Cell for Holding Radioactive Solutions. Length in direction of x-ray beam, 1 cm; volume, 0.4 cm^3; tape seal not shown.

Determination of X-Ray Absorption-Edge Height by Extrapolation. – Heretofore, the height of the x-ray absorption edge has been calculated from intensity measurements made at one point on each side of the edge. In order to minimize the effects of possible shifts in instrumental parameters, it is now proposed to obtain this height more directly by making several measurements of intensity on each side of the edge and then extrapolating to the theoretical wavelength of the edge. An IBM 1604 computer program was written to test the accuracy of various assumed algebraic forms of the x-ray intensity (I) vs wavelength (λ) curve. Thus far, the most successful form appears to be $\log I = \lambda^3$, I being corrected for scattering and resolving-time losses.

Analysis of Solids by X-Ray Fluorescence. – With the help of R. B. Bullock,[1] an IBM 1604 computer program is being developed to use a modified form of Burnham, Hower, and Jones's procedure[2] for analyzing multicomponent alloys by x-ray fluorescence.

8.1.*b* Atomic Absorption

Cyrus Feldman

Determination of Ruthenium. – The atomic absorption of solutions of ruthenium in dilute HNO_3 was investigated by use of the 50-cm end-fed absorption tube, hollow cathode, and detection equipment described previously.[3,4] Study of all variable parameters showed that the best results are obtained under the following operating conditions: spectral line Ru 3498.9 A (first order); 50-μ slit; Beckman medium-bore H_2-O_2 burner, fed with 3 lb of H_2 and 20 lb of air; center section of absorption tube surrounded by a 12-in. electric tube furnace operated at 900°C; and hollow cathode charged with compacted Ru metal powder. Under these conditions, the minimum detectable concentration of Ru is ~0.5 μg/ml. The slope of the analytical curve ranges from ~0.09 optical density unit per μg/ml at 1.5 μg/ml to ~0.07 at 4.5 μg/ml.

Determination of Selenium. – The chemical state of Se in a flame – and thus the flame absorption coefficient for Se resonance radiation – depends strongly on the oxidizing power of the flame (see previously observed behavior of Sn[3]). Experiments in which the absorption tube was used without the furnace showed that Se 1960.3 A (the resonance line of Se) is not absorbed when a 100-μg/ml solution of Se is aspirated into a H_2-O_2 flame. However, in a H_2-air flame, absorption of the line can be detected at a Se concentration of 1 μg/ml. A commercial selenium hollow cathode was used in this work.

8.1.*c* Flame Photometry

T. C. Rains

Cyrus Feldman

Isolation and Flame Photometric Determination of Barium. – In the flame photometric determination of barium at the 0- to 50-μg/g level in salts of alkali metals or alkaline-earth elements, the barium must be isolated from the major component to increase the concentration of the barium and to remove spectral interferences. All the calcium must be removed, since even traces of it (~1 μg/ml) give intense CaO bands, which interfere with the most sensitive Ba line (553.5 μ).

Korkisch and Tera[5] indicate that the nitrate form of Dowex 1 retains barium more strongly than it retains alkali metals or other alkaline-earth elements if the solvent is methanol (90 vol %)–5 N HNO_3 (10 vol %). Tests showed that traces of barium can be so retained but that the low solubility of the samples (e.g., KNO_3) in this medium limits the usefulness of the procedure.

The coprecipitation of barium with $PbCrO_4$ was studied briefly by Reynolds,[6] although individual results and conditions were not given in detail. This procedure appeared interesting and was

[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 Analysis," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1963*, ORNL-3537, pp. 60–62.

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

[5] J. Korkisch and F. Tera, "Separation of Thorium by Anion Exchange," *Anal. Chem.* **33**, 1264 (1961).

[6] S. A. Reynolds, "Determination of Alkaline-Earth Eleme ts," *Anal. Chem. Div. Ann. Progr. Rept. Dec. 31, 1962*, ORNL-3397, pp. 75–78.

investigated further. As much as 300 μg of Ba was coprecipitated quantitatively (~99%) with 10 mg of Pb as $PbCrO_4$ at pH $\geqq$ 6.2; contamination from alkali metals or other alkaline-earth elements was negligible (see Table 8.2). Because of the serious spectral interference of calcium, it probably would be necessary to repeat the coprecipitation if much calcium were present. Under the same conditions, 1 mg of Fe^{3+} or 10 mg of PO_4^{3-} does not affect recovery of the barium, but 1.5 mg of Al^{3+} significantly reduces the recovery.

To circumvent the effect of aluminum, barium was first coprecipitated with calcium oxalate. According to Peters,[7] precipitation of barium as the oxalate is quantitative from 33 vol % ethyl alcohol solutions. The tracer ^{133}Ba was used to test the coprecipitation as oxalate of 10 μg of Ba with 10 mg of Ca in 30 vol % methanol. The results (Table 8.3) indicate that recovery is essentially complete in this medium at pH 3. The oxalate so obtained is dissolved in a mixture of nitric and perchloric acids, and the solution is subjected to the $PbCrO_4$ procedure (see above).

The Ba-bearing $PbCrO_4$ is dissolved in a mixture of nitric acid and hydrogen peroxide, and the solution is diluted to volume with an acetone-water-glycerol solution. The final diluted solution is analyzed for barium with a Jarrell-Ash flame photometer; the 553.6-mμ Ba line is used.

[7]C. A. Peters, "The Estimation of Calcium, Strontium, and Barium as the Oxalates," *Am. J. Sci.* 12 (4), 216 (1901).

Table 8.2. Contamination of $PbCrO_4$ Precipitate by Various Elements Under Conditions That Give Complete Coprecipitation of Barium

Major Element	Amount Present (mg)	Amount Found in $PbCrO_4$ Precipitate (μg)
Li	80	<3
Na	200	<3
K	320	50
Mg	60	<8
Ca	180	20
Sr	165	520

Table 8.3. Effect of pH on the Coprecipitation of Barium with Calcium Oxalate in the Presence of 30 vol % Methanol

pH	Recovery of Ba (%)
0.5	77
1	98.4
2	99.2
3	99.5
3[a]	90.0

[a]No methanol used.

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

A. E. Cameron

8.2.*a* Disposable Spectrographic-Excitation Chamber for Radioactive Samples

J. A. Carter

A disposable spectrographic-excitation chamber was designed, tested, and used for determining impurities in ^{147}Pm and ^{151}Sm. Figure 8.2 shows its general design. The body of the chamber is a 1½-in.-ID glass cross. Plexiglas tubes, machined as shown, are slipped onto each of the four glass cross ports to serve as molds for mounting the brass electrode guides and the glass or quartz lenses with epoxy. Excitation chambers used for very long exposures or with high currents must have water-cooled electrode guides, as shown in Fig. 8.2C. Water cooling is not required, however, for exposure durations of 3 min at 5 amp. Air-intake and exhaust connections are provided by sealing ¼-in. copper tubing adjacent to each electrode guide. The chamber is lightweight and compact and is easily fitted to an electrode stand so that the analytical gap may be adjusted during an exposure.

Samples of ^{147}Pm and ^{151}Sm were analyzed successfully for 50 trace elements by use of the disposable excitation chamber and M-11 gas-mask filters for containment. Excitation chambers of this general design may be used for studies in controlled atmospheres.

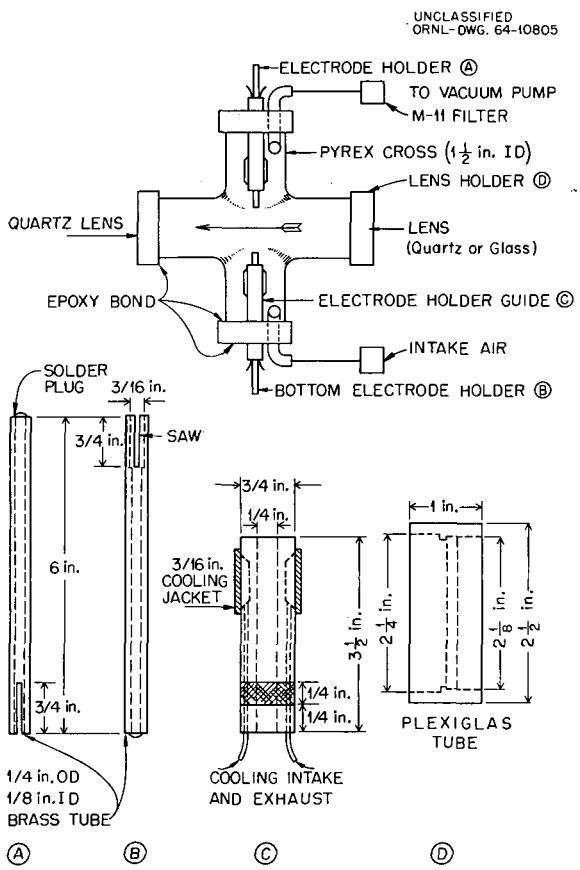

Fig. 8.2. Disposable Spectrographic Excitation Chamber for Radioactive Samples.

8.2.*b* Determination of Certain Impurities in Lithium Fluoride Powder by Spark Excitation

J. A. Carter

A rapid technique was developed that utilizes the Paschen Direct Reader for determining Ca, Mg, Na, Mn, Ni, Ce, Gd, and Lu in lithium fluoride powder. The lithium fluoride is cemented to a ⅛-in.-thick by ½-in.-diam graphite disk with acrylic spray. This disk is mounted on a conventional rotating disk holder, axis horizontal, for excitation by means of a high-voltage ac spark. Calibration standards were prepared by mixing the appropriate weights of the elements to be determined with high-purity lithium fluoride. For the internal standard, the Li 3232.61 A line is used. Table 8.4 gives the useful concentration range for each element and the analytical lines employed. This technique was used in over 1000 analyses for the Reactor Chemistry Division.

Table 8.4. Element Concentration Range in Lithium Fluoride by a Rotating Disk–AC Spark Technique

Element	Wavelength (A)	Useful Range (μg per g of LiF)
Na	5889.95	10–2000
Mg	2852.13	10–2000
Ca	4226.73	10–2000
Mn	2593.73	10–2000
Ni	3101.55	50–2000
Ce	4186.60	100–2000
Gd	3646.20	50–2000
Lu	2615.42	10–2000
Li[a]	3232.61	

[a]Internal standard.

9. Mass Spectrometry

A. E. Cameron

9.1 TRU MASS SPECTROMETRY

A. E. Cameron

R. E. Eby

The new double-magnetic-deflection mass spectrometer was put in service in Building 9735 on August 1, 1964. The vacuum system is entirely of metal, and pumping is done with VacIon pumps. The magnets and the vacuum-system assembly are in a laboratory arranged for containment of alpha radioactivity (see Fig. 9.1). As with the other two double-stage instruments in use in the Referee Analysis Laboratory, ion detection is by secondary electron multiplier, and pulse counting is used instead of more conventional current measurement. Spectra are stored in a 400-channel analyzer, from which they are read out on magnetic, printed, or punched tape. The sample size required is of the order of 1 to 2 ng (0.5 μc), depending on the radioactivity of the material. In addition to the advantages of small sample size, the equipment also permits the measurement of rare isotopes down to a concentration as low as 1 ppm. All samples of highly radioactive alpha emitters are now being analyzed on this instrument.

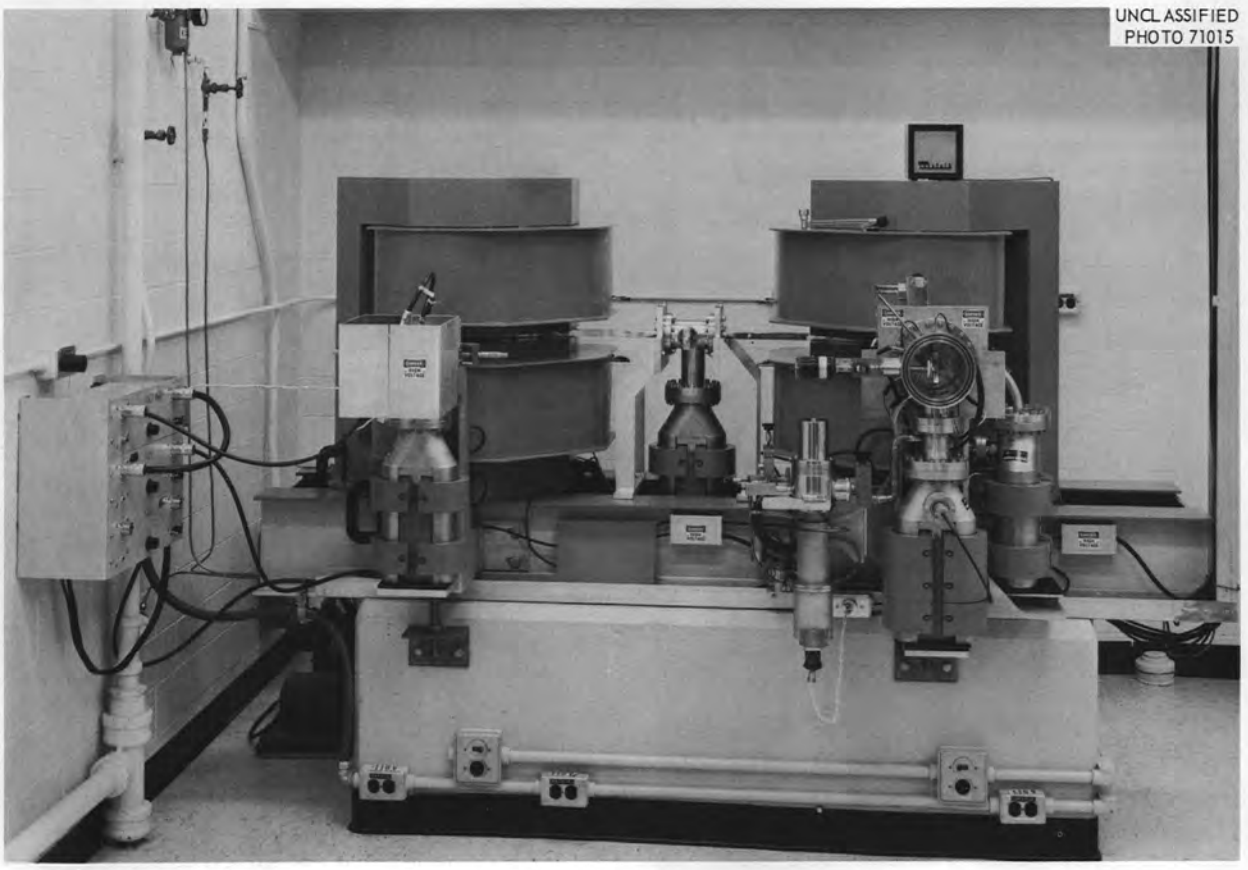

Fig. 9.1. TRU Mass Spectrometer in Alpha-Containment Laboratory.

9.2 ISOTOPIC COMPOSITION OF ATMOSPHERIC NEON

A. E. Cameron

J. R. Walton

The published results of several mass spectrometric determinations of the isotopic composition of atmospheric neon are not in good agreement. The ready availability of ^{20}Ne and ^{22}Ne of high isotopic and chemical purity and in adequate quantity makes "calibrated" measurement of the isotopic composition possible by correcting for bias in the mass spectrometer. Several standards were carefully prepared from the separated isotopes, and the bias was determined. The results for six samples of neon obtained from commercial suppliers agreed with each other. The agreement indicates either that no fractionation occurred in the preparation of the samples or that the same degree of fractionation occurred in different plants. Results obtained thus far indicate that the isotopic composition (atom percent) of atmospheric neon is: ^{20}Ne, 90.514 ± 0.031; ^{21}Ne, 0.266 ± 0.005; ^{22}Ne, 9.220 ± 0.029. The calculated atomic weight is 20.1794 ± 0.0018; this value can be compared with the value 20.183, which is based on gas density measurements.

10. Infrared Spectroscopy

Lucy E. Scroggie

Infrared spectroscopy was used to determine the effect of gamma radiation on such compounds as butyl-α-methylbenzylphenol, 2-thenoyltrifluoroacetone, dimethylglyoxime, sodium dimethylglyoxime, *o*-benzylphenol, di-*sec*-butylphenylphosphonate, and ion-exchange resins. Fractions of the products from the nitric acid degradation of diethylbenzene contained alkyl and aryl nitro, ketone carbonyl, and hydroxyl groups, as indicated by infrared absorption spectra.

The hydrolysis of uranium carbides and thorium carbides with water or nitric acid yields mixtures of two types: water-soluble acidic solids and ether-soluble waxes. The infrared spectra of the waxes revealed the alkyl and alkene units of hydrocarbon polymers. The yellow-to-brown solids that were soluble in water and methanol were mixtures of polybasic organic acids, which are difficult to separate by any means. Oxalic acid was identified and removed by vacuum sublimation. The residue apparently consisted of mellitic acid (benzenehexacarboxylic acid) and minor amounts of benzenepentacarboxylic acid; some samples contained hydroxyl groups and nitrate ions in small quantities. Repeated attempts to separate these acids by paper chromatography with several solvents were unsuccessful.

Table 10.1 lists the various types of samples investigated by infrared spectroscopy. Most studies involved the detection and/or identification of impurities in commercial solvents, process streams, or compounds (inorganic and organic) prepared at ORNL; the identification of commercial products; or the characterization of organic compounds. In addition, semiquantitative or quantitative determinations were made of ethylene glycol–water in engine oil, four isomers of phenylbutanol in reaction mixtures, Cellulube in hexane extracts of air samples, and phenol in di-*sec*-butylphenylphosphonate.

Table 10.1. Types of Samples Examined by Infrared Spectroscopy

Organic	
2-Thenoyltrifluoroacetone–xylene	N,N′,N″-Trimethylborazine
Butyl-α-methylbenzylphenol–Amsco	Long-chain tertiary amine
Dimethylglyoxime	Epoxy resin
Sodium dimethylglyoxime	Di-2-ethylhexylphosphoric acid
Diethylbenzenes	Diphenyl ether
Soybean oil–2-ethylhexanol	Alkyl phthalate
o-Benzylphenol–diisopropylbenzene	Tributyl phosphate
Engine oils	Extracts of LiCl solutions
Irradiated ion-exchange-resin effluents	Degraded pump oil
3,9-Diethyltridecanol-6	Di-sec-butylphenylphosphonate
3,9-Diethyltridecanone-6 oxime	Aqueous extract of tributyl phosphate
3,9-Diethyltridecanamine-6	Iodinated-hydrocarbon vapors
Commercial corrosion inhibitor	Methyl isocyanide
Commercial solvents	Trioctylarsine oxide
Commercial odorants	Uranium carbide dissolution products
Phenylbutanol isomers	Thorium carbide dissolution products
Commercial lubricant vapors	Oil smears
Commercial rust remover	
Inorganic	
KCl (single crystals and ingots)	SiF_4-SiF_3CH_3-$SiF_2(CH_3)_2$ mixture
NaCl (ingots)	$Mg(BrO_3)_2$
$KF \cdot UF_5$	KIO_4-KIO_3 mixtures
$LiF \cdot UF_5$	$KClO_4$-$KClO_3$ mixtures
$RbF \cdot UF_5$	GeO_2 glass
LiF (single crystals)	

11. Optical and Electron Microscopy

M. T. Kelley

T. E. Willmarth

T. G. Harmon H. W. Wright

11.1 ELECTRON MICROSCOPY OF RADIOACTIVE MATERIALS

11.1.a Electron Microscope Facility for Studying Radioactive Materials

A contract was let to the H. K. Ferguson Company for modification of room 201, Building 3019, to house the Philips EM 100B electron microscope. The installation of this microscope and its associated equipment will be the first step toward establishing the much-discussed facility for electron microscopy of radioactive materials of interest in nuclear engineering. Preliminary preparation of radioactive samples will be done in hot cells and will be coordinated with other

uses of the cells by the Analytical Chemistry Division. Final preparation of samples for electron microscopy will be done in the analytical laboratories in Building 3019.

Certain powder samples of 100-r/g radioactivity were prepared and examined successfully,[1] and replica techniques for some radioactive surfaces were developed.[2] However, each radioactive sample received for the first time presents specific problems and requires special testing and the development of certain procedures before it can be examined properly in the electron microscope. The facility will likely be in limited operation by January 1965 and in full operation later that year.

[1]M. T. Kelley *et al.*, "Optical and Electron Microscopy," *Anal. Chem. Div. Ann. Progr. Rept. Dec. 31, 1961*, ORNL-3243, pp. 51–54.

[2]Staff of the Metallurgy Division, *Technical Function, Operation of the High Radiation Level Examination Laboratory, Bldg. 3525*, ORNL-CF-61-1-75, pp. 49–50 (Jan. 31, 1961).

11.2 NUCLEAR SAFETY PROGRAM

Electron microscopy was done for those research groups studying simulated fallout of radioactive particles from possible accidents at experimental or industrial reactors. Studies were made to determine the best procedures for collecting and sampling radioactive particulates. The particulates used in the study were produced on a laboratory scale and in large-scale mockup tests

Fig. 11.1. Representative Particles Collected on Millipore Filter from Simulated Rapid Fuel Burnup. Magnification, 91,500×.

simulating such accidents. Materials collected on a variety of filters, in electrostatic and thermal precipitators, and in impactors were examined in the electron microscope to determine the size and shape of individual particulates and the nature of agglomeration, if it occurred. Figure 11.1 shows the chainlike agglomerates that form under certain conditions. Selected-area electron diffraction was used to identify the chief constituents of such structures. A technique for electron autoradiography of such radioactive particles and agglomerates is being developed; the technique is similar to that described by Caro, Van Tubergen, and Kolb.[3]

[3]L. A. Caro, R. P. Van Tubergen, and J. Kolb, "High Resolution Autoradiography I. Methods," *J. Cell Biol.* 15(2), 173 (1962).

11.3 ELECTRON OPTICAL INSTRUMENTATION

The Philips EM 200 electron microscope discussed earlier[4] is in operation; resolution better than 6 A has been achieved with it.

11.4 ELECTRON AND OPTICAL MICROSCOPY AND ELECTRON DIFFRACTION RESEARCH ASSISTANCE TO OTHER LABORATORY DIVISIONS

In their research problems requiring optical and electron microscopy and electron diffraction, assistance was given to the Chemistry, Chemical

[4]M. T. Kelley *et al.*, "Optical and Electron Microscopy," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1963*, ORNL-3537, pp. 68–70.

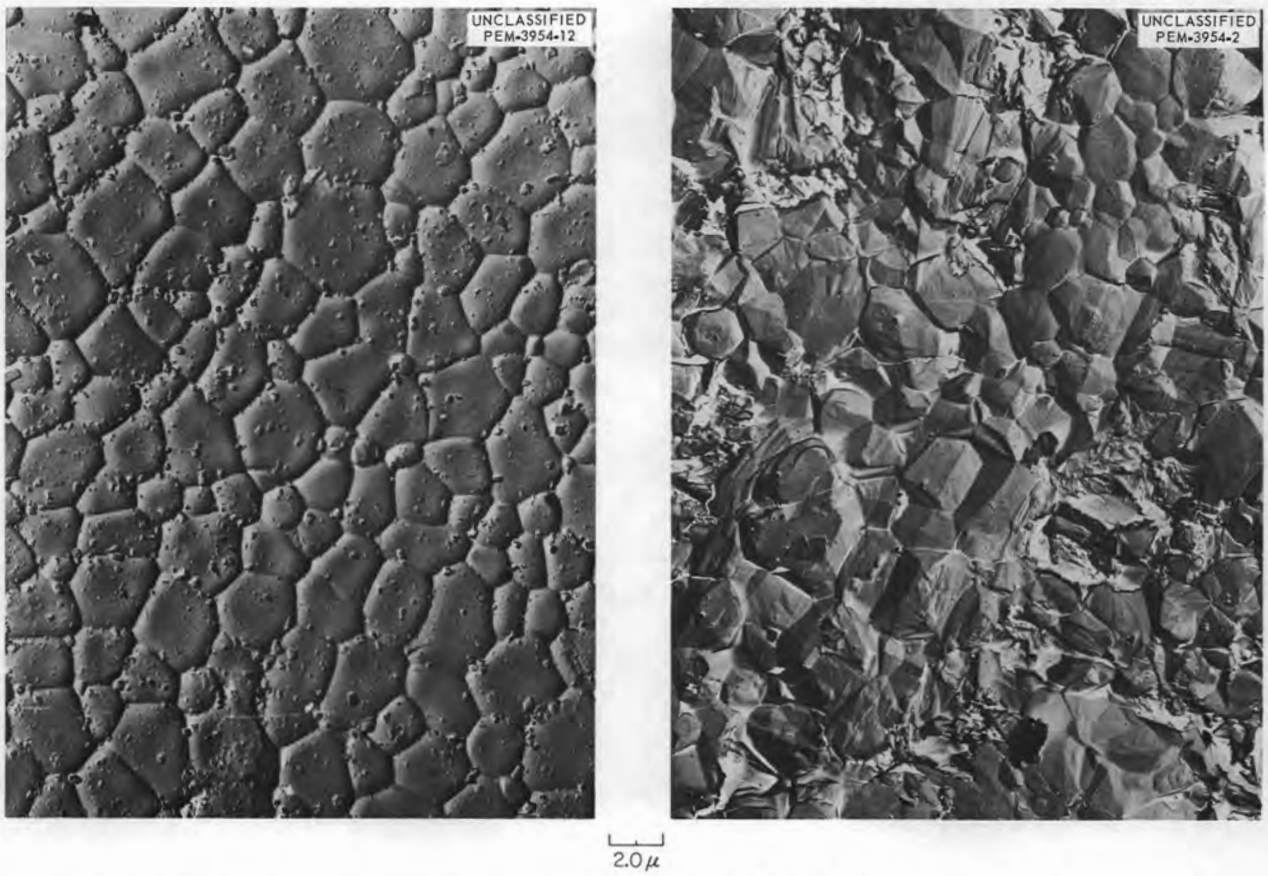

Fig. 11.2. Surface (left) and Internal Structure (right) of Thoria Sol-Gel Beads Formed in the Presence of a Cationic Surfactant. Carbon replica, palladium shadowcast.

Technology, Instrumentation and Controls, Isotopes, Operations, Physics, Reactor Chemistry, Health Physics, and Metals and Ceramics Divisions. Of particular interest was the electron microscopy done relative to the development of thoria-urania sol-gel beads.[5] Figure 11.2 shows typical results of the studies of the structures of such materials. Other materials of special interest in nuclear science were also studied. These materials included: films formed on cadmium, zinc, indium, silicon, stainless steel (302 and 304), and Y-Al alloys; sintered particles of Al_2O_3 and Fe_3O_4; particles of Ce-Al alloy, hydrocarbons, alpha- and gamma-irradiated solutions of $AgNO_3$ (ref. 6), and pine wax produced in an electric field; molybdenum that contained zirconium oxide and thorium oxide particles; niobium foils that had been exposed to liquid potassium; ammonium tungstate; pyrolytic graphite; sodium fluoride; cerium oxide; diatomaceous earth; and thoria sols.

The Health Physics and Chemistry Divisions were assisted in the preparation of aluminum semireflectors for use in research spectrophotometers. The Physics Division was aided in preparing and fabricating special (7-cm^2) semiconductor (germanium and silicon) radiation detectors for use in experiments on neutron and charged-particle fission and on fission-oriented nuclei.[7]

11.5 ULTRASTRUCTURE STUDIES OF EXCHANGE RESINS

D. H. Freeman,[8] with T. E. Willmarth's assistance, studied the ultrastructure of certain exchange resins by means of the high resolution of the Philips EM 200 electron microscope.

[5]T. E. Willmarth, "A Microscopic Study of Thoria Sol-Gel Beads," presented at Micro 64 (International Microscopical Society), Chicago, Ill., July 1964 (to be published).

[6]H. A. Mahlman and T. E. Willmarth, "Radiolytic and Photolytic Reduction of Aqueous Silver Nitrate Solutions," *Nature* **202**, 590 (1964).

[7]J. W. T. Dabbs, F. J. Walter, and G. W. Parker, "Modification of Cryostat and Associated Equipment," *Phys. Div. Ann. Progr. Rept. Jan. 31, 1964*, ORNL-3582, p. 123.

[8]Summer employee, Washington State University, Pullman.

12. Nuclear and Radiochemical Analyses

J. C. White

W. S. Lyon

12.1 NUCLEAR PROPERTIES OF RADIONUCLIDES

12.1.*a* Gamma-Ray Branching in the Decay of ^{141}Ce

J. S. Eldridge

An early value for the branching ratio for the 0.145-Mev gamma transition in the decay of ^{141}Ce was 0.43 gamma/dis.[1] However, the adopted value for the branching of the 0.435-Mev negatron group (70%)[2] and the total conversion coefficient for the 0.145-Mev gamma ray (α_T = 0.513) indicate that the branching ratio should be 0.48 gamma/dis.

Isotopically pure ^{141}Ce was obtained, as described in Sect. 12.2.*a*, by double neutron capture on ^{139}La followed by negatron decay of the 3.5-hr ^{141}La product. Absolute disintegration rates were determined for several sources with a 4π beta-gamma coincidence counter. Gamma-ray

[1]W. S. Lyon, "Isotopic Analysis: Cerium-141 and Cerium-144," *Nucl. Sci. Eng.* **4**, 709 (1958).

[2]"Ce^{141}. Ground-State Decay," *Nuclear Data Sheets*, vol. 5, Natl. Acad. Sci.–Natl. Res. Council, Washington, D.C., 1962.

intensity was determined with a 3 × 3 in. NaI(Tl) detector. The branching ratio observed for the 0.145-Mev gamma-ray transition in the decay of ^{141}Ce was 0.493 ± 0.006 gamma/dis. This value is in much better agreement with that predicted from negatron branchings and total conversion coefficients (0.48 gamma/dis) than with the value reported previously (0.43 gamma/dis).

12.1.*b* Gamma Transitions in the Decay of ^{79}Kr

J. S. Eldridge

Several irradiations of enriched ^{78}Kr were carried out in the ORR to produce ^{79}Kr for use in studies of the nuclear properties of ^{79}Kr and of its production techniques. Nuclear data obtained previously with a conversion-electron spectrograph indicate that the energy of the highest-energy gamma transition in the decay is 0.833 Mev.[2]

Gamma-ray spectra collected over a period of several days showed the existence of 1.11- and 1.33-Mev gamma rays in addition to at least one other weak transition; these gamma rays decayed with a 35-hr half-life. Decay measurements taken over a period of 13 days in a high-pressure ionization chamber yielded a preliminary value of 35 hr for the half-life of ^{79}Kr. Figures 12.1 and 12.2 give the high-energy and low-energy portions, respectively, of the gamma-ray spectrum for this nuclide.

Future irradiations are scheduled to obtain decay-scheme parameters necessary for the assay of ^{79}Kr, which has potential applications in engineering and biological experiments. This work was done jointly with R. S. Pressley.[3]

[3]Isotopes Division.

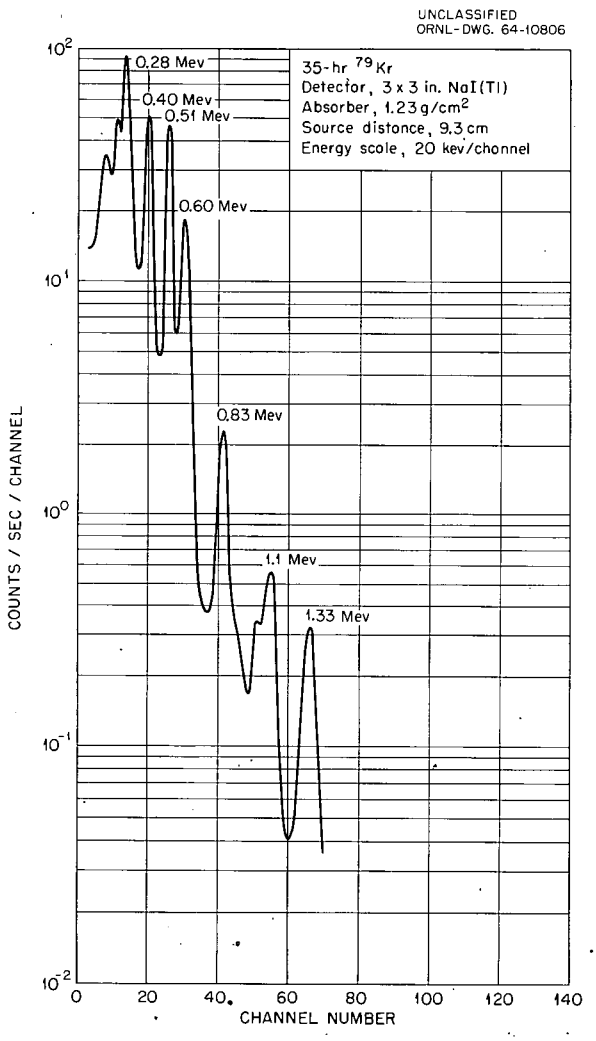

Fig. 12.1. High-Energy Gamma-Ray Spectrum of ^{79}Kr.

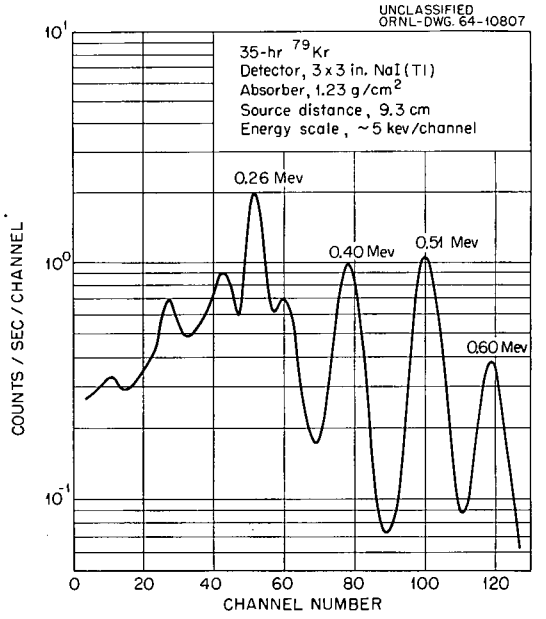

Fig. 12.2. Low-Energy Gamma-Ray Spectrum of ^{79}Kr.

12.1.c Decay of ^{99}Mo-^{99m}Tc

Peter Crowther[4] J. S. Eldridge

The decay parameters of ^{99}Mo-^{99m}Tc were determined by liquid-liquid extractions, 4π beta-gamma and gamma-gamma coincidence counting, and absolute gamma-spectrometry measurements. Gamma-ray energies and intensities were obtained with a 3 × 3 in. NaI(Tl) spectrometer. The relative intensities of the 0.74- and 0.78-Mev gamma rays were measured with a lithium-ion-drifted germanium diode detector; these gamma rays were unresolved in the NaI(Tl) spectra. The observed numbers of gamma photons per ^{99}Mo disintegration with energies of 0.142, 0.181, 0.372, 0.740, 0.780, and 0.93 Mev were 0.076, 0.068, 0.013, 0.124, 0.044, and 0.004 respectively. Gamma-gamma coincidence measurements confirmed the existence of the 0.93-Mev gamma ray in the ^{99}Mo decay. A quantitative separation of ^{99m}Tc, followed by determinations of x- and gamma-ray emission rates, gave the sum of the internal conversions of the 0.140- and 0.142-Mev gamma rays as α_T = 0.099 ± 0.015. At equilibrium, the radioactivity of the ^{99m}Tc daughter was determined to be 0.964 that of the ^{99}Mo radioactivity. A paper on this work was submitted to *Nuclear Physics* for publication.[5]

12.1.d New Isomer of Bromine: ^{82m}Br

J. F. Emery

The decays of ^{82m}Br were studied by gamma-ray scintillation spectroscopy and beta proportional counting. This new isomer decays with a half-life of 6.20 ± 0.05 min and excites a highly converted gamma-ray transition of 46 ± 2 kev in ^{82}Br; α_K is 268. The total combined beta branching is 2.4%.

The thermal-neutron activation cross section and resonance integral for ^{82m}Br are 3.0 and 34 barns, respectively, and the corresponding values for the total production of ^{82}Br are 3.23 ± 0.2 and 41.3 ± 1 barns. This work was discussed,[6] and a paper was accepted for publication in the *Journal of Inorganic and Nuclear Chemistry*.

12.1.e Studies of Rare-Earth-Element Alpha Emitters

T. H. Handley

A study of rare-earth-element alpha emitters and a search for new alpha emitters, especially of low branching ratio, were made. The radionuclides were produced in either the ORIC or the 86-in. cyclotron by bombarding selected enriched stable isotopes with 75-Mev alpha particles or 20-Mev protons. The radionuclides were separated by ion exchange on Dowex 50 and were eluted with α-hydroxyisobutyric acid. A new alpha emitter with a decay energy of 2.63 ± 0.03 Mev was discovered and was assigned to the nuclide ^{148}Eu. The measured ratio of alpha to electron capture branching was $(9.4 \pm 2.8) \times 10^{-9}$, which corresponds to a partial alpha half-life of $(1.6 \pm 0.5) \times 10^7$ years. By use of a Coulomb barrier penetration formula and the measured alpha decay energy, a half-life of $(6.2\ ^{+6.7}_{-3.3}) \times 10^6$ years was calculated. The agreement, within experimental error, between the measured and calculated partial alpha half-lives indicates that the ground-state spin and parity assignments are probably the same for ^{148}Eu as for its daughter ^{144}Pm. A paper on this subject is scheduled for publication in the December issue of *The Physical Review*.[7]

This work was done jointly with K. S. Toth, I. R. Williams, and E. Newman.[8]

12.1.f Properties of Nuclear Levels in a Number of Even-A Nuclei ($184 \leq A \leq 192$)

Benjamin Harmatz[9] T. H. Handley

A large accumulation of internal-conversion-electron data has made possible the study of the properties of high-energy nuclear levels in the transition region of mass numbers 184 to 192. Reasonably detailed schemes that show the nuclear levels were constructed from these measurements and led to the following even-even

[4] P. Crowther, temporary alien employee, South African Atomic Energy Board, Pelindaba, Pretoria, South Africa.

[5] P. Crowther and J. S. Eldridge, "Decay of ^{99}Mo-^{99m}Tc," submitted for publication in *Nuclear Physics*.

[6] J. F. Emery, "Br^{82m}: A New Bromine Isomer," presented at the 148th National American Chemical Society Meeting, Chicago, Ill., Aug. 30–Sept. 4, 1964.

[7] K. S. Toth *et al.*, "New Rare Earth Alpha Emitter, ^{148}Eu," *Phys. Rev.*, in press (1964).

[8] Each from the Electronuclear Division.

[9] Electronuclear Division.

nuclei: $^{184m}Re \rightarrow {}^{184}W$, $^{186}Ir \rightarrow {}^{186}Os$, $^{188}Ir \rightarrow {}^{188}Os$, $^{190}Ir \rightarrow {}^{190}Os$, and $^{192}Ir \rightarrow {}^{192}Os$. Parameters relevant to rotational configurations and branching ratios of the de-exciting transitions were evaluated. The low-energy spectra, with their associated vibrational and rotational modes of excitation, were confirmed and extended. Triple isomerism was observed in ^{190}Ir including isomeric transitions of $M3$ (26 kev) and $M4$ (148 kev) character. The metastable state of ^{184}Re is partially de-excited by an 83-kev isomeric transition of $M4$ multipolarity. A paper on this subject was published.[10]

12.1.*g* Studies of Neutron-Deficient Hafnium Radionuclides

T. H. Handley

The decays of neutron-deficient hafnium radionuclides ($168 \leqq A \leqq 173$) produced in ORIC by (α, xn) reactions on ytterbium stable isotopes were studied with high-resolution spectrographs and gamma scintillation spectrometers. The hafnium radionuclides were separated from the ytterbium target by solvent extraction with thenoyltrifluoroacetone (TTA). The carrier-free radionuclides were electroplated onto fine platinum wire. Over 60 transitions in the 100-kev energy range were resolved. Assignment of the resolved decay schemes to particular radionuclides will be based on relative activation yield, determined by use of enriched isotopes of ytterbium plus bombarding energies, and on decay rates of conversion lines in a series of photographic exposures. The detailed analysis of conversion spectral lines and of relative gamma scintillation spectra will make possible a study of the properties of nuclear-energy levels in lutetium isotopes.

This work was a continuation of previous work done with the 86-in. cyclotron. Bombarding with 75-Mev alpha particles produces radionuclides previously unavailable with 20-Mev proton bombardment. These studies were done jointly with B. Harmatz.[9]

[10]B. Harmatz and T. H. Handley, "Properties of Nuclear Levels in a Number of Even-Mass Nuclei ($184 \leqq A \leqq 192$)," *Nucl. Phys.* **56**, 1 (1964).

12.1.*h* Half-Life Measurements

S. A. Reynolds

Unpublished decay data for ^{140}La were analyzed according to the least-squares method both by the IBM 7090 computer with the CLSQ code and by hand calculation.[11] The half-life values determined were 40.2 ± 0.1 and 40.1 ± 0.1 hr respectively. The former result was chosen because the CLSQ program weights the early high-precision points more than the later ones, and the hand calculation involves taking logarithms, which appears to simulate a weighting in the reverse sense. Literature values are 40.0 to 40.3 hr, with a weighted mean of 40.2 hr.[12]

Direct measurement of the gamma decay of four ^{137}Cs sources has been in progress for five years. The data acquired thus far have been treated by the CLSQ code, and the half-life value so obtained, which is the present tentative value, is 30.6 ± 0.7 years (95% C.L.). A recent value based on specific activity is 30.8 ± 0.3 years.[13]

12.1.*i* Redetermination of the Half-Life of ^{232}U

W. S. Lyon

The value 71.4 ± 0.6 years was determined for the half-life of ^{232}U by measuring the heat evolution of a weighed sample of uranium oxide whose uranium content was 99.24% mass 232. The value 72.1 ± 0.5 years was obtained by counting the alpha disintegration rate. A mean between these values, 71.7 ± 0.9 years, at an estimated 95% confidence level is proposed for the half-life of ^{232}U. A paper on this subject was published.[14] This work was done jointly with J. M. Chilton,[15] R. E. Leuze,[15] and R. A. Gilbert.[16]

[11]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).

[12]"La^{140}, Ground-State Decay, Sheet 1," *Nuclear Data Sheets*, vol. 5, Natl. Acad. Sci.–Natl. Res. Council, Washington, D.C., 1962.

[13]R. E. Lewis, ORNL Isotopes Division, personal communication to S. A. Reynolds, July 1964.

[14]J. M. Chilton *et al.*, "A Redetermination of the Half-Life of Uranium-232," *J. Inorg. Nucl. Chem.* **26**, 395 (1964).

[15]Chemical Technology Division.

[16]Chemistry Division.

12.1.*j* Half-Life of ^{76}As

J. F. Emery

The half-life of ^{76}As was recalculated by use of computer programs to analyze the decay data. The new value for the half-life of ^{76}As is 26.05 ± 0.10 hr; values reported in the literature range from 26.1 to 26.8 hr.[17]

12.2 MEASUREMENT OF RADIOACTIVITY

12.2.*a* Epithermal Neutron Irradiation of Lanthanum to Produce ^{136}Cs

J. S. Eldridge

High-purity lanthanum was irradiated in a high epithermal neutron flux facility of the ORR to produce ^{136}Cs by the (n,α) reaction. Cerium-141 and ^{140}Ba were produced in addition to ^{136}Cs. The ^{141}Ce could have been produced by two paths:

$$\begin{array}{ccccc} {}^{139}\mathrm{La} & \xrightarrow{(n,\gamma)} & {}^{140}\mathrm{La} & \xrightarrow{(n,\gamma)} & {}^{141}\mathrm{La} \\ & & \downarrow \beta^- & & \downarrow \beta^- \\ & & {}^{140}\mathrm{Ce} & \xrightarrow{(n,\gamma)} & {}^{141}\mathrm{Ce} \end{array}$$

Since an appreciable quantity of ^{141}Ce was produced and the (n,γ) cross section of ^{140}Ce is only 0.5 barn, one concludes that ^{140}La has a high cross section. The ^{141}Ce was isolated in high radiochemical purity and in carrier-free form by thenoyltrifluoroacetone (TTA) extraction.

It was not expected that ^{140}Ba would be produced in detectable quantities, as it can be made by only an (n,p) reaction on ^{140}La; its production here indicates a high (n,p) as well as a high (n,γ) cross section for ^{140}La.

This work will be repeated to measure cross sections of some of the radioactive species involved and to study the feasibility of producing ^{141}Ce by these two paths.

This work was done jointly with H. A. O'Brien.[18]

[17] D. Strominger, J. M. Hollander, and G. T. Seaborg, "Table of Isotopes," *Rev. Mod. Phys.* **30** (2), pt. 2, 585 (1958); esp. p. 644.

[18] Isotopes Division.

12.2.*b* Gamma-Ray Spectroscopy with Lithium-Drifted Germanium Diode Detectors

J. S. Eldridge

A lithium-drifted germanium diode detector of the type described by Tavendale and Ewan[19] was used in several analytical applications. The high resolution inherent in this device separates gamma rays of closely similar energies, which are unresolved in conventional NaI(Tl) spectroscopy. A study of the decay of ^{99}Mo-^{99m}Tc (see Sect. 12.1.c) required a means of determining the abundance of 0.78-Mev gamma rays in ^{99}Mo. Figure 12.3 indicates the gamma-ray spectrum of this radionuclide and shows that the 0.74- and 0.78-Mev gamma rays are unresolved. Figure 12.4 shows the gamma-ray spectrum of this nuclide obtained with a germanium diode detector. The

[19] A. J. Tavendale and G. T. Ewan, "A High Resolution Lithium-Drifted Germanium Gamma-Ray Spectrometer," *Nucl. Instr. Methods* **25**, 185 (1963).

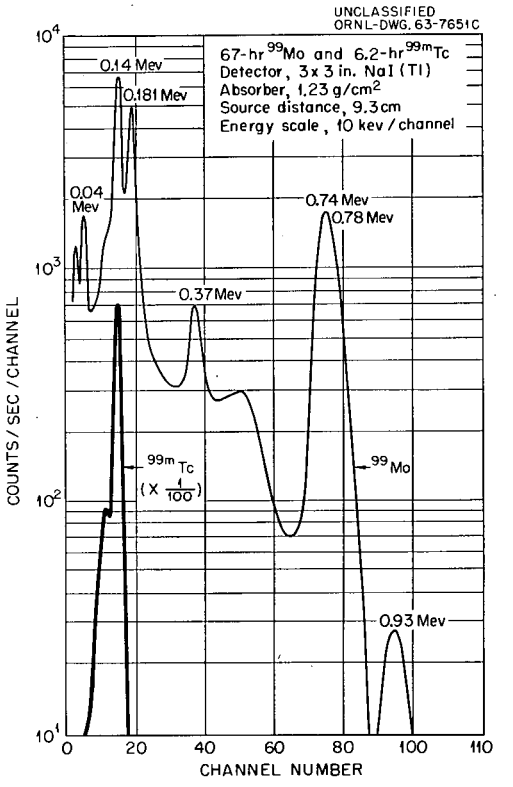

Fig. 12.3. Gamma-Ray Spectra of ^{99}Mo (upper curve) and ^{99m}Tc.

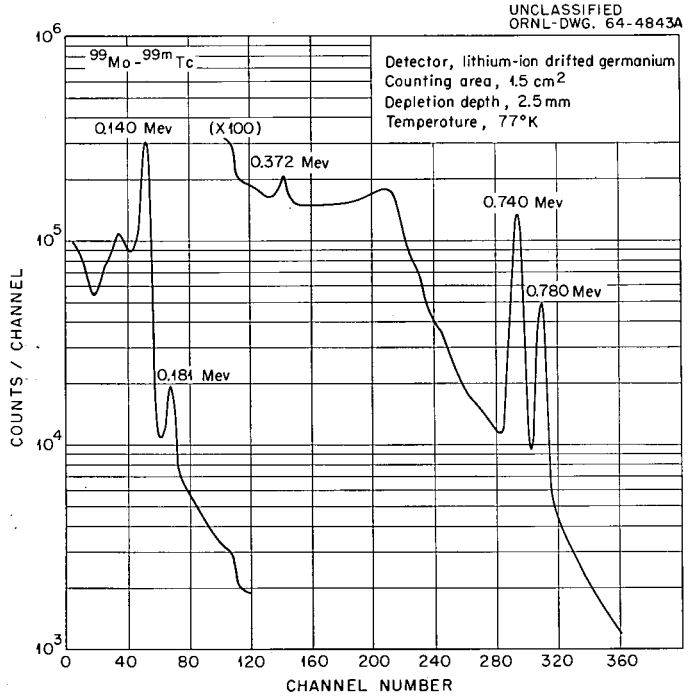

Fig. 12.4. Gamma-Ray Spectrum of ^{99}Mo-^{99m}Tc Obtained with a High-Resolution Lithium-Drifted Germanium Diode Detector.

0.74- and 0.78-Mev photopeaks are clearly resolved. Determination of the branchings in ^{99}Mo through the 0.74- and 0.78-Mev transitions permitted the calculation of equilibrium concentrations of ^{99m}Tc.

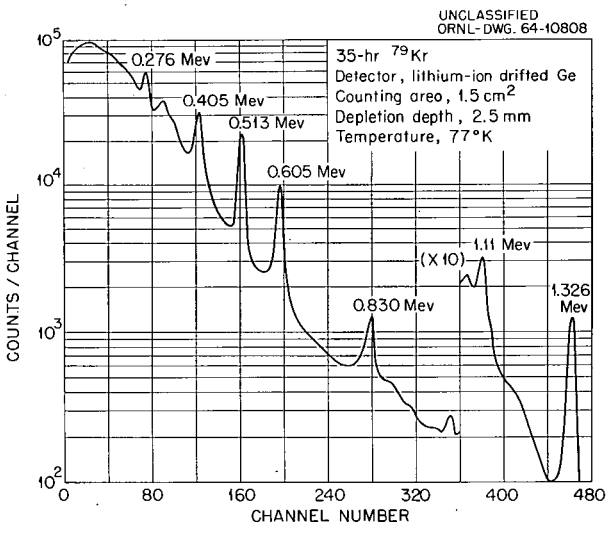

Fig. 12.5. Gamma-Ray Spectrum of ^{79}Kr Obtained with a High-Resolution, Lithium-Drifted Germanium Diode Detector.

Figure 12.5 is a gamma-ray spectrum of 35-hr ^{79}Kr obtained with a lithium-ion-drifted germanium detector. Because of its high resolution, this detector permits the determination of precise energy values more easily than does NaI(Tl) spectroscopy. Comparison of Fig. 12.5 with Fig. 12.1 illustrates the utility of the high-resolution detector; in Fig. 12.1 the 1.1-Mev photopeak is only partly resolved, whereas in Fig. 12.5 it is resolved into three gamma rays.

Mixtures of ^{95}Zr-^{95}Nb and ^{59}Fe-^{60}Co that are unresolved in conventional NaI(Tl) spectroscopy were successfully separated with the germanium diode detector.

12.2.c Effect of Gamma Spectrometer Live Time on Precision of Measurements

S. A. Reynolds

The effect of live time on the performance of a typical 256-channel analyzer was studied briefly.

The study required three ^{137}Cs sources (A, B, and C, provided by J. F. Emery) that had precisely known ratios of radioactivity (1:2.5:5). Spectra of these sources were taken at four different distances, and the spectra of B and C were compared with that of A. The correction for random-summing loss was estimated as follows. It was assumed that, in the region of the sum spectrum up to twice the energy of the Compton maximum, every second count represented the loss of a count from the primary peak (peak/total is ~0.5). Above this energy level, every sum count indicated the loss of a peak count. Thus, the correction was found to be +0.2% per 1000 counts/sec in the peak. The data were grouped and averaged in order to show the effects of different parameters or operating conditions. None of these have much effect; the results are reasonably accurate even at live time as small as 10%, although making measurements at such a small live time is not recommended.

12.2.*d* International Atomic Energy Agency (IAEA) Intercomparison of Determinations of ^{137}Cs

J. S. Eldridge Peter Crowther[4]

To clarify existing discrepancies among several laboratories in which different assay methods are used to determine ^{137}Cs, the International Atomic Energy Agency (IAEA) Division of Research and Laboratories conducted an interlaboratory intercomparison. Results from ORNL by three assay methods were 8.91, 8.99, and 9.04 $\mu c/g$; the mean of the results from all the participating laboratories was 9.06 $\mu c/g$. Participation in this intercomparison has helped to improve the assay methods used to measure this important radionuclide.

12.2.*e* Absolute Determination of ^{125}I in Clinical Applications

J. S. Eldridge Peter Crowther[4]

A sum-coincidence method for the absolute determination of ^{125}I was studied with several detectors. Four detector types gave absolute disintegration-rate values that were accurate to better than 1%. Effects of distance, sample volume, and detector resolution on the absolute method were studied. Detection efficiencies were measured in six measurement systems and gave values ranging from 18 to 47%. Surface effects in NaI(Tl) detectors that cause distorted spectra were explained. A paper on this subject was published.[20]

12.2.*f* Determinations of ^{232}U by Gamma Spectrometry

S. A. Reynolds

The gamma spectra were taken for a number of samples of ^{233}U that had been enriched in a calutron. Most of the gamma radiations were of low energy and arose in the decay of ^{233}U. The high-energy gammas were of major interest; they are associated with the ^{208}Tl daughter of contaminant ^{232}U,[21] measured through the 2.6-Mev radiation of ^{208}Tl.[22] The peaks characteristic of ^{208}Tl were seen, as well as those of ^{209}Tl and ^{213}Bi which are daughters of ^{233}U. The samples were enclosed in a variety of containers, principally shipping cans ~7 in. high and metal chambers. A simple estimate of the effective distance *h* of a source was made by counting its container both while upright and while inverted. Uranium-232 in concentrations from one to several hundred parts per billion was measured in ^{233}U samples that were 0.1 g to many grams in size.

Uranium-232 was found in each of several samples of enriched ^{235}U; levels of ^{232}U of ~0.1 to 0.7 ppb were measured in ^{235}U. Thus, a hitherto unreported finding[23] by ORGDP personnel in 1960 was confirmed. Uranium-232 is not formed in significant quantity in nature, but on irradiation of uranium it is produced both from thorium impurity and from the ^{231}Pa daughter of ^{235}U.

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[20] J. S. Eldridge and P. Crowther, "Absolute Determination of ^{125}I in Clinical Applications," *Nucleonics* **22**(6), 56 (1964).

[21] S. A. Reynolds, "Radioactivity Associated with Uranium, Thorium, and Plutonium," pp. 562–72 in vol. 2 of *Progress in Nuclear Energy, Series III, Process Chemistry*, ed. by F. R. Bruce, J. M. Fletcher, and H. H. Hyman, Pergamon, New York, 1958.

[22] S. A. Reynolds, "Determination of U^{232} in U^{233}," *Anal. Chem. Div. Ann. Progr. Rept. Dec. 31, 1958*, ORNL-2662, p. 57.

[23] C. A. Kienberger, ORGDP, personal communication to S. A. Reynolds, July 1964.

12.3 RADIOCHEMICAL STUDIES

12.3.*a* Trialkyl- and Hexaalkylphosphorothioic Triamides as Extractants for Metal Ions. Selective Extraction of Hg^{2+}, Ag^{+}, and Cu^{+}

T. H. Handley

N,N′,N″-Trihexylphosphorothioic triamide (thio-THPA) and N,N′,N″-hexabutylphosphorothioic triamide (thio-HBPA), which are nitrogen analogs of the neutral esters of phosphorothioic acid, were evaluated as reagents for extracting metal ions from solutions of aqueous mineral acids. Of 33 metal ions studied, only Cu^{+}, Hg^{2+}, Ag^{+}, Pd^{2+}, and Au^{3+} are extracted in significant amounts. The extracted species are formed rapidly. From aqueous solutions 0.1 to 6 *M* in acid and with a 2.62 m*M* solution of thio-THPA or thio-HBPA in $CHCl_3$, the metal ions Hg^{2+}, Ag^{+}, and Cu^{+} are extracted quantitatively. The D_{Hg} (distribution ratio of Hg) is independent of the concentration of HNO_3 or H_2SO_4 in the aqueous phase; the D_{Ag} depends on NO_3^- concentration. Mercury(II) combines with thio-THPA in a mole ratio of 2 to 1. The metal-ion concentration, the type of organic solvent, and the substituent group of the trialkylphosphorothioic triamides are significant variables. Structures are postulated for the complex species formed between the extractant and the metal ions. A paper[24] on this subject is scheduled for publication in the December issue of *Analytical Chemistry*.

12.3.*b* Metal Di-*n*-butyl Phosphorodithioates as Extractants for Metals

T. H. Handley

The metal dialkyl phosphorodithioates have greater chemical stability than the corresponding acids and, accordingly, are less subject to oxidation and hydrolysis. Therefore, they were considered as possible extractants and were found to show the following definite order of replacement with respect to one another: $Pd^{2+} > Au^{3+} > Cu^{+} > Hg^{2+} > Ag^{+} > Cu^{2+} > Bi^{3+} > Pb^{2+} > Cd^{2+} > In^{3+} > Zn^{2+}$. When one metal ion is to be separated from another, selectivity is improved by the proper choice and use of a metal dialkyl phosphorodithioate as the extractant. However, the behavior of the dialkyl phosphorodithioates of certain metal ions, for example, Hg^{2+}, Ag^{+}, and Pd^{2+}, is more complicated than the simple replacement of one metal ion for another. At high loading, a complex species is formed that may contain two different metal ions or a quantity of a single metal ion which is greater than that represented by a hydrogen atom replacement reaction. Therefore, the use of the dialkyl phosphorodithioate of Hg^{2+}, Ag^{+}, or Pd^{2+} as an extractant may not always improve selectivity.

Experimental evidence of the complicated behavior of the alkyl phosphorodithioates of certain metal ions was obtained in studies of the extraction of Ag^{+}. A series of experiments was made in which a CCl_4 solution of $[(C_4H_9O)_2P(S)S]_2Hg$ or $(C_4H_9O)_2P(S)SAg$ was used to extract Ag^{+} from an acid aqueous phase. A Scientific Communication[25] on this subject is to be published in *Analytical Chemistry*.

12.3.c A New Approach to the Separation of Trivalent Actinide Elements from Lanthanide Elements. Selective Liquid-Liquid Extraction with Tricaprylmethylammonium Thiocyanate

F. L. Moore

Preferential liquid-liquid extraction of the anionic thiocyanate complexes of the trivalent actinide elements with tricaprylmethylammonium thiocyanate dissolved in xylene or other solvents affords a new improved method for their separation from the lanthanide elements. The order of extractability is Cf > Be > Am, Cm >> Yb > Tm > Eu > Pm > Y > Ce > La. Various dilute acids markedly enhance the separation factors. Among the advantages of this method are the high single-stage separation factors possible and the elimination of the neutron hazard and the corrosion problems associated with lithium chloride–hydrochloric acid systems. The method has several useful analytical and process

[24] T. H. Handley, "Trialkyl- and Hexaalkylphosphorothioic Triamides as Extractants for Metal Ions. Selective Extraction of Mercury(II), Silver(I), and Copper(I)," *Anal. Chem.* **36**, in press (1964).

[25] T. H. Handley, "Use of Metal Di-*n*-butyl Phosphorodithioates as Extractants for Metals," *Anal. Chem.* **36**, in press (1964).

applications. A paper on this subject was published.[26]

12.3.*d* Rapid Separation and Determination of Technetium

Gerald Goldstein

Extraction with cyclohexanone, first suggested by Boyd and Larson,[27] was applied to the determination of technetium in fission products. About 98% of the technetium can be extracted from 0.1 to 6 *N* H_2SO_4 solutions. There are very few interferences. Reducing agents can prevent the complete extraction of TcO_4^-; a few elements, notably rhenium in the form of ReO_4^-, are also extractable.

Recently, considerable interest has been shown in the use of technetium as a burnup monitor because of its low rate of diffusion and volatility and because of the high fission yield of the mass-99 chain (6.06% for ^{235}U fission). For short neutron irradiations (a few minutes) the number of fissions occurring in a uranium sample was determined by allowing the sample to cool for 20 to 24 hr, during which time ^{99m}Tc grows in from its ^{99}Mo parent. The ^{99m}Tc is then separated by cyclohexanone extraction, and its disintegration rate is determined by gamma spectrometry. From this value, the number of atoms of ^{99}Mo present at the end of the irradiation and, consequently, the number of fissions can be calculated. Gamma spectra of the fission product solution and of the extract (Fig. 12.6) demonstrate that the 140-kev ^{99m}Tc transition is easily measured in the extract. Results for several irradiations of natural uranium are shown in Table 12.1.

Technetium can also be separated rapidly and efficiently from long-lived fission products ($^{141,144}Ce$, ^{140}Ba-^{140}La, ^{95}Zr-^{95}Nb, $^{103,106}Ru$, and ^{137}Cs) by a single extraction from 2.5 *N* H_2SO_4, followed by one wash with a volume of 2.5 *N* H_2SO_4 equal to the volume of the extract. The decontamination factor is 10^5 to 10^6; about

[26] F. L. Moore, "A New Approach to Separation of Trivalent Actinide Elements from Lanthanide Elements. Selective Liquid-Liquid Extraction with Tricaprylmethylammonium Thiocyanate," *Anal. Chem.* **36**, 2158 (1964).

[27] G. E. Boyd and Q. V. Larson, "Solvent Extraction of Heptavalent Technetium," *J. Phys. Chem.* **64**, 988 (1960).

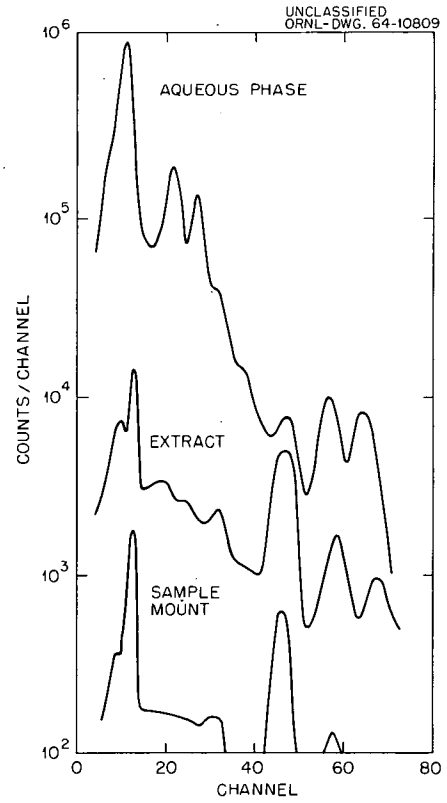

Fig. 12.6. Gamma Spectra of Short-Lived Fission Product Solution and Separated ^{99m}Tc. (~10 kev per channel.)

Table 12.1. Results of Determination of Number of Fissions

1.00 mg of natural uranium irradiated

Duration of Irradiation (min)	^{99m}Tc[a] (dis/sec)	^{99m}Mo[b] (atoms)	Fissions[c] (number/min)
	$\times 10^{-3}$	$\times 10^{-9}$	$\times 10^{-9}$
1	2.35	1.17	1.17
2	4.83	2.39	1.19
3	6.89	3.41	1.14

[a] Corrected to time of separation.

[b] At end of irradiation.

[c] 1.16×10^9 calculated from average flux value.

Table 12.2. Results of Separation of ^{99}Tc from Long-Lived Fission Products

^{99}Tc (μg) Added	Recovered[a]	Recovery (%)	Relative Standard Deviation of Recovery (%)
0	~0.01		
5.84	5.47	93.7	0.6
14.6	13.6	93.2	1.2
29.2	27.0	92.5	1.4
29.2[b]	26.9	92.3	0.7

[a]Average of four determinations.
[b]No fission product activity added.

93% of the technetium is recovered. Table 12.2 shows the results of extractions of technetium spikes added to 10 μc of fission product radionuclides. In this case, ^{99}Tc was measured by the liquid-scintillation method.

12.3.e Separation of Trace Metals from Uranium by Cellulose Column Chromatography

L. C. Bate R. A. A. Muzzarelli[28]

By cellulose column chromatography, submicrogram quantities of 13 metal ions were separated from 50 g of uranyl nitrate dissolved in ethyl ether (95%)–nitric acid (5%) solution. The absorption and elution of the trace metals was followed with radioisotopes. A KCNS–ethyl ether (60%)–methanol (40%) eluent removed In, Zn, Co, Cu, Cr, Mn, Ba, Fe, and Ag; a KCNS-acetone eluent removed Sr and Cd; and KCNS–HCl (30%)–acetone (70%) eluted Sb and Cs. All these ions except barium were completely removed by the eluents.

[28]Alien guest. Present address: Université de Sherbrooke, Faculté des Sciences, Sherbrooke, Quebec, Canada.

12.3.f Chemical Effects of Isomeric Transitions. Separation of Tellurium Isomers in Different Chemical Media

R. L. Hahn

Study of the chemical changes that follow isomeric transitions in some tellurium radionuclides was continued.[29] The chemical separation of the nuclear isomers of ^{127}Te and ^{129}Te in different media was investigated. The variation of $f_j(^{127}Te)$ with pH in aqueous solutions of HCl, HNO_3, $HClO_4$, and NaOH was measured; f_j is the probability that, if the parent metastable-state radionuclide is in the form of telluric acid, the daughter ground-state radionuclide will be found as tellurous acid because of chemical interactions in substance j. The relation between f_j and pH is essentially the same for the different acids. For 6 N acid solutions, f_j is close to unity; it decreases with increase in pH, and at pH 6.0 it approaches the value for H_2O (0.760 ± 0.017). For NaOH solutions, f_j increases with a decrease in pH and also approaches the value for H_2O (see Fig. 12.7). The variation of f_j with pH can be expressed by the relation

$$f_j = \exp(x_j)/[1 + \exp(x_j)] ,$$

with

$$x_j = m_j(\mathrm{pH}) + b_j ;$$

m_j and b_j are parameters obtained from the data. Also, relative f_j values for ^{127}Te and ^{129}Te were determined in solid telluric acid, water, and 6 N solutions of HCl, $HClO_4$, and NaOH. Although the absolute f_j values for these nuclides change from substance to substance, the ratios of the f_j's remain fairly constant (~1). Thus, f_j appears to be mainly a function of the chemical – not the nuclear – properties of the isomer-separation process. A paper on this subject was published.[30]

[29]R. L. Hahn, "Chemical Effects of Isomeric Transitions: Separation of the Isomers of ^{127}Te, ^{129}Te, and ^{121}Te," *J. Chem. Phys.* **39**, 3482 (1963).

[30]R. L. Hahn, "Chemical Effects of Isomeric Transitions: The Separation of Tellurium Isomers in Different Chemical Media," *J. Chem. Phys.* **41**, 1986 (1964).

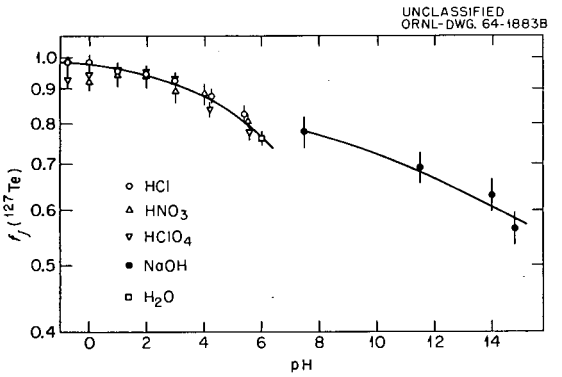

Fig. 12.7. Variation of f_j (^{127}Te) with pH for Various Substances j. The solid curves are fits to the data calculated with the equations: $f_j = \exp(x_j)/[1 + \exp(x_j)]$. For the acids, $x = -0.404$ (pH) + 3.59; for NaOH, $x = -0.129$ (pH) + 2.25.

12.3.*g* Yields of Krypton and Xenon from Fission of Uranium

R. L. Hahn

Work on the determination of yields of krypton and xenon from fission of uranium was continued.[31] Changes were made in the vacuum system so that 10-year ^{85}Kr could be used to determine the chemical yield of the gas-handling procedure for krypton. Auxiliary studies were carried out to check calculations of the solid angle subtended by a sphere with respect to a right circular cylinder; agreement to within 3% was obtained between the theoretical and experimental results.

12.3.*h* Ore Selection for Project Coach

S. A. Reynolds

Exploratory studies were made to acquire analytical data relevant to methods of ore selection for Project Coach,[32] which is concerned with recovery of transplutonium elements formed by an explosion of a nuclear device in a salt bed. Initially, it was assumed that a simple external reading of the gross gamma radiation from ore would suffice to select the material to be processed. A more complicated method involving measurement of spontaneous-fission gammas or neutrons would be feasible but was shown to be unnecessary.

The early part of the experimental program included the taking of gamma spectra of samples from the Gnome underground test presumed to be reasonably representative of Coach "ore." Spectra of a typical aqueous leach solution and water-insoluble solid are shown in Fig. 12.8. All the samples contained the same radionuclides, but in different proportions; and all included both fission products and neutron-induced nuclides. Although the levels of radioactivity of the various samples were very different, the spectra did not show meaningful differences in the behavior of the elements present. This observation was substantiated by consideration of earlier radiochemical analytical results.

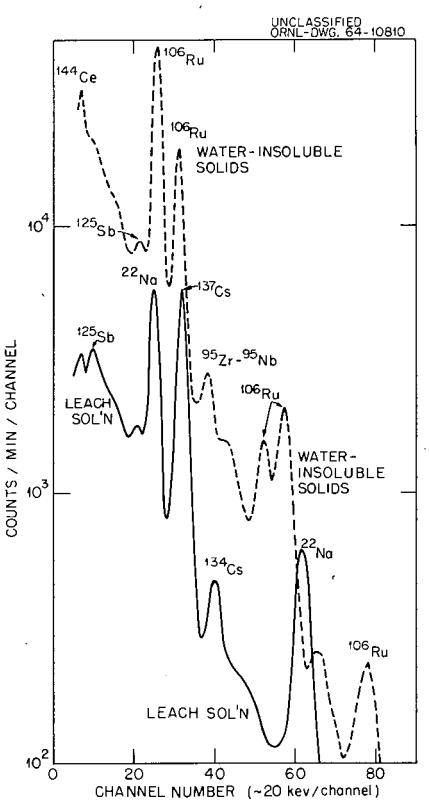

Fig. 12.8. Gamma Spectra of Samples from the Gnome Underground Test.

[31] R. L. Hahn, "Determination of the Yields of Krypton and Xenon Nuclides in Uranium Fission," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1963*, ORNL-3537, p. 76.

[32] "Project Coach," *Chem. Tech. Div. Ann. Progr. Rept. May 31, 1964*, ORNL-3627, pp. 256–58.

The analyses consisted primarily in determining gross gamma radioactivity, plutonium, and gross transplutonic elements (TrPu) in 48 samples of Gnome material. After exhaustive leaching with dilute HCl, the acidity was adjusted to ~1.5 *M*, and Pu was determined by double TTA extraction under the usual conditions. The TrPu, as a group, were determined by oxidizing the raffinates from the Pu extraction and performing $La(OH)_3$ and LaF_3 precipitations. The Pu and TrPu values were corrected according to ^{144}Ce recovery, based on gamma spectra.

Various correlations were explored. Thus, good correlation was observed between TrPu and Pu radioactivities and between TrPu and Ce. Correlation between TrPu and gross gamma, though not so good, was real, as shown by computation of correlation coefficients. The ratio of TrPu to Pu alpha radioactivities increased during a period of a few months, and both the magnitude of the ratio and its increase were consistent with ~1% ^{241}Pu being present in the post-shot Pu. Decay of the gross gamma radioactivity fitted reasonably well the equation[33]

$$A = Kt^{-1.5} ,$$

said to be applicable from 100 days to 3 years post-shot.

The TrPu-Ce correlation was used as the basis for a field method for ore selection. A gamma spectrometer could be used to screen batches of ore, but a less expensive device could also serve. A mockup of such a "gamma probe" was made from a scintillation counter in a lead shield with a hole, ~2 in. in diameter, in the top. A 1.8-g/cm^2 Al absorber (to absorb betas) was placed over the hole, and 1-g portions of ten selected samples were counted. Next, a 0.9-g/cm^2 Pb absorber (~3 half-thicknesses for ^{144}Ce gamma rays) was added, and the samples were re-counted. The *difference* between the two counts of each sample is proportional to its soft-gamma content, largely ^{144}Ce. The differences were compared with TrPu values; the correlation was very good. Therefore, it is concluded that TrPu content can be estimated in the field by use of a simple scintillation device with a lead filter.

[33]J. E. Watson, *An Analysis of Calculated and Measured Fission Product Activities*, BRL-1239 (February 1964).

12.3.*i* Search for Natural Transuranic Radioactivity

S. A. Reynolds

A small-scale study, related to a problem in cosmology, is of inherent interest and possible significance in low-level work. A "surplus" alpha radioactivity was reported in some natural materials. The surplus was ascribed to ^{247}Cm (ref. 34) and to ^{244}Pu by other investigators,[35] who reported detecting it in a mineral fraction of Conway (New Hampshire) granite. A solution resulting from leaching Conway granite with dilute nitric acid was furnished by F. J. Hurst.[15] A TTA extraction for Pu was made and none was found. The limit of detection was such that the finding does not contradict that of Cherdyntsev and Mikhailov,[34] who reported the mysterious activity as only a few percent of that of ^{238}U, but the finding is inconsistent with the second report.[35] The ^{247}Cm postulate was shown to be invalid because the half-life of ^{247}Cm is too short,[36] and the ^{244}Pu postulate was retracted.

12.3.*j* Low-Level Analyses

S. A. Reynolds C. L. Burros[37]

Required cooperative effort in low-level analyses has continued as previously reported,[38] and results appear to be very satisfactory; a few examples follow. The study of air-filter samples included identification of the radionuclides in an April "dustfall" as old fission products. In several coal samples, the upper-limit radioactivity levels of gross beta, Ra, U, Th, and K and the ash content were determined. Comparison of these data

[34]V. V. Cherdyntsev and V. F. Mikhailov, "A Primordial Transuranium Isotope in Nature," *Geochem. (USSR)* (English Transl.) **1**, 1 (1963).

[35]R. D. Cherry, K. A. Richardson, and J. A. S. Adams, "Unidentified Excess Alpha-Activity in the 4.4-MeV Region in Natural Thorium Samples," *Nature* **202**, 639 (1964).

[36]P. R. Fields *et al.*, "Possible Existence of Curium in Nature. II," *Phys. Rev.* **131**, 1249 (1963).

[37]Radioisotopes-Radiochemistry Group.

[38]S. A. Reynolds and C. L. Burros, "Low-Level Radiochemical Studies," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1963*, ORNL-3537, p. 81.

with published values[39] showed that the level of radioactivity correlates with the percentage of ash. Four 50-g disks of metallic lead were analyzed by gamma spectrometry and low-background beta counting; the Ra, Th, and U contents were negligible, but one of the samples contained 80 dis min^{-1} g^{-1} of ^{210}Pb.

12.4 LIQUID SCINTILLATORS

12.4.*a* Liquid Scintillators That Contain 1-Methylnaphthalene

Gerald Goldstein W. S. Lyon

Liquid scintillators containing 1-methylnaphthalene were prepared, and their properties were determined. Commercially available 1-methylnaphthalene was purified by chromatography on acidic-grade activated alumina. Solutions that contain 1 g of POPOP per liter of the purified 1-methylnaphthalene have a relative pulse height (gamma excitation) equal to that of conventional xylene- or toluene-based scintillators. Pulse height is proportional to the Compton recoil energy. Possibly 1-methylnaphthalene can be used in large-volume scintillators, because of its high flash point (112°C) and high density (1.02 g/cc), as a secondary solvent in xylene- and *p*-dioxane-based scintillators, in studies of energy-transfer processes, and in distinguishing particles of different kinds by the pulse-shape discrimination method. A paper on this subject was published.[40]

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

H. H. Ross H. L. Holsopple

The formulation of an efficient liquid detector for thermal neutrons, with the neutron target as an integral part of the system, is often a difficult problem. Practical systems should have a high detection efficiency, low gamma sensitivity, fast response time, large pulse-height outputs from thermal-neutron reactions, and good chemical stability. Also, it is desirable that the solutions be easy to prepare.

Because neutrons are uncharged, they cannot be detected in a liquid scintillator as a result of primary ionization. However, advantage can be taken of various neutron interactions to produce light output from these systems. Fast and epithermal neutrons are observed in conventional liquid scintillators as a result of neutron-proton recoil reactions with the solvent. Thermal neutrons are detected by allowing them to undergo an exoergic nuclear reaction with a suitable target; the resulting reaction fragments can then interact with the liquid scintillator to produce a measurable output pulse.

The compound, N,N′,N″-trimethylborazine, is an efficient solvent in the liquid-scintillation process. Additionally, the natural ^{10}B content of the compound enables the system to be used for the detection of thermal neutrons according to the reaction $^{10}B(n,\alpha)^{7}Li$. The system is unusual in that the compound serves as both the neutron target and the scintillator solvent. When compared with conventional boron-loaded scintillators, the borazine system exhibits a number of significant advantages. An article on this work was accepted for publication in *Nuclear Instruments and Methods.*[41]

12.4.*c* Absolute Counting of Beta Emitters by the Liquid-Scintillation Method

Gerald Goldstein

Extrapolation methods for absolute counting of beta emitters in liquid scintillators have been reported from time to time in the past ten years and have now been examined in some detail with the Packard Tri-Carb liquid-scintillation spectrometer. In this method, the integral counting rate is measured at various pulse-height-discriminator settings. The logarithm of the count rate is plotted vs the discriminator setting, and the line obtained

[39] M. Eisenbud and H. G. Petrow, "Radioactivity in the Atmospheric Effluents of Power Plants That Use Fossil Fuels," *Science* **144**, 288 (1964).

[40] G. Goldstein and W. S. Lyon, "Liquid Scintillators Using 1-Methylnaphthalene," *Intern. J. Appl. Radiation Isotopes* **15**, 133 (1964).

[41] H. H. Ross and H. L. Holsopple, "N,N,N-Trimethylborazine as a Liquid Scintillator Solvent for Thermal Neutron Detection," to be published in *Nuclear Instruments & Methods.*

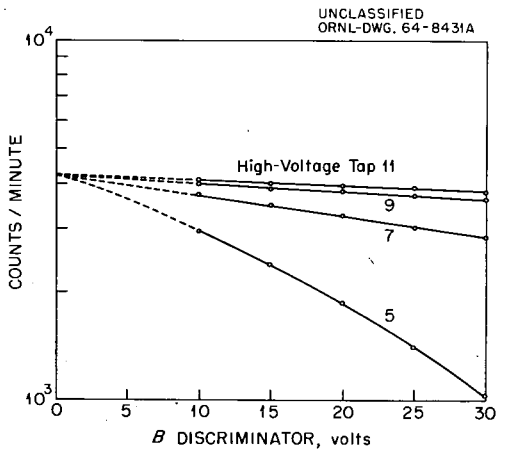

Fig. 12.9. Results of Extrapolation Method Applied to ^{99}Tc.

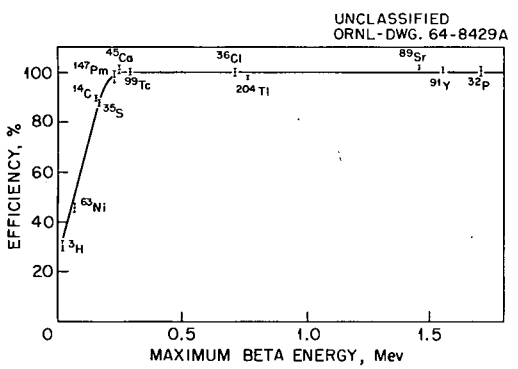

Fig. 12.10. Efficiency of the Extrapolation Method.

is extrapolated to zero discriminator setting (Fig. 12.9) to obtain the absolute disintegration rate. By counting radioisotopes previously standardized by other methods, it was found that the efficiency is 100% if the maximum beta energy is greater than 200 kev (Fig. 12.10). The instrumental and sample-solution variables were evaluated; the results will be reported in detail elsewhere.

This method has some significant advantages. If the maximum beta energy is greater than 200 kev, quenching agents have virtually no effect on the extrapolated value, as shown for ^{99}Tc in Table 12.3. They do, however, increase the slope of the straight line. If two beta groups, or beta emitters, are present, the sum of both can be determined by using instrument settings suitable for the lower-energy group. Initial experiments with a beta-gamma emitter (^{60}Co) indicate that the gamma radiation does not cause any difficulty.

If one uses a computer program[42] to establish the best straight line and the intercept, the relative standard deviation of replicate measurements is usually about 1%.

Potential applications of the method include decay-scheme studies and standardization of radionuclide preparations. As an example of the former, the half-life of ^{99}Tc was remeasured[43] by

[42] M. H. Lietzke, *A Generalized Least-Squares Program for the IBM-7090 Computer*, ORNL-3259 (Apr. 4, 1962).

[43] G. Goldstein, "Specific Activity of ^{99}Tc by Liquid Scintillation Counting," *Chem. Div. Ann. Progr. Rept. June 20, 1964*, ORNL-3679, pp. 19–21.

Table 12.3. Effect of Quenching Agents on ^{99}Tc Measurements

Quencher Added[a]	Intercept (counts/min)	Standard Deviation of the Intercept (counts/min)	Slope
	$\times 10^{-4}$	$\times 10^{-4}$	$\times 10^{3}$
None	4.34	0.03	−3.00
Chloroform	4.28	0.02	−4.31
Cyclohexanone	4.25	0.03	−4.20
Pyridine	4.27	0.02	−3.13
Tetraphenylarsonium chloride[b]	4.25	0.03	4.13

[a] 1 v/v %.

[b] 0.1 *M* solution in $CHCl_3$.

the specific activity method, the disintegration rate being determined by the extrapolation technique, and was found to be $(2.14 \pm 0.05) \times 10^5$ years.

Further studies of this technique and additional applications of it are in progress.

12.5 APPLICATION OF COMPUTERS

12.5.*a* Electronic Resolution of Gamma-Ray Spectra

J. S. Eldridge

Standard-response curves from 42 radionuclides were collected for the "library."[44] A computer program was written to recall the pooled master spectra in a variety of paper-tape formats. Plans are under way for the dissemination of the standard data in paper-tape and graphical forms. It is anticipated that other laboratories will be able to use the spectral data as well as the spectral resolution program[45] by requesting a copy of the library from a distribution center.

Final validation of the spectral data will be made by independent analysis of each radionuclide cataloged in the library. This validation will make use of the program for spectral resolution.[45]

12.5.*b* Computer Program for Accumulating Standard Gamma-Ray Spectra and for Quality-Control Measurements

F. F. Dyer

Efforts were redirected from the program previously mentioned[46] to writing a program for the accumulation of standard gamma-ray spectra. This program, written in FORTRAN 63 for the CDC 1604-A, is similar to the one reported by Eldridge[47] but is tailored for activation-analysis problems and is designed to be used with other programs that run on the 1604-A. Three spectra of each of four standards (12 total spectra) can be summed by the program. The resulting spectrum, which contains a much larger number of counts per channel than the original separate spectra, is very precise. Before summation, each spectrum is changed to a standard pulse-height scale (gain). The method reported by Heath[48] is used; the position of a well-resolved photopeak is found, and the spectrum is then shifted so that the photopeak appears in a preselected position. Alternatively, the program can be used to compute values of chi-square for each channel for a total of ten spectra. Used in this manner, the program permits quality-control checks to be made on gamma-ray spectrometers.

12.6 APPLICATION OF RADIOISOTOPES TO ANALYTICAL CHEMISTRY

12.6.*a* Radio-Release Method for the Evaluation of Atmospheric Pollution. Sulfur Dioxide

H. H. Ross

Atmospheric pollution has recently become a problem of national concern. Therefore, the determination of sulfur-bearing air pollutants, primarily sulfur dioxide, is being studied carefully. The high-sensitivity radio-release method was chosen as a new approach to the determination of sulfur dioxide. This method is based on the selective reaction of the sought constituent with a radioactive reagent of known specific activity. During the reaction, radioactivity is released as a gas or as an easily separable chemical species. The amount of the sought constituent is then calculated from the amount of radioactivity released, the specific activity of the reagent, and the stoichiometry of the reaction. Alternatively, the calculations can be avoided by use of standard samples.

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[44] J. S. Eldridge, "Electronic Resolution of Gamma-Ray Spectra," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1963*, ORNL-3537, p. 85.

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

[46] F. F. Dyer and E. Ricci, "Computer Program for the Calculation of Absolute Activities, Fluxes, and Cross Sections," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1963*, ORNL-3537, p. 86.

[47] J. S. Eldridge, "Electronic Resolution of Gamma-Ray Spectra," *Anal. Chem. Div. Ann. Progr. Rept. Dec. 31. 1962*, ORNL-3397, p. 88.

[48] R. L. Heath, *Data Analysis Techniques for Gamma-Ray Scintillation Spectrometry*, IDO-16784 (May 29, 1962).

The method is potentially one of the most useful radioisotopic analytical techniques. In addition to sensitivity, it has the advantages of selectivity and speed and it readily lends itself to automated systems.

The new method for sulfur dioxide determination is based on the release of radioiodine according to the reaction

$$5SO_2 + 2KI^*O_3 + 4H_2O \rightarrow K_2SO_4 + 4H_2SO_4 + I_2^* .$$

The reaction is carried out in solution by forcing air through a bubbler train that contains a basic solution of potassium iodate. After the sample has reacted, the solution is acidified, and the liberated radioiodine is extracted. The radioactivity of this solution is determined, and the amount of sulfur dioxide is calculated.

A paper on this work was given[49,50] and will be published.[50]

12.6.*b* Application of Radioisotopes to the Determination of Sodium, Lithium, and Phosphorus

W. J. Ross

Sodium. – The standard gravimetric method for determining sodium as sodium zinc uranyl acetate was evaluated with the help of ^{24}Na tracer. A modified and accelerated version was developed whereby sodium tracer is included for yield correction, and the reagent is preheated to improve the purity of the precipitate and to increase the recovery of sodium. The time per analysis is decreased significantly, and the accuracy is increased, thereby enhancing the usefulness of this method for routine analyses.

Lithium. – A similar attempt was made to accelerate the determination of lithium by incorporating ^{131}I in the KIO_4 reagent used in the classical gravimetric method. Such a radioactive reagent has been useful in determining yield and in verifying that the Li/I mole ratio in the precipitate is 4.2 ± 0.2. However, the routine use of such a radioreagent cannot be recommended, because it is difficult to maintain complete dissolution of KIO_4.

Phosphorus. – Phosphorus-32 was used to establish the degree of recovery of small amounts (<10 mg) of phosphorus as $Mg_2P_2O_7$ in the standard gravimetric method. By elimination of the need for quantitative recovery, this usually lengthy procedure was performed in less than 8 hr without loss of precision or accuracy.

12.6.c Separation of Sodium

W. J. Ross

A program was initiated to develop a rapid and efficient method for removing gross amounts of ^{24}Na from neutron-activated samples, especially biological and geological materials, so that minor gamma radionuclides can be identified and measured. Essentially quantitative retention of sodium has been achieved from ammoniacal and slightly acid solutions by such solid ion exchangers as zirconium phosphate, zirconium antimonate, and Dowex 50, even in the presence of complexing agents such as fluoride, tartrate, and EDTA. Inasmuch as many other elements are absorbed under the same conditions, these exchangers do not effectively isolate sodium.

The most promising method for removing ^{24}Na is by isotopic exchange. In batch-type equilibrations, essentially quantitative exchange of ^{24}Na in 80 to 90% HCl has been achieved with solid NaCl. Exchange occurs during the dissolution and recrystallization of NaCl and is decreased in very dilute or very concentrated HCl systems. Initial experiments with 38 elements showed that only Cl, Mn, Cd, and Ba are retained on the solid NaCl to an extent that exceeds 7%. For the removal of sodium from solutions, this approach, as well as other possibilities, will be studied during the coming year in an effort to broaden the scope of usefulness of neutron activation analysis.

[49] H. H. Ross, "Radio-Release Methods for the Evaluation of Atmospheric Pollution," presented at the 12th Anachem Conference, Detroit, Mich., Oct. 21–23, 1964.

[50] H. H. Ross and W. S. Lyon, "Radio-Release Methods for the Evaluation of Atmospheric Pollution: SO_2," presented at the International Atomic Energy Agency Symposium on Radiochemical Methods of Analysis, Salzburg, Austria, Oct. 19–23, 1964 (to be published in the proceedings of the Symposium).

12.7 ACTIVATION ANALYSIS

12.7.a Study of Precision in Activation Analysis

F. F. Dyer J. F. Emery

Activation analysis is often used to compare the trace-element concentrations of a number of samples, for example, samples taken to ascertain homogeneity of a standard or those that pertain to criminal investigation. The correct interpretation and the general utility of the results depends markedly on experimental errors. Because experimental errors in activation analyses have not been studied adequately, it seemed worth while to begin a study of the precision of these methods. Thus far in this study, measurements have been made only on elements that produce radionuclides with half-lives of a few minutes or less. Samples that contained known amounts of selenium and silver, which give 17- and 24-sec activities, respectively, are typical of those studied. For these samples, the relative standard errors are usually about ±1% at the 95% confidence level. Some of the findings have been described.[51]

[51] F. F. Dyer and J. F. Emery, "Precision of Activation Analysis of Short-Lived Nuclides," presented at the Eighth Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Oct. 6–8, 1964.

12.7.b Application of Activation Analysis to Forensic Science

F. F. Dyer J. F. Emery

Ammunition Primers. – Ammunition primers contain large amounts of compounds of barium and antimony. These elements usually deposit in detectable amounts on the hands of one who fires a gun.[52–54] However, the total deposit per firing, as well as the ratio of barium to antimony, is variable. If the ratio were constant, it could be used to indicate the type or brand of ammunition fired. Since this ratio has not been measured for unfired primers, a limited study was made to determine it. The primers used in several brands of .38-caliber ammunition were analyzed, and the residues left in some of the primer caps after firing were analyzed. Table 12.4 gives the results. The data indicate that the barium-to-antimony ratio varies with brand of ammunition

[52] J. D. Buchanan and R. R. Ruch, "Applications of Neutron-Activation Analysis in Scientific Crime Detection," *Quarterly Report for Period Ending October 31, 1962*, GA-3664 (Oct. 31, 1962).

[53] V. P. Guinn, R. R. Ruch, and S. C. Bellanca, "Application of Neutron-Activation Analysis in Scientific Crime Detection," *Quarterly Report for Period Ending January 31, 1963*, GA-4041 (Jan. 31, 1963).

[54] R. R. Ruch, S. B. Madigan, and V. P. Guinn, "Applications of Neutron-Activation Analysis in Scientific Crime Detection," *Quarterly Report for Period Ending July 31, 1963*, GA-4576 (July 31, 1963).

Table 12.4. Ratio of Barium to Antimony in Primer Material Before and After Firing

Type of Sample	Brand of Ammunition	Number of Cartridges Analyzed	Measured Ba/Sb Ratio[a,b]
Primer	Federal	5	2.42 ± 0.17[c]
Primer	Federal	5	1.37 ± 0.34[c]
Primer	Remington-Peters	5	3.45 ± 0.50
Burned primer	Western	3	1.57 ± 0.55[d]
Burned primer	Remington-Peters	3	4.46 ± 0.90[d]

[a] The errors given are 95% confidence limits.

[b] No measurements were made of burned Federal primer.

[c] Three measurements were made for each cartridge.

[d] One measurement was made for each cartridge.

and that this ratio is smaller in the original primer than in the residue left in the cap after the firing.

Because cartridges from only a few boxes of ammunition were analyzed, it is not known how constant the ratio remains from one batch to another of a given brand. The second conclusion is tentative because of the overlap of the 95% confidence intervals for burned and unburned primers. Additional analyses should show definite differences.

Gunshot Residues. – A preliminary study of the firing of a .38-caliber revolver showed that in many instances visible particles are deposited on the shooter's hand. A few of these particles contained significant quantities of barium and antimony. A total of 21 additional firings were made to determine whether the barium-to-antimony ratio of the particles and that of the unfired primer could be correlated. Of 50 particles analyzed, only five contained barium and/or antimony, and their barium-to-antimony ratios did not correspond to that of the primers.

This work was done in cooperation with J. F. Gallagher.[55]

12.7.c Analysis of Bond Paper

J. F. Emery F. F. Dyer

The aluminum and titanium contents of 27 sheets from eleven brands of bond paper were measured. Five samples from each sheet were analyzed, determinations were made nondestructively, and gamma-ray spectra were resolved with a least-squares computer program. The results indicate that each sheet of paper is homogeneous, but differences among some brands are very pronounced. Elements that produce longer-lived radionuclides will be measured subsequently.

12.7.d Wool Analysis

L. C. Bate W. B. Healy[56]

The trace-element content was determined on wool from twin wethers raised in two areas on pastures whose trace-element compositions reflect soil differences.[57,58] Nine elements (Mn, Cu, Zn, Al, Se, Mo, W, V, and Au) were measured quantitatively, and four others (Ag, Hg, Sb, and Cr) were detected. The amounts of five of the elements (Mn, Zn, Cu, Se, and W) present in the wool depended on which soil area the animal was raised on. The trace-element content of the wool was compared with that of the internal organs; the results indicate the possibility of following the animal's nutrition by monitoring the trace-element content of the wool. This work was discussed,[59] and a paper was submitted to the *New Zealand Journal of Agricultural Research.*[60]

12.7.e Absorption of Trace Elements by Hair

L. C. Bate

Certain elements (Au, Se, and Hg) are absorbed by hair and cannot be removed subsequently by a detergent wash. It seemed possible that significant fractions of the elements normally found on hair might be surface contaminants and that washing with a detergent might not remove them. Therefore, a thorough study of this possibility seemed necessary.

The absorption of Au^{3+}, Zn^{2+}, Se^{2+}, Cr^{3+}, Cr^{6+}, I^-, As^{3+}, As^{5+}, Br^-, Cu^{2+}, Na^+, Ag^+, Hg^{2+}, Sb^{3+}, Co^{2+}, Fe^{2+}, Ca^{2+}, Ba^{2+}, Sr^{2+}, Mn^{2+}, K^+, Cl^-, and P^{5+} was investigated. Some of the cations are believed to be absorbed as anionic species. Most of them are normally found by activation analysis of hair. In most experiments, 100 mg of hair was placed in 15 ml of an aqueous solution that contained the element to be absorbed. The amount of absorption was measured by radioactive tracers. The composition of the solution approximated the chemical composition of perspiration, and all the absorptions were made at a number of pH values

[55]Federal Bureau of Investigation, Laboratory Division, Washington 25, D.C.

[56]Soil Bureau, Department of Scientific and Industrial Research, Lower Hutt, New Zealand.

[57]W. B. Healy, T. G. Ludwig, and F. L. Losee, "Soils and Dental Caries in Hawke's Bay, New Zealand," *Soil Sci.* **92**, 359 (1962).

[58]L. C. Bate and W. B. Healy, "Wool Analysis," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1963*, ORNL-3537, p. 98.

[59]L. C. Bate and W. B. Healy, "Determination of Trace Elements in Wool," presented at the Eighth Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Oct. 6–8, 1964.

[60]W. B. Healy, L. C. Bate, and T. G. Ludwig, "Neutron Activation Analysis of Wool," submitted for publication, *New Zealand Journal of Agricultural Research.*

similar to those of perspiration. All the elements are absorbed except the alkali metals. The extent of absorption depends on the element as well as on the pH of the solution.

Removal of the absorbed elements with complexing agents is now being tried; EDTA appears to be effective in removing some of the absorbed elements.

12.7.*f* Hair Analysis

L. C. Bate W. B. Healy[56]

Analysis of three groups of hair samples was completed.[61] The samples were from local people, from residents of Hasting and Napier, New Zealand, and from children and pigs that had kwashiorkor (before- and after-recovery samples). In general, the results are similar, and most of the differences apparently are due to differences in diets. All the hair samples were analyzed after being cleaned with a nonionic detergent.[62]

A paper[62] on washing of hair with nonionic detergents was accepted for publication in the *Journal of Forensic Science*.

12.7.*g* Applied Neutron Activation Analysis

J. F. Emery

A number of samples were analyzed by neutron activation analysis for the Botany Department of the University of Florida. Some 30 solutions of $(NH_4)_2CO_3$, enriched to various degrees in ^{18}O, and about 20 CO_2-filled capsules were analyzed for ^{18}O by the reaction $^{18}O(n,\gamma)^{19}O$ ($t_{1/2}$ = 29 sec). Mercury residues from the vacuum system used to prepare the samples complicated the analysis of the liquid samples, and ^{41}Ar ($t_{1/2}$ = 110 min) masked the ^{19}O radioactivity in the gas samples.

Other materials analyzed were: ORR fuel elements for surface contamination by ^{235}U, air filters for selenium, crystalline ZrO_2 for ^{238}U, ^{58}Ni metal for cobalt, solutions of $BeSO_4$ for aluminum, and a germanium oxide crystal for aluminum, vanadium, and chloride.

The fluoride contents of K_2TaF_7 and $CsTaF_6$ were determined by neutron activation analysis; the results were 33.95 ± 0.52 and 26.50 ± 0.28 wt % respectively.

12.7.*h* Activation Analysis of Uranyl Nitrate Solutions

W. J. Ross

Activation analysis was used successfully to determine 0.1 to 10 ppm of Cd, Cr, Co, Mo, Cu, and Mn in an IAEA round-robin sample that contained 50 mg of U per milliliter. A method was developed whereby these trace constituents could be determined by gamma spectrometry after "preseparation" of uranium, neutron activation of the residual components, and chemical separation of the individual nuclides. The preseparation was required to prevent the formation of dangerous and overshadowing amounts of gamma activity in uranium during activation; it consisted in the extraction of uranium from 0.5 *M* HNO_3 with a cyclohexane solution of tri-*n*-octylphosphine oxide (TOPO). Essentially none (10% of Mo) of the trace elements were removed from the aqueous phase during this extraction. An amount of arsenic equivalent to 1 ppm of the sample was detected in the cadmium fraction and represented the only extraneous element that may have been added with the reagents before activation. The results obtained by this method were in excellent agreement with those obtained by other methods used in this program.

12.7.*i* Activation Analysis of High-Purity Materials

W. J. Ross

Samples of high-purity beryllium, aluminum, and iron were analyzed for trace amounts of 62 possible elemental impurities by measuring the gamma activities of the radionuclides produced by neutron activation. Ten elements were determined by nondestructive techniques after neutron bombardment of a portion of the sample for 10 sec and rapid

[61] L. C. Bate and W. B. Healy, "Hair Analysis," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1963*, ORNL-3537, p. 96.

[62] L. C. Bate, "The Use of Activation Analysis in Procedures for the Removal and Characterization of the Surface Contaminants of Hair," presented at the meeting of the American Academy of Forensic Science, Chicago, Ill., Feb. 28–29, 1964 (to be published in the proceedings of the Meeting).

measurement of the induced short-lived radionuclides. A second portion of the sample was activated for 20 min and was analyzed for the gamma-emitting radionuclides of chlorine, bromine, and iodine. Forty-nine elements were determined in a third portion of the sample after a 16-hr irradiation. The induced radionuclides were divided into six groups by a series of rapid chemical separations, and each gamma activity was identified, counted, and compared with that of a standard radionuclide. A paper on this subject was published.[63]

12.7.*j* Neutron-Absorption Analysis with Isotopic Sources

J. E. Strain W. J. Ross

The neutron-absorption analysis of synthesized organo-boron compounds for boron was demonstrated on compounds in which the boron is so tightly bound that the conventional pyrolytic analysis for boron is impossible.

Total neutron-absorption cross section of reactor construction materials was determined by use of a large sample ($\sim$7000 cm^3) and two 2-curie ^{241}Am-Be neutron sources. The limit of cross-section measurement was found to be equivalent to ± 2 μg equivalents of B per cm^3. This concentration is within the purity limits presently imposed in reactor material acceptance criteria for beryllium and aluminum. The total cross-section analysis was proved to be practical for determining H_2O, D_2O, Be, graphite, and Al. The analysis is nondestructive and requires less than 30 min.

12.7.*k* Helium-3 Activation Analysis

E. Ricci R. L. Hahn

Markowitz and Mahony[64] and Demildt[65] have shown that ^{3}He-activation analysis for oxygen in beryllium, thorium, Mylar, and actinide elements is not only feasible but is also very sensitive (fractions of ppb). Extension of the technique to other elements of low atomic weight by the use of short-lived as well as long-lived radionuclides is being attempted. The problem is complicated by the possibility of interferences; that is, the same radionuclide may be produced from two or more different elements. When excitation functions are known, however, it should be possible to reduce interferences by selectively varying the energy of the ^{3}He ions.

About 80 irradiations were performed at the 5.5-Mev Van de Graaff accelerator with ^{3}He ions of energies from 3 to 10 Mev on foils of Mylar (C, O, H), nylon (C, O, H, N), Teflon (C, F), and beryllium. Data were obtained for the excitation functions of reactions leading to the following radionuclides: ^{11}C (20.5 min), ^{13}N (10 min), ^{14}O (72 sec), ^{15}O (124 sec), ^{17}F (66 sec), and ^{18}F (111 min). Thick targets of SiO_2 and ZrO_2 were irradiated with 10-Mev ^{3}He ions to establish sensitivities for the oxygen determination. The results indicate that ultimate sensitivities of about 2×10^{-10} g may be obtained if ^{15}O or ^{17}F is counted with the foil 10 cm from the detector. These data are taken with a 1-min irradiation in a $^3He^{2+}$ current of 25 μa. Analogously, for an irradiation of 110 min, a sensitivity of about 3×10^{-11} g of oxygen may be obtained with ^{18}F. Thick-target neutron yields were also determined; Table 12.5 shows the results. A fraction of the

Table 12.5. Neutron Yields for Reactions Induced on Thick Targets by ^{3}He Ions of 5 and 10 Mev

^{3}He current = 25 μa

Target	Neutron Yield (10^{10} neutrons/sec)	
	5 Mev	10 Mev
Be	1.4	9.5
Li	1.0	5.0
B	0.70	5.3
C	0.098	0.93
O	0.016	0.58

[63] W. J. Ross, "Activation Analysis of High Purity Materials," *Anal. Chem.* **36**, 1114 (1964).

[64] S. S. Markowitz and J. D. Mahony, "Activation Analysis for Oxygen and Other Elements by Helium-3-Induced Nuclear Reactions," *Anal. Chem.* **34**, 329 (1964).

[65] A. C. Demildt, "Microactivation Analysis for Oxygen in the Actinide Metals," *Anal. Chem.* **35**, 1228 (1963).

neutron output (10-Mev ^{3}He) from Be, Li, and B targets had energies greater than 20 Mev, as determined by the $^{12}C(n,2n)^{11}C$ reaction. The above data show that a ^{3}He-beam generating machine could also be used as an effective neutron source for activation analysis. Results are encouraging, and work is being continued in this field.

12.8 14-Mev NEUTRON GENERATOR

12.8.*a* Output Spectrum from 14-Mev Neutron Generators; Its Rapid Estimation and Its Influence on Cross-Section Measurements and Activation Analysis

E. Ricci

Neutron generators that use the reaction $^3H(d,n)^4He$ are being employed extensively in 14-Mev neutron cross-section determinations and in activation analysis. The frequent assumption that the generated neutrons are monoenergetic is not always valid; consequently, the nuclear chemist must know the neutron spectrum to appraise his results and conclusions. A simple approximate method to estimate such spectra was developed. The neutrons impinging on a sample are divided into four groups, and the energy range of each of these groups is obtained from calculations of scattering; the corresponding neutron percentages result from probability considerations. The influence of the 14-Mev neutron spectrum on flux measurements, cross-section determinations, and activation analysis was studied. From these studies, means to appraise irradiation geometries and to calculate and correct for experimental errors were developed. A paper on this work was accepted for publication in the *Journal of Inorganic and Nuclear Chemistry*.[66]

[66]E. Ricci, "Output Spectrum from 14-MeV Neutron Generators: Rapid Estimation and Influence in Cross-Section Measurements and Activation Analysis," to be published in the *Journal of Inorganic & Nuclear Chemistry*.

12.8.*b* Catalog of 14-Mev Neutron Reactions

J. E. Strain W. J. Ross

The gamma-ray spectra and reaction cross sections for the $Z = 3$ through $Z = 30$ elements were published.[67] Work is continuing on the remaining elements ($Z > 30$).

12.8.*c* Titanium Irradiations at the 14-Mev Neutron Generator

E. Ricci

Work on (n,p) and $(n,np) + (n,d)$ cross sections of titanium isotopes[68] was resumed after the neutron spectrum produced by the new stainless steel target assembly of the generator was estimated. The calculation of the neutron spectrum was made by using the modified method reported in Sect. 12.8.*a* of this report. A suitable irradiation geometry was found at which 92% of the neutrons have energies of 14.8 ± 0.1 Mev. A test irradiation showed that enough data for the cross-section calculations can be obtained by running three successive 2-hr irradiations of double samples (natural Ti-^{47}Ti, natural Ti-^{49}Ti, and ^{47}Ti-^{49}Ti) sandwiched between aluminum flux monitors. This work is in progress.

12.8.*d* Determination of Oxygen with 14-Mev Neutrons

J. E. Strain

To increase the sensitivity of the determination of oxygen in metals with 14-Mev neutrons, an inert-gas system was fabricated. This system prevents sample contamination, reduces background, and permits the use of larger samples. It is possible to prepare the sample, irradiate it, and count the induced radioactivity without exposing the sample to either oxygen or moisture. The totally inert system makes possible the analysis of alkali metals without using sealed irradiation

[67]J. E. Strain and W. J. Ross, *14-Mev Neutron Reactions*, ORNL-3672 (in press, 1964).

[68]E. Ricci, "Irradiation of Titanium with 14-Mev Neutrons," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1963*, ORNL-3537, p. 100.

containers. The sensitivity expected for an 18-g sample is $\leq$10 ppm.

12.8.e Miscellaneous Uses of the 14-Mev Neutron Generator

J. E. Strain

Fast-neutron activation analysis was performed on a variety of samples for O_2, N, P, Mn, As, and Se. By the technique of delayed-neutron counting, the analysis of highly radioactive reactor fuel elements for residual fissionable materials was demonstrated. In this evaluation of the technique, the neutron generator is pulsed on for 10 sec and then is turned off; next, a high-efficiency $^{10}BF_3$ neutron counter is turned on to count delayed neutrons. The paraffin moderator, in which the counting tubes are embedded, serves to moderate the 14-Mev neutrons for activation and to thermalize the delayed neutrons for counting. The limit of detection was 0.1 mg of ^{235}U.

In cooperation with the Health Physics Division and the Isotopes Division, a program of evaluating tritium targets for neutron production was initiated. To date, Ti-^{3}H targets have been studied extensively. The optimum target of this type contains 4 mg of Ti per square centimeter and about 20 curies of ^{3}H in a 3.17-cm diameter. This combination produces a half-life of 1400 μa-hr and an initial neutron yield of 9×10^7 neutrons sec^{-1} μa^{-1} at a deuteron energy of 150 kev. Targets in which Y, Er, and Zr are used separately to absorb the ^{3}H are now being evaluated.

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13. Inorganic Preparations

M. T. Kelley D. E. LaValle
R. B. Quincy

The preparation of anhydrous fused or sublimed salts continued, but on a reduced scale. For the Ion Spectroscopy Laboratory of the Metals and Ceramics Division, $MgCl_2$, $SnCl_2$, $SbCl_3$, $NiCl_2$, NaCl, KCl, and RbCl were prepared in 100-g quantities, and some in multiple batches. For use in these solvent media, the following salts were provided: $V_2(SO_4)_3 \cdot xH_2O$, $LiBF_4$, $Ni(BF_4)_2 \cdot 8H_2O$, $V(BF_4)_3 \cdot xH_2O$, and Li_3PO_4. These compounds were prepared by either known methods or those described in previous reports.

Other anhydrous, fused, or sublimed salts were prepared for the High Temperature and Structural Chemistry Group of the Chemistry Division; they were SrI_2, BaI_2, YCl_3, $LaCl_3$, and KCl-LiCl eutectic. In other work for this group, K_2S in 30-g quantities was prepared by two different methods involving reaction of the elements. In one method, the vapors of sulfur were allowed to react with molten potassium at 200°C in a double-chambered, evacuated, sealed tube. In the other, finely ground sulfur was caused to drop in small portions into liquid potassium at 200°C in a tube whose outlet opened to a stream of inert gas. The products of both methods were essentially the same; their melting points (~945°C) were about 35°C higher than that previously reported for the product from the liquid ammonia method.[1] For the Inorganic and Physical Chemistry Group, pound quantities of high-purity KCl and LiCl were prepared, and for the Radiation Chemistry Group solid lithium hypobromite, stable at room temperature, was prepared successfully.

Series of preparations were made for the Salt Separation Chemistry of Protactinium from Irradiated Thorium Group of the Reactor Chemistry Division. These included multiple 100-g batches

[1] G. Brauer, ed. (R. F. Riley, translation ed.), *Handbook of Preparative Inorganic Chemistry*, 2d ed., vol. 1, p. 360, Academic, New York, 1963.

of $AlCl_3$. Fusion of this salt without explosion was finally achieved through the removal of all absorbed, extraneous, volatile materials such as chlorine and CCl_4 by sublimation through aluminum chips at 450°C. Like amounts of $ThCl_4$ and $ZrCl_4$ were prepared by repeated sublimations of commercially obtained materials. Also, 100 g of anhydrous $MgCl_2$ was prepared by the reaction of NH_4Cl on MgO and subsequent sublimation of the resultant product. Also for this group several preparations of UCl_3 were made by the reaction of HCl gas on UH_3. Purification of the products by the iodine sublimation method of Gregory[2] was attempted but was only partly successful. Finally, a product of 98% purity was obtained by use, with some refinements, of the apparatus described by Johnson, Butler, and Newton.[3]

In work for the Neutron Diffraction Studies Group of the Solid State Division, 20 g of DyP of practically 100% purity was made by a new[4] method in which dysprosium metal at 750°C in an alundum boat is exposed to phosphorus vapors in a sealed system. Preparatory to making pure HoS, an investigation of the holmium-sulfur system is under way because of the lack of information on this system in the literature. In other work for the Solid State Division, high-purity KCl is being produced on an almost routine basis for the Research and Development on Pure Materials Group.

For the High-Temperature Materials Group of the Metals and Ceramics Division, several more 10-g preparations of K_2O were made and like amounts of the niobates of lithium and potassium.

The work for the Physics Division was limited to the preparation of more alloys of the rare-earth metals and the treatment of various alloys of manganese with chromium, vanadium, and iron at 1000°C in highly purified hydrogen. Both types of work were for the Neutron Diffraction Group.

Miscellaneous preparations included: 50 g of $MoCl_5$ for the Physical Metallurgy Laboratory of the Metals and Ceramics Division, 5 g of IBr for the Tandem Routine Operations Group of the Physics Division, 10 g of ICl for the Characterization, Control, and Simulation of Accident-Released Fission Products Group of the Reactor Chemistry Division, and 10 g of Li_3Sb for the Radiation Detection Group of the Instrumentation and Controls Division.

[2]N. W. Gregory, *Preparation and Properties of the Uranium Halides*, MDDC-1755 (declassified Feb. 18, 1948).

[3]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).

[4]M. T. Kelley and D. E. LaValle, "Inorganic Preparations," *Anal. Chem. Div. Ann. Progr. Rept. Dec. 31, 1961*, ORNL-3243, pp. 77–78.

14. Organic Preparations

J. C. White

P. F. Thomason

H. L. Holsopple

Several organic compounds were custom synthesized to fill research needs of the Chemical Technology, Chemistry, and Analytical Chemistry Divisions. These compounds, prepared and purified on request, were not readily available from commercial sources.

14.1 COMPOUNDS PREPARED

14.1.*a* Methyl Isocyanide

Methyl isocyanide (25 g) was synthesized for the Chemical Separation of Isotopes Group of the

Chemistry Division. The compound was prepared by decomposing a solid complex of methyl iodide and silver cyanide with an aqueous solution of potassium cyanide. Methyl isocyanide was separated by distillation through a packed column and was purified by passing the vapors, under vacuum, through appropriate contaminant-removing reagents. Infrared spectroscopy showed that the synthesized material is essentially pure methyl isocyanide. This work is reported in detail elsewhere.[1]

14.1.*b* N,N′,N″-Trimethylborazine

A quantity of N,N′,N″-trimethylborazine was prepared as a new liquid-scintillator solvent to be used in thermal-neutron detection. The reaction of monomethylammonium chloride and lithium borohydride gives N,N′,N″-trimethylborazine according to the reaction

$$3CH_3NH_3Cl + 3LiBH_4 \rightarrow B_3N_3H_3(CH_3)_3 + 9H_2 + 3LiCl .$$

1,2-Bis-(2-methoxyethoxy)ethane was used uniquely as the solvent in which this reaction occurred. Elemental analysis of the product gave the following results (in weight percent):

Component	Theoretical	Found
Carbon	29.4	29.3
Hydrogen	9.87	9.67
Boron[a]	26.5	25.1

[a]Determined by neutron absorption.

The product was further characterized by its refractive index (1.4396) and boiling point (134°C), which agree closely with the values 1.4404 and 133°C, respectively, that are given in the literature.[2] The details of this preparation are given elsewhere.[3,4]

[1]H. L. Holsopple, *Synthesis of Methyl Isocyanide*, ORNL-TM-777 (Jan. 27, 1964).

[2]W. V. Hough *et al.*, "The Preparation, Identification and Characterization of N-Trialkylborazoles," *J. Am. Chem. Soc.* **77**, 864 (1955).

14.1.*c* 6-Amino-3,9-diethyltridecane

A long branched-chain primary amine was needed by the Chemical Technology Division in their extraction studies. To provide this, first a C_{17} alcohol, 3,9-diethyltridecanol-6, was oxidized to the corresponding ketone with chromic anhydride according to the general method of Mosher and Langerak.[5] Then this ketone was reacted with hydroxylamine hydrochloride and sodium carbonate to give 6-oxime-3,9-diethyltridecane (1.5 kg); the reaction was

$$2C_{17}H_{34}O + 2NH_2OH \cdot HCl + Na_2CO_3 \rightarrow 2C_{17}H_{34}NOH + 2NaCl + CO_2 + 3H_2O .$$

Vigorous refluxing of reactants for 1 hr was necessary to complete the preparation.

6-Amino-3,9-diethyltridecane was then prepared by submitting the oxime to hydrogenation over Raney nickel catalyst for ~1 hr at 125°C under 90 atm of hydrogen. The product was purified by fractional vacuum distillation and characterized by elemental analysis.

The hydrogenation apparatus consists primarily of an American Instrument Company shaker assembly and a bomb assembly cold-tested at 12,000 psi. High-pressure valves, tubing, and fittings were required to transmit hydrogen from the cylinder to the bomb assembly and back through a transducer to a continuous pressure recorder. Thermocouples linked the bomb assembly to a continuous temperature recorder. Power to heat the shaker assembly was supplied through a 20-amp Variac and was maintained by a single-set Wheelco temperature controller. Figure 14.1 is a photograph of the bomb assembly and its parts.

The shaker assembly rocks the bomb through an angle of ~30°, that is, the bomb placed on its side is rocked in such a way that one end is alternately 15° above and 15° below horizontal.

[3]H. L. Holsopple and L. E. Scroggie, *Synthesis and Infrared Absorption Spectra of N,N′,N″-Trimethylborazine*, ORNL-TM-860 (May 8, 1964).

[4]H. H. Ross and H. L. Holsopple, "N,N′,N″-Trimethylborazine as a Liquid Scintillator Solvent for Thermal Neutron Detection," *Nucl. Instr. Methods*, in press.

[5]W. A. Mosher and E. O. Langerak, "Oxidation of Some Aliphatic Alcohols with Chromic Acid," *J. Am. Chem. Soc.* **71**, 286 (1949).

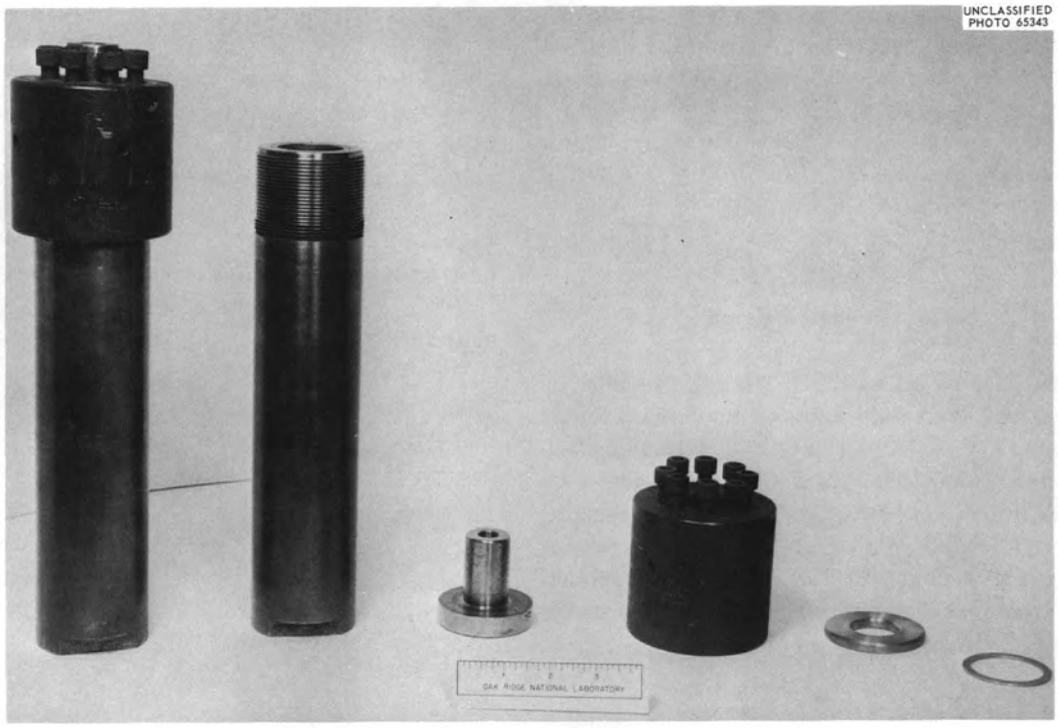

Fig. 14.1. Bomb Assembly and Its Parts.

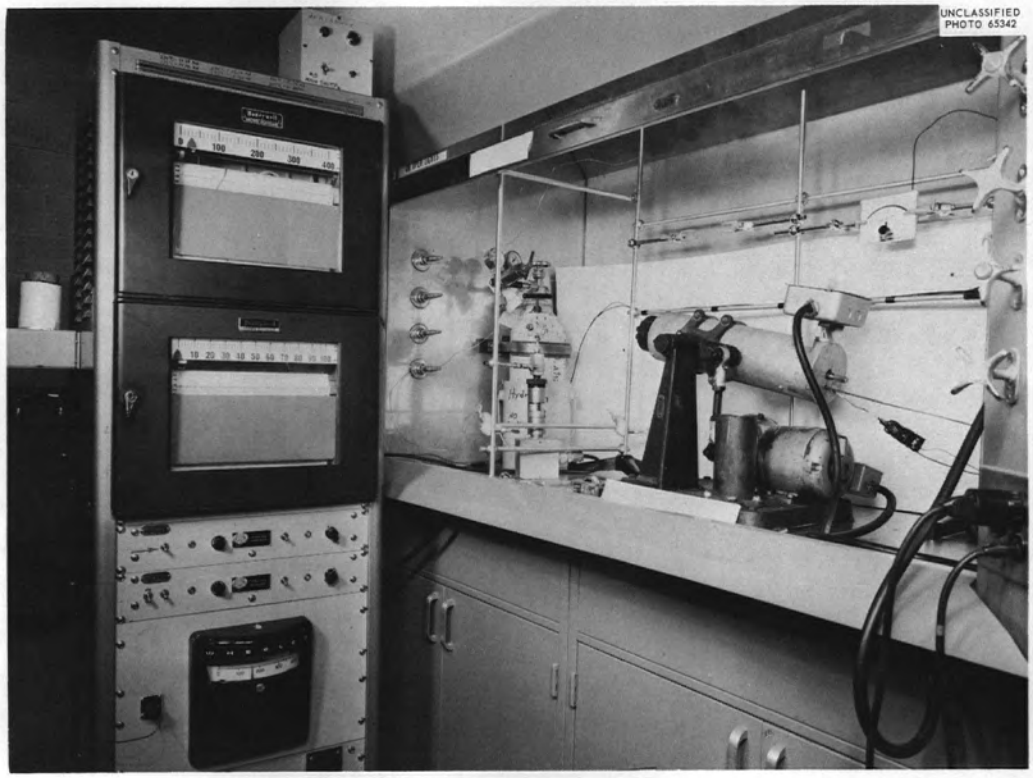

Fig. 14.2. Apparatus for Pressure Reactions.

The rate of oscillation varies from 36 to 38 oscillations/min. Movement is obtained from an $\frac{1}{8}$-hp 1725-rpm electric motor. The heater receptacle is integral with the shaker assembly, the temperature being controlled from the instrument panel. Figure 14.2 shows the apparatus ready for operation.

14.1.*d* Monoacid Esters

A new monoacid ester, monoheptyldecyltetrapropenylsuccinate, was prepared for the Chemical Technology Division. Heptyldecyl alcohol (0.5 mole) and tetrapropenylsuccinic anhydride (0.5 mole) were reacted to give 260 g of the ester. Monoheptyldecylmaleate (550 g) was prepared for the Chemical Technology Division by an analogous reaction between heptyldecyl alcohol and maleic anhydride.

14.1.*e* α,α'-Dipiperidyl

α,α'-Dipiperidyl (15 g) was synthesized for the Isotope Separations Group of the Chemistry Division. α,α'-Dipyridyl was reduced with hydrogen (platinum oxide as catalyst) in a 250-ml hydrogenation bomb at 100 atm and 150°C. A moderate excess of 5 *N* HCl was used to dissolve the dipyridyl. The quantity of hydrogen used agreed with the absorption of 12 hydrogen atoms. The solution that remained after the reduction was separated from the platinum and made basic; the dipiperidyl was extracted into diethyl ether. The ether was removed at reduced pressure, and the liquid α,α'-dipiperidyl was filtered through a fine-fritted funnel.

The phenyl isothiocyanate addition product, $C_{10}H_{20}N_2 \cdot 2C_6H_5NCS$, of α,α'-dipiperidyl was crystallized from ethyl alcohol and had a melting point of 178°C, which is the same as the literature value.[6] Elemental analysis of the α,α'-dipiperidyl gave the following results (in weight percent):

Component	Theoretical	Found
Carbon	71.4	71.4
Nitrogen	16.6	16.0

[6]C. R. Smith, "Dipiperidyls," *J. Am. Chem. Soc.* **50**, 1936 (1928).

14.2 DETERMINATION OF ALDEHYDES AND KETONES

An acidimetric method for the determination of aldehydes and ketones with hydroxylamine hydrochloride and pyridine as reagents was used successfully on carbonyl samples from the Chemical Technology Division. Pyridine forces the formation of oxime to completion and renders the initial reaction mixture neutral to bromophenol blue, thus eliminating the need of a blank titration.

14.3 PURIFICATION OF α-METHYLNAPHTHALENE

α-Methylnaphthalene was purified by fractional distillation for use in studies being made by the Radiochemistry Group of the Analytical Chemistry Division.

14.4 CURRENT PROGRAMS

High-purity ^{10}B diborane is needed by the Physics Division. Boron trifluoride enriched to 96% ^{10}B will be reacted with lithium aluminum hydride in 1,2-bis-(2-methoxyethoxy)ethane as solvent; the ^{10}B diborane will be collected by fractional condensation.

Octyldecyl sulfoxide may be prepared by treating an aqueous alcoholic solution of octyldecyl chloride with sodium sulfide to form octyldecyl sulfide, which can then be oxidized to the sulfoxide with 30% hydrogen peroxide or chromic anhydride.

Preliminary studies have been made of the feasibility of preparing a quantity of chlorophosphonazo III. This reagent, a structural analog of arsenazo III, is of interest to the analytical service groups for possible use in the photometric determination of calcium and magnesium. The possibility of synthesizing other azo-type chromogenic reagents such as dibromoarsenazo II and lumogallion will be investigated.

Syntheses of tetra(perfluorovinyl) tin and perfluoroacrylic acid are being attempted. The

vinyltin compound is being prepared by reacting bromotrifluoroethylene with tin chloride and magnesium in tetrahydrofuran. The Grignard reagent prepared from bromotrifluoroethylene reacts according to the equation

$$4CF_2{=}CFMgBr + SnCl_4 \rightarrow (CF_2{=}CF)_4Sn + 2MgBr_2 + 2MgCl_2 .$$

Perfluoroacrylic acid will be prepared from the same Grignard reagent.

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	Finance and Materials	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	Armour	General Electric	Miscellaneous	Savannah River Laboratory	Special Projects	Westinghouse	Y-12	Total
Mass Spectrometry Analyses	2	4	12	50	2,484	440	4	1610		50		120	7,400	1,851	4	18	230		4,579	4,158	10	34			972				454	24,486
Optical and Electron Microscopy	3	2	18		176	73				41		5	21	110		3	193		114	340	6								24	1,129
Process Analyses																														
General Analyses Laboratory	9	17	15	93	29,604	891	33		467	733		4	212	7,365	85	643	34	285	2,129	3,182	402	82			14					46,299
General Hot Analyses Laboratory	11	13			14,774					55			876	58		3230			223	1,472			9					101		20,822
High-Level Alpha Radiation Laboratory	3	4			9,902					5			545							25						12		47	21	10,564
Materials Testing Laboratory	5	10	45	1	11,497	695	2		20	211	3		118	1,204	25	28	19	138	1,228	4,620	18									19,887
Radioisotopes-Radiochemistry Laboratory	9	18	164	39	7,559	26			28	3,838			11,736	1,828	153	5861			2,848	13,042	505			1019	51					48,724
Reactor Engineering Services Analytical Chemistry Laboratory	1	3	6		114	30				258	16	9	12	519		25			3,404	1,185		6							116	5,704
Spectrochemical Laboratory	2	2	200		625	85	59		430				10,856	327	50		518		1,130	2,177	976	50							614	18,101
X-Ray and Spectrochemical Analyses	9	6	142	5	5,451	861			4094	15,076	30		654	1,666		16	76	30	714	1,344	464									30,638
Total	54	79	602	188	82,186	3101	98	1610	5039	20,267	49	138	32,430	14,928	317	9824	1070	453	16,369	31,545	2381	172	9	1019	1037	12		148	1229	226,354

15. Quality Control

C. K. Talbott Bobby G. Eblen G. R. Wilson

An improvement occurred in the overall quality level of the analytical work for the 12-month period July 1963 through June 1964. The overall quality level increased from 94% to 96.5%; the number of control results increased 15%. The marked improvement in the quality level in the General Hot Analyses Laboratory is due mostly to the increase in the number of coulometric determinations of uranium. The 2S, or 95% confidence, limit for this determination is now at 1% and should be reduced to 0.5%. The number of control programs decreased by one as a result of the discontinuation of the colorimetric determinations of chloride and copper and the addition of the volumetric determination of cadmium.

Table 15.1 shows the distribution of these control tests by laboratories. The number of control tests and their specific characteristics are shown more appropriately in Table 15.2.

Table 15.1. Distribution by Laboratories of Control Tests for July 1963 Through June 1964

Laboratory	Number of Control Results		Quality Level (%)[a]	
	Total	Outside Fixed Limits	1963	1964
General Hot Analyses	1395	61	88	95.6
Materials Testing	1262	49	97	96.1
General Analyses	2149	69	94	96.8
Reactor Engineering Services	379	10	96	97.4
Total	5185	Total 189	Av 94	Av 96.5

[a]Control results within prescribed 2S limits.

Table 15.2. Distribution by Methods of Control Results for July 1963 Through June 1964

Type of Method	Constituent	Number of Control Programs		Number of Control Results	
Colorimetric (spectrophotometric)	Aluminum	1		173	
	Chromium	2		158	
	Iron	2		261	
	Manganese	1		53	
	Mercury	1		8	
	Molybdenum	1		85	
	Nickel	3		200	
	Niobium	1		174	
	Thorium	4		473	
	Uranium	3	19	720	2305
Coulometric	Uranium	2	2	1228	1228
Fluorometric	Uranium	3	3	470	470
Gravimetric	Carbon	3		589	
	Molybdenum	1		27	
	Nickel	1		56	
	Silicon	1	6	4	676
Potentiometric	Uranium	1	1	21	21
Volumetric	Aluminum	1		132	
	Cadmium	1		3	
	Chromium	1		60	
	Nitrate	2		117	
	Phosphorus	1		54	
	Sulfate	1		69	
	Sulfur	1	8	50	485
Total			39		5185

16. Mass Spectrometric Analysis

A. E. Cameron J. R. Sites

The Mass Spectrometry Laboratory continued to analyze sweep gas from experiments in the ORR, helium from trailers for the liquid-helium plant and the Reactor Chemistry Division, special argon from cylinders for ORNL Stores, fission product off-gases for the Solid State Division, rare-gas products of the thermal-diffusion plant, argon-helium mixtures for the Kilorod Program, and special gas mixtures for several divisions. The enriched ^{14}C content of some CH_4 was determined for the Isotopes Division, as were the chemical purity and isotopic composition of young fission product Kr and Xe. The accelerator tank gas for three Van de Graaff generators was analyzed for the High Voltage Laboratory. The ^{18}O content of CO_2 samples was measured for the Biology Division. The CF_4, Kr, and Xe contents of radioactive trapped off-gases from a reactor capsule test in Idaho were determined. Various phases of the SNAP program were supported by certifying the purity of gas from cylinders and by analyzing the gaseous products of experiments. The carbon isotope ratios in acetylene and benzene samples were determined for ORINS.

The relative isotopic abundances were determined for uranium-inventory samples, uranium-burnup experiments, uranium fuel-element certification, and separated stable-isotope cyclotron targets. Radioactive samples of ^{10}Be, ^{36}Cl, ^{59}Ni, ^{90}Sr, ^{131}I, ^{147}Pm, and ^{151}Sm were analyzed. The Yb contaminant present at the parts-per-million level in a separated-calcium-isotope crystal was characterized.

A total of 950 samples were analyzed for the Stable Isotope Separation program. This work included the isotopic analysis of the following 34 elements: B, Ba, Ca, Cd, Cr, Cu, Er, Fe, Ga, Gd, Ge, Hf, Hg, Ir, K, Mg, Mo, Ni, Pb, Rb, Ru, S, Se, Si, Sm, Sn, Ta, Te, Ti, U, W, Yb, Zn, and Zr.

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17. Spectrochemical Analysis Laboratory

A. E. Cameron

J. A. Carter Zell Combs

About 1200 samples were analyzed in the Spectrochemical Analysis Laboratory, and over 18,000 results were reported. Thirty-three percent of the samples originated in the Isotopes Division; they included the radioisotopes ^{147}Pm and ^{151}Sm and the stable isotopes of B, Ba, Ca, Cd, Cl, Cr, Cu, Dy, Er, Ga, Gd, Hf, Ir, Fe, K, Pb, Li, Mg, Mo, Rb, Ru, Sm, Se, Si, S, Ta, Te, Tl, Sn, Ti, W, Yb, Zn, and Zr. Lithium fluoride accounted for 208 samples, 162 samples were metals for identification or specification testing, and over 100 samples contained beryllium.

The Wadsworth and Ebert spectrographs were relocated; as a result, one arc-spark stand serves both instruments. This arrangement provides wavelength coverage sufficient to determine 60 to 65 trace elements with one exposure; previously, three exposures were required.

18. Process Analyses

L. T. Corbin

18.1 HIGH-LEVEL ALPHA RADIATION LABORATORY

J. H. Cooper

The High-Level Alpha Radiation Laboratory reported 10,557 results during the past year. Of these, 95% were for the Chemical Technology Division and 5% were for the Isotopes Division.

The usual procedure for determining ^{231}Pa alpha radioactivity is to measure the gross alpha activity and to determine the percent of ^{231}Pa alpha activity by alpha pulse-height analysis. Whenever dissolved solids prevent the preparation of good counting plates or whenever high ^{232}U content masks the ^{231}Pa alpha peak in alpha spectrometry, an alternative procedure must be followed. Uranium and iron are extracted from 9 *M* HCl into either diisobutylcarbinol or tributyl phosphate and are separated from the Pa if the Pa is first complexed with fluoride ion. If no solids are present, an aliquot of the aqueous phase can then be counted and pulsed for ^{231}Pa. If solids are present, fluoride can be complexed with borate and the Pa extracted into diisobutylcarbinol, counted, and pulsed.

The Savannah River Laboratory was assisted in a study of the penetration of plutonium into the aluminum cladding of fuel elements. Small chips of the aluminum were analyzed by extracting the plutonium into TTA and counting the alpha radioactivity. Anion exchange separation of the plutonium did not give quantitative recovery, but two successive extractions with TTA did.

Samples of plutonium sols were analyzed for ammonia and nitrate. A micro Kjeldahl distillation apparatus was installed in a glove box, and the nitrogen was distilled from samples of plutonium that contained several microcuries of alpha activity.

18.2 GENERAL ANALYSES LABORATORY

W. R. Laing

A total of 46,273 results were reported during the past year. Several new or modified methods were placed in service. A Leco inert-gas-fusion apparatus was used to determine oxygen in uranium nitride. Cesium was extracted from a solution of its ores into 4-sec-butyl-2-(α-methylbenzyl)phenol (BAMBP), stripped with 0.25 *N* HCl,[1] and measured by flame spectrophotometry. A WO_3 (40%)–Na_2WO_4 (60%) flux was used to determine F^- in solids and solutions that contained Ta. Gas-release tests were made on sol-gel thoria at temperatures up to 2200°C. The upper pressure for permeability measurements was extended from 1.5 atm to 6.0 atm. Work continued on the determination of free HF in solutions that contain $H_4NbO_2F_5$ and HF; thorium was used as the titrant, but the trisodium salt of 4,5-dihydroxy-3-(*p*-sulfophenylazo)-2,7-naphthalenedisulfonic acid (SPADNS) was found to be a better indicator than Thoron.

Several methods recently put into service have resulted in considerable cost savings. A Technicon AutoAnalyzer was used to determine both uranium and the synthetic detergent, ABS. For the determination of uranium, it was operated at a rate of 60 samples/hr, with a relative standard deviation slightly less than that obtained by standard spectrophotometry. The operating rate for the ABS determination was 20 samples/hr, compared with 4 samples/hr when done by hand. The relative standard deviation was the same in both cases.

In many samples, a volumetric EDTA titration was used in place of gravimetry to determine thorium; use of the volumetric method resulted in a 50% saving of time.

Location of another vendor of crucibles, funnels, and stoppers for the vacuum-fusion apparatus has resulted in a 45% reduction in the cost of these supplies.

A more rapid method for the measurement of coating densities of AVR fuel particles was developed. The density of the coated particle is measured; then the particle is burned in oxygen, and the CO_2 and oxide residue are weighed. From

[1] W. J. Ross and J. C. White, "Determination of Cesium and Rubidium After Extraction with 4-sec-Butyl-2(α-methylbenzyl)phenol," *Anal. Chem.* 36, 1998 (1964).

a knowledge of the density of the core material, the density of the coating is calculated.

Computer programs were coded for pycnometer density, total anion, permeability, and porosity measurements. The coding of programs for quality control and surface-area measurements is planned.

18.3 GENERAL HOT ANALYSES LABORATORY

C. E. Lamb

The preparation for handling, treating, and analyzing radioactive materials originating from new programs was continued as one of the major efforts by the General Hot Analyses Laboratory. Special programs requiring such work exist in the Reactor Chemistry, Reactor, and Chemical Technology Divisions. The programs include Curium Recovery, Power Reactor Fuel Processing, ^{231}Protactinium Cleanup from ^{232}Uranium Production, and Close-Coupled Process Development.

The initial phase of the development of methods and equipment for the MSRE program was completed. The work and prototype equipment were moved from the mockup area to the High-Radiation-Level Analytical Laboratory (HRLAL), and the training of personnel was started concurrently with the fabrication of equipment for use in the hot cells.

For analysis of samples from the Curium Recovery Program, several new methods with related equipment were selected and tested in a hot cell. They included the determination of aluminum[2] in the presence of rare-earth elements by titration with EDTA to a mixed-indicator (bromcresol–methyl red) end point, free acid[3] by potentiometric titration with LiOH in LiCl solution, chloride[4,5] by titration with $AgNO_3$ and by spectrophotometric measurement using mercuric chloranilate ($HgC_6Cl_2O_4$) as the chromogenic agent, nitrate[6] by spectrophotometric measurement using sulfanilic acid and α-naphthylamine, and rare-earth elements[7] in the presence of aluminum by EDTA titration to a xylenol orange end point.

At the request of the Power Reactor Fuel Processing Group, a quartz tube furnace[8] (Fig. 18.1) to be used within a hot cell was fabricated for the determination of carbon in graphite, pyrolytic-carbon-coated fuel particles, and solutions of dissolved carbon. The samples were burned in an oxygen atmosphere, and the gas was passed through a solution of $KMnO_4$ (when samples contained nitrates) and a trap of Drierite, and then absorbed in an Ascarite trap. The carbon content was calculated from the increased weight of the trap. The method was unsatisfactory for samples of low carbon content; the lower limit was not established.

In cooperation with the Metals and Ceramics and the Reactor Chemistry Divisions, a glass-and-platinum electrolysis assembly, supplied by J. L. Cook,[9] was tested in a hot cell for the disintegration of irradiated graphite spheres that contain particles of pyrolytic-carbon-coated uranium fuels. The spheres were electrolytically disintegrated in 8 *M* HNO_3 at 4 to 5 amp and 4 to 5 v; the disintegration required 12 to 24 hr. An analysis for the uranium in the supernatant liquid then made possible the calculation of the number of fractured particles leached by the nitric acid solution. Also for the Reactor Chemistry Division, graphite was dissolved completely in a hot cell under reflux conditions using fuming nitric-perchloric acid.

Materials submitted by organizations outside ORNL were analyzed; these included UO_2-ThO_2 and UO_2-ZrO_2 pellets from Westinghouse and a UO_2SO_4 solution from the I.I.T. Research Institute.

An Alpha Facility that consists of 65 lin ft of glove boxes was constructed in Building 3019. The Facility was designed for use in handling alpha-emitting materials removed from the hot

[2]H. Flaschka *et al.*, "The EDTA Titration: Applications II," *Chemist-Analyst* 47, 22 (1958).

[3]H. Kubota and D. A. Costanzo, "Reactions in Concentrated Lithium Chloride Solution: Determination of Free Acid and Hydrolyzable Cation," *Anal. Chem.* 36, in press (1964).

[4]F. J. Miller, "Chlorine (Chloride), Potentiometric Silver Nitrate Titration Method," Method Nos. 1 212020 and 9 00712020 (1-29-59), *ORNL Master Analytical Manual*; TID-7015, suppl. 2.

[5]J. E. Barney II and R. J. Bertolacini, "Colorimetric Determination of Chloride with Mercuric Chloranilate," *Anal. Chem.* 29, 1187 (1957).

[6]L. Procházková, "Bestimmung der Nitrate im Wasser," *Z. Anal. Chem.* 167, 254 (1959).

[7]H. Flaschka *et al.*, "The EDTA Titration: Applications IV," *Chemist-Analyst* 47, 78 (1958).

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[9]Metals and Ceramics Division.

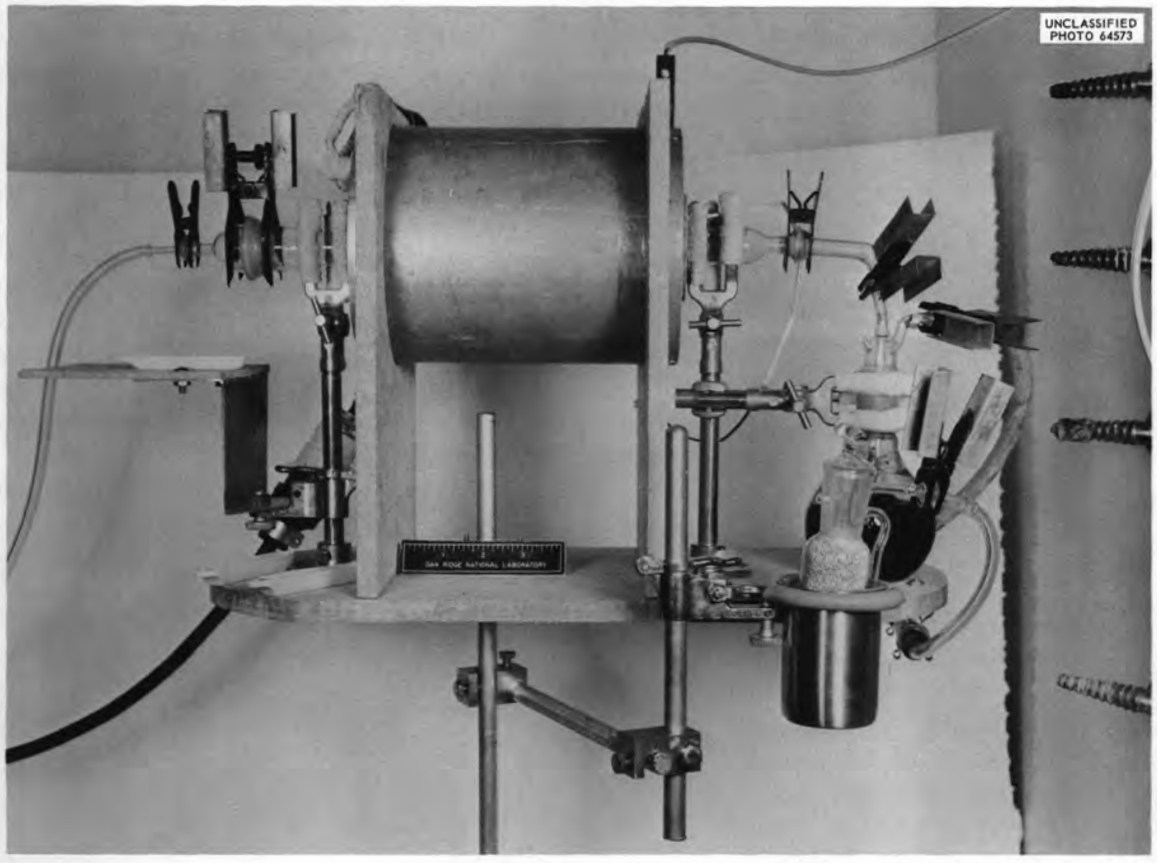

Fig. 18.1. Quartz Tube Furnace for Combustion of Carbon Samples.

cells after the radiation level had been reduced to a safe limit and also other samples low in total radiation but relatively high in alpha emitters.

Four alien guests (two from Germany, one from Italy, and one from France) were trained in analytical chemistry hot-cell operations.

18.4 MATERIALS TESTING LABORATORY

L. J. Brady

The Materials Testing Laboratory, with a staff of 15 employees, reported the results of about 19,900 analyses during the past year. Of this total, about 58% were for the Chemical Technology Division, 23% for the Reactor Chemistry Division, 6% for the Metals and Ceramics Division, and 6% for the Reactor Division.

Specification tests of the flush salts and charge material for the MSRE included the determination of major components as well as traces of iron, chromium, and nickel. In addition, oxygen and total sulfur were determined in many fused fluoride salt samples.

The determination of the thorium content of granite samples and of acid leach solutions was continued.

The rare-earth-element content of samples derived from extraction studies was determined, with arsenazo as the chromogenic agent. Calibration curves were prepared after solutions were tested that contained known amounts of the same mixture of rare-earth elements as those that were being tested in the extraction process. No preferential extraction of specific rare-earth elements was expected during the process.

Samples of potassium nitrate were analyzed for nitrite. They had been subjected to radiation in

the Van de Graaff generator, and the quantity of nitrite produced was used to estimate the energy input to the salts being tested.

Nitrogen was determined in refractory samples of uranium nitride and boron nitride. After the sample was dissolved in aqueous solution or by fusion with sodium hydroxide, the ammonia was distilled into a solution of boric acid and then titrated.

The carbonate content of samples of thorium oxide and rare-earth slurries or sols that contained less than 0.1% carbonate was determined by gas chromatography. The carbon dioxide was evolved from an acid solution of the sample and was swept into a sampling loop with helium. The loop was maintained at liquid-nitrogen temperature, and the solid carbon dioxide was retained in the capillary loop by a section of powdered glass held in place with small plugs of glass wool. After the carbon dioxide was collected, the stopcocks on the sampling loop were closed, and the loop was allowed to warm to room temperature. The gaseous carbon dioxide was then measured with a gas chromatograph.

A number of samples of tellurium that had been collected on nickel-wool filters were analyzed to determine their tellurium content. The entire sample was dissolved in acid under reflux. The tellurium was precipitated from solution by reducing it to the metal with sulfur dioxide and hydrazine hydrochloride. The tellurium metal was filtered from the solution, dried, and weighed. Precautions were necessary to prevent loss of tellurium during the dissolution.

For the Plant and Equipment Division, a series of compatibility tests was conducted. They were designed to determine the tendency of various lubricating oils to form highly viscous sludge when kept in contact with antifreeze mixtures based on ethylene glycol. Attempts were being made to circumvent difficulties encountered in the past when antifreeze was allowed to come in contact with the oils lubricating engines used to drive air compressors. Some of these engines had been damaged as the result of abnormal wear on the bearings.

The carbon and nitrogen contents of a number of dietary foods and of plant materials were determined for the Health Physics Division; macrocombustion methods were used.

18.5 RADIOISOTOPES-RADIOCHEMISTRY LABORATORY

E. I. Wyatt

The Radioisotopes-Radiochemistry Laboratory had an increase in personnel and in the volume and variety of analytical work. All the intermediate-level radiochemical work is now performed in Building 9204-1 instead of Building 3550. The field of low-level radiochemical analysis now includes tritium determinations. Gamma-ray spectrometric analysis was expanded to include the hot-cell work in Building 3525. The use of computer programs to resolve spectrometer data was increased, the objective being to resolve complex gamma-ray spectra from the Curium, the TRU, and the Nuclear Safety Pilot Plant Programs. A second anticoincidence beta proportional counter and an additional multichannel analyzer were acquired. The latter is being used in conjunction with a lithium-drifted germanium diode detector to obtain better resolution of gamma spectra than is given by NaI detectors.

The mass distribution of the fission products formed by the epicadmium-induced fission of ^{237}Np was measured. The fission products were determined radiochemically. A report of this work is being prepared for publication. The half-lives of ^{141}Ce and ^{144}Ce were measured by means of a beta proportional counter; values of 31.9 ± 0.3 and 284.2 ± 1.0 days, respectively, were obtained.

18.6 HIGH-RADIATION-LEVEL ANALYTICAL FACILITY (HRLAF)

L. G. Farrar

In the High-Radiation-Level Analytical Facility (HRLAF) contamination difficulties in handling dilutions removed from the cells for hot-laboratory work made necessary the use of rigid monitoring techniques for all materials removed from the hot cells. The number of radiation-monitoring instruments used in the HRLAF has almost doubled over the past year.

Two hot cells sealed in glove-box fashion for work with high-level alpha-emitting materials were

operated successfully, with minor modifications for access and viewing. The two containment cells were used to separate ^{233}U from its daughter nuclides, to separate americium and curium from fission products, and to clean up 45 g of ^{231}Pa. These operations involved the use of ion exchange, solvent extraction, precipitation, and filtration under hot-cell and glove-box conditions.

The radioactivity of samples received in the HRLAF over the past year has increased by a factor of at least 10. Because of the unusual nature of recent samples, longer storage of hotter samples has caused the breakdown of the gasket material in the viewing window of the storage cell. Under irradiation, the gasket material yields a phosphate compound that precipitates as lead phosphate in the oil of the viewing window and thus causes cloudiness. The oil has been changed twice to reduce cloudiness, but it is expected that a complete cleaning of the window will soon be necessary.

High-level contamination has been experienced in cells used to check MSRE analytical procedures with irradiated salt samples. Additional containment of crushed salt samples is being used in an effort to decrease general contamination in the cell.

More frequent changes of manipulator boots resulted in a decrease in the alpha contamination of manipulators. A manipulator-decontamination facility was completed; it will greatly facilitate the repair of contaminated manipulators. During routine maintenance of manipulators, all units were fitted with quick-disconnect replaceable master-slave hands and relays to decrease manipulator repair time. Routine maintenance in the HRLAF has included repairs to 123 manipulators and the decontamination of 11 work cells.

Greater use is being made of transfer carriers to decrease contamination of carriers shipped to other hot facilities at ORNL. An effort has been made to standardize transfer carriers as much as possible.

Spread of fission products as a result of the operation of a flame photometer in cell 6 of the HRLAF was diminished by separating the fission products from the samples before the sample is burned in the cell.

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19. Reactor Engineering Service Laboratory

A. E. Cameron

C. K. Talbott

The Reactor Engineering Service Analytical Chemistry Laboratory made ~5700 analyses on about 1160 samples. Of these, about 50% were submitted by the Reactor Division, 30% by the Reactor Chemistry Division, 8% from the Metals and Ceramics Division, and the remainder were derived from ten other sources. The samples consisted principally of water; high-alloy steel; solutions of HCl, HNO_3, KOH, $CdSO_4$, $CdCl_2$, $Cd(NO_3)_2$, $Sm_2(SO_4)_3$, and low-level waste; Al; bromates of Ba, Ca, and Sr; and corrosion scale samples. Analyses were made less frequently on a variety of materials such as ThO_2, UO_2, gas samples, plant material, and plastic and insulation products. About 160 cylinders of mixed gases were prepared during this period. Methods of analyses included volumetric analysis, spectrophotometric and potentiometric titrations, gravimetry, thermogravimetry, spectrophotometry, and gasometric analysis. Physical measurements were made on many of the samples; the measurements included pH, conductance, particle-size distribution, specific gravity, and zeta potential.

Part C. ORNL Master Analytical Manual

20. ORNL Master Analytical Manual

M. T. Kelley

Helen P. Raaen

20.1 CUMULATIVE INDEXES TO THE *ORNL MASTER ANALYTICAL MANUAL*

Cumulative indexes to the *ORNL Master Analytical Manual* were first issued in 1963. The purpose of the indexes and the arrangement of their content has been explained.[1] The indexes were updated to indicate the changes made in the content of the *Manual* during 1963. The updated cumulative indexes are entitled "Indexes to the *Oak Ridge National Laboratory Master Analytical Manual* (1953–1963), TID-7015 (Indexes), Revision 1." The document is available from the Office of Technical Services, United States Department of Commerce, Washington, D.C. 20230; the price is $2.50. The indexes were issued as a companion document to Supplement 6 of TID-7015.

The computer work required for the preparation of the updated indexes was supervised by Ann S. Klein.[2]

20.2 MAINTENANCE OF THE *ORNL MASTER ANALYTICAL MANUAL*

The sixth supplement to the reprinted form of the *ORNL Master Analytical Manual* was published. It contains the new methods and the revisions issued in 1963. This supplement, designated TID-7015 (Suppl. 6), is available from the Office of Technical Services, Department of Commerce, Washington, D.C. 20230, at a price of $5.00.

The new Section 4, "Mass Spectrometric Methods," and also the Subsections 9 02, "Molten-Salt Reactor Experiment (MSRE) Methods," and 9 06, "Transuranium (TRU) Process Methods," were added to the *Manual*. Section 4 is a reprinted form of ORNL-3528, *Isotopic Mass Spectrometry of the Elements*, by E. J. Spitzer and J. R. Sites; it consists primarily of one-page guide sheets for the mass spectrometric analysis of 64 elements. Subsections 9 02 and 9 06 contain four and ten methods, respectively, that are being evaluated for use in the MSRE and TRU work.

Altogether, 106 new methods were added to the *Manual*; 4 of these were for the purpose of record only. Revisions were made to 46 methods; 1 obsolete method was 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, methods-writing assignments were made to persons in the Analytical Chemistry Division.

[1] Helen P. Raaen, "Cumulative Indexes to the *ORNL Master Analytical Manual*," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1963*, ORNL-3537, p. 129.

[2] Technical Information Division.

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
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2	Dyer, F. F.	"Reactor Neutron Flux: Characteristics and Uses," pp. 14–32 in *Guide to Activation Analysis*, ed. by W. S. Lyon, Jr.	Van Nostrand, Princeton, N.J., 1964
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4	Hahn, R. L.	"Radiation Detectors and Counting Statistics," pp. 67–79 in *Guide to Activation Analysis*, ed. by W. S. Lyon, Jr.	Van Nostrand, Princeton, N.J., 1964
5	Koirtyohann, S. R., 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
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8	Ricci, E.	"Practical Examples of Activation Analysis," pp. 116–42 in *Guide to Activation Analysis*, ed. by W. S. Lyon, Jr.	Van Nostrand, Princeton, N.J., 1964
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[1]Present address: Union Carbide Corp., Nuclear Division, Tuxedo, N.Y.
[2]Alcohol and Tax Unit, Treasury Department, Washington, D.C.
[3]Chemistry Department, Temple University, Philadelphia, Pa.
[4]Temporary alien employee, South African Atomic Energy Board, Pretoria, South Africa.

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36	Horton, C. A., B. C. Pal[6]	"The Structure of 3-Methyladenine, and Methylation of 6-Dimethylaminopurine"	*J. Chem. Soc.* **1964**, 400
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[5]Scripps Institute of Oceanography, LaJolla, Calif.

[6]Research participant in the Biology Division from Chemistry Department, Tuskegee Institute, Tuskegee, Ala.

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[7]Chemistry Department, University of Tennessee, Knoxville.

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[13]Present address: Jarrell-Ash Company, Waltham, Mass.

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73	Burns, J. F.	*Trip Report, July 16–Aug. 15, 1963*	Sept. 12, 1963 (unpublished report)
74	Cameron, A. E.	*Report of Foreign Travel – July 1–30, 1963*	Aug. 19, 1963 (unpublished report)
75	Carter, J. A., W. S. Pappas,[14], R. R. Wright,[14] C. W. Weber[14]	"Preparation and Recovery of Cesium Compounds" in *The Joint National Institutes of Health–Atomic Energy Commission Zonal Centrifuge Development Program Semiannual Report for Period July 1–December 31, 1963*	ORNL-3656, p. 49 (August 1964)
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79	Holsopple, H. L., Lucy E. Scroggie	*Synthesis and Infrared Absorption Spectra of N,N′,N″-Trimethylborazine*	ORNL-TM-860 (May 8, 1964)
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[14]ORGDP, Oak Ridge, Tenn.

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86		*Statistical Quality Control Report, January Through June, 1964*	Aug. 12, 1964 (unpublished report)
87		*Statistical Quality Control Report, Analytical Chemistry Division, January Through September, 1963*	Nov. 11, 1963 (unpublished report)
88		*January Through December, 1963 – Statistical Quality Control Report, Analytical Chemistry Division*	Feb. 18, 1964 (unpublished report)
89	Lyon, W. S.	"Research on Activation Analysis Problems" in *Isotopes Technology Development Program*	TID-7681, p. 13 (February 1964)
90		"Electronic Resolution of Gamma-Ray Spectra" in *Isotopes Technology Development Program*	TID-7681, p. 17 (February 1964)
91	Manning, D. L.	*Report of Foreign Travel*	Oct. 26, 1964 (unpublished report)
92	Raaen, Helen P. (ed.)	*Annual Progress Report for Period Ending November 15, 1963, Analytical Chemistry Division*	ORNL-3537 (Feb. 10, 1964)
93		*Oak Ridge National Laboratory Master Analytical Manual, Supplement 6*	TID-7015, suppl. 6 (June 1964)
94	Raaen, Helen P.	*Report on Foreign Travel*	Jan. 17, 1964 (unpublished report)
95	Raaen, Helen P., R. P. Clark*	*Fabrication of the Vertical-Orifice Teflon Capillary of a Teflon Dropping-Mercury Electrode*	ORNL-3654 (August 1964)
96	Raaen, Helen P., Ann S. Klein* (eds.)	*Indexes to the Oak Ridge National Laboratory Master Analytical Manual (1953–1963)*	TID-7015, Indexes, revision 1
97	Reynolds, S. A.	"Radioisotopes in Alkaline-Earth Analysis" in *Second Annual Oak Ridge Radioisotope Conference, April 19–22, 1964*	TID-7689, p. 81 (April 1964)
98	Ross, H. H.	"A Unique Scintillator for Neutron Detection Using Lithium-6 Enriched Isotope" in *Isotope Technology Development Program*	TID-7681, p. 15 (February 1964)
99	Ross, H. H., R. E. Yerick[10]	"A Unique Scintillator for Neutron Detection" in *Second Annual Oak Ridge Radioisotope Conference, April 19–22, 1964*	TID-7689, p. 103 (April 1964)
100	Scroggie, Lucy E.	*A Literature Survey of Sensitive Methods for the Determination of Dissolved Gases and Impurities in Reactor Coolant Water*	ORNL-TM-724 (Nov. 11, 1963)

101	Shaffer, J. H.,* W. R. Grimes,* G. M. Watson,* D. R. Cuneo,* J. E. Strain, M. J. Kelly*	"Recovery of Uranium and Protactinium from Molten Fluoride Systems by Precipitation as Oxides" in *Proceedings of the Protactinium Chemistry Symposium, Gatlinburg, Tenn., April 25–26, 1963*	TID-7675, p. 86 (February 1964)
102	Strain, J. E., A. P. Litman*	*Trip Report to General Atomic, September 28, 1964*	Oct. 30, 1964 (unpublished report)
103	Spitzer, E. J., J. R. Sites	*Isotopic Mass Spectrometry of the Elements*	ORNL-3528 (Dec. 3, 1963)
104	White, J. C.	*Determination of Traces of Iron(III) in NaF-LiF-KF-UF_4 with Thiocyanate*	ORNL-TM-742 (Sept. 20, 1955)
105		*Analytical Chemistry Research and Development Group Monthly Summary – December, 1963*	Jan. 9, 1964 (unpublished report)
106		*Analytical Chemistry Research and Development Group Monthly Summary – January, 1964*	Feb. 7, 1964 (unpublished report)
107		*Monthly Summary – February, 1964, Analytical Chemistry Research and Development Group*	Mar. 17, 1964 (unpublished report)
108		*Analytical Chemistry Research and Development Group Monthly Summary – March, 1964*	Apr. 17, 1964 (unpublished report)
109		*Monthly Summary – April, 1964, Analytical Chemistry Research and Development Group*	May 11, 1964 (unpublished report)
110		*Analytical Chemistry Research and Development Group Monthly Summary – May, 1964*	June 16, 1964 (unpublished report)
111		*Analytical Chemistry Research and Development Group Monthly Summary – June, 1964*	July 10, 1964 (unpublished report)
112		*Analytical Chemistry Research and Development Group Monthly Summary – July, 1964*	Aug. 7, 1964 (unpublished report)
113		*Monthly Summary – August, 1964, Analytical Chemistry Research and Development Group*	Sept. 8, 1964 (unpublished report)
114		*Analytical Chemistry Research and Development Group Monthly Summary – September/October 1964*	Nov. 6, 1964 (unpublished report)
115	Young, J. P.	*Report of Foreign Travel*	Oct. 28, 1964 (unpublished report)

New Methods Issued to *ORNL Master Analytical Manual*

	AUTHOR(S)	TITLE	NUMBER(S)	DATE
116	Apple, R. F.	"Dissolution of Irradiated, High-Fired, Refractory Nuclear Materials"	1 0902 9 00902	4-16-64
117		"Zirconium in MSRE Fuel, Amperometric Cupferron Titration Method"	9 021201	4-15-64
118		"Chromium in MSRE Fuel, Amperometric Ferrous Sulfate Titration Method"	9 021202	6-24-64
119		"Fluoride in MSRE Fuel, Pyrolysis Method"	9 021203	7-6-64

120		"Uranium in MSRE Fuel, Polarographic Method"	9 021204	7-13-64
121	Brady, L. J.	"Nitrate (Nitrogen), Volumetric Ferrous Ammonium Sulfate Method"	1 215671	10-14-63
122	Hill, F. M., F. L. Layton	"Carbon in Metals and Metallic Oxides, Leco Conductometric Method"	1 211793 9 00711793	6-27-63
123	Horton, A. D.	"C_2 Through C_5 Paraffins and Olefins, Gas Chromatographic Method"	1 221003 9 00721003	10-7-63
124		"C_4 Through C_8 Paraffins and Olefins, Gas Chromatographic Method"	1 221004 9 00721004	10-3-63
125	Kubota, H.	"Niobium, Spectrometric Hydroquinone Method"	1 215510 9 00715510	3-24-64
126	Layton, F. L.	"Sulfur (Sulfide and Nonsulfide), Indirect Polarographic Method"	1 218123 9 00718123	10-10-63
127	Lynn, E. C., F. M. Hill	"Aluminum and Thorium, Volumetric Disodium Dihydrogen Ethylenediaminetetraacetate (EDTA) Method"	1 210272 1 218772 9 00710272 9 00718772	4-22-64
128	Lyon, W. S.	"Aluminum Absorption Studies"	2 0915 5 10105 9 0733004	9-10-63
129		"Gamma Ionization Chamber"	2 00360 5 00360 9 00360	9-12-63
130		"Geiger-Mueller Counter"	2 00355 5 00355 9 00355	9-10-63
131	Manning, D. L.	"Thorium, Amperometric Disodium Dihydrogen Ethylenediaminetetraacetate—Iron(II) Indicator Method"	1 218773 9 00718773	7-13-64
132	Moore, F. L.	"Gross Alpha Radioactivity in Transuranium (TRU) Process Solutions, Direct-Measurement Method"	9 062301	6-30-64
133		"Gross Alpha Radioactivity in Transuranium (TRU) Process Solutions, Lanthanum Fluoride Precipitation—Proportional Alpha Counting Method"	9 062302	6-30-64
134		"Gross Alpha Radioactivity in Transuranium (TRU) Process Solutions, Triisooctylamine (or Tricaprylamine) Extraction—Proportional Alpha Counting Method"	9 062303	6-30-64
135		"Alpha Spectrometry of Transuranium (TRU) Process Solutions, Silicon Diode Detector Method"	9 062304	7-1-64
136		"Americium in Transuranium (TRU) Process Solutions, Ammonium Persulfate Oxidation—Proportional Alpha Counting Method"	9 062305	7-1-64
137		"Plutonium Alpha Radioactivity in Transuranium (TRU) Process Solutions, Lanthanum Fluoride Precipitation—Proportional Alpha Counting Method"	9 062306	7-1-64

138		"Plutonium Alpha Radioactivity in Transuranium (TRU) Process Solutions, Thenoyltrifluoroacetone (TTA) Extraction—Proportional Alpha Counting Method"	9 062307	7-2-64
139		"Transplutonium Alpha Radioactivity in Transuranium (TRU) Process Solutions, Triisooctylamine Extraction—Proportional Alpha Counting Method"	9 062308	7-2-64
140		"Transplutonium Alpha Radioactivity in Transuranium (TRU) Process Solutions, Lanthanum Fluoride Precipitation—Proportional Alpha Counting Method"	9 062309	7-2-64
141	Morton, J. E.	"Mercury-197m_2, 197, Product Analysis Guide"	9 0733492 9 0733804	6-22-64 4-13-64
142		"Strontium-89 and Strontium-90 in Aqueous Solutions, Di-2-Ethylhexylphosphoric Acid—Amsco Extraction Method"		
143	Parker, H. A.	"Bismuth-210, Product Analysis Guide"	9 0733111	6-19-64
144		"Thulium-170, Product Analysis Guide"	9 0733882	6-22-64
145	Parker, H. A., J. E. Morton	"Calcium-47, Product Analysis Guide"	9 0733152	6-19-64
146	Raaen, Helen P.	"Preparation of Computer Input Information for Annual Revision to Cumulative Indexes for the *ORNL Master Analytical Manual*"	07	4-10-64
147	Reynolds, S. A.	"Gamma Scintillation Counter"	2 00365 5 00365 9 00365	5-17-63
148		"Decay Studies"	2 0914 5 10104 9 0733003	5-22-63
149	Rickard, R. R.	"Iodine Adsorbed on Charcoal, Neutron Activation Analysis (Isotopic Carrier) Method"	5 11391	9-20-63
150		"Ruthenium Radioactivity in Aqueous Solutions, Pyridine Extraction—Gamma Counting Method"	2 21734 9 062310	7-20-64
151		"Nickel, Neutron Activation Analysis (Fast Neutron—Isotopic Carrier) Method"	5 115450	7-21-64
152	Rubin, I. B.	"Separation of Krypton and Xenon from Contaminating Gases, Molecular Sieve Absorption Method"	1 00710 9 00654	10-9-63
153	Spitzer, E. J., J. R. Sites	"General Operating Conditions for Isotopic Mass Spectrometry of the Elements"	4 0001	2-20-64
154		"Aluminum, Isotopic Mass Spectrometry"	4 0102	2-20-64
155		"Antimony, Isotopic Mass Spectrometry"	4 0104	2-20-64
156		"Barium, Isotopic Mass Spectrometry"	4 0108	2-20-64
157		"Boron, Isotopic Mass Spectrometry"	4 0112	2-20-64
158		"Bromine, Isotopic Mass Spectrometry"	4 0113	2-20-64
159		"Cadmium, Isotopic Mass Spectrometry"	4 0114	2-20-64

160	"Calcium, Isotopic Mass Spectrometry"	4 0115	2-20-64
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176	"Iridium, Isotopic Mass Spectrometry"	4 0140	2-20-64
177	"Iron, Isotopic Mass Spectrometry"	4 0141	2-20-64
178	"Lanthanum, Isotopic Mass Spectrometry"	4 0143	2-20-64
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217	"Zirconium, Isotopic Mass Spectrometry"	4 0198	2-20-64

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	AUTHOR(S)	TITLE	NUMBER(S)	DATE
218	Bate, L. C., G. W. Leddicotte[1]	"Particle-Size Distribution in Thorium Oxide, Neutron Activation—Sedimentation Method"	5 10200	R. 5-22-63
219	Canada, D. C.	"Total Plutonium, Thenoyltrifluoroacetone Extraction Method"	2 31621 9 008621	R. 3-24-64
220	Connelly, M. V.	"Boron (Borate), Potentiometric Mannitol—Sodium Hydroxide Titration Method"	1 211220 9 00711220	R. 5-15-63
221		"Ion-Exchange Separation of Boron from Other Elements"	1 00701	R. 5-16-63
222	Corbin, L. T., G. W. Leddicotte,[1] S. A. Reynolds, P. F. Thomason	"Safety"	1 0020 2 0050 5 0050 9 0055	R. 5-16-63
223	Lynn, E. C.	"Magnesium, Volumetric Versene Method"	1 214770 9 00714770	R. 6-26-64
224	Raaen, Helen P.	"Organization of the *ORNL Master Analytical Manual* and Numbering of the Methods"	00	R. 2-20-64
225		"Typing and Printing Methods for the *ORNL Master Analytical Manual*"	03	R. 8-1-63

226		"Writing Instrumental Methods for the *ORNL Master Analytical Manual*"	05	R. 10-16-63
227		"Use and Care of Polyethylene"	1 001005	R. 10-16-63
228	Reynolds, S. A.	"Apparatus, General"	2 0010 5 10110	R. 5-16-63
229		"Reagents"	2 0040 5 10140	R. 5-16-63
230	Wyatt, E. I.	"Antimony-125 CF, Product Analysis Guide"	9 0733042	R. 6-14-63
231		"Arsenic-77, Product Analysis Guide"	9 0733064	R. 6-14-63
232		"Barium-131, Product Analysis Guide"	9 0733085	R. 6-14-63
233		"Bromine-82, Product Analysis Guide"	9 0733131	R. 7-1-63
234		"Cadmium-115*m* HSA, Product Analysis Guide"	9 0733141	R. 7-1-63
235		"Cerium-141,144, Product Analysis Guide"	9 0733181	R. 3-1-64
236		"Cesium-134, Product Analysis Guide"	9 0733193	R. 4-23-64
237		"Chromium-51, Product Analysis Guide"	9 0733211	R. 8-15-63
238		"Cobalt-58, Product Analysis Guide"	9 0733225	R. 8-15-63
239		"Copper-64, Product Analysis Guide"	9 0733231	R. 8-15-63
240		"Gold-198, Product Analysis Guide"	9 0733331	R. 8-20-63
241		"Gold-199, Product Analysis Guide"	9 0733332	R. 6-22-64
242		"Iodine-131, Product Analysis Guide"	9 0733391	R. 8-20-63
243		"Iridium-194, Product Analysis Guide"	9 0733402	R. 8-20-63
244		"Iron-55,59, Product Analysis Guide"	9 0733411	R. 8-22-63
245		"Mercury-203, Product Analysis Guide"	9 0733491	R. 8-22-63
246		"Molybdenum-99, Product Analysis Guide"	9 0733501	R. 8-23-63
247		"Neodymium-147—Promethium-147, Product Analysis Guide"	9 0733511	R. 9-9-63
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249		"Palladium-109, Product Analysis Guide"	9 0733591	R. 9-10-63
250		"Potassium-42, Product Analysis Guide"	9 0733641	R. 9-10-63
251		"Praseodymium-142, Product Analysis Guide"	9 0733650	R. 9-11-63
252		"Praseodymium-143, Product Analysis Guide"	9 0733651	R. 9-11-63
253		"Promethium-147, Product Analysis Guide"	9 0733661	R. 9-11-63
254		"Rubidium-86, Product Analysis Guide"	9 0733721	R. 9-12-63
255		"Ruthenium-103,106, Product Analysis Guide"	9 0733731	R. 9-12-63
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257		"Scandium-56, Product Analysis Guide"	9 0733751	R. 9-12-63
258		"Selenium-75, Product Analysis Guide"	9 0733761	R. 9-12-63
259		"Silver-110*m*, Product Analysis Guide"	9 0733781	R. 7-1-63
260		"Silver-111, Product Analysis Guide"	9 0733783	R. 7-7-63
261		"Sodium-24, Product Analysis Guide"	9 0733792	R. 7-1-63

262		"Tin-113, Product Analysis Guide"	9 0733891	R. 7-1-63
263	Wyatt, E. I., R. R. Rickard	"Iodine Activity in Aqueous Solutions"	2 21391 9 0733391	R. 8-20-63

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	AUTHOR(S)	TITLE	NUMBER(S)	DATE
264	Farrar, L. G.	"Uranium, Copper, and Nickel in Homogeneous Reactor Fuel, Coulometric Titration Method"	9 082228	6-4-63
265	Kubota, H.	"Monobutylphosphoric Acid, Dibutylphosphoric Acid, and Orthophosphoric Acid in Tributyl Phosphate, Volumetric Sodium Hydroxide Titration Method"	1 221006 9 00721006	4-21-64
266	Laing, W. R., H. G. Davis	"Total Gases in Solids – Release Measurement and Collection for Mass Spectrometric Analysis"	1 106 9 00606	2-6-64
267	Rubin, I. B.	"Carbon in Aqueous and Nitric Acid Solutions"	1 211794	11-15-64

ORAL PRESENTATIONS

Eighth Conference on Analytical Chemistry in Nuclear Technology

More than 300 scientists, including 21 representatives from 8 foreign countries, attended the Eighth Conference on Analytical Chemistry in Nuclear Technology, which was held October 6–8, 1964, 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 L. J. Brady, Cyrus Feldman, H. L. Holsopple, W. R. Laing, W. S. Lyon, A. S. Meyer, and J. R. Sites.

Fifty-six papers were presented at the meeting, nine of which were presented by members of the ORNL staff.

In addition to the usual program, meetings were also held simultaneously by a Subcommittee of the National Research Council on Radioactivity Standards, by a group on beryllium exchange analysis, by another group on the exchange of analyses on uranium, and by a large number of participants in an impromptu meeting on the analysis of alkali metals.

The emphasis on the determination of trace constituents of nuclear and reactor materials was by far the most important aspect of the conference. This emphasis, in addition to giving consideration to the determination of a wide range of constituents in many materials, extended to the important problem of determining impurities in alkali metals commonly used as reactor coolants and to a serious discussion of the problems involved in the determination of the degree of burnup in reactor fuels.

The Ninth Conference in this series is tentatively scheduled to be held at the Mountain View Hotel, Gatlinburg, Tennessee, on October 12–14, 1965.

At Meetings of Professional Societies

	AUTHOR(S)	TITLE	PRESENTED AT
268	Bate, L. C.	"Individual Identification by Activation Analysis of Hair and Its Surface Contamination"	American Academy of Forensic Science, Chicago, Ill., Feb. 28–29, 1964
269	Bate, L. C.,[15] W. B. Healy[16]	"Determination of Trace Elements in Wool"	8th Annual Conference, Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Oct. 6–8, 1964
270	Belew, W. L.,[15] D. J. Fisher, M. T. Kelley	"Second Derivative DC Polarography as a Technique in Quantitative Polarographic Measurements"	12th Anachem Conference and Instrument Exhibit, Wayne State University, Detroit, Mich., Oct. 21–23, 1964
271	Biggers, R. E.,[15] D. A. Costanzo	"A Generalized Computer Program to Obtain Reaction Rate Data by the Analysis of Spectrometric Absorption Data from Dynamic Multicomponent Systems"	Symposium on Molecular Structure and Spectroscopy, Department of Physics and Astronomy, Ohio State University, Columbus, June 15–19, 1964
272	Cameron, A. E.	"Commentary on ORNL Double-Magnet Mass Spectrometer"	8th Annual Conference, Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Oct. 6–8, 1964
273	Crowther, P.,[4,15] J. S. Eldridge	"Decay of Molybdenum-99–Technetium-99*m*"	10th Annual Meeting, American Nuclear Society, Philadelphia, Pa., June 15–17, 1964
274	Dyer, F. F.,[15] L. C. Bate, J. F. Emery	"Application of Nuclear Analysis to Forensic Science"	6th Annual Contractor's Meeting, Division of Isotopes Development, U.S. Atomic Energy Commission, Washington, D.C., Nov. 9–10, 1964
275	Dyer, F. F.,[15] J. F. Emery	"Precision of Activation Analysis of Short-Lived Nuclides"	8th Annual Conference, Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Oct. 6–8, 1964
276	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
277	Eldridge, J. S.,[15] P. Crowther[4]	"Evaluation of I^{125} Measurement Techniques for Clinical Applications"	2d Annual Meeting, Oak Ridge Radioisotope Conference, Gatlinburg, Tenn., Apr. 19–22, 1964
278	Emery, J. F.	"Br^{82m}: A New Bromine Isomer"	148th Meeting, American Chemical Society, Chicago, Ill., Aug. 30–Sept. 4, 1964
279	Feldman, C.	"Atomic Absorption Analysis"	Research Committee Meeting, Union Carbide Nuclear Corp., Buffalo, N.Y., May 25–26, 1964

[15]Speaker.

[16]Soil Bureau, Department of Scientific and Industrial Research, Lower Hutt, New Zealand.

280		"The Place of Atomic Absorption in Tissue Analysis"	12th Anachem Conference and Instrument Exhibit, Wayne State University, Detroit, Mich., Oct. 21–23, 1964
281	Feldman, C., T. C. Rains,[15] N. Marion Ferguson	"Chemical Isolation and Flame Photometric Determination of Barium"	8th Annual Conference, Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Oct. 6–8, 1964
282	Fisher, D. J.	Discussion following Chapter III	Robert A. Welch Foundation Conference on Chemical Research, VII. Modern Developments in Analytical Chemistry, Houston, Tex., Nov. 18–20, 1963
283		"Controlled Potential D.C. Polarography"	The Society for Analytical Chemistry and the Polarographic Society, London, England, July 28, 1964
284		"Circumventions of Chemical Measurement Difficulties Through Systems Design"	147th Meeting, American Chemical Society, Philadelphia, Pa., Apr. 5–10, 1964
285	Fisher, D. J.,[15] W. L. Belew, M. T. Kelley	"Recent Developments in DC Polarography"	Third International Congress of Polarography, Southampton, England, July 19–25, 1964
286		"A Simple Quasi Reference Electrode....Applications in Controlled-Potential Polarography and Voltammetry and in Chronopotentiometry"	Third International Congress of Polarography, Southampton, England, July 19–25, 1964
287	Goldberg, G.	"The Rapid Determination of Low Concentrations of Oxygen in Potassium and NaK by a Modified Amalgamation Technique"	15th Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, Pittsburgh, Pa., Mar. 2–6, 1964
288		"The Rapid Determination of Low Concentrations of Oxygen and Hydrogen in Alkali Metals by a Modified Amalgamation Technique"	8th Annual Conference, Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Oct. 6–8, 1964
289	Goldstein, G.	"Absolute Counting of Beta Emitters by the Liquid Scintillation Method"	8th Annual Conference, Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Oct. 6–8, 1964
290	Kelley, M. T.	"The Accurate and Precise Determination of Uranium, with Special Reference to Coulometry"	International Atomic Energy Agency Panel on Analytical Chemistry of Nuclear Materials, Vienna, Austria, Mar. 23–26, 1964
291	Kubota, H.,[15] D. A. Costanzo	"Reactions in Concentrated Lithium Chloride Solutions: Determination of Free Acid and Hydrolyzable Metal"	12th Anachem Conference and Instrument Exhibit, Wayne State University, Detroit, Mich., Oct. 21–23, 1964
292	Lyon, W. S.	"Research on Activation Analysis Problems"	5th Annual Contractor's Meeting, Division of Isotopes Development, U.S. Atomic Energy Commission, Washington, D.C., Dec. 2–3, 1963

293		"Electronic Resolution of Gamma-Ray Spectra"	5th Annual Contractor's Meeting, Division of Isotopes Development, U.S. Atomic Energy Commission, Washington, D.C., Dec. 2–3, 1963
294	Lyon, W. S.,[15] J. S. Eldridge, P. Crowther[4]	"Experimental Study of Artifacts and Errors Encountered in Gamma-Ray Spectrometry for Radiochemistry and Activation Analysis"	International Atomic Energy Agency's Symposium on Radiochemical Methods of Analysis, Salzburg, Austria, Oct. 19–23, 1964
295	Mamantov, G.,[7,15] D. L. Manning	"Voltammetry and Chronopotentiometry of Iron in Molten Fluorides"	Southeastern Regional American Chemical Society Meeting, Charlotte, N.C., Nov. 14–16, 1963
296	Manning, D. L., J. M. Dale, G. Mamantov[7,15]	"Polarography in Molten Fluorides"	Third International Congress of Polarography, Southampton, England, July 19–23, 1964
297	Manning, D. L.,[15] J. M. Dale, G. Mamantov[7]	"Voltammetric Measurements in Molten Fluoride Salt Systems"	15th Meeting, Comite International de Thermodynamique et de Cinetique Electrochimiques, London, England, Sept. 21–26, 1964
298	Morton, J. E.,[15] H. A. Parker, E. I. Wyatt, L. T. Corbin	"Shielded Facility for Use in the Analysis of Radioisotopes"	11th Hot Laboratory and Equipment Conference, New York, Nov. 18–22, 1963
299	Perone, S. P.,[15,17] T. R. Mueller	"The Application of Derivative Techniques to Stationary Electrode Polarography"	148th Meeting, American Chemical Society, Chicago, Ill., Aug. 30–Sept. 4, 1964
300	Peters, D. G.,[8,15] W. D. Shults	"Chronopotentiometry of Plutonium in Mineral Acid Media"	148th Meeting, American Chemical Society, Chicago, Ill., Aug. 30–Sept. 4, 1964
301	Reynolds, S. A.	"Current Work at ORNL"	Taft Sanitary Engineering Center, Public Health Service, Cincinnati, Ohio, Apr. 8, 1964
302		"Radioisotopes in Alkaline-Earth Analysis"	2d Annual Oak Ridge Radioisotope Conference, Gatlinburg, Tenn., Apr. 19–22, 1964
303		"Recent Radiochemical Work at ORNL"	Seminar, Northeastern Radiological Health Laboratory, Public Health Service, Winchester, Mass., Aug. 27, 1964
304		"Nondestructive Determination of Uranium-232 and Thorium-228 by Gamma Spectrometry"	8th Annual Conference, Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Oct. 6–8, 1964
305	Reynolds, S. A.,[15] C. L. Burros	"Some Experiences in Environmental Analyses at ORNL"	9th Annual Meeting, Health Physics Society, Cincinnati, Ohio, June 14–18, 1964
306	Ricci, E.	"Output Spectra of 14-Mev Neutron Generators. Rapid Estimation and Influence in Activation Analysis"	10th Annual Meeting, American Nuclear Society, Philadelphia, Pa., June 15–17, 1964

[17] Department of Chemistry, Purdue University, Lafayette, Ind.

307	Ross, H. H.	"Radioisotopes in Chemical Analysis"	Annual Lecture Series, Rochester Section, American Chemical Society, Rochester, N.Y., Nov. 12, 1964
308		"Radio-Release Methods for the Evaluation of Atmospheric Pollution"	12th Anachem Conference and Instrument Exhibit, Wayne State University, Detroit, Mich., Oct. 21–23, 1964
309		"A Unique Liquid Scintillator for Neutron Detection Using Lithium-6 Enriched Isotope"	5th Annual Contractor's Meeting, Division of Isotopes Development, U.S. Atomic Energy Commission, Washington, D.C., Dec. 2–3, 1963
310	Ross, H. H.,[15] T. H. Handley, W. J. Ross	"Application of Isotopes to Analytical Chemistry"	6th Annual Contractor's Meeting, Division of Isotopes Development, U.S. Atomic Energy Commission, Washington, D.C., Nov. 9–10, 1964
311	Ross, H. H.,[15] R. E. Yerick[10]	"A Unique Scintillator for Neutron Detection"	2d Annual Meeting, Oak Ridge Radioisotope Conference, Gatlinburg, Tenn., Apr. 19–22, 1964
312	Ross, H. H.,[15] W. S. Lyon	"Radio-Release Methods for the Evaluation of Atmospheric Pollution: SO_2"	International Atomic Energy Agency's Symposium on Radiochemical Methods of Analysis, Salzburg, Austria, Oct. 19–23, 1964
313	Ross, W. J.,[15] J. E. Strain	"Reactions Observed with 14-Mev Neutrons"	8th Annual Conference, Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Oct. 6–8, 1964
314	Strain, J. E.	"Explanation and Demonstration of Neutron Activation Analysis"	AEC Exhibit, National Science Fair, Baltimore, Md., May 6–8, 1964
315		"Isotopic and Accelerator Neutron Sources"	ORINS, Activation Analysis Course, Oak Ridge, Tenn., Apr. 8 and 10, 1964
316	Strain, J. E.,[15] W. S. Lyon	"The Use of Isotopic Neutron Sources for Chemical Analysis"	International Atomic Energy Agency's Symposium on Radiochemical Methods of Analysis, Salzburg, Austria, Oct. 19–23, 1964
317	Willmarth, T. E.	"A Microscopical Study of the Structure of Spherical Thoria Sol-Gel Beads"	"Micro 64" (International Electron Microscopy Society), Chicago, Ill., July 1964
318		"Analytical Electron Microscopy – Past and Present at Oak Ridge National Laboratory"	Louisiana Electron Microscopy Society, New Orleans, La., Oct. 2, 1964
319	Young, J. P.	"Direct Spectrophotometric Studies of Redox Reactions in Molten Fluoride Salts"	15th Meeting, Comite International de Thermodynamique et de Cinetique Electrochimiques, London, England, Sept. 21–26, 1964

320		Seminar, Dept. de Physico-Chimie, Centre d'Etudes Nucleaires de Saclay, Gif-sur-Yvette, France, Sept. 1, 1964
321	"Spectrophotometric Observations of the Reaction of Molten Lithium Fluoride–Sodium Fluoride–Potassium Fluoride with Several Active Metals"	145th Meeting, American Chemical Society, New York, N.Y., Sept. 8–13, 1963
322	"The Role of Serendipity in Research"	King's College Chemical Society, King's College, Wilkes-Barre, Pa., May 14, 1964

Under the ORNL Traveling Lecture Program

	LECTURER	LECTURE TITLE	PRESENTED AT
323	Cameron, A. E.	"Geological Age Determination by Isotope Measurements"	Western Carolina College, Cullowhee, N.C., Jan. 6, 1964
324			North Carolina State College, Raleigh, Jan. 8, 1964
325			Tennessee Polytechnic Institute, Cookeville, Feb. 13, 1964
326			Appalachian State Teachers College, Boone, N.C., Apr. 30, 1964
327			Rice University, Houston, Tex., May 6, 1964
328			Stephen F. Austin State College, Nacogdoches, Tex., May 7, 1964
329	Raaen, Helen P.	"How a Sewing Needle and Teflon Are Advancing Polarography"	Texas Woman's University, Denton, Tex., Mar. 20, 1964
330	Ross, H. H.	"Modern Methods in Liquid Scintillator Technology"	Georgia Institute of Technology, Atlanta, Oct. 15, 1964
331	Thomason, P. F.	"Controlled-Potential Coulometry: New Analytical Tool in the Nuclear Energy Program"	Birmingham-Southern College, Birmingham, Ala., Apr. 21, 1964
332	White, J. C.	"The New Image of Analytical Chemistry in Science"	Purdue University, Lafayette, Ind., Nov. 11, 1964

Patents

	AUTHOR(S)	TITLE	PATENT NO(S).	ISSUED
333	Shaffer, J. H.,* J. E. Strain, D. R. Cuneo,* M. J. Kelly*	"Separation of Protactinium from Molten Salt Reactor Fuel Composition"	3,110,555	Nov. 12, 1963

Indexes

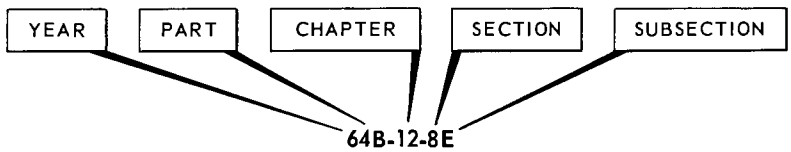
Helen P. Raaen Ann S. Klein[1]

Indexes are a part of the Analytical Chemistry Division annual report for the first time this year. The numbering system used will make possible the preparation of cumulative indexes to this and future annual progress reports from the Division. This section of the report contains a key-word index and an author index.

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. 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.

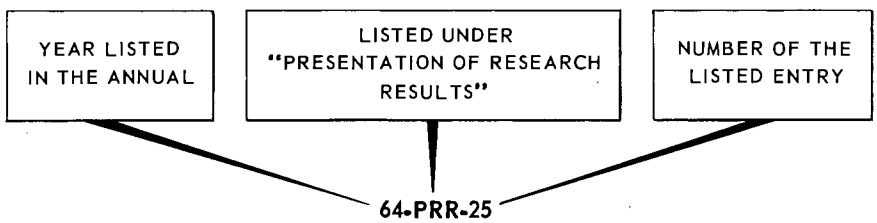
The Author Index is an alphabetical listing of authors, together with number entries that specify the material to which each author contributed. This index includes both the authors of material in the body of the report and the authors of the entries listed in the Presentation of Research Results. In 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 64B-12-8E, where 64 indicates the year of publication of the annual report, B designates a major part or division of the report, 12 is the chapter number, 8 is a primary section within the chapter, and E is a subsection (designated in the subsection heading with e instead of E); 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 64-PRR-25, where 64 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,



[1]Technical Information Division.

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.

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.

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