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ANALYTICAL CHEMISTRY DIVISION
ANNUAL PROGRESS REPORT
FOR PERIOD ENDING OCTOBER 31, 1966

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UC-4 – Chemistry

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ANALYTICAL CHEMISTRY DIVISION
ANNUAL PROGRESS REPORT
For Period Ending October 31, 1966

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JANUARY 1967

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Preface

Analytical chemistry at ORNL is centralized within the Analytical Chemistry Division. Consequently, the activities of the Division reflect to a considerable extent the diversity of the research program at ORNL. The general mission of the Division is to provide complete analytical support for these programs – research in analytical chemistry, development of new and improved methods for analysis, and performance of service analyses.

The research and development efforts within the Division continue to be application oriented, with emphasis on providing, where feasible, instantaneous automatic analyses. Many of the research and development programs in analytical chemistry for the MSRE and for the bioengineering programs, as well as a great deal of the more fundamental effort in the instrumental chemistry research program, are directed toward this goal. Constant attention in given in the service laboratories to assure a high standard of operation by institution of new or improved methods of analysis and more efficient and refined instrumentation.

The increase in effort within the Division in analytical biochemistry is noteworthy and significant. Assistance to the Laboratory's large biology program is being made in research, development, and service activities, with particular emphasis on instrumentation for automated analysis. It is anticipated that a continued increase in effort will be required to provide analytical support in this area. The Division considers that the ability to provide analytical support to such diversified research projects within the Laboratory is a most important function. Attaining and maintaining this capability continues to be the major challenge to analytical chemistry.

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Summary

PART A. ANALYTICAL RESEARCH

1. Analytical Instrumentation

The instrumental design work of the Analytical Instrumentation Group was continued with an increased emphasis on solid-state operational-amplifier modules. New prototype analytical instruments that use these modules include: (1) a fast, controlled-potential and controlled-current cyclic voltammeter; (2) a solid-state controlled-potential coulometric titrator, designed to update the vacuum-tube instruments in present use within the Division; and (3) a square-wave polarograph that also uses integrated-circuit operational amplifiers and logic modules.

Evaluation programs were continued in the field of high-precision dc polarography by use of the ORNL model Q-2792 controlled-potential dc polarograph-voltammeter for single-cell studies and of the controlled-potential differential dc polarograph for two-cell studies. Values of $S \leq 0.1\%$ for replicate determinations were achieved. Two open-literature papers and a Ph.D. thesis based on this work have been accepted. A precision "drop hammer" for accurate control of the mercury drop time and high-performance active filter networks have been developed for, and installed in, the Q-2792 instrument.

The circuit of the ORNL model Q-2564 coulometric titrator was modified; this change should eliminate a possible error when high-concentration solutions are titrated in the cell. Revision of an existing spectrographic instrument will produce a double-beam flame spectrophotometer for atomic absorption work. This instrument will compensate for variation in the intensity of the hollow-cathode light source.

Apparatus for automatic cutoff at the end point in a constant-current method for determining Cl^- was designed and installed. One-second, parallel-T filter networks were calculated for, and installed in, an ORNL model Q-1988-FES polarograph being used in fundamental studies with the Teflon D.M.E. An up-to-date circuit drawing of the model Q-1988-FES was prepared.

Graphitar 14 was evaluated as a material of construction for electrodes in voltammetry. Chemical evaluation of an ac-dc controlled-potential polarograph is in progress.

Work continued in chronopotentiometry with a previously described instrument designed to compensate for extraneous currents; mercury-pool electrodes and planar-mercury electrodes are used. Work with solid electrodes is planned also. A paper on the theory of programmed-current derivative polarography was accepted for publication.

Design work on the following remotely operated analytical instruments and apparatus for use in the analytical hot cells of the Transuranium Processing Plant has been completed: (1) an analytical balance; (2) a solvent extractor; (3) an ORNL model Q-2564 high-sensitivity coulometric titrator; (4) a model VIIA flame spectrophotometer; and (5) a special version of the ORNL model Q-1728 velocity-servo potentiometric tritrator that features a newly designed elevator and titrant-delivery unit.

Automatic equipment for the transfer of aliquots from a Technicon AutoAnalyzer product stream to filter-paper disks is being devised for the Bioanalytical Development Group. This equipment will be used in the routine assay of tRNA samples.

2. Effects of Radiation on Analytical Methods

The anodic electrode reactions of the I_2-I^- system in acid and neutral media at the quiescent pyrolytic-graphite electrode (P.G.E.) have been established. The feasibility of the use of these reactions for analytical purposes was evaluated.

A chronopotentiometric study of the I_2-I^- system at the P.G.E. and the glassy-carbon electrode

(G.C.E.) has been carried out. It was found that the system undergoes essentially the same reactions at either electrode; the results have been published.

The halide ions were shown to undergo stepwise oxidation at the P.G.E. in dimethyl sulfoxide. The characteristic features of each oxidation step and the differences existing for each halide ion were determined.

The reaction between Zr and alizarin red S was studied by voltammetric and spectrophotometric techniques. The complex species formed was identified, and the reaction kinetics were clarified.

A voltammetric method was developed for determining micro amounts of Zr. The method is based on the anodic reactions of alizarin red S and its Zr complex at the rotating P.G.E. The method is relatively simple, accurate, and free of interferences.

Acid chloride solutions under irradiation will dissolve the metals Au, Zr, W, Pt, and Nb; Ta is only slightly affected.

A deaerated $FeSO_4$ solution sealed into glass tubes was demonstrated to be suitable as a high-temperature dosimeter up to 250°C; it has a G value of 8.1.

It has been established that the relatively high rate of loss of volatile solutes from concentrated solutions of LiCl is due to the very high activity coefficients of H^+ in such solutions. The possibility is being pursued that the radiolytic acid depletion which occurs in acid chloride solutions under irradiation can be counteracted by the addition of aldehydes or chloral hydrate, both of which release acid as a product of radiolysis.

3. Analytical Chemistry for Reactor Projects

Equipment was developed for the determination of oxide by hydrofluorination and was applied to the analysis of highly radioactive MSRE fuel that contained 50 to 70 ppm of oxide. Members of the Division participated in extensive engineering studies to determine the nature and source of organic deposits that were interfering in MSRE operations; a method was developed for the continuous measurement of hydrocarbons in MSRE off-gas.

Analytical molten-salt development efforts are being concentrated on in-line analysis for the Molten-Salt Breeder Experiment (MSBE). Research on the analytical chemistry of molten salts has indicated that in-line methods for UF_3, UF_4, most corrosion products, and oxide are feasible. A facility to test practical analytical transducers is being planned. Diffusion coefficients and activation energies for the U(IV) → U(III) reduction wave in MSRE fuel were determined, and the wave was found to be reproducible to better than 1% over short periods. An improved voltammeter is being built to exploit this wave for in-line determinations. Continued voltammetric studies indicated that Fe, Ni, and possibly Cr can be determined in MSBE-type salts. Ionic Fe and Ni were actually measured in slightly radioactive fuel withdrawn from the MSRE. A wave for the voltammetric determination of oxide was found to be masked by a U oxidation wave.

A more detailed spectrum of UF_3 in MSRE fuel solvent was obtained, and the absence of interferences from corrosion products was confirmed. Investigation of unusual oxidation states of rare-earth-metal fission products indicated possible interference from Sm(II) but none from Eu(II). An intense absorption peak in the ultraviolet spectrum of U(IV) was found to be suitable for monitoring part-per-million concentrations of U in coolant salt streams. A modified optical system was ordered to improve measurement with captive-liquid cells.

Transmission spectra were obtained for the solid fluoride salts CrF_2, FeF_2, K_3CrF_6, UF_4, and $LiUF_5$; reflectance spectra were recorded for CrF_3, K_3CrF_6, UF_4, and $LiUF_5$. These spectra are being interpreted and compared with molten-salt spectra to aid in the understanding of the nature of these ions in molten-salt solvents.

Spectra of small bits of other solid material have also proved to be of value. A shift in charge-transfer absorption was found to cause certain rare-earth-metal germanomolybdates to change color on heating. Spectra of 0.1-mm^2 areas of deposits of metal on film yielded information of value in the interpretation of densitometric determinations of the metal deposits.

A cryogenic-trapping technique was developed to follow the release of ^3H and ^{85}Kr during various steps of the shear-leach process. An x-ray fluorescence method was found to be too insensitive to monitor UF_6 and PuF_6 in process gas streams of the Fluidized-Bed Volatility Pilot Plant. Spectrophotometric methods are being evaluated for these in-line determinations.

Analytical assistance to the Nuclear Safety Program includes the development of in-line instrumentation to monitor gases generated by in-pile burnup tests and a new program to determine organic atmospheric contaminants that would contribute to problems in trapping of radioiodine.

4. Special Research and Development Activities

Pyrochromatograms of ion exchange resins and of vinylbenzene-divinylbenzene indicated that the divinylbenzene in the pyrolyzate is an index of cross-linking for the copolymer but not for the resin.

A pyrolysis–gas chromatographic technique was used to study the cause of swelling of UAl_3-Al reactor fuel elements prepared by powder metallurgical techniques. Pyrochromatograms of a mold-release compound, fuel-element compacts, and the powder components indicate that the gases released during swelling are of composition similar to that of the gases generated by hydrolysis of uranium carbides.

The polarographic studies of the use of the Teflon D.M.E. have indicated that the retraction of mercury into the capillary is greater for Teflon D.M.E.'s than for glass D.M.E.'s. This retraction may contribute to a lower S/N ratio for the Teflon D.M.E.'s. Vertical-orifice Teflon D.M.E.'s that permit rapid polarography are being studied; they may have future use in the analysis of radioactive solutions or process streams. Also, a vertical-orifice Teflon D.M.E. is being used to study the polarography of U in aqueous HF.

The polarographic study of Ni in concentrated solutions of LiCl used in the Tramex process was completed, and the results were published.

A voltammetric method for the determination of Al by oxidation of the Al–Solochrome Violet RS complex and use of a rotated P.G.E. was developed and published.

The anodic behavior of the halide ions in dimethyl sulfoxide was studied, and the results were published.

The results of a voltammetric study of I_2 in aqueous medium by use of the P.G.E. were published.

Several "specific-ion" electrodes were evaluated, because they have possibility for use in radioactive solutions and in process streams. A monovalent-cation electrode and a Pungor-N.I.L. "iodide" electrode show that concentrations of appropriate ions can be measured to within about ±5%.

The ultraviolet spectra were recorded for special interference filters and for solutions of 4-sec-butyl-2-(α-methylbenzyl)phenol (BAMBP) in several diluents.

An attempt to obtain the spectrum of Cf failed, because the quantity of ^{249}Cf ($\sim 5\ \mu g$) was insufficient.

Infrared spectrometry was used to examine or record reference spectra for more than 500 samples that were chiefly either commercial products that contained impurities or organic compounds synthesized at ORNL.

In NMR studies, spectra were recorded of phosphonium halides, barium ethylbenzenesulfonate, and special lipid samples. The NMR spectra obtained for the Biology Division included a number of samples of sRNA and Gramicidin A. Other NMR work was done on various oximes and Cs-BAMBP complex.

A potentiometric method was developed for the determination of free acid in Tramex process solutions.

The Willard-Winter method for the separation of F^- was modified to permit microgram quantities of F^- to be determined spectrophotometrically in samples that contain NO_3^-

A polarographic method for the determination of NO_3^- in the Tramex process solutions was developed.

Attempted potentiometric titrations of Tc(VII) with various reducing titrants were unsuccessful. Preliminary experiments indicate that the reduction of Tc(VII) with a solution of $NaAsO_2$ can be followed spectrophotometrically.

A potentiometric titration method for the determination of Pu is under study. It consists in the oxidation of Pu to Pu(VI) with Ag(II), reduction of Pu(VI) with excess Fe(II), and titration of the unreacted Fe(II) with Ce(IV).

The extraction in hexone and spectrophotometric determination of U in situ is under investigation.

A modified pyrohydrolysis separation and spectrophotometric determination of F^- has been used for the analysis of HFIR target dissolver solutions.

Several existing analytical methods were adopted to particular problems; these including the determinations of Ru, Al_2O_3, KO_2, amines, Sn(II), and free S.

A rapid method was developed for the measurement of density of single particles of reactor fuels by the density-gradient technique.

5. Analytical Biochemistry

Analytical assistance to the Biology and Chemical Technology Divisions was continued in two programs: the Macromolecular Separations Program and the Body Fluids Analysis Program. The major effort has been in the analysis of transfer ribonucleic acids (tRNA's). Conditions were established for the quantitative determination of phenylalanine- and leucine-accepting tRNA's, and we are now working on the tyrosine-, alanine-, and valine-accepting compounds. To meet an increasing demand for routine assays, a double-labeling technique was developed in which two tRNA's are determined simultaneously by reacting them with ^{14}C- and ^{3}H-labeled amino acids. In addition, instrumentation is being developed for automating the assay procedure. A proposed gas chromatographic method for amino alcohols, which can be obtained by reduction of aminoacyl-tRNA esters, has been tested with the available amino alcohols. Impurities in tRNA preparations are now being determined also. A column chromatographic method was developed for analysis of the hydrolysis products of tRNA.

The fluorescence and the polarization of the fluorescence of some metal chelates in the presence of nucleic acids and similar compounds were studied. The effect on intensity depends on the type of nucleic component, the analytical conditions, and the instrumentation used.

In the Body Fluids Analysis Program some 100 ultraviolet-absorbing components have been separated from human urine by anion exchange chromatography. Of these, about 25 have been identified. A large column is being prepared for isolating sufficiently large amounts of the compounds for chemical identification. Improvements are being made in the instrumentation to miniaturize the system and to shorten the analysis time.

Gas chromatographic studies of the pyrolysis products of the constituents of nucleic acid have continued and were extended to include some nucleosides and nucleotides. Compounds can be identified from the relative amounts of the low-molecular-weight products. Work on the high-molecular-weight pyrolysis products is in progress.

6. X-Ray and Spectrochemical Analyses

Computer codes were written, and tested for x-ray absorption-edge analysis and electron-probe analysis. The latter has been used for metals, ceramics, and biological materials. With suitable standards and visual interpolation, the Li_2CO_3-matrix method of spectrographic semiquantitative analysis gave results correct to within a factor of 1.5 in 71% of the cases. The germanium-graphite-matrix method gave superior sensitivity with volatile, easily reduced metals. For cobalt in solution, a detection limit of 0.01 μg/ml was reached by the atomic-absorption method.

A Tc channel was added to the Paschen Direct Reader; Cr-Tc alloys were analyzed successfully by means of the Direct Reader.

7. Mass Spectrometry

With the MS-7 spark-source mass spectrograph, some 440 analyses were made on about 50 types of samples. The samples included very pure metals, multipass zone-refined Nb alloys, and reactor-construction metals. A new technique of semiquantitative analysis was introduced in which the test solution is evaporated onto a metal substrate and Sr is used as an internal standard; as little as 0.1 ng of element per electrode can be detected. A program was written to use the IBM 360/75 computer to calculate impurity concentrations.

With the double-stage mass spectrometer used as a mass separator, a batch of ^{251}Cf was obtained.

Studies of the effect of O_2 contamination on the surface ionization of UF_4 showed why the measured ion intensities fail to obey the Saha-Langmuir equation at temperatures below 2300°K. Also, the first ionization potential of U was determined to be 6.26 ± 0.02 ev by a surface-ionization comparison technique.

8. Optical and Electron Microscopy

Improvements in methods and equipment have made possible the processing of highly radioactive samples in the Building 3019 facility for the electron microscopy of irradiated materials. Research assistance to the Nuclear Safety Program was continued, and support has been given

in a study of *Escherichia coli* polysomes by the Biology Division. For the Microsphere Development Program, electron microscopy and electron diffraction were used to study the hydrous sols of uranium, the hydroxide sols of the lanthanide elements, and the physical structures of lanthanide sol-gel beads and of lanthanide oxide microspheres. Also, optical and electron microscopy and electron diffraction services were provided to other divisions on a wide variety of materials of interest in nuclear technology.

9. Nuclear Radiochemical Analyses

Gamma-ray transitions in the decay of ^{105}Ru, ^{116}In, ^{141}Ce, ^{147}Nd, and ^{188}Re were studied. Cross sections, resonance integrals, and nuclear properties of the radioelements formed have been measured for the target nuclides ^{123}Te, ^{140}La, and ^{139}La. The latter has been used as a source for the production of ^{141}Ce by double neutron capture, as has been ^{186}W for production of ^{188}W to be used for an ^{188}Re radioisotope generator.

Nuclear spectroscopy of the radioactivities from neutron-deficient Os, Rh, Hf, and rare-earth elements has continued, and the alpha-particle and gamma-ray spectra of ^{149}Gd and ^{149}Eu were measured. The cooperative isotopes program has included half-life measurements, technical assistance, quality control, updating of published nuclear data, and investigation of certain customer complaints concerning ^{14}C and ^{131}I products. Study of release of $^{3}H_2$ from ^{3}H-labeled luminous paints continues.

The standardization of ^{197}Hg has been studied. A paper that describes assay methods for all radioisotopes produced at ORNL has been published. Lithium-drifted germanium detectors (6 and 40 cc) for gamma spectrometry and silicon detectors for alpha spectroscopy have been investigated. Thick-target yields from ^{3}He particles on B, N, and Na and angular distribution of recoil nuclei from ^{3}He-particle irradiation of carbon were measured and correlated with theory. A new solvent-extraction method for Am was devised, and other radiochemical methods for this element were evaluated. The isolation of trivalent actinide-lanthanide elements by extraction of anionic complexes with a quaternary amine has been demonstrated, as has separation and purification of Bk by TTA extraction. A method for the rapid separation of ^{95}Nb from ^{95}Zr was reported, and a new liquid-liquid extraction system for trivalent actinides and lanthanides by use of high-molecular-weight amines was developed. Characteristics of radioactive liquid wastes were studied. The adsorption of trace elements on human hair and their elution from it were evaluated. Computer programs and the library of absolutely assayed gamma-ray spectra continue to be used in assay problems. A program AREA LSQ was written to combine features of two others, and a program THIKSIG was developed to handle yield calculations in charged-particle reactions. A prototype of the Ross beta-excited-light-source spectrometer was built, and testing was begun. A study of emission characteristics of a number of phosphors emitting in the 600- to 800-mμ range was undertaken. Use of radioisotopes, activation analysis, or both in a number of water problems is under investigation: determinations of O_2, PO_4^{3-}, and micro amounts of elements and tracing water and sediment movement.

A method to measure the total absorption cross section of Al_2O_3 and ThO_2 by neutron absorption is under development; an ^{241}Am-Be neutron source is being used. Progress in the field of nucleonics was reviewed in an article for *Analytical Chemistry*. The sensitivities of 15 low-*Z* elements with 18-Mev ^{3}He particles have been measured, and the interactions of these particles with four low-*Z* nuclides have been studied closely and compared with those predicted from theory.

The Texas Nuclear Corporation 14-Mev neutron generator was modified to produce 2.5 ma of beam current, and a new Kaman generator was installed in a facility for the determination of O_2 in alkali and refractory metals. Fast-neutron activation has been used to determine ^{2}H in H_2O, ^{16}O in metals, and atom ratios in NaO_2, UF_2, and ^{18}MgO. In addition, the generator has been used to monitor leached fuel elements for residual ^{235}U content. A study is under way of the effect of Lucite surrounding the generator-beam area on the yield of 14-Mev neutron reactions.

10. Inorganic Preparations

Most of the programs of the preceding year were continued. These included the preparation of fused salts for the Chemistry Division and the preparation of high-purity KCl, rare-earth-metal alloys, and alkali-metal superoxides for the Solid

State Division. A new program, the preparation of alkali-metal phosphate and germanate glasses, was initiated, and a series of spinels and 150 g of lithium-6 salicylate were prepared for the Solid State Division. For the Physics Division, $^{7}LiClO_3$, $Cu(ClO_3)_2 \cdot 6H_2O$, and a solid solution of $FeBr_3$ in $CrBr_3$ were prepared. The Metals and Ceramics Division was provided with $TeCl_4$, $TiCl_3$, and anhydrous NiI_2. Some dilutions of a previously prepared 20 mole % solid solution of $^{235}UCl_3$ in $LaCl_3$ were made for the Isotopes Division.

11. Organic Preparations

As in past years, the principal function of the Organic Preparations Laboratory has been to support ORNL research divisions by supplying, through custom synthesis, organic compounds needed for experimental activities. These compounds included: benzyltributylphosphonium bromide, benzyltrioctylphosphonium bromide, tetra-*n*-butylphosphonium iodide, tri-*n*-butylsulfonium iodide, triisoamyl phosphate, tri-*n*-amyl phosphate, ^{14}C-labeled propionic acid, 1,3-ethylpentyl-4-ethyloctyl bromide, *m*-aminobenzylamine, and barium *p*-ethylbenzenesulfonate.

In addition to preparative work, a number of compounds were purified for use in research work that required high-purity reagents. Divisions using the services of the Organic Preparations Laboratory were: Analytical Chemistry, Chemistry, Chemical Technology, Health Physics, Isotopes, Metals and Ceramics, Neutron Physics, Reactor Chemistry, and the K-25 Technical Division.

PART B. SERVICE ANALYSES

12. Mass Spectrometric Analyses

The Mass Spectrometry Service Laboratory reported some 7700 results. Most of the analyses were made on separated stable isotopes for the Isotopes Division. Assistance was given in determinations of the neutron-capture cross sections of ^{63}Ni and ^{171}Tm. Also, the isotopic abundances of ^{36}Cl and ^{40}K were measured to determine accurately the neutron flux used in the irradiation of a sample of KCl at the Savannah River Plant.

The Transuranium Mass Spectrometry Laboratory made 63% more analyses than in the previous year. Many of the samples are now received as electrodeposits on tungsten or platinum wire. This form facilitates their handling and reduces the probability of spreading contamination.

13. Spectrochemical Analysis Laboratory

The number of analyses reported from the Spectrochemical Analysis Laboratory increased by 23% over the previous year. The Paschen Direct Reader was used extensively for the quantitative analysis of many unusual alloys, metals, and mixtures.

14. Process Analyses

The process analyses laboratories performed 135,000 analyses, a 12% decrease under the past year. The analytical laboratories and cell block in the Transuranium Processing Plant were put into service. Brief statements of the new developments in each laboratory follow.

The High-Level Alpha Radiation Laboratories measured physical properties of plutonia-urania microspheres. An improved procedure for making sources for isotopic analyses was instituted. Plutonium was determined in several interlaboratory-check samples.

The General Analyses Laboratories continued work to develop an improved vacuum-fusion analyzer. A Leco carbon analyzer was modified to give increased sensitivity. Improved methods and apparatus were used for density measurement, determinations of Si and Cd, and purification of mercury. A biochemistry laboratory for the analysis of tRNA was put in operation.

The High-Radiation-Level Analytical Laboratories continued to supply analytical support to other ORNL divisions, particularly the Reactor, Isotopes, and Chemical Technology Divisions. Reductions in manpower and in work were effected as a result of the completion of the Curium Recovery Program. A glove-box facility is being prepared for use with the Plutonia Sol-Gel Program. Radioactive samples from the MSRE power-level runs were analyzed; only minor problems were encountered, and all results were within the anticipated limits.

The Radioisotopes-Radiochemistry Laboratories received most of their samples from various

nuclear-safety programs. A new radiochemical procedure for Te was developed. The half-lives of the following radioisotopes were either determined or redetermined: ^{133}Ba, ^{124}Sb, and ^{129}Te. The decay properties of ^{98}Tc and the fission yield of ^{98}Tc from ^{235}U were determined.

The High-Radiation-Level Analytical Facility continued to analyze materials having high levels of alpha radioactivity. Containment and waste-removal operations continued to be improved. In-cell decontamination procedures have been used to reduce the hazards associated with normal cell operations and maintenance.

The conceptual design of the High-Level Alpha Laboratories was completed.

The Statistical Quality Control Program increased during the year; some 5000 control tests were made. The quality of the work remained about the same in one participating laboratory and decreased in the other.

PART C. ORNL MASTER ANALYTICAL MANUAL

15. ORNL Master Analytical Manual

The cumulative indexes to the *ORNL Master Analytical Manual* were updated to make the indexes cumulative for the years 1953 through 1965. The updated indexes are available from the Clearinghouse for Federal Scientific and Technical Information; they are designated TID-7015 (Indexes), Revision 3.

The eighth supplement to the reprinted form of the *Manual* (TID-7015, Suppl. 8) was issued; it includes fourteen new methods and revisions to twenty-three methods. Six additional methods, not included in Suppl. 8, were written for record only; eight methods were discontinued. The Table of Contents to the *Manual* was revised.

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

Part A. Analytical Research

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

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

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1. Analytical Instrumentation

M. T. Kelley
D. J. Fisher
R. W. Stelzner

1.1 CONTROLLED-POTENTIAL AND CONTROLLED-CURRENT CYCLIC VOLTAMMETER

T. R. Mueller H. C. Jones

A controlled-potential, controlled-current cyclic voltammeter was designed around solid-state operational amplifiers for use with solid electrodes in the investigation of the electroanalytical chemistry of molten-salt systems. The instrument has the following capabilities:

1. linear, single or cyclic potential-sweep voltammetry between arbitrary potentials (+3 v to −3 v maximum) at sweep rates between 0.005 and 500 v/sec,
2. Cottrell-type measurements with selectable initial and step (0.01- to 1-v steps) potentials,
3. cyclic chronopotentiometry with selectable switching potentials and with initial-potential control,
4. maximum current of 100 ma in any mode of operation,
5. an accurate time base (0 to 10 v in 20 msec to 2000 sec),
6. facilities for recording output on an *X-Y* recorder, oscilloscope, or digital recorder (external analog-to-digital conversion).

The prototype has been constructed and is being tested and evaluated.

1.2 SOLID-STATE CONTROLLED-POTENTIAL COULOMETRIC TITRATOR

H. C. Jones

The prototype of a controlled-potential coulometric titrator designed to update models now in use in the Analytical Chemistry Division is being fabricated. This instrument will contain Philbrick Researches Inc. solid-state operational amplifiers. Its cell-current capability will be ~100 ma. This titrator should provide stable, reliable performance and should not require frequent or complex maintenance.

1.3 DOUBLE-BEAM FLAME SPECTROPHOTOMETER FOR ATOMIC ABSORPTION

H. C. Jones

The Spectrographic Intensity-Ratio Analog Computer[1] (SIRAC) is being converted for use as a double-beam flame spectrophotometer for atomic absorption. A beam splitter will be used to divert a portion of the light from a hollow-cathode source. This split light beam, falling on a multiplier phototube, will be used to furnish a reference signal to compensate for fluctuations in the hollow-cathode source. The SIRAC circuitry has been modified to reduce the generation of internal noise and to permit connection of the multiplier phototubes now in use. This spectrophotometer will be evaluated as soon as the optical system has been completed and assembled.

1.4 THEORY OF PROGRAMMED-CURRENT DERIVATIVE CHRONOPOTENTIOMETRY

W. D. Shults T. R. Mueller

A communication[2] that presents a generalized theoretical basis for derivative chronopotentiometry was published.[3] Experimental verification of the work is anticipated.

1.5 SQUARE-WAVE POLAROGRAPH

G. C. Barker[4] R. W. Stelzner

The square-wave polarograph,[5] designed at AERE, Harwell, is being constructed at the Woolwich Outstation of the Analytical Sciences Division of the AERE.

[1]H. C. Jones, D. J. Fisher, M. T. Kelley, and R. E. Weekley, "Spectrographic Intensity-Ratio Analog Computer," *Anal. Chem. Div. Ann. Progr. Rept. Dec. 31, 1959*, ORNL-2866, p. 3.

[2]W. D. Shults and T. R. Mueller, "Theory of Programmed-Current Derivative Chronopotentiometry," *J. Electroanal. Chem.* 12, 354 (1966).

[3]T. R. Mueller and W. D. Shults, "Derivative Chronopotentiometry," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1965*, ORNL-3889, p. 8.

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

[5]G. C. Barker and R. W. Stelzner, "A Square-Wave Polarograph," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1965*, ORNL-3889, p. 10.

1.6 APPARATUS FOR PRECISE CONTROL OF THE DROP TIME OF THE DROPPING-MERCURY ELECTRODE (D.M.E.) IN POLAROGRAPHY

W. L. Belew H. C. Jones

When the drop time of the dropping-mercury electrode (D.M.E.) is controlled precisely, several advantages may be realized.[6] The mercury flow rate and drop time can be adjusted independently of each other, thus making possible the matching of $m^{2/3}t^{1/6}$ values for different capillaries. Better precision and sensitivity may result from controlled drop times, since the average-current circuits in the polarograph can be tuned more accurately to the drop time. Drop times obtained at low flow rates with a controlled-drop-time capillary can be shorter than those of a capillary having natural drop times; thus the polarograms can be recorded more rapidly. Because of the possibility of attaining these advantages, work was continued on the development of a practical apparatus to control the drop time of the D.M.E.

To use the drop-time-control apparatus ("drop hammer") in analytical applications for a long period of time, it is necessary for the drop hammer to meet the following criteria:

1. The drop hammer should detach drops reliably without mechanical adjustment for each individual capillary.
2. The drop hammer should operate for long periods of time with minimum service or adjustment. If the device must be adjusted each time a series of polarograms is to be run, its value in routine analytical applications is in doubt.
3. The polarographic current should remain diffusion controlled and should not be increased by undue stirring in the solution.

The two major problems in designing a drop hammer to fulfill these criteria are: eliminating the capillary vibrations, which cause stirring in the solution, and controlling and reproducing precisely for an extended period of time the small displacement of the capillary necessary to detach

[6]W. L. Belew, T. R. Mueller, and H. C. Jones, "Controlled-Potential DC Polarograph-Voltammeter, ORNL Model Q-2792," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1965*, ORNL-3889, p. 5.

the drops. A drop hammer was designed and fabricated that reduces capillary vibration to a minimum; it is being evaluated at present. Preliminary data indicate that reproducible drop times are obtained with the drop hammer for different capillaries without adjustment of the hammer. With use of the drop hammer, polarographic measurements were repeated with excellent precision (S = 0.1%) for drop times from 0.125 to 1.00 sec. For the same capillary with natural drop times, S = 0.3%. The low noise level achieved with the drop hammer for polarograms of 1×10^{-6} M Pb^{2+} indicates that the sensitivity will be equal to that obtained with selected capillaries having natural drop times and that the noise level will not be as dependent on capillary selection.

The drop hammer will be evaluated further to determine its mechanical dependability, the effect of drop-time control on polarographic diffusion constants, and the effect of its use on the precision and sensitivity achieved in polarography.

1.7 IMPROVED AVERAGING FILTERS FOR POLAROGRAPHIC INSTRUMENTATION

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

Active-network filters that remove the current oscillations arising from the growth and fall of the mercury drops in the polarographic technique have been designed mathematically. These electronic filters represent a compromise among criteria for maximum attenuation, minimum delay, and zero overshoot. Butterworth, third-order time-averaging, third-order zero-overshoot, and third- and sixth-order Paynter filters were constructed, tested, and rejected. A fourth-order, active-network, low-pass filter followed by a dual parallel-T active network (tuned to the fundamental and second harmonic of the drop frequency) constitutes the best system found to date.

The new filter system, which has been installed in an ORNL model Q-2792 controlled-potential dc polarograph-voltammeter,[7] achieves a better signal-to-noise ratio, closer adherence to the mathematical time-derivative of the polarographic curve, and superior resolution.

[7]W. L. Belew, T. R. Mueller, and H. C. Jones, "Controlled-Potential DC Polarograph-Voltammeter, ORNL Model Q-2792," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1965*, ORNL-3889, pp. 4–5.

1.8 EVALUATION OF A CONTROLLED-POTENTIAL AC-DC POLAROGRAPH

R. W. Stelzner T. M. Florence[8]

A controlled-potential ac-dc polarograph[9] designed at ORNL is being evaluated for operational characteristics on chemical systems.

1.9 EVALUATION OF GRAPHITAR 14 AS A WORKING ELECTRODE FOR VOLTAMMETRIC ANALYSIS

T. R. Mueller

The appraisal of a material for fabricating stationary electrodes for voltammetry was mentioned previously.[10] Graphitar[11] 14 has been used for some time as a plunger in the remotely operated ORNL model Q-1348 pipetter for handling corrosive liquids. Its resistance to chemical attack, low porosity, and excellent lapping characteristics make it ideal for this application. These properties, in addition to its good electrical conductivity, made it attractive as a possible working electrode for voltammetric analysis. Graphite and carbon electrodes have been used successfully in several forms for voltammetric analysis. Graphitar appeared to have the desirable properties of these forms with the added advantage of ease of fabrication into practical electrodes.

Several polished electrodes of Graphitar 14 were prepared, and their electrochemical behaviors were investigated in KCl and H_2SO_4 solutions. The reductions of potassium ferricyanide in 1 M KCl and of ferric sulfate in 0.5 M H_2SO_4 were studied at these electrodes; also, the anodic and cathodic charging curves in the supporting electrolyte alone were observed. The reductions of the electroactive species at the 1 mM level were reproducible within ±1% on a short-term basis.

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

[9]R. W. Stelzner, "Controlled-Potential AC Polarography," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1963*, ORNL-3537, pp. 6–7.

[10]W. L. Belew, T. R. Mueller, and H. C. Jones, "Controlled-Potential DC Polarograph-Voltammeter, ORNL Model Q-2792," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1965*, ORNL-3889, p. 4.

[11]Trademark of the United States Graphite Co. for a resin-impregnated high-density mixture of carbon and graphite.

However, gradual etching of the electrodes rendered them unusable after a day. Results with solutions of KCl alone showed changes of several hundred percent in the residual current during one day. Photomicrographs of electrode surfaces exposed to 1 *M* KCl for 48 hr showed severe etching and large voids. Etching with 0.5 *M* H_2SO_4 was evident visually but was less severe. In all the experiments, the electrode potential was controlled and was kept below those potentials at which graphite is known to oxidize.

It was concluded that, although there may be some combination of solvent and electrolyte in which these electrodes might be superior to other forms of graphite electrodes, Graphitar 14 seems to offer little promise as a practical inert electrode for voltammetric analysis.

1.10 CHRONOPOTENTIOMETRY WITH COMPENSATION FOR EXTRANEOUS CURRENTS

T. R. Mueller W. D. Shults

The method of chronopotentiometry with compensation for extraneous currents and some applications of it have been reported.[12–14] The previous work was done with platinum electrodes. Extensive refinements were made in the instrument. Then an attempt was made to apply the technique to mercury-pool electrodes. The experiments were complicated by the difficulties associated with the design of cells suitable for the pool electrodes. Cells of the type used by Kuempel and Schaap[15] have been procured and will be used to investigate several electrochemical systems with planar-mercury electrodes. Additional studies with solid electrodes are being planned.

[12]W. D. Shults, T. R. Mueller, F. E. Haga, and H. C. Jones, "Chronopotentiometer with Compensation for Extraneous Currents," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1965*, ORNL-3889, p. 1.

[13]D. G. Peters, W. D. Shults, and T. R. Mueller, "Evaluation of Technique of Chronopotentiometry with Compensation for Extraneous Currents," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1965*, ORNL-3889, p. 2.

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

[15]J. R. Kuempel and W. B. Schaap, "Construction and Evaluation of Planar Mercury Pool Electrodes," *Anal. Chem.* **38**, 664 (1966).

1.11 CONTROLLED-POTENTIAL DIFFERENTIAL DC POLAROGRAPHY

W. D. Shults H. C. Jones
D. J. Fisher M. T. Kelley
W. B. Schaap[16]

The study of controlled-potential differential dc polarography, mentioned first in the last annual report,[17] was completed. The work was both presented orally[18] and published in thesis form.[19] This work is to be described in a series of articles in the open literature; two papers have been accepted and two others are planned.

The first paper[20] in the series deals with instrumentation, apparatus, and techniques. The abstract from the paper follows:

"This is the first in a series of papers dealing with controlled-potential differential direct-current polarography. The design of a controlled-potential differential polarograph that utilizes nonsynchronized dropping-mercury electrodes is given. The instrument incorporates circuitry to remove "drop oscillations" from conventional or differential polarograms. A gain adjustment is included in one channel so that effective equalization of the capillary characteristics of the dual electrodes can be accomplished electronically. The validity of this gain adjustment technique is evaluated. The performance of the instrument when utilized with each of the several techniques comprising differential polarography--the ΔE-differential, subtractive, and comparative polarographic techniques--is illustrated."

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

[17]W. D. Shults, D. J. Fisher, W. B. Schaap, and H. C. Jones, "Controlled-Potential Differential DC Polarography," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1965*, ORNL-3889, pp. 10–12.

[18]W. D. Shults, "Controlled-Potential Differential DC Polarography, presented at Analytical Group Seminar, Chemistry Department, Indiana University, Bloomington, April 4, 1966.

[19]W. D. Shults, *Controlled-Potential Differential DC Polarography* (Ph.D. thesis), Indiana University, June, 1966.

[20]W. D. Shults, D. J. Fisher, H. C. Jones, M. T. Kelley, and W. B. Schaap, "Controlled-Potential Differential DC Polarography I. Instrumentation, Apparatus, Techniques," *Zeitschrift fuer Analytische Chemie*, in press.

The second paper[21] in the series deals with the ΔE-*differential* polarographic technique; the abstract follows:

"A brief theoretical and experimental study is presented of the polarographic technique in which a constant voltage difference (ΔE) is maintained between two electrodes immersed in the same or identical solutions throughout the potential scan in order to obtain a derivative-like curve of differential current (Δi) *vs.* electrode potential (E). A comparison of the curves obtained by this technique with the "true" first derivative of the polarographic wave, as observed by operational-amplifier computation, is also given."

A third paper in this series will present the results of our studies of *comparative* polarography. Single- and dual-cell comparative techniques were described in the previous annual report, and some results were given.[17] Further work with comparative polarography included: experimental evaluation of both techniques at the 10^{-5} M concentration level, mathematical treatments of the determinate and statistical errors associated with the comparative technique, experimental verification of these error analyses, and derivation of an expression that gives the optimum "compensation fraction" (i.e., the optimum concentration of standard solution relative to that of the unknown solution) for a given set of experimental conditions.

The *subtractive* polarographic technique was studied further to establish its true worth relative to that of other polarographic techniques. Solutions that contained Cd^{2+}, Cu^{2+}, O_2, Tl^+, In^{3+}, and some mixtures of these were studied by subtractive polarography as well as by conventional and first-derivative polarography. The significant conclusions drawn from these studies are that: subtractive polarography affords improved sensitivity, selectivity, and resolution over that of conventional polarography, the sensitivity and selectivity of the subtractive and first-derivative polarographic techniques are comparable, and the resolution of subtractive polarography is superior to that of first-derivative polarography. These studies will be reported in the fourth paper in the series dealing with differential polarography.

1.12 HIGH-PRECISION DC POLAROGRAPHY

E. S. Wolfe[22] W. L. Belew
D. J. Fisher R. W. Stelzner
H. C. Jones

The relative advantages and the practical limitations of several high-precision one- and two-cell methods of dc polarography are being investigated and elucidated. Null-point, two-cell differential titration and one-cell, electrical-null methods were considered but have not been evaluated experimentally.

The use of the second cell in two-cell comparative polarographic methods has been justified on the basis that it compensates for common-mode errors and signal drifts and provides for scale expansion. It appears, however, that the compensation is achieved only to a first approximation. To closely match errors and drifts of the two cells over a reasonable time interval, exact control and matching of experimental conditions is required for both cells. Hence, one-cell methods, such as comparative amperometric dc polarography,[23] are also of practical interest for achieving highly precise and accurate polarographic measurement of concentration. High precision ($S \leq 0.1\%$) has been obtained both by one- and two-cell dc polarographic methods on replicate measurements of a sample. Because of their greater operational simplicity, one-cell methods are preferred. The primary problem with one- and two-cell methods is to consistently and reliably obtain high precision over a long time interval (days). Studies were made and are continuing of reproducibility of the instrument performance and of the diffusion current as affected by the designs and behaviors of the cell and the D.M.E. Further work on the development of several high-precision dc polarographic methods and the required instrumentation is in progress.

[21] W. D. Shults and W. B. Schaap, "Controlled-Potential Differential DC Polarography II. The ΔE-Differential Technique," *Zeitschrift fuer Analytische Chemie*, in press.

[22] ORNL Student Technical Assistant from the University of North Carolina, Chapel Hill.

[23] D. J. Fisher, D. Thiele, W. D. Shults, and W. L. Belew, "Comparative Amperometric DC Polarography: A New High-Precision and High-Accuracy Method," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1965*, ORNL-3889, pp. 3–4.

1.13 REMOTELY OPERATED INSTRUMENTATION FOR THE ANALYTICAL HOT CELLS OF THE TRANSURANIUM PROCESSING PLANT

1.13.a Analytical Balance

W. L. Maddox

A Voland model 750D analytical balance was modified for use in the analytical hot cells of the Transuranium Processing Plant. To it were added: an electro-optical system that indicates the beam position on a remote meter, a synchro drive for the 100-mg weighing chain, special large knobs to facilitate master-slave manipulation of the zero adjustment and of weights greater than 100 mg, and electric motors to drive the enclosure door and the beam-pan-weight arrestment.

1.13.b Titrant-Delivery Unit and Elevator for the ORNL Model Q-1728 Potentiometric Titrators

W. L. Maddox

Design was completed of a new remotely operated titrant-delivery unit and an associated elevator for use with the ORNL model Q-1728 series potentiometric titrators. Two of the new delivery units and one of the elevators were built, tested, and put into service in the analytical cells of the Transuranium Processing Plant. The drawings for these are: ORNL-Dwgs. 66-2700–66-2704 for the delivery unit and 66-2705 and 66-2706 for the elevator. The drawings will be forwarded to the Division of Technical Information Extension for publication in the *Engineering Materials List*, TID-4100.

1.13.c ORNL Model Q-2564 High-Sensitivity Coulometric Titrator

H. C. Jones

An additional ORNL model Q-2564 high-sensitivity coulometric titrator[24–27] was fabricated, checked out, and delivered for use in the analytical hot cells of the Transuranium Processing Plant. Six of these instruments (including the prototype) are now in use at ORNL, two each in the General Hot Analyses and High-Level Alpha Radiation Laboratories, one in the Methods Development Group, and this one in the Transuranium Processing Plant.

1.13.d Model VIIA Flame Spectrophotometer

H. C. Jones

A model VIIA flame spectrophotometer was fabricated, checked out, and installed in the analytical hot cells of the Transuranium Processing Plant. Except for minor circuit modifications, the model VIIA is essentially the same as the model VII.[28] The flame housing is located inside a glove box and is bolted through the end of the glove box to a bracket that carries the chopper motor and monochromator. A quartz window in the end of the glove box allows passage of the light beam from the flame housing to the monochromator. The instrument will be used to determine lithium.

1.13.e Solvent-Extraction Apparatus

T. R. Mueller

The remotely operated solvent-extraction apparatus[29,30] consists of a shaker for safely effecting rapid and intimate contact between the immiscible phases in liquid-liquid extractions and a phase-separation device that uses disposable sample vials and syringes and does not require visual observation of the interface between phases.

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

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

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

[27] H. C. Jones and W. D. Shults, "High-Sensitivity Coulometric Titrator, ORNL Model Q-2564," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1965*, ORNL-3889, p. 6.

[28] 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.

[29] "Summary," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1965*, ORNL-3889, p. xiii.

[30] T. R. Mueller, "Remotely Operated Solvent Extraction Apparatus," *Transuranium Quarterly Progr. Rept. Oct. 31, 1965*, ORNL-3965, p. 88.

The apparatus is in use in a hot cell in the analytical laboratories of the Transuranium Processing Plant. In addition to its primary design function, the phase-separation device has been used both as a pipetter and to separate liquids from solids.

1.14 MODIFICATIONS TO THE ORNL MODEL Q-2564 HIGH-SENSITIVITY CONTROLLED-POTENTIAL COULOMETRIC TITRATOR

T. R. Mueller H. C. Jones

When the ORNL model Q-2564 coulometric titrator[31] is used to analyze concentrated solutions, the initial cell-current demand may exceed the capability of the potentiostat. There are several consequences of this situation. The potentiostat is forced to its voltage limit, potential control in the cell is lost, and excessively large currents are drawn through the reference electrode. When the overload condition is removed by sufficient electrolysis, the limited potentiostat may remain in this condition for some time because of the long time-constant of its slow channel. Since the concentration of the electroactive species of interest is now too low to consume all the current being forced through the cell, the potential of the counter electrode with respect to the S.C.E. must increase to a value that corresponds with an additional electrode process. Therefore, analytical results will be high, and the reference electrode, depending on its size and type, may be damaged.

To minimize these difficulties, fast overload-recovery circuitry was added to the potentiostat, and the reference electrode was coupled into the potentiostat with a high-input-impedance amplifier. With these modifications, the potentiostat recovers within about 10 msec, and the current through the reference electrode is limited to 150×10^{-12} amp. Detailed circuit modifications will be published after field testing is completed.

1.15 ADDITION OF 1-sec FILTER CIRCUITS TO THE ORNL MODEL Q-1988-FES POLAROGRAPH

R. W. Stelzner W. L. Belew
Helen P. Raaen[32]

Symmetrical parallel-T filter networks, tuned for a mercury drop time of 1.0 sec, and low-pass *RC* networks were designed for the ORNL model Q-1988-FES polarograph.[33] The calculated delay-time of the filter system is 1.60 sec. The circuits were installed in the instrument being used for studies of the Teflon D.M.E.

1.16 APPARATUS FOR DEAD-STOP END-POINT DETERMINATION OF CHLORIDE

H. C. Jones T. R. Mueller

A Philbrick Researches Inc. solid-state operational amplifier is used to terminate the cell current and to stop the timer in the constant-current determination of chloride. The amplifier is used as a voltage-crossing detector. It compares an adjustable fixed reference potential from its own power supply with a signal potential obtained from a Leeds and Northrup pH indicator. The pH indicator receives its input from the indicating electrodes in the cell. A relay connected to the output of the amplifier terminates the cell current and stops the timer.

1.17 CIRCUIT DIAGRAM FOR ORNL MODEL Q-1988-FES POLAROGRAPH

W. L. Belew

A circuit diagram was drawn that shows the modifications necessary to convert a model Q-1988-A polarograph[34] to a model Q-1988-FES polarograph.[35] The necessary modifications are: faster scan rates, duel 0.5-sec parallel-T (tuned to the fundamental and second harmonic of the drop frequency) average-current circuits, a faster derivative circuit, and an inverter-gain switch.

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[31] H. C. Jones, W. D. Shults, and J. M. Dale, "High-Sensitivity Controlled-Potential Coulometric Titrator," *Anal. Chem.* 37, 680 (1965).

[32] Methods Development Group.

[33] W. L. Belew, "Modification of the ORNL Model Q-1988A Controlled-Potential, Derivative Polarograph for Use with a Rapidly Dropping Mercury Electrode," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1963*, ORNL-3537, pp. 1–2.

[34] M. T. Kelley, D. J. Fisher, W. D. Cooke, and H. C. Jones, "Controlled-Potential and Derivative Polarography," pp. 158–182 in *Advances in Polarography*, ed. by I. S. Longmuir, vol. 1, Pergamon, Oxford, 1960.

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

1.18 INSTRUMENTATION FOR ANALYTICAL BIOCHEMISTRY

W. L. Maddox

The Bioanalytical Development Group has proposed a system for the routine assay of tRNA samples by use of the Technicon AutoAnalyzer (Sect. 5.1.e). Equipment will be designed in the Analytical Instrumentation Group for automatically transferring aliquots from the product stream of the AutoAnalyzer to filter-paper disks.

A receiver for the disks was designed, and the prototype of it has been fabricated. Means of actuating a sampler and coupling it to the product stream are now being investigated.

1.19 ELECTRON EXCHANGE BETWEEN COPPER(I) AND COPPER(II) IN ACETONITRILE

S. E. Manahan[36]

The electron-exchange reaction

$$Cu^*(I) + Cu(II) \rightleftharpoons Cu^*(II) + Cu(I)$$

was investigated in acetonitrile by use of ^{64}Cu tracer. After the exchange reaction, the Cu(I) and Cu(II) were separated by precipitation of Cu(I) as $Cu(CH_3CN)_4ClO_4$ from solution at $-40°C$.

In all cases exchange was complete within the time of separation. If it is assumed that exchange is first-order in respect to both Cu(I) and Cu(II), the exchange rate, R, is described by the expression

$$R = k[Cu(I)][Cu(II)] ,$$

where k is the rate constant. A minimum value of 0.3 F^{-1} sec^{-1} was determined for k. A note on this work was submitted for publication.[37]

[36]ORAU Research Participant, summer 1966; Assistant Professor of Chemistry, Department of Chemistry, University of Missouri, Columbia.

[37]S. E. Manahan, "Electron Exchange between Copper(I) and Copper(II) in Acetonitrile," submitted for publication in the *Journal of Physical Chemistry*.

2. Effects of Radiation on Analytical Methods

J. C. White

2.1 VOLTAMMETRY OF THE IODINE SYSTEM IN AQUEOUS MEDIUM AT THE PYROLYTIC-GRAPHITE ELECTRODE (P.G.E.)

F. J. Miller[1] H. E. Zittel

The voltammetry of the iodine system in aqueous medium at the pyrolytic-graphite electrode (P.G.E.) was studied, and a paper on the work has been published;[2] the abstract follows:

"The voltammetric behavior of the I_2-I^- system was studied with the P.G.E. At the P.G.E., a voltage scan of a solution of I^- in acid or neutral medium shows three voltammetric waves that correspond to three successive oxidations. The peak potentials of these waves occur at about +0.5, +0.9, and +1.2 v *vs.* S.C.E. A solution of I_2 in the same medium exhibits two anodic waves that correspond to the latter two seen when I^- is the electroactive species. The data indicate that the observed anodic waves result from the stepwise reaction

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

The variables that affect these electrode reactions have been studied. It was established that the

[1]Methods Development Group.

[2]F. J. Miller and H. E. Zittel, "Voltammetry of the Iodine System in Aqueous Medium at the Pyrolytic Graphite Electrode," *J. Electroanal. Chem.* 11, 85 (1966).

first anodic wave is suitable for the determination of I^-. The second anodic wave has not been reported previously; on the basis of the data obtained, an electrode reaction for this wave is proposed."

2.2 CHRONOPOTENTIOMETRY OF THE IODINE SYSTEM AT THE PYROLYTIC-GRAPHITE ELECTRODE (P.G.E.) AND GLASSY-CARBON ELECTRODE (G.C.E.)

H. E. Zittel F. J. Miller[1]

A chronopotentiometric study of the iodine system at the P.G.E. and the glassy-carbon electrode (G.C.E.) was concluded; the results are given in an article accepted for publication.[3]

It was determined that the anodic reactions of the I_2-I^- system at both the P.G.E. and G.C.E. follow the scheme

$$I^- \longrightarrow \tfrac{1}{2}I_2 + e\,, \qquad (2.1)$$

$$\tfrac{1}{2}I_2 + H_2O \longrightarrow IO^- + 2H^+ + e\,, \qquad (2.2)$$

$$IO^- + 2H_2O \longrightarrow IO_3^- + 4H^+ + 4e\,. \qquad (2.3)$$

The effects of variables such as $[H^+]$, $[I^-]$, and $[I_2]$ were established.

The deviation of measured τ and $E_{1/2}$ values from theoretical values based on the above reactions was shown to be attributable to: oxidation of I^-, which follows an alternate-path reaction; adsorption of an electroactive species on the P.G.E. or G.C.E. during reactions (2.1) and (2.2); and a chemical reaction that follows reaction (2.2) and causes the corresponding τ value to be decreased.

2.3 ANODIC REACTIONS OF THE HALIDE IONS IN DIMETHYL SULFOXIDE AT THE PYROLYTIC-GRAPHITE ELECTRODE (P.G.E.)

H. E. Zittel F. J. Miller[1]

The effect of radiation on aqueous solutions of halide ions has been of continuing interest. This effect is almost invariably deleterious. Therefore, the use of nonaqueous media in the determination of halide ions is being considered. A study of the reaction of halide ions in such media was begun. Since dimethyl sulfoxide is a common nonaqueous solvent for inorganic substances, it was selected for the first study.

The anodic reactions of the halide ions in dimethyl sulfoxide at the P.G.E. were studied. The Cl^-, Br^-, and I^- ions demonstrate one-, two-, and three-step oxidations respectively. The electrode reaction $X^- \rightarrow \tfrac{1}{2}X + e$ is complicated by a catalytic reaction that causes considerable enhancement of the diffusion currents for Br^- and Cl^-. The charge-transfer value, αn_a, for all three primary reactions is ~ 0.5.

The anodic reactions of the halide ions in dimethyl sulfoxide at the P.G.E. are postulated to be

$$X^- \longrightarrow \tfrac{1}{2}X_2 + e\,, \qquad (2.4)$$

$$\tfrac{1}{2}X_2 \longrightarrow X^+ + e\,. \qquad (2.5)$$

The nascent halogen formed in reaction (2.4) may react with dimethyl sulfoxide to give an irreversible-catalytic-irreversible type of coupled reaction that leads to a multistep charge transfer. It was shown by studies of peak current as a function of voltage scan rate that the catalytic reaction is slow for I^-, intermediate for Br^-, and fast for Cl^-.

An article on this work was accepted for publication.[4]

2.4 VOLTAMMETRIC AND SPECTROPHOTOMETRIC STUDIES OF THE ZIRCONIUM–ALIZARIN RED S COMPLEX

H. E. Zittel T. M. Florence[5]

At a rotated P.G.E., alizarin red S (i.e., sodium alizarin-3-sulfonate) produces well-formed two-electron-change oxidation and reduction waves. The Zr–alizarin red S complex is oxidized at a potential that is 0.35 v more positive than that

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[3]H. E. Zittel and F. J. Miller, "Chronopotentiometry of the Iodine System at the Pyrolytic-Graphite and Glassy-Carbon Electrodes," *J. Electroanal. Chem.* (in press).

[4]H. E. Zittel and F. J. Miller, "Anodic Reactions of the Halides in Dimethyl Sulfoxide at the Pyrolytic-Graphite Electrode," *Anal. Chim. Acta* (in press).

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

of the free dye. A mixture of alizarin red S and its Zr complex exhibits two discrete oxidation waves. In the ranges of acidity and Zr concentration studied, a stable, stoichiometric 1:1 complex is formed between Zr and alizarin red S. Both the voltammetric and spectrophotometric data indicate that Zr chelates with alizarin red S through the two phenolic oxygens rather than through one phenolic oxygen and the quinone group. Spectrophotometric and kinetic studies showed that the reaction proceeds by way of a hydrolyzed species, probably $Zr(OH)_2^{2+}$. The equilibrium constant for the reaction

$$Zr^{4+} + 2H_2O + H_2A \rightleftharpoons Zr(OH)_2A + 4H^+ ,$$

where H_2A represents alizarin red S, was found to be 4.2×10^4, and the corresponding mixed stability constant,

$$\beta_{121} = \frac{[Zr(OH)_2A]}{[Zr]\,[OH]^2\,[A]} ,$$

was calculated as $\log \beta_{121} = 49.0$ at 25°C and $\mu = 1.56\ M$. The acid dissociation constants of alizarin red S were determined spectrophotometrically to be pK_1^a (β-OH) = 5.49 ± 0.01 and pK_2^a (α-OH) = 10.85 ± 0.03 at 25°C and $\mu = 0.5\ M$.

The results of this study were submitted for publication.[6]

2.5 VOLTAMMETRIC METHOD FOR THE DETERMINATION OF ZIRCONIUM

H. E. Zittel T. M. Florence[5]

Alizarin red S and similar hydroxyanthraquinone dyes have been used for the spectrophotometric determination of various metals. Zirconium is one of the few metals that form a colored complex with alizarin red S in highly acid solutions. The complex is very susceptible to radiation damage, because the quinone structure is readily attacked by reducing radicals. Therefore, a voltammetric method for determining Zr as the Zr–alizarin red S complex was developed. This method depends on the chemical reactivity of functional groups not readily affected by radiation.

Alizarin red S shows both anodic and cathodic reactions at the P.G.E. A solution that contains a mixture of free alizarin red S and its Zr complex exhibits two discrete oxidation waves. The Zr concentration can be determined by measuring either the height of the wave for the complex or the decrease in height of the free-dye wave. Zirconium in concentrations as low as 0.1 ppm can be determined with a simple polarographic apparatus; the precision and accuracy of the method compare favorably with those of other methods available for the determination of traces of Zr. Since the procedure is carried out in strongly acid medium, very few interferences occur. Of those ions normally present with Zr, PO_4^{3-} is the only serious interference. The method is based on the oxidation of the α- and β-hydroxy groups of alizarin red S to the quinone structure.

The results of this study were submitted for publication.[7]

2.6 DISSOLUTION OF SOME INERT METALS IN IRRADIATED ACID CHLORIDE SOLUTIONS

Hisashi Kubota

The more inert metals are frequently used to contain chemical process solutions, whereas noble metals are used as electrode materials. The behaviors of six relatively inert metals – Au, Zr, W, Pt, Nb, and Ta – in irradiated 12 *M* HCl solution, which is known to produce Cl_2, were investigated. Their behaviors in irradiated 12 *M* HCl, nonirradiated 12 *M* HCl, and nonirradiated 12 *M* HCl saturated with Cl_2 were compared. No dissolution of any of these metals was observed in nonirradiated 12 *M* HCl, which was expected. Chlorine-saturated nonirradiated 12 *M* HCl dissolves measurable amounts of Au, Zr, W, and Pt; Nb and Ta remained unaffected. All these metals except Ta dissolve measurably in irradiated 12 *M* HCl; Ta is attacked very slowly.

The rate of dissolution of each metal is a function of the total Cl_2 produced and is nearly independent of the surface area. Thus, the rate is faster in HCl solution of higher concentration, because the rate of Cl_2 formation is higher in it.

[6]H. E. Zittel and T. M. Florence, "Voltammetric and Spectrophotometric Study of the Zirconium–Alizarin Red S Complex," submitted for publication in *Analytical Chemistry*.

[7]H. E. Zittel and T. M. Florence, "A Voltammetric Method for the Determination of Zirconium," submitted for publication in *Analytical Chemistry*.

The rates of dissolution of six metals immersed as foils in a large volume of irradiated 12 *M* HCl are given below.

Metal	Rate of Dissolution: Milligrams/10^6 rads	Rate of Dissolution: Micromoles/10^6 rads
Au	2.50	12.7
Zr	1.12	12.4
W	1.10	6.02
Pt	1.09	5.58
Nb	0.03	0.03
W	Negligible	Negligible

Although the absolute rates of dissolution are not large, the amounts of the dissolved metals are enough to interfere in analytical procedures, particularly colorimetric and electrometric.

This radiolysis is a convenient way to prepare dilute standard solutions of these metals, except Zr, in acid chloride medium free of nitrates. Zirconium is partially converted to an insoluble oxide during the dissolution.

2.7 HIGH-TEMPERATURE CHEMICAL DOSIMETRY

Hisashi Kubota

The discrepancy between the low *G* values of deaerated $FeSO_4$ dosimeters reported previously[8] and values published in the literature was corrected. It was found that sealing the tube which contained dosimetry solution at a point wet with part of the solution caused the low values. Deaerating the solution by alternately freezing and thawing it under vacuum and introducing argon gas into the tube just before the thawing stage leaves the inner wall of the radiation tube above the dosimetry solution dry for the subsequent sealing operation. The *G* value obtained for a dosimeter prepared in this way was 8.07 ± 0.07 at 25°C, which compares favorably with the normally accepted value, 8.2.

The responses of this dosimeter to radiation dose at 150, 200, and 250°C differ very little (Fig. 2.1). The response at 250°C is slightly low; however, these values are within the standard deviation at each dose level. Thus, a single line was drawn to represent the response of this dosimeter in the temperature range 150 to 250°C. The slope of this line is equivalent to a *G* value of 8.1. The response begins to decrease as the dose is increased. This effect is possibly the same as that described by Hochanadel and Ghormley for an air-saturated $FeSO_4$ dosimeter up to 72°C.[9]

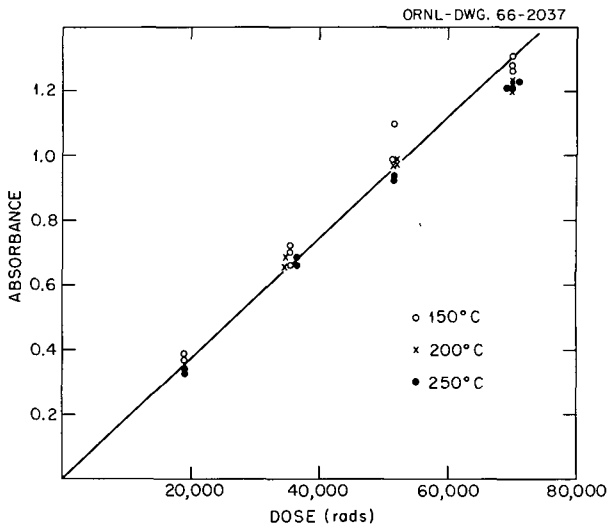

Fig. 2.1. Response of Deaerated Ferrous Sulfate Dosimeter at Elevated Temperature.

The $FeSO_4$ dosimeter has been in use since January, when the first spent-fuel elements were stored. The readings to date agree well with those taken with a phosphate-glass dosimeter and also with the values predicted from theoretical calculations.

2.8 GAMMA RADIOLYSIS OF ACID CHLORIDE SOLUTIONS

Hisashi Kubota

Alcohols other than methanol were tested as possible agents to scavenge the Cl_2 produced in gamma-irradiated acid chloride solutions. The polyhydroxy alcohols ethylene glycol and glycerol are more effective than methanol. When either is

[8] H. Kubota, "High-Temperature Chemical Dosimetry," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1965*, ORNL-3889, p. 16.

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

added at 5% concentration, the Cl_2 is completely removed after a small induction period. Phenol and catechol were also tested on the basis that the products of their reactions with Cl_2 would be more stable to further reaction once they had scavenged the Cl_2. Although they are effective in removing Cl_2, they tend to produce discolored products, presumably polymeric material.

When formaldehyde in 0.2 *N* HCl–10 *M* LiCl is irradiated, the HCl concentration decreases, but two weak acids are produced, the more abundant and stronger of which is HCO_2H. Formic acid is sufficiently strong that it cannot be differentiated from HCl by titration in water or in 10 *M* LiCl. The acidity functions of HCl and of HCO_2H in water and in 10 *M* LiCl were determined by use of the indicators *o*- and *p*-nitroaniline. The depression in the absorbance of these indicators by HCO_2H in 10 *M* LiCl is greater than that by HCl in water. This fact indicates that HCO_2H is as strong an acid in 10 *M* LiCl as HCl is in water and therefore should be as effective in suppressing the hydrolysis of many cations.

The very high activity coefficients of H^+ in 10 *M* LiCl would possibly cause a measurable loss of volatile acid from this medium when left exposed to the atmosphere, as is the case in the transuranium processing operations. Solutions of HCl and HCO_2H were prepared in water and in 10 *M* LiCl and were placed in flasks open to air. Their acidities were checked with time. In 10 *M* LiCl the HCl concentration decreased from 0.20 to 0.17 *N* in 4 days and to 0.14 *N* in 12 days; the HCO_2H concentration decreased from 0.20 to 0.19 *N* in 4 days and to 0.18 *N* in 12 days. In water no detectable change in the concentrations of these acids occurred. The relative ease with which the acids escape from a concentrated salt solution, even at room temperature, is a fact to be considered in process work in which concentrated salt solutions are used.

The use of an aqueous solution of chloral hydrate as a dosimeter for low-level x rays or gamma rays has been proposed; its usefulness depends on its decrease in pH with increase in absorbed radiation dose.[10–12] The decrease in pH is attributed to the formation of HCl as a radiolytic product. Chloral hydrate would then be a suitable reagent to place in 10 *M* LiCl solutions that undergo acid depletion by radiolysis, since one of its radiolytic products, HCl, will compensate for the acid being lost. The gaseous product from the radiolysis of chloral hydrate solution is mostly CO_2. The CO_2 escapes readily from the LiCl medium and will not form complex products that could possibly contaminate the system. The use of this reagent as a possible counteractant to the loss in acid is being pursued further.

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[10] H. L. Andrews and P. A. Shore, "X-Ray Dose Determinations with Chloral Hydrate," *J. Chem. Phys.* 18, 1165 (1950).

[11] K. Sugimoto and S. Musha, "Radiation Chemistry of Aqueous Chloral Hydrate Solutions – Application to Continuous Dosimetry and Roentgenometric Titration," *Bull. Univ. Osaka Prefect.*, Ser. A 11, 67 (1962).

[12] W. Boyd, L. E. Brownell, and M. A. Farvar, "Chloral Hydrate: 800 Rad Dosimeter," *Nucleonics* 24(5), 6 (1966).

3. Analytical Chemistry for Reactor Projects

J. C. White
A. S. Meyer

3.1 MOLTEN-SALT REACTOR EXPERIMENT

3.1.a Determination of Oxide in MSRE Salts

R. F. Apple J. M. Dale
A. S. Meyer

An apparatus for the determination of oxide in radioactive molten fluoride salts, together with the necessary sample-handling equipment, was designed, constructed, and installed in a hot cell of the High-Radiation-Level Analytical Laboratories (HRLAL). The apparatus was checked out with simulated fuel-salt samples and was then used to analyze salt samples taken from the reactor at maximum reactor power. The selected method is based on the reaction

$$O^{2-} + 2HF \longrightarrow 2F^- + H_2O ,$$

which occurs when a molten-salt sample is purged with an H_2-HF gas mixture. The amount of water evolved is taken as a measure of the quantity of oxide in the molten-salt sample. The prime considerations in the design of the apparatus were that it must be compatible with the existing sampling technique and must minimize the chances of contamination by the moist atmosphere of the hot cell. Because large (50-g) samples can be remelted and purged in the original sample ladle, the atmospheric exposure during transfer is limited to the upper surface of the frozen salt.

Preliminary studies[1] performed with the Karl Fischer titration demonstrated that BeO, UO_2, and ZrO_2 added in standard amounts were rapidly hydrofluorinated by HF in H_2 at partial pressures greater than 0.2 atm. For more convenient measurements in the hot cell, the water remaining in the hydrofluorinator effluent subsequent to the removal of HF with an NaF trap is measured with a P_2O_5 water electrolysis cell (Beckman Instruments Inc. moisture-monitor cell with rhodium electrodes). The integrated function, cell current vs time, serves as a measure of the water generated by hydrofluorination. Because there is a maximum water-influx rate beyond which the cell can be damaged, it was necessary to divide the effluent gas stream with a capillary splitter and to pass only about 5% of the stream through the cell. Figure 3.1 is a schematic flow diagram that shows the essential features of the hot-cell apparatus and the prototype bench-top apparatus.

Known amounts of ZrO_2, added to a 50-g melt of prehydrofluorinated MSRE salt, were recovered quantitatively (101 ± 2%). Also, the quantitative evolution of oxide was confirmed by the lack of evolution of additional water from terminal (oxide-depleted) melts when the hydrofluorinating gas was enriched or the melt temperature was varied.[2] Table 3.1 shows the reproducibility of the combined sampling and analysis techniques; the results are from analyses of successive samples withdrawn in copper ladles from a 6-kg reservoir of MSRE salt. The first four samples were transferred to the hydrofluorinator in a dry box, whereas

[1] "Development and Evaluation of Methods for the Analysis of the MSRE Fuel," *MSR Program Semiann. Progr. Rept. Feb. 28, 1965*, ORNL-3812, p. 162.

[2] "Development and Evaluation of Methods for the Analysis of the MSRE Fuel," *MSR Program Semiann. Progr. Rept. Feb. 28, 1966*, ORNL-3936, p. 157.

Table 3.1. Results of Determination of Oxide in Molten Simulated MSRE Fuel

Sample No.	Oxide (ppm)
1	350
2	345
3	340
4	340
5	375[a]
6	360[a]
7	405[b]

[a] Exposed to 100%-humidity atmosphere for at least 24 hr.

[b] Exposed to room atmosphere for two weeks.

ORNL-DWG 65-8855C

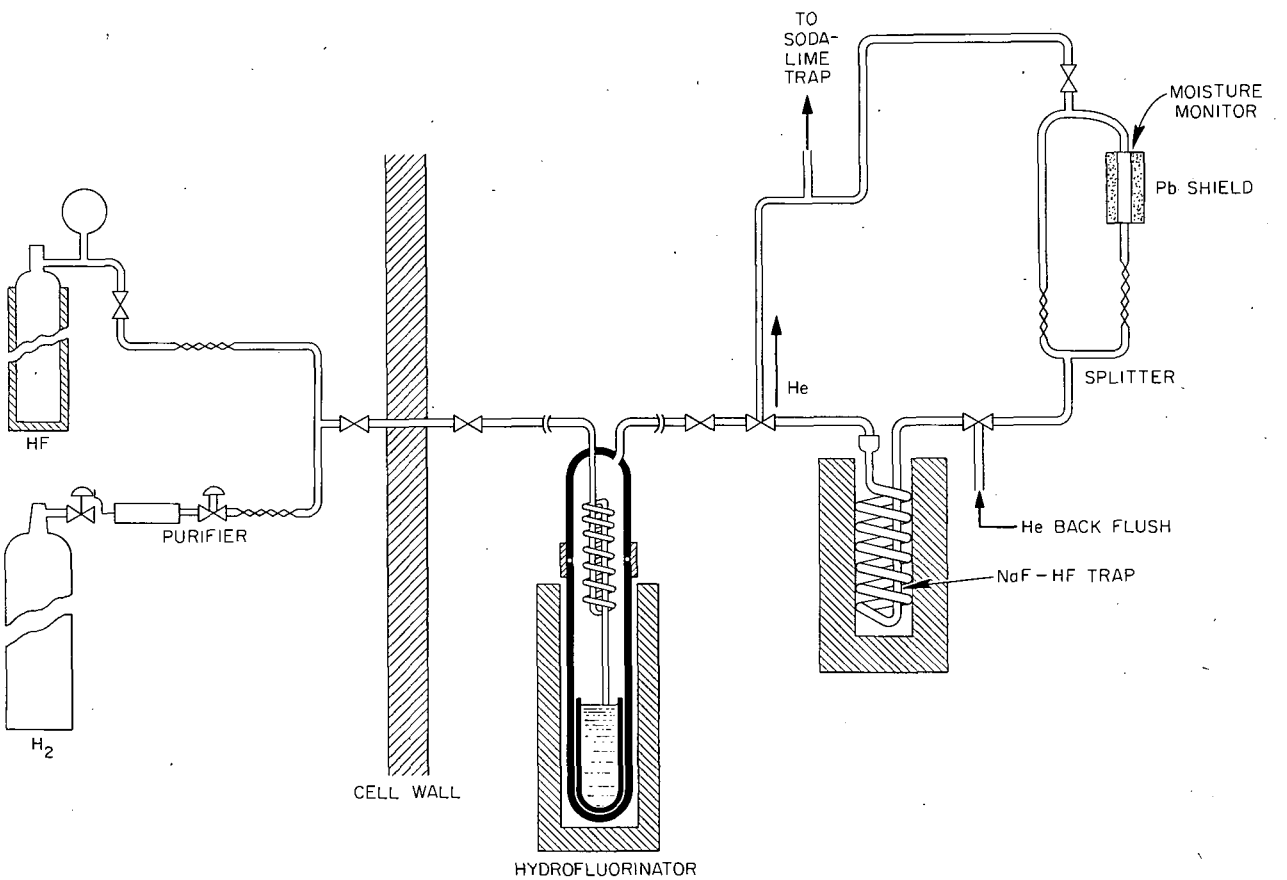

Fig. 3.1. Schematic Flow Diagram of the Apparatus for the Determination of Oxide in MSRE Fuel by Hydrofluorination.

the last three were deliberately exposed to the atmosphere for the indicated periods. To remove surface contamination, all samples were purged initially with a hydrofluorinating gas mixture before analysis. The relatively small increase in oxide on prolonged exposure confirms the effectiveness of this technique for removing surface contamination that results from brief exposures during hot-cell transfers.

The prototype apparatus was also used to analyze samples of fuel and flush salt withdrawn from the reactor during startup and zero-power operations. Figure 3.2 shows the results of these analyses and of the analyses made by the $KBrF_4$ method[3] during the same period. The results of the $KBrF_4$ analyses parallel those of the hydrofluorination method but are slightly higher because of atmospheric contamination of the pulverized samples.

A disassembled hydrofluorinator for analyses in a hot cell is shown in Fig. 3.3. From left to right are shown a sample ladle, a nickel liner, the hydrofluorinator top, the hydrofluorinator bottom, and a yoke that clamps the top and bottom of the hydrofluorinator to seal the system with a Teflon O-ring. The hydrofluorinator is loaded by suspending the ladle from the tip of the delivery tube and inserting it in the hydrofluorinator bottom, which contains the liner. When the assembly is clamped together, the delivery tube is spring loaded against the surface of the salt for premelting hydrofluorination. As the salt melts, the delivery tube is driven to the bottom of the ladle

[3]G. Goldberg, A. S. Meyer, Jr., and J. C. White, "Determination of Oxides in Fluoride Salts by High-Temperature Fluorination with Potassium Bromotetrafluoride," *Anal. Chem.* 32, 314 (1960).

for efficient purging. The salt is confined to the liner by eccentric ring baffles on the delivery tube. When the analysis is completed, the solidified sample together with the ladle, liner, and delivery tip can be removed and discarded; thus, the more expensive components of the hydrofluorinator are conserved.

Figure 3.4 shows the assembled hydrofluorinator positioned in its furnace and coupled to the valve compartment with an argon-driven ball-joint disconnector. The valve compartment also contains the sodium fluoride trap, the capillary effluent splitter, the electrolytic cell, and a soda-lime trap for HF disposal. These hot-cell components were designed with the assistance of J. H. Evans.[4]

Components located outside the hot cell include hydrogen purification equipment, a mixing station to control the composition of the hydrofluorinating gas, and a control console for controlling the furnace temperature and gas flows. These external components are equipped with interlocks and other safety devices to prevent excessive flows of gases to the hot cell.

[4]Equipment Design Section of the General Engineering and Construction Division.

The results of analyses performed in the hot cell are listed in Table 3.2. The standard deviation of these results is less than 8 ppm. This value does not, however, necessarily indicate the precision of the method, since the oxide content of the fuel may have changed during the period of reactor operation over which the samples were

Table 3.2. Oxide Concentrations of Coolant and Fuel Salt from the MSRE

Sample		Oxide Concentration
Type	Number	(ppm)
Coolant salt	1	25
Fuel salt	2	49
	3	53
	4	52
	5	47
	6	67
	7	59
	8	66
	9	56

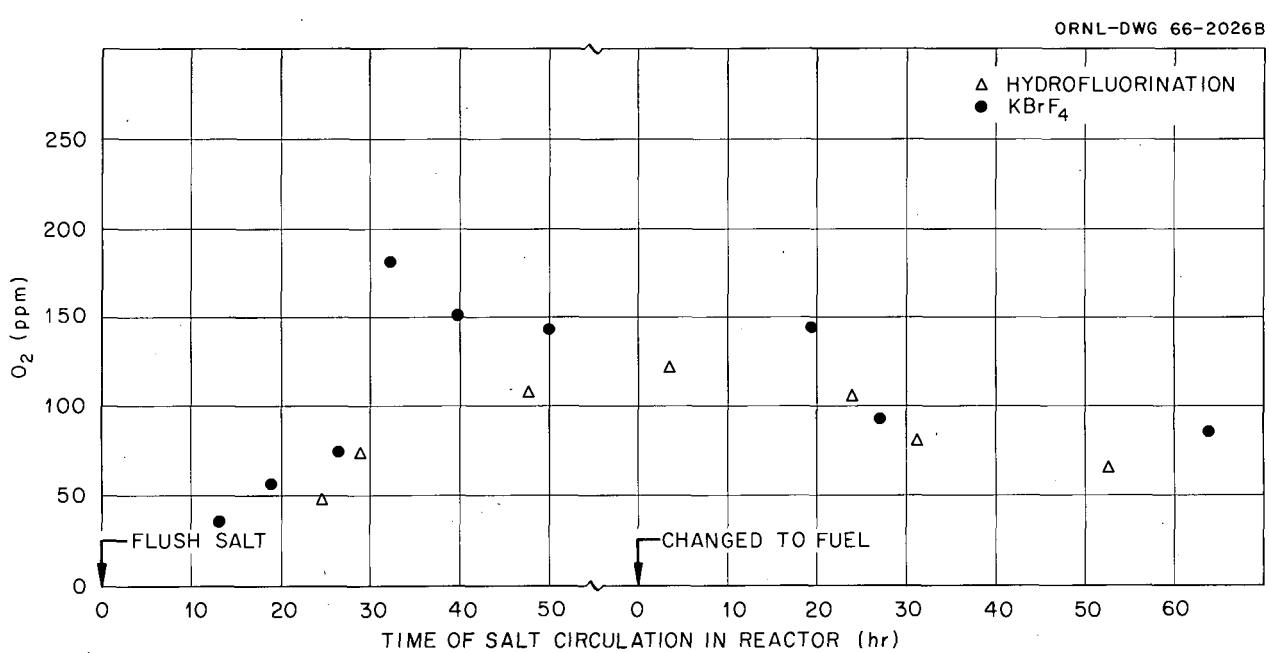

Fig. 3.2. Results of Hydrofluorination and $KBrF_4$ Analyses for Oxide in Flush and Fuel Salts During Startup of the MSRE.

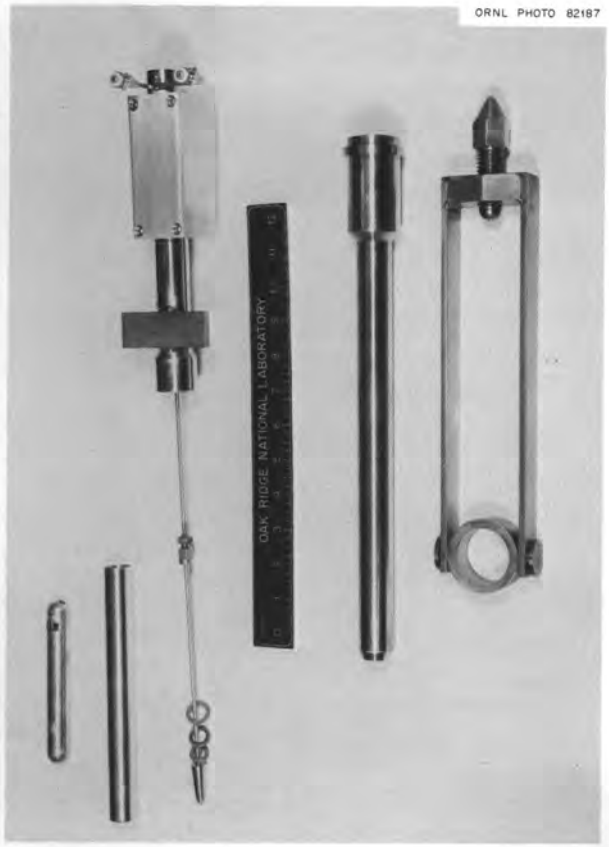

Fig. 3.3. Disassembled Hydrofluorinator.

taken. Also, the increase in oxide from sample 5 to sample 6 coincides with the opening of the reactor sampling station for maintenance and may reflect contamination by residual moisture in this system. These samples were withdrawn at power levels from 0 to 7.5 Mw; their maximum radioactivity was 1000 r/hr at 1 ft. To determine whether the oxygen might be removed from the samples by F_2 produced by radiation damage to the fuel during cooling, one sample (No. 7) was stored for 24 hr before it was analyzed; no significant reduction in oxide content occurred.

A standardization technique based on measuring the water derived from the 550°C hydrogenation of weighed SnO_2 standards was developed to verify the performance of the equipment. Oxide recoveries of 96.1 and 95.6% were obtained; the interim between the analyses was four months. This slight negative bias is attributed to momentary interruption in the flow of gas through the moisture-monitor cell. Bench tests have shown that these interruptions of flow occur only during electrolysis and are probably caused by bubbles of H_2 and O_2 formed under the electrolyte film of partially hydrated P_2O_5. A special regenerating technique in which a 3% solution of H_3PO_4 in acetone is used was developed to provide a desiccant coating sufficient to absorb all the water from a gas stream and still give minimal interruptions of flow. With cells regenerated in this manner, the recovery of oxide from standard samples of SnO_2 was 99.6 ± 1.3%.

3.1.b Analysis of MSRE Helium for Hydrocarbons

C. M. Boyd C. A. Horton
A. D. Horton A. S. Meyer

The Analytical Chemistry Division, together with members of the Reactor and the Reactor Chemistry Divisions, participated in various experiments to determine possible sources of the organic deposits that have caused plugging of valves and filters

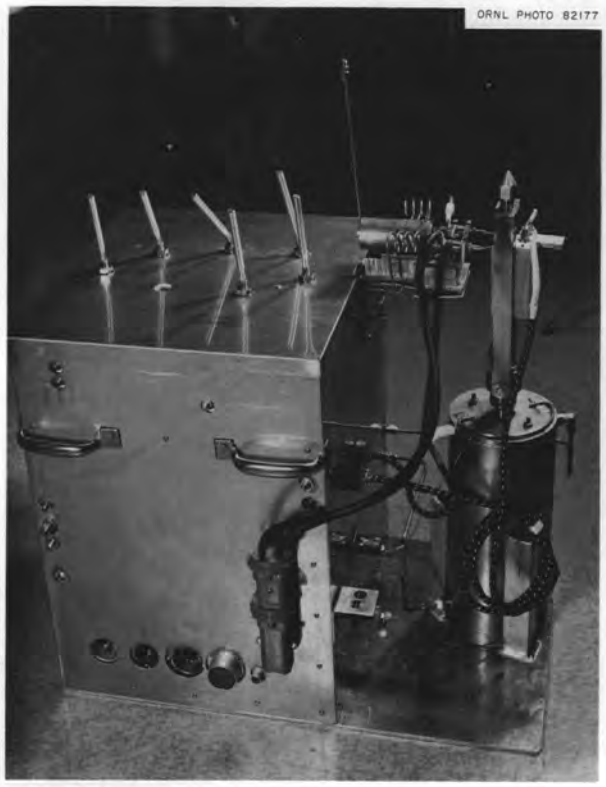

Fig. 3.4. Hot-Cell Apparatus for Determination of Oxide in MSRE Salts.

in the MSRE off-gas system. The analytical support included: (1) the installation of a continuous hydrocarbon analyzer to monitor gas streams from Reactor Chemistry Division experiments designed to simulate oil leaks into the MSRE pump, to evaluate trapping systems for reducing hydrocarbon concentration, and to monitor hydrocarbons in the off-gas from the Y-12 pump test loop; (2) the determination, by gas chromatography and by selective chemical reactions, of individual hydrocarbons in "grab" samples from the above experiments and in a sample trapped from an MSRE nonradioactive purge gas; and (3) the development of a technique to measure the concentrations of hydrocarbons collected on an experimental charcoal trap by pyrolysis of a sample of the charcoal followed by gas chromatographic analysis of the pyrolyzate. By sampling the charcoal bed at various depths, it is possible to determine the distribution of individual hydrocarbons as a function of trap length. Table 3.3 shows the distribution of hydrocarbons on a spent charcoal trap from experiments by the Reactor Chemistry Division. In contrast with the monotonically decreasing distribution of hydrocarbons along the length of a relatively fresh trap, these data show the successive displacement of lighter hydrocarbons by heavier fractions until a breakthrough of C_6 hydrocarbons occurs.

The continuous hydrocarbon monitor proved to be the most useful means of analysis, particularly in measurements at the Y-12 test loop, where it was possible to relate the hydrocarbon concentration to an operating variable (pressure drop across the shaft annulus) and thus to distinguish between possible locations of oil leaks. It was concluded that the leak was through the shield plug seals rather than down the shaft annulus.

Gas chromatographic analyses of the hydrocarbons derived from the injection of measured quantities of oil into the pump tank indicate that, at least in the absence of radioactivity, any oil that enters the pump bowl is cracked almost completely to yield methane, ethane, and unsaturated hydrocarbons lighter than C_5. None of these compounds was effectively trapped on charcoal at 100°C. Conversely, in the Reactor Chemistry Division experiments in which the oil was injected into an empty nickel furnace, the cracking was incomplete and gave a substantial yield of $>C_6$ hydrocarbons, which are trapped with relatively high efficiency.

A thermal-conductivity method to measure continuously the total hydrocarbon concentration in the radioactive off-gas of the MSRE was developed in collaboration with S. S. Kirslis.[5] In this method the off-gas sample is passed over copper oxide at 700°C to convert hydrocarbons to CO_2 and H_2O. This oxidized stream is passed through one side of a thermal-conductivity detector and thence to a trap that contains Ascarite and $Mg(ClO_4)_2$ to yield a stream of inert gases, which is directed

[5]Reactor Chemistry Division.

Table 3.3. Results of the Determination of Hydrocarbons in a Charcoal Trap by Pyrolysis at 475°C and Gas Chromatography

Increment of Trap Length (in.)	Hydrocarbon (wt %)						
	$C_{<6}$	C_6	C_7	C_8	C_9 and C_{10}	$C_{>10}$	Total
0–0.5	0.51	0.25	0.25	0.25	2.10	14.30	17.76
0.5–1.0	0.33	0.22	0.35	1.62	5.59	9.39	17.50
1.0–1.5	0.30	0.31	1.40	3.95	4.48	3.13	13.57
1.5–2.0	0.19	0.77	4.20	3.30	0.59	0.27	9.32
2.0–2.5	0.25	1.87	2.61	0.98	0.08	0.06	5.85
2.5–3.0	0.32	2.29	1.06	0.32	0.01	0.02	4.02
3.0–3.5	0.39	2.73	0.26	0.05	0.01	0.01	3.45

through the reference side of the thermal-conductivity cell. In bench-top tests the response of this apparatus was linear to total hydrocarbons in concentration up to 1000 ppm; the limit of detection was below 10 ppm. A similar system with the trap and copper oxide furnace designed for a one-year operation of the MSRE will be installed in the gas-sampling station of the MSRE.

3.2 ANALYTICAL METHODS FOR THE IN-LINE ANALYSIS OF MOLTEN FLUORIDE SALTS

3.2.a Status of In-Line Methods of Analysis

R. F. Apple A. S. Meyer
J. M. Dale J. P. Young[6]

The rapid acquisition of data on the compositions of the fuel, coolant salt, and cover gas is highly desirable in the operation of a fluid-fuel reactor. For the operation of the MSRE, in which the fuel is effectively isolated and is of near constant composition, the delays involved in sampling, sample transport, and hot-cell analysis are tolerable. In future breeder reactors such as the proposed Molten-Salt Breeder Experiment (MSBE), which will incorporate integral fuel processing with takeoff and makeup streams, the instantaneous acquisition of data on the composition of process streams is essential to safe and efficient operation. Data can be obtained instantaneously only by in-line analysis. Accordingly, our fundamental studies of the analytical chemistry of molten-salt systems are being evaluated carefully to determine which techniques justify further development for practical application to in-line analysis.

Process analyses that would be of major value in the operation of the MSBE include determinations of: major constituents, UF_3, corrosion products, and oxide in a salt of composition similar to that of MSRE fuel; uranium, protactinium, and corrosion products in the blanket salt ($LiF-BeF_2-ThF_4$); traces of uranium in depleted fuel-processing streams; and traces of fuel components in the coolant salt (composition not yet selected).

The following techniques appear to be the most suitable for in-line applications: electrometric methods, including rapid-scan voltammetry, for the determination of uranium and certain corrosion products and for continuous measurement of the potential of the fuel against a reference electrode (an Ni-NiF reference electrode is under study); spectrophotometric measurement, in a windowless captive-liquid cell, of UF_3, traces of UF_4, and possibly protactinium; and a continuous countercurrent hydrofluorination of a salt stream to liberate water and hydrogen in stoichiometric proportion to oxide content and reducing power. All these techniques are at a state of development that justifies their being tested under simulated in-line conditions.

These tests will require a special facility, the basic function of which will be to deliver a continuous stream of molten salt of known composition. The most practical facility commensurate with the space available in a California hood consists of a 20-kg-capacity salt reservoir fitted with a stirrer; ports for sampling, addition of solid and molten constituents, purge streams, and electrodes for purification; and a helium gas lift to continuously transfer a stream of salt to an elevated constant-level vessel. This constant-level vessel will furnish a gravity-fed stream through various analytical apparatuses and then back to the salt reservoir. The facility will also be used to test flow equipment such as capillaries, orifices, and freeze valves that will be needed to control the flow of salt. From tests with an aqueous solution of zinc chloride in a gas lift made of Teflon (to simulate the density and nonwetting properties of fuel, respectively), it appears practical to transfer salt at an adequate flow to a height of 6 to 8 ft. Similar tests must be performed with molten fuel salt to provide design data for the facility.

3.2.b Electroanalytical Chemistry of Molten Fluoride Salts

D. L. Manning Gleb Mamantov[7]

The determination of corrosion products in MSRE fuel was investigated by controlled-potential voltammetry; a three-electrode system (indicator, quasi-reference, and isolated counter electrodes) was used. The technique offers excellent possi-

[6]Research Group.

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

bility for the in-line determination of iron and nickel in reactor process streams but fails to resolve the wave for Cr(II) → Cr from the neighboring wave for U(IV) → U(III). However, at the low concentration of UF_4 expected in the MSBE fuel (0.2 vs 0.8 mole % in MSRE fuel), it may still be possible to measure chromium by derivative techniques.

The voltammetric technique was applied to samples from the MSRE to determine the oxidation states of iron and of nickel, which appear to be present in the fuel in concentrations above those predicted to exist in equilibrium with INOR-8 at the observed concentrations of chromium. Concentrations of ionic iron and nickel of only about 10 and 1 ppm, respectively, were determined by voltammetric scans of remelted samples that had been withdrawn from the MSRE before it was operated at power. These values compare with total concentrations (determined chemically) of 125 and 45 ppm, respectively, and indicate that the major portions of these contaminants are probably present as finely divided metals. Thus, the concentrations of these corrosion products in true ionic solution are more consistent with thermodynamic predictions.

Electrochemical reduction and oxidation of U(IV) in $LiF\text{-}BeF_2\text{-}ZrF_4$ (64.0-34.0-1.8 mole % and 65.6-29.4-5.0 mole %) were investigated by rapid-scan voltammetry and chronopotentiometry. Well-defined and reproducible current-voltage curves and potential-time curves were obtained at concentrations of U(IV) as high as 0.8 mole % (MSRE fuel). With the use of a platinum indicator electrode, the relative standard deviation of peak current for 41 determinations over a period of 36 days was 2.0%; considerably better precision ($S = 0.76\%$ for 8 determinations) was obtained over a 2-hr period. Other indicator electrodes tested included pyrolytic-graphite, molybdenum, tungsten, and tantalum. Of the electrodes tested, platinum and platinum-rhodium(10%) gave the best reproducibility.

At 500°C the reduction of U(IV) at platinum is a reversible one-electron process as determined from Nernstian log plots and from the diagnostic criteria of linear-sweep voltammetry. Also, from chronopotentiometry, a plot of the product of current density and transition time ($i_o\tau$) vs ($\tau^{1/2}$) yielded a straight line, which relationship is in agreement with the theory for a reversible electrode process. From the slope of the line the diffusion coefficient for U(IV) was calculated to be about 1.5×10^{-6} cm^2/sec, in good agreement with the value obtained by voltammetry, (1.5 to 2.0) $\times 10^{-6}$ cm^2/sec. The effect of temperature on the diffusion coefficient (D) was determined over the temperature range 480 to 600°C. From a plot of log D vs $1/T$, the activation energy that corresponds to U(IV) → U(III) was found to be ~11 kcal/mole.

Additional data are being collected to evaluate further the precision of the voltammetric measurements at different levels of U(IV) concentration. From these data it is hoped to obtain a better assessment of this approach as an analytical method for in-line determination of U(IV) in molten fluorides.

So that electrodes having more reproducible areas can be used, a new voltammeter is being designed and fabricated by T. R. Mueller[8] that will measure a 20-fold higher current (100 ma) than can be measured with existing voltammeters. With present equipment the area of the electrode is limited; thus, slight changes in melt level introduce a significant change in reduction current. The new instrument will also provide more rapid voltage scan (up to 500 v/sec) to minimize flow effects in an in-line cell.

An anodic wave due to the oxidation of oxide or hydroxide impurities, or both, in one melt at about +1.3 v vs the quasi-reference electrode, which had been considered for oxide determination, was found to be masked in MSRE salts. The large anodic wave in the presence of uranium apparently results from the oxidation U(IV) → U(V) followed by the catalytic disproportionation and reduction of U(V). Although this uranium interference appears to eliminate the possibility of the voltammetric determination of oxide, it does not necessarily preclude the controlled-potential electrolysis of oxide to elemental oxygen or carbon oxides, or both, which can be measured by gas chromatography.

3.2.c Spectrophotometric Studies of Molten-Salt Reactor Fuels

J. P. Young[6]

Studies pertaining to the continuous spectrophotometric determination of U(III) in circulating

[8]Analytical Instrumentation Group.

MSBR fuels have continued. A general description of the optical design of a facility for performing this determination is given elsewhere.[9] The facility will be used in conjunction with a commercial instrument, the Cary model 14H recording spectrophotometer. The optical problems involved have been discussed further and in detail with the designer of this spectrophotometer, Applied Physics Corporation, and have culminated in a purchase order issued to them for the development of suitable sample-space optics. The resulting apparatus is to be delivered in six months and will provide optimum light-gathering power and optical design for use with a double-convex lens-shaped drop of liquid. The apparatus will be used with existing instrumentation and will be compatible with the optical-path extension required in the final proposed reactor installation.

More detailed studies of the spectrum of U(III) in molten $2LiF \cdot BeF_2$ have provided better resolution of the various absorptions in the complex ultraviolet absorption peaks of U(III). As reported before,[9] the maximum absorption occurs at 360 mμ, but shoulders are observed at about 310, 445, and 508 mμ. These shoulders might be of analytical value if as-yet-unknown interferences prevent the use of the absorption at 360 mμ. On the basis of calculations made to estimate the probable valence states of fission products, it is expected that dissolved fission products will be in one of their more normal valence states, and if so, these ions will cause no interference at their expected concentrations.

Studies of the spectra of lower-valent rare-earth-metal ions in molten fluoride salts have been initiated. A preliminary spectrum of Sm(II) has been obtained in molten $2LiF \cdot BeF_2$. A broad absorption peak is exhibited by Sm(II); the peak has a maximum at 325 mμ and a shoulder at ~470 mμ. The molar absorptivity is not yet known exactly but is greater than 200. Spectra of Sm(II) were observed for solutions prepared by adding $SmCl_2$, SmF_2, or Sm metal to molten $2LiF \cdot BeF_2$. Samarium(II) in aqueous solution is reported to exhibit some six overlapping peaks in the region 200 to 600 mμ, with major peaks at 310 and 560 mμ.[10] Cursory studies of Sm(II) in molten $2LiF \cdot BeF_2$ indicate strong absorption below 300 mμ. These and other divalent rare-earth-metal ions will be studied to determine possible interference with the proposed continuous spectral monitor for U(III). If Sm(II) were present in the MSBR fuels, it could cause errors in the U(III) determination.

Although it has been assumed from other spectral studies, mainly on the solvent LiF-NaF-KF, that corrosion-product ions will not interfere with the proposed determination, this assumption had not been verified experimentally until this period. The molar absorptivities of Fe(II), Cr(II), and Ni(II) at their wavelengths of maximum absorbance are 5 at 1020 mμ, 6 at 760 mμ, and 10 at 432 mμ, respectively. Although these data are for the maximum absorbance, the spectra of Fe(II), Cr(II), and Ni(II) exhibit two peaks, one peak, and three peaks, respectively, from which their coordination can be inferred. None of these dissolved species will interfere at concentration levels of 10 to 100 times that expected in the fuel salt. In general, the spectra of these 3*d* ions can be interpreted as arising from essentially octahedral coordination in the case of Ni(II) and Cr(II) and distorted octahedral symmetry in the case of Fe(II). A cursory spectral study of Cr(III) was made. The molar absorptivity at its wavelength of maximum absorbance, 706 mμ, is ~7; Cr(III) exhibits two other less intense peaks. Chromium(III) is not expected to be present in the fuel salt.

In a study of the absorption spectrum of several different solutions of Fe(II) in molten $LiF\text{-}BeF_2$, an extraneous peak was noted at 432 mμ. The position of the peak indicated the presence of Ni(II) as an impurity in the melt. Based on a subsequent study of the Ni(II) spectra in these melts, a spectrophotometric determination of Ni(II) in these molten salts was possible. The results of this determination, compared with those from wet-chemical analysis of the same samples, follow:

Sample	Ni(II) (wt %)	
	Molten-Salt Spectra	Wet-Chemical
1	0.21	0.19
2	0.18	0.22

[9] "Spectrophotometric Studies of Molten-Salt Reactor Fuel," *MSR Program Semiann. Progr. Rept. Aug. 31, 1965*, ORNL-3872, p. 145.

[10] F. D. S. Butement, "Absorption and Fluorescence Spectra of Bivalent Samarium, Europium and Ytterbium," *Trans. Faraday Soc.* **44**, 617 (1948).

These melts were made in graphite containers but were stirred with a nickel stirrer. It would seem that this high nickel contamination, found originally by means of the molten-salt spectra, may have arisen from the stirrer.

An extremely sensitive absorption peak attributed to U(IV) has been found at 235 mμ; it has not been reported previously in any molten-salt solvent. The molar absorptivity of this peak is ~1500. This peak may correspond to the peak observed at 207 mμ[11] in the absorption spectrum of U(IV) in 1.0 *M* $^2HClO_4$. A possible analytical application of this absorption would be the continuous spectrophotometric monitoring of coolant salt for leakage of uranium-bearing fuel salt. Based on the absorbance at 235 mμ, in the absence of interfering ions, it should be possible to detect U(IV) in concentrations as small as 2 ppm by use of a cell having a 1-cm path length.

3.3 SPECTROPHOTOMETRIC STUDIES OF SOLID FLUORIDE SALTS

J. P. Young[6]

As pointed out previously,[12] to understand the nature of the spectra of solutes dissolved in molten-salt solvents, it is advantageous to know the spectral characteristics of the solid-solute species. The estimation of U(III) in solid samples by reflectance techniques[12] has been of help to persons in the Reactor Chemistry Division in their attempts to prepare pure UF_3.

In cooperation with G. W. Clark,[13] the apparatus for obtaining spectra of small crystals[14] was modified so that the spectra of heated solids can be obtained. The apparatus fits in the high-temperature cell assembly.[15] The transmission spectra of crystalline CrF_2 and FeF_2 were obtained over the temperature ranges 25 to 540°C and 25 to 450°C respectively. The results of these studies indicate that the differences between the spectra of these 3*d* ions in the crystals at room temperature and the spectra of these ions in molten LiF-BeF_2 are a function of increase in temperature rather than change of phase. The spectra indicate that crystalline CrF_2 changes from a distorted octahedral symmetry at room temperature to a higher symmetry, possibly octahedral, at 540°C. In the melt, Cr(II) exhibits the same higher symmetry. On the other hand, the symmetry of the FeF_2 does not change significantly either on being heated or dissolved, although its spectrum shifts gradually to lower energies on heating. These results will be published.

The reflectance spectrum of CrF_3 and the reflectance and transmission spectra of K_3CrF_6 were obtained at room temperature. Slight differences between the spectra of these two compounds are due to different coordination. The spectrum of Cr(III) in molten 2LiF-BeF_2 is similar to that of Cr(III) in solid K_3CrF_6. By means of the small-crystal holder,[14] the spectrum of crystalline K_3CrF_6 was obtained on a crystal only 0.2 mm in diameter.

The reflectance and transmission spectra of crystalline $LiUF_5$ were obtained and compared with previously reported[12] reflectance and transmission spectra of crystalline UF_4. Uranium in $LiUF_5$ is 9-coordinated,[16] whereas uranium in UF_4 is 8-coordinated. The spectra of U(IV) in these two compounds are similar but not identical, because the coordinations of the compounds differ.

3.4 SPECTROPHOTOMETRIC STUDIES OF SOLID MATERIALS

J. P. Young[6]

The spectral studies of solid materials were continued. Primarily, the studies have involved absorption measurements of solids mounted in variations of the small-crystal holder.[14] The results obtained with solid fluorides are discussed in Sect. 3.3; nonfluoride solids are discussed below.

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[11] D. Cohen and W. T. Carnall, "Absorption Spectra of Uranium(III) and Uranium(IV) in $DClO_4$ Solution," *J. Phys. Chem.* **64**, 1933 (1960).

[12] J. P. Young, "Absorption and Diffuse-Reflectance Spectra of Solid Materials," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1965*, ORNL-3889, p. 13.

[13] Metals and Ceramics Division.

[14] J. P. Young and G. W. Clark, "Apparatus for the Spectrophotometric Study of Small Crystals," *Rev. Sci. Instr.* **37**, 234 (1966).

[15] J. P. Young and J. C. White, "A High-Temperature Cell Assembly for Spectrophotometric Studies of Molten Fluoride Salts," *Anal. Chem.* **31**, 1892 (1959).

[16] G. D. Brunton, "The Crystal Structure of $LiUF_5$," *Acta Cryst.*, in press.

3.4.a Spectra of Rare-Earth-Metal Germanomolybdates

The spectral study of several rare-earth-metal germanomolybdates discussed previously[12] was continued, with particular emphasis on the effect of temperature. C. B. Finch[13] observed that heating causes the rare-earth-metal germanomolybdates to undergo reversible color changes (generally, colorless to yellow, blue to green, etc.). Spectra of these crystals were obtained over the temperature range 25 to 300°C by means of the heated small-crystal holder (Sect. 3.3). From the spectra of Eu_2GeMoO_8, Dy_2GeMoO_8, and Y_2GeMoO_8, it was apparent that the change in color results from a shift of some type of charge-transfer or LaPorte-allowed absorption, probably in the germanomolybdate configuration, to lower energies at elevated temperatures. This reversible shift contributes an intense yellow color to the crystals at higher temperatures. Of interest, also, were the shifts of the *f-f* transitions of Eu and Dy ions in the crystals. Yttrium germanomolybdate exhibits no such *f-f* spectra. On the basis of these results, the crystal structure of these compounds is being reinvestigated. The change in *f-f* spectra with temperature, particularly in the case of Dy(III), beautifully demonstrates the operation of selection rules in spectral transitions. Assignments of essentially all the 20 some absorption peaks seen in the 300- to 800-mμ region of the spectrum are possible. The increase in the population of higher-energy ground states with increasing temperature is easily seen, and the energy difference between these ground-state levels is very apparent.

3.4.b Spectral Studies of Metal Deposits on Film

J. P. Young[6]

The technique of obtaining spectra through a 0.1-mm aperture was used in a study for A. L. Southern.[17] The question was raised as to whether interference effects might cause an error in densitometric measurements of metals deposited on transparent films. The metal deposition is caused by preferential scattering from metal crystals under the influence of a particle beam. By scanning the resultant film through a slit of ~0.1 mm aperture with a densitometer, a profile of the preferential scattering is obtained. In collaboration with Southern, a simple micro manipulator was designed to move films across a 0.1-mm aperture and thus to obtain a spectrum from 300 to 750 mμ as a function of distance along the film. The technique was satisfactory for the measurement of a zinc deposit. However, with a gold deposit, it appears that more reliable results might be obtained if filters are used to eliminate the transmission effects of the gold film. None of the results obtained for either type of film indicate problems caused by optical interference effects; however, none of the films examined have shown visual evidence of such effects.

[17]Solid State Division.

3.5 FUEL PROCESSING

3.5.a Determination of ^3H and ^{85}Kr Released During the Processing of ThO_2-UO_2 Reactor Fuel

C. M. Boyd

An apparatus was designed and tested for the determination of ^3H and ^{85}Kr released during the processing of ThO_2-UO_2 reactor fuel. This apparatus was used by the Chemical Technology Division to follow the behaviors of ^3H and ^{85}Kr during the simulated processing of irradiated ThO_2-UO_2 fuel. Details and results of these tests have been reported.[18]

The system (Fig. 3.5) includes a mechanical roughing pump and a mercury diffusion pump for evacuating the fuel-shearing chamber and also the apparatus used to transfer, convert, and separate the gases. The transfer of the released gases from the chamber in a hot cell to the apparatus in a glove box was accomplished with a Toepler pump aided by the addition of controlled amounts of hydrogen. The gases were cryogenically pumped from a storage bulb through CuO at 800°C to convert ^3H to ^3H_2O; the ^3H_2O, Kr, and Xe were adsorbed on 13X molecular sieves

[18]J. H. Goode, *Hot-Cell Evaluation of the Release of Tritium and ^{85}Krypton During Processing of ThO_2-UO_2 Fuels*, ORNL-3956 (June 1966).

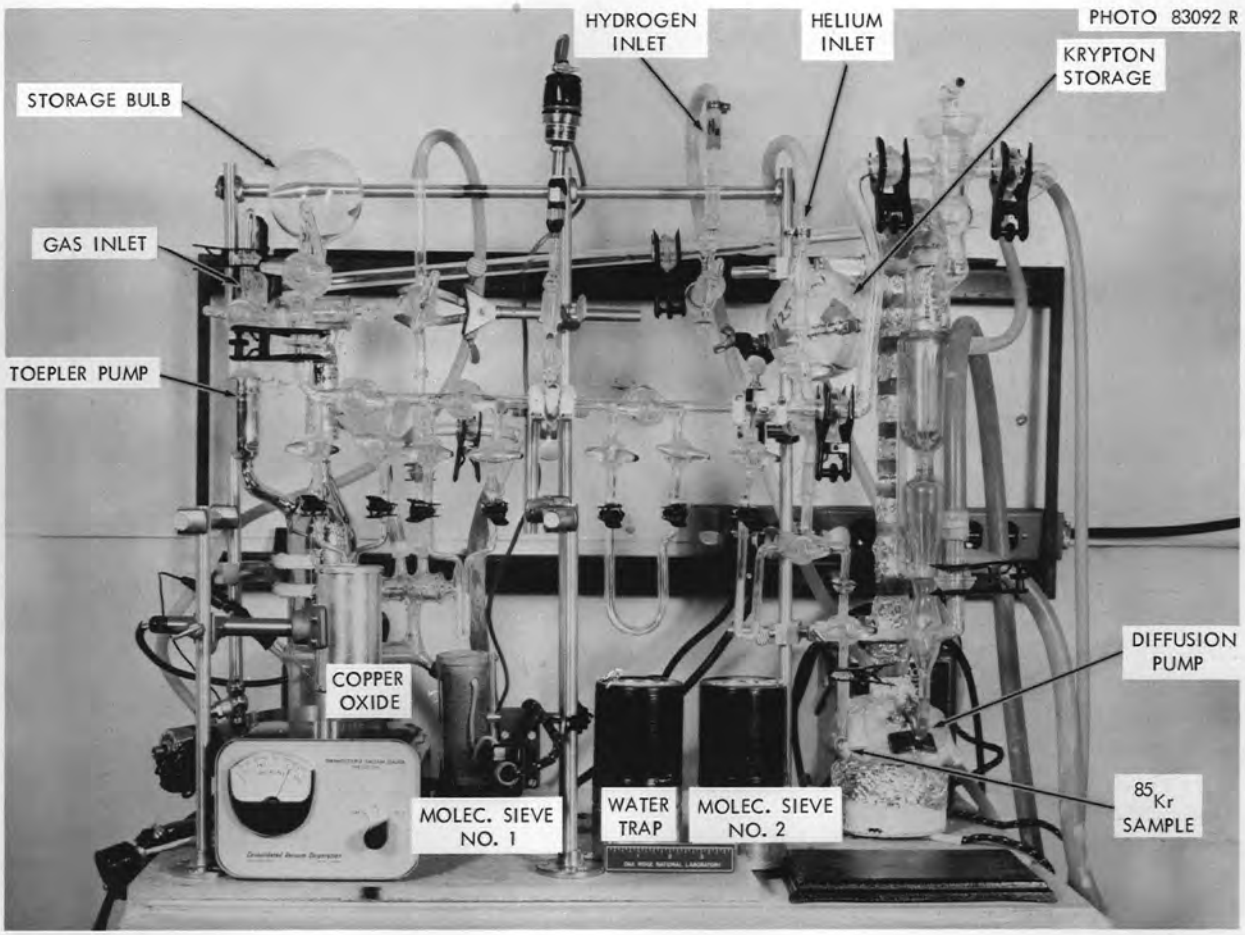

Fig. 3.5. Apparatus for the Separation of ^{3}H and ^{85}Kr.

at liquid-nitrogen temperature. Unadsorbed He was pumped off, and the molecular sieves were heated to 400°C to desorb $^{3}H_2O$, Kr, and Xe. The $^{3}H_2O$ was frozen out in a liquid-nitrogen-cooled trap, and the Kr and Xe were adsorbed on molecular sieves at liquid-nitrogen temperature. The $^{3}H_2O$ was taken up in a scintillation liquid and was determined by beta scintillation counting. The Kr and Xe were flushed from the heated molecular sieves into a storage bulb, from which aliquots were taken for ^{85}Kr determinations by gamma counting.

The recovery efficiency of the system was determined to be >95% by crushing, in the fuel-shearing chamber, a glass capsule that contained an $^{3}H_2$-H_2 standard. With modifications the apparatus was also used to measure ^{3}H released during the dissolution of the sheared fuel samples.

3.5.b Determination of UF_6 and PuF_6 in Gases from the Fluidized-Bed Volatility Pilot Plant

C. M. Boyd
A. S. Meyer
C. A. Horton
W. F. Peed[19]

Methods are needed to determine when the processes for the volatilization of fissionable material from spent UO_2 fuel elements can be terminated economically. An effective method for such process control is to measure continuously the concentration of UF_6 and PuF_6 in the effluent from the fluidized-bed reactor or in any equivalent process stream. To determine when production rates have become insignificant,

[19] Thermonuclear Division.

low concentrations (ppm) of UF_6 and PuF_6 must be detected in the presence of high concentrations (percent) of their fluorinating reagents (BrF_5 and F_2, respectively) in nitrogen. Other constituents of these process streams include: reagent contaminants, CF_4, SF_6, HF, and COF_2; volatile fission products, principally MoF_6; and traces of transuranium isotopes of high alpha radioactivity.

The measurement of fluorescent x radiation excited by an isotopic source is a convenient and selective technique for process-control determination of heavy elements.[20] This technique was evaluated with simulated process samples by exciting the spectra of aqueous solutions of uranium with a ^{57}Co source and measuring the characteristic spectra with both scintillation and solid-state detectors. Even with the most optimistic estimates of reduced backscattering from gaseous samples, the calculated limit of detection for the in-line measurement of U is about 1%.

From published spectra the suitability of spectrophotometric methods was evaluated. In both the infrared and ultraviolet regions, substantial interferences from both reagents and expected contaminants exist at the absorption maxima of all the expected products. A direct spectrophotometric measurement is possible only for UF_6 in the near ultraviolet (~500 mμ), and the detection limit is of the order of hundreds of parts per million. A differential spectrophotometric method was proposed in which the product is trapped selectively to provide a stream of reagents and contaminants to use in a reference absorption path. Spectral regions for potential use in differential measurements include 325 mμ, 8.6 μ, and 16.1 μ for UF_6 and 213 mμ, 8.8 μ, and 16.3 μ for PuF_6.

Any methods developed for these Fluidized-Bed Pilot Plant streams should also be applicable to comparable process streams in the MSBE fuel-processing system.

3.6 NUCLEAR SAFETY

3.6.a Analysis of Purge Gas from In-Pile Meltdown Experiments

R. F. Apple A. S. Meyer

An in-line analytical system is being developed for the continuous determination of the major gases generated by the in-pile meltdown of various types of fuel elements in air and in mixed atmospheres.[21] It is necessary to measure, during a 5-min interval, variations of H_2, O_2, H_2O, CO, and CO_2 contents in purge gas at low flow rates (100 to 800 cc/min) that also contain volatile fission products. A system of commercially available components has been designed to accomplish this rapid in-line analysis. The analytical instrumentation includes a dual infrared analyzer for CO and CO_2, an electrolytic moisture monitor for H_2, and electrochemical analyzers for H_2 and O_2. Components were selected on the basis of speed of response, compactness and low internal volume of the transducers, and compatibility of the signals with an existing digital data-acquisition system.

Currently, a process infrared analyzer is being evaluated experimentally that has been modified especially for this application. After laboratory tests are completed, the analyzer will be installed in a shielded hood at the ORR and operated during an appropriate meltdown experiment to determine the effects of radiation on the measurement.

3.6.b Determination of Organic Pollutants in Air

A. D. Horton

A program was undertaken to study the distribution of atmospheric contaminants that can react with radioiodine to form compounds such as methyl iodide which are difficult to trap. The purposes of this investigation are to develop methods of sampling and analyzing the air, to collect statistical data to be used as a guide in the design of a model program for the general monitoring of reactor atmospheres, and to determine what contaminants are present in reactor atmospheres in sufficient concentrations to justify the experimental measurement of their yield of radioiodine compounds in simulated nuclear accidents.

Currently, samples are being taken at random locations around ORNL by means of an evacuated

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[20] H. R. Bowman, E. K. Hyde, S. G. Thompson, and R. C. Jared, "Application of High-Resolution Semiconductor Detectors in X-Ray Emission Spectrography," *Science* **151**, 562 (1966).

[21] W. E. Browning, Jr., C. E. Miller, Jr., B. F. Roberts, R. P. Shields, W. H. Montgomery, O. W. Thomas, A. F. Roemer, and B. D. Neely, "Simulated Loss-of-Coolant Accident Experiments in the ORR," *Nucl. Safety Program Semiann. Progr. Rept. Dec. 31, 1965*, ORNL-3915, pp. 3–8.

40-liter bomb-type sampler. Concentrates, obtained by drawing 2.5-liter aliquots of these samples through a liquid-nitrogen-cooled trap packed with silicone oil (0.15% SE-30) on 40- to 50-mesh glass beads, are analyzed by gas chromatography. Hydrocarbons, probably from automobile exhausts, have been found in part-per-billion concentrations in all these samples. The concentrations of the contaminants vary widely, both with sample point and time; therefore, an extensive program will be required to collect sufficient data for statistical generalizations.

4. Special Research and Development Activities

J. C. White

4.1 GAS CHROMATOGRAPHY

A. S. Meyer

4.1.a Pyrolysis–Gas Chromatographic Determination of Cross-Linking in Vinylbenzene-Divinylbenzene Copolymers and in Ion Exchange Resins Based on Vinylbenzene and Divinylbenzene

A. D. Horton

Preliminary work was completed on a method to determine percent cross-linking in vinylbenzene-divinylbenzene (VB-DVB) copolymers and in ion exchange resins based on these compounds. Pyrolysis of the resin and gas chromatographic determination of the percent DVB should give a direct determination of percent cross-linking, which is directly proportional to the DVB content. Data were obtained in the following way. The resins, chosen at random, were dried overnight at 105°C; the copolymers were used as received. Samples were pyrolyzed in the Honaker-Horton pyrolyzer[1] between 500 and 800°C; an Apiezon L column was used for the resins, and a Carbowax 20M column for the copolymers.

For lack of a standard sample of DVB, the method was checked by dividing the area of the (*m*- + *p*-DVB) peak of the pyrochromatogram of 1%-cross-linked VB-DVB copolymer (C) by the area of the corresponding peak for 16%-cross-linked copolymer (D). Values so obtained are shown in Table 4.1. The ratio C/D was about 10% lower than theoretical. When the same criterion was applied to two ion exchange resins, Dowex 1-X4 and 1-X10, no direct relationship was found between the ratios of percent cross-linking and the percent cross-linking values given by the supplier; Table 4.2 gives the data. However, the ratios of VB peak areas of the chromatograms were approximately proportional to the ratios of the given percent cross-linking.

Although VB content is theoretically unrelated to percent cross-linking in an ion exchange resin, the VB concentrations in Dowex 1-X4 and 1-X10 seem to indicate the percent cross-linking. Further study is needed to determine whether this relationship is purely fortuitous. The concentrations of other components that result from pyrolysis of the resins vary with the percent cross-linking, and a direct relationship might be found by comparing the corresponding percents of one of these components in resins having different cross-linking.

The results might be improved by obtaining essentially water-free resins and copolymers and pure VB and DVB standards.

4.1.b Pyrolysis and Gas Chromatographic Determination of Palm Oil

A. D. Horton

The gaseous products of the pyrolytic decomposition of palm oil were determined by use of the Honaker-Horton pyrolyzer[1] at 500 and 590°C.

[1]C. B. Honaker and A. D. Horton, "A Simple Pyrolyzer for Use with Gas Chromatography," *J. Gas Chromatog.* **3**, 396 (1965).

Table 4.1. Results of the Pyrolysis–Gas Chromatographic Analysis of VB-DVB Copolymers

T (°C)	Peak Area VB / Sample Wt (in.^{2}/g)		A/B	Peak Area (m- + p-DVB) / Sample Wt (in.^{2}/g)		C/D[a]
	1% Cross-Linked (A)	16% Cross-Linked (B)		1% Cross-Linked (C)	16% Cross-Linked (D)	
675	525.9	358.0	1.47	2.4	44.1	0.054
725	532.4	358.9	1.48	2.3	40.8	0.056
	491.8	329.4	1.49	2.2	38.8	0.057
800	340.0	234.4	1.45	2.5	4.2	0.60
	315.4	231.9	1.36	2.4	3.9	0.62

[a]Theoretical, 0.062.

Table 4.2. Results of the Pyrolysis–Gas Chromatographic Analysis of Dowex 1 Ion Exchange Resins

T (°C)	Peak Area VB / Sample Wt (in.^{2}/g)		E/F[a]	Peak Area (m- + p-DVB) / Sample Wt (in.^{2}/g)		G/H
	Dowex 1-X4 (E)	Dowex 1-X10 (F)		Dowex 1-X4 (G)	Dowex 1-X10 (H)	
500	30.8	59.4	0.52	7.0	6.9	1.01
	30.5	57.1	0.53	6.9	6.7	1.03
725	31.9	60.9	0.52	12.7	12.7	1.00

[a]Theoretical, 0.40.

These gases were suspected to be the cause of swelling of components fabricated by powder metallurgy in which palm oil was used as a mold release.

Pyrolyzates in the C_1-to-C_4 range were determined by use of the squalane-modified medium-activity silica-gel column and of the 13X molecular-sieve column. Standards were used to identify the compounds. Table 4.3 gives the weight percent of each of the gaseous products of the pyrolysis.

4.1.c Nondestructive Desorption of Organic Compounds from Inorganic Solids and Their Subsequent Determination by Gas Chromatography

A. D. Horton

The pyrolyzer developed by Horton and Honaker[1] was used to desorb organic gases from U-Al alloys at 590°C. This technique was applied to samples of alloys of various compositions that were used to form reactor fuels by powder metallurgy processes at 590°C. No organic mold release was in contact with the samples, but swelling occurred in some cases when the powder was processed.

Gases were desorbed from the samples by inserting them into the pyrolyzer at 590°C for 1 hr in a stream of helium flowing at the rate of 60 cc/min. The gases were collected in a stainless steel U-tube trap filled with glass beads and immersed in liquid nitrogen. The trap was then connected to the inlet of the gas chromatograph, heated to 200°C, and backflushed into the chromatographic column with helium at 60 cc/min.

A column that contained medium-activity silica gel modified with 3 wt % squalane was used to determine C_1 to C_4 hydrocarbons plus iso- and n-pentane. With a high-activity silica-gel column,

CO_2, CS_2, and H_2S were measured; with a 13X molecular-sieve column, H_2, CH_4, and CO were determined. A separate sample was used for each column. Table 4.4 gives the results obtained from several different alloys. The predominant gas was CO_2; other nonhydrocarbons present were H_2 and CO. The gases H_2S and CS_2 were not detected; the limit of their detection is 10 ppm. The predominant hydrocarbon was ethylene. The concentration of olefins was greater than that of straight-chain or isomeric hydrocarbons.

Water was added to one sample of UAl_3(88.73%)–Al 101-A(11.27%) prior to inserting the boat into the hot zone of the pyrolyzer. About two-thirds of the ethane was released before the sample was heated. This occurrence is typical of the hydrolysis of UC_n, where $n > 1$, and indicates that uranium carbides may be the source of the hydrocarbons found in the alloys.

Table 4.3. Gaseous Products of the Pyrolysis of Palm Oil

Identity	Weight Percent	
	500°C	590°C
H_2	0.001	0.2
CH_4	0.2	1.3
C_2H_4	<0.05	2.1
C_2H_6	0.05	0.6
C_3H_6	0.2	2.9
C_3H_8	<0.05	<0.05
C_4H_8	0.1	1.5
C_4H_{10}	0.05	0.1
C_5H_{12}	0.1	0.1
CO	0.1	0.5
CO_2	<0.05	<0.05

Table 4.4. Compounds Desorbed from U-Al Alloys at 590°C

Compound[a]	Amount of Compound Found (wt ppm)			
	UAl_3	Al X-8001(2.778 parts)–UAl_3(2.901 parts)	Al 101-A	Al 101-A(11.27%)–UAl_3(88.73%)
H_2	10.7	8.3	13.5	6.9
CO	78.8	119.8	135.0	105.8
CO_2	131.7	349.1	243.8	485.0
CH_4	48.1	15.8	16.3	12.6
C_2H_6	8.8	3.4	6.3	2.5
C_2H_4	43.3	11.7	20.1	14.4
C_3H_8	10.0	2.3	2.5	2.3
C_2H_2	0.2	0.3	0.8	0.09
iso-C_4H_{10}	0.8	0.1	0.2	0.07
n-C_4H_{10}	2.1	0.3	0.8	0.7
C_3H_6	24.5	6.3	10.2	9.4
iso-C_5H_{12}	0.4	<0.02	0.03	0.2
n-C_5H_{12}	0.5	0.2	0.2	0.3
n-C_4H_8	14.0	3.1	4.3	3.9
iso-C_4H_8	4.3	1.4	2.0	1.8
Total	378.2	522.1	456.03	645.96

[a]Arranged in order of elution from the squalane-modified silica-gel column.

4.2 POLAROGRAPHY AND VOLTAMMETRY

P. F. Thomason

W. D. Shults

4.2.a Polarographic Studies with the Teflon Dropping-Mercury Electrode (D.M.E.)

Helen P. Raaen

Polarographic studies with the Teflon dropping-mercury electrode (D.M.E.) were continued. A general article on this work was published.[2] Oscilloscopic techniques were used in the study of signal-to-noise ratios (S/N) of Teflon D.M.E. capillaries relative to that of a typical reference glass D.M.E. capillary. In this way, interesting characteristics of various types of Teflon D.M.E.'s were demonstrated. Vertical-orifice rapid Teflon D.M.E.'s were evaluated for obtaining fundamental polarographic data. With one of these, a study was begun of the polarography of uranium in aqueous hydrofluoric acid.

Signal-to-Noise Ratios (S/N) of Glass and Teflon D.M.E.'s (D. J. Fisher,[3] W. L. Belew,[3] Helen P. Raaen). – The study was continued of the signal-to-noise ratios (S/N) of both horizontal- and vertical-orifice Teflon capillaries relative to that of a typical reference glass D.M.E. capillary.[4] As a means to study the possibility of charge effects as contributors to noise, the characteristics of individual drops were observed with an oscilloscope. The S/N of Teflon D.M.E.'s was found to decrease with mechanical disturbance of the joint between the Teflon and glass segments. For this reason, joints formed by means of a Swagelok connector and by heat-shrinkable plastic tubing were used, in addition to those of the original design (Araldite adapters held together with a rubber band). The joint of the original design was the best.

The oscilloscopic traces indicate that the retraction of mercury occurs to a greater extent for Teflon than for glass capillaries. It is believed that this characteristic of Teflon capillaries may be the cause of the somewhat lower S/N ratio for Teflon than for glass capillaries. With respect to the life of a mercury drop, as it is usually measured in seconds, this retraction becomes evident at the beginning of the life of the drop as a so-called "delay time" during which the current may decrease to zero or near zero. Whether the reverse-current spike and the delay time are reproducible has not been determined. The retraction of the mercury may also affect the reproducibility of the charging and faradaic currents, which immediately follow the delay time. D. J. Fisher[3] and R. W. Stelzner[3] have suggested that since all these effects occur early in the life of a drop, it should be possible to avoid them by observing the current for only a relatively short segment of time late in the life of a drop, that is, to use a modified Tast type of polarographic circuit. Increased precision and sensitivity might thereby be gained. Such an auxiliary circuit can be added to the circuit of the Q-1988-FES polarograph without loss of any advantages achieved with that instrument. Also, addition of a modified Tast circuit should not preclude the use of a vertical-orifice capillary for rapid polarography. Further study of S/N ratios of glass and Teflon D.M.E. capillaries is being postponed until this addition to the polarograph can be made.

Evaluation of Vertical-Orifice, Rapid Teflon D.M.E.'s for Obtaining Fundamental Polarographic Data (Helen P. Raaen). – A vertical-orifice D.M.E. makes possible rapid polarography, a decided advantage in the analysis, for example, of radioactive solutions or process streams. Polarography with a vertical-orifice Teflon D.M.E. may be directly applicable to problems of accountability, and perhaps also of corrosion, in the Fluidized-Bed Volatility Pilot Plant.

Experimental work with the vertical-orifice, rapid Teflon D.M.E.'s[5] was resumed. Capillary characteristics (t, m, w) of six combinations of the available vertical-orifice Teflon-capillary segments and glass-capillary segments were measured. The objective was to determine whether any combination

[2]Helen P. Raaen, "Instrumentation for Polarography of Glass-Corroding Media," *Analysis Instrumentation 1965* (Proceedings of the Eleventh Annual ISA Analysis Instrumentation Symposium Held May 26–28 at Montreal, Canada), Plenum, New York, 1966.

[3]Analytical Instrumentation Group.

[4]D. J. Fisher, W. L. Belew, Helen P. Raaen, and Bonnie C. Johnson, "Signal-to-Noise Ratios (S/N) of Glass and Teflon D.M.E. Capillaries," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1965*, ORNL-3889, pp. 28–29.

[5]Helen P. Raaen, "Vertical-Orifice Rapid Teflon D.M.E.," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1964*, ORNL-3750, p. 21.

of these segments provides a D.M.E. having m within the range in which polarographic maxima of the second kind do not occur (about 2 to 4 mg/sec) and also having t not greater than 0.5 sec, which was the upper limit of the quadruple, parallel-T, RC filter of the Q-1988-FES polarograph. No combination gives suitable values for both m and t; usually, if one is suitable the other is too large.

A combination that has a small t (<0.5 sec), a large m (>12 mg/sec), and a large w (>4 mg) at a reasonable h was selected for use in determining whether, at the large m, a significant maximum of the second kind occurs. The D.M.E. selected consists of a 117-μ-diam Teflon segment and a 100-μ-diam, 123-mm-long glass segment. The polarographic wave of the $Tl^+ \rightarrow Tl^0$ reduction was used to detect the maximum. A significant maximum does occur. It can be suppressed almost completely with Triton X-100 in concentration not less than about 0.02 v/v %, which is relatively high. By use of 0.02 v/v % Triton X-100, satisfactory polarograms were recorded; values measured from the polarograms for $E_{1/2}$ and n of the $Tl^+ \rightarrow Tl^0$ reduction agree with those reported in the literature. Thus it appears that the use of a suitable maximum suppressor in adequate concentration may be the means of securing satisfactory performance from a vertical-orifice Teflon D.M.E. having large m and w values.

A second D.M.E. (67-μ-diam Teflon segment–72-μ-diam, 211-mm-long glass segment) having a lower m value (~2 mg/sec) was tested. Its t value at the maximum h attainable with the polarographic standtube in use was ~1 sec, hence the filter circuitry of the Q-1988-FES polarograph was modified to accommodate a 1-sec drop time. Polarograms were then recorded of Tl^+ in 0.1 M KCl–1 mM HCl, of Cd^{2+} in 0.1 M KCl–1 mM HCl, and of Pb^{2+} in 1 M HCl and in both 1 M and 8 M HF. A maximum of the first kind was present on the wave for Pb^{2+} in 1 M HCl and on that for Tl^+. Triton X-100 in about 0.01% concentration suppresses the maximum. The waves for Pb^{2+} in 1 M HF (see Fig. 4.1) and in 8 M HF and for Cd^{2+} appear to have no maxima and to be analytically useful. With this D.M.E., the precision of drop formation in the chloride media that did not contain maximum suppressor was not as good as is desired. In the chloride medium that contained maximum suppressor and in the aqueous HF media, drop formation was precise.

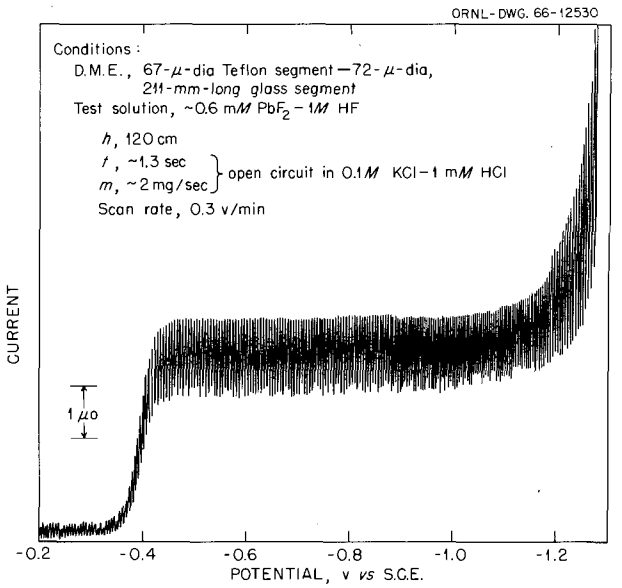

Fig. 4.1. Polarogram of Pb^{2+} in 1 M HF Obtained with a Vertical-Orifice Teflon D.M.E.

A third D.M.E. is being studied that consists of a 72-μ-diam Teflon segment and a 70-μ-diam, 98-mm-long glass-capillary segment. Except for the shorter length of the glass segment, the dimensions are about the same as those of the second D.M.E. The range of h values and thus of m and t are thereby increased; this flexibility will be needed in applied work. The polarograms obtained with this D.M.E. also show the first-kind maxima. These maxima appear to be characteristic of this type of D.M.E. and not to result from imperfections in the internal geometry of a particular Teflon segment, which imperfections could cause irregular mechanical disturbances in the mercury drops. This particular D.M.E. is being evaluated further to determine its suitability for rapidly obtaining reproducible and theoretically correct polarograms.

Polarography of Uranium in Aqueous Hydrofluoric Acid (Helen P. Raaen). – Study of the polarography of uranium in aqueous hydrofluoric acid was begun. With a vertical-orifice, rapid Teflon D.M.E. (72-μ-diam Teflon segment–70-μ-diam, 98-mm-long glass segment), polarograms were recorded for solutions of uranyl nitrate (mM and less) in 1 M HF without maximum suppressor. The test solutions were bridged to an S.C.E. through 0.1 M KCl–1 mM HCl. The polarograms show two interesting uranium waves.

A broad wave occurs whose $E_{1/2}$ is about −0.55 v vs S.C.E. The wave is reproducible and shows no evidence of maxima. For this wave, the relation between the diffusion current, and also first-derivative peak height, and uranium concentration (C) is linear for the range of uranium concentration studied (i.e., ~0.2 to 1 mM). The linearity of these relations and the magnitude of the wave ($i_d/C \cong 0.04$ μa μg^{-1} ml under the conditions used) indicate that the wave may be useful in practical analyses. From the width of the slightly unsymmetrical first-derivative peak at half-peak height, the electron change (n) associated with the wave was calculated to be 0.3. The slight dissymmetry of the peak and the fact that n is not an integer suggest that the electrode process is irreversible and possibly consists of more than one reaction.

Another very large, reproducible, and apparently complex wave exists whose $E_{1/2}$ is in the vicinity of +0.3 v vs S.C.E.; it is readily distinguishable from the mercury-dissolution wave. The magnitude of this wave ($i_d/C \cong 0.2$ μa μg^{-1} ml^{-1}) indicates that it may be a catalytic wave. Because the test solutions contained nitrate, the wave was thought possibly to be a uranium-catalyzed nitrate wave. However, the addition of sulfamic acid to the test solution to destroy nitrites did not affect the height of the wave but shifted the wave to a slightly more negative position. The relation of diffusion current to uranium concentration appears not to be linear; the first-derivative peak is unsymmetrical. The limited study of this wave provides no further information about it.

These initial results indicate that a comprehensive study of the polarography of uranium in aqueous hydrofluoric acid will be fruitful, both in the acquisition of fundamental electrochemical and reaction-mechanism data and in the development of practical methods of analysis. The work will be extended to such a study.

4.2.b Polarography of Nickel in Concentrated Chloride Media

T. M. Florence[6]

Concentrated solutions of LiCl are used in the Tramex process for the separation of actinide elements from fission products, and so the chemistry of various elements in this medium is important from the analytical chemistry viewpoint. In concentrated chloride media, Ni^{2+} is reduced at the D.M.E. at far more positive potentials than in dilute chloride solutions. The positive shift in half-wave potential increases with increasing ionic strength and is also greater when the cation of the supporting electrolyte has a high hydration number. The reduction in overpotential is due to the formation of a chloride complex of nickel, $Ni(H_2O)_5Cl^+$, which has a stoichiometric stability constant of 0.094 ± 0.009 at an ionic strength of 10.0 M. This nickel species is not formed in solutions of low ionic strength. In anhydrous methanol saturated with LiCl, nickel is present as the tetrachloro complex, $NiCl_4^{2-}$, which has polarographic behavior similar to that of the monochloro complex. From current-potential curves recorded at a rotated pyrolytic-graphite electrode, the voltammetric behavior of nickel can be studied in the absence of specific adsorption of the chloride ion.

The results of this work were published.[7]

4.2.c Voltammetric Determination of Aluminum by Oxidation of Its Solochrome Violet RS Complex at the Rotated Pyrolytic-Graphite Electrode (P.G.E.)

T. M. Florence[6] F. J. Miller H. E. Zittel[8]

A new highly sensitive method was developed for the determination of traces of aluminum. At a rotated pyrolytic-graphite electrode, both Solochrome Violet RS (5-sulfo-2-hydroxybenzene-azo-2-naphthol) and its aluminum complex produce well-formed oxidation waves. In an acetate buffer of pH 4.7 these waves are separated by 0.35 v. The concentration of aluminum can be determined by measuring either the height of the aluminum-complex wave or the decrease in height of the free-dye wave. Aluminum can be determined at concentrations as low as 10 ppb by use of simple polarographic equipment. A relative standard deviation of 3% is obtained at the 400-ppb aluminum concentration level and 5% at the 40-ppb level.

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

[7]T. M. Florence, "Polarography of Nickel in Concentrated Chloride Media," *Australian J. Chem.* **19**, 1343 (1966).

[8]Research Group.

Of 30 ions tested, only Fe^{3+}, Co^{2+}, Ti^{4+}, and V^{5+} interfere significantly in the recommended procedure.

This method has been published.[9]

4.3 STUDIES OF SPECIFIC-ION ELECTRODES

P. F. Thomason

W. D. Shults

J. A. Bell[10]

The performances of several "specific-ion" electrodes were evaluated: a "cation" electrode, an "iodide" electrode, and a "halide" electrode.

By a Nernstian expression, the potential developed at such electrodes generally can be related to the concentration of the ion being measured; hence, a linear relationship between potential and *p*Ion is to be expected. In practice, the response of the cation electrode is related to the sum of the concentrations of the univalent cations present in the solution, including the H^+. Furthermore, the electrode responds differently to various cations. Therefore, electrode response for the cation electrode is given by the modified Nernst equation

$$E = E^0 + 0.059 \log ([A] + K_{AB}[B]) ,$$

where [A] and [B] represent the concentrations of two different univalent cations and K_{AB} is a "selectivity coefficient" denoting the response of the electrode to ion A relative to ion B. The selectivity coefficient for the H^+ relative to other univalent cations is so large that the electrode is effectively a pH electrode when the pH of the solution is low. With solutions of moderate to high pH (e.g., pH > 6 for Na^+ or pH > 8 for Li^+), the electrode response to H^+ becomes insignificant relative to its response to other univalent cations. The usual practice then is to measure cation concentrations in media of high fixed pH. Adjustment of the pH of the solution without addition of cations that cause electrode response can be difficult. Anion exchange with a hydroxide-form resin was a satisfactory technique.

The cation electrode performs more satisfactorily for measurement of Na^+ than for measurement of Li^+. The potential-pNa curve at pH > 8 is virtually linear over a pNa range from 0 to 4, and the slope (−0.055 v/pM) is about equal to the theoretical value (−0.059 v/pM). The potential-pLi curve under the same conditions is not linear, nor does it have the correct slope, but the curve is usable with calibration over a pLi range from 0 to 4. This behavior is consistent with the experimentally determined value of K_{NaLi} of 20, that is, the electrode "sees" Na^+ about 20 times better than it sees Li^+. Analysis of synthetic transuranium-process-type solutions of LiCl-HCl for lithium content gave results accurate to approximately ±3%. The electrode appears to offer promise primarily for monitoring aqueous solutions of alkali-metal hydroxides of moderate concentration.

The Pungor-N.I.L. (National Instrument Laboratories, Cambridge, Mass.) iodide electrode consists of a silicone-rubber membrane impregnated with a precipitate of AgI. This electrode responds quickly and reproducibly to I^-, the potential-pI curve being linear and of correct slope over the pI range from 1 to 4. Electrode response is somewhat slower, and the *E*-pI curve exhibits some curvature at lower I^- concentrations; however, the electrode is usable with calibration to pI 6. The iodide electrode can be used to measure I^- concentrations in dilute acid media (HCl, HNO_3, or H_2SO_4) even when large amounts of other halide ions (Cl^-, Br^-) are present.

The "silver billet" electrode was tested for direct measurement of Cl^- concentration. The potential-pCl curve was linear and of the proper slope over the pCl range from 0 to 3.5. Some curvature was observed at lower Cl^- concentrations, but the electrode is useful with calibration to pCl 5. Other halide ions (Br^-, I^-) interfere.

The results obtained with these three electrodes indicate that specific-ion electrodes can be used to measure concentrations of appropriate ions with the convenience commonly associated with a pH measurement, the error being only about ±5%. Evaluation of the performance of "divalent ion," "phosphate," and "fluoride" specific-ion electrodes is planned.

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[9] T. M. Florence, F. J. Miller, and H. E. Zittel, "Voltammetric Determination of Aluminum by Oxidation of Its Solochrome Violet RS Complex at the Rotated Pyrolytic Graphite Electrode," *Anal. Chem.* 38, 1065 (1966).

[10] ORAU Student Trainee from Presbyterian College, Clinton, S.C.

4.4 SPECTROMETRY

P. F. Thomason

W. D. Shults

4.4.a Ultraviolet Absorption Spectrometry

J. R. Lund P. F. Thomason

The ultraviolet absorption spectra of 13 interference filters were recorded with the Cary model 14 spectrophotometer. A program was written in FORTRAN 63 to correct absorbances for any attenuation or cell-balance value, to convert the corrected absorbances to percent transmittance, and then to print out percent transmittance vs wavelength at 10-A intervals for all values of percent transmittance from 0.1 to 100% over the spectral range from 2000 to 7000 A.

Ultraviolet absorption spectrometry was used to study samples from some of the solvent-extraction processes being investigated in the Chemical Technology Division. Spectra were obtained of solutions of 4-*sec*-butyl-2(α-methylbenzyl)phenol (BAMBP) in Amsco 125-82, BAMBP in *n*-octane, $AuCl_3$ in aqueous 1 *M* chloride, and Au in *n*-octane–dimethyl sulfoxide (0.1 *M*). As a diluent and reference solution for quantitative spectrophotometry, purified *n*-octane was shown to be better than Amsco, which contains impurities that vary from batch to batch.

The Cary model 14 spectrophotometer was also used to measure and to record the spectra of 2-ethylnaphthalene and of a number of other compounds whose properties as scintillators are being investigated by members of the Health Physics Division.

4.4.b Visible Spectrometry – Attempt to Obtain the Absorption Spectrum of Californium

D. A. Costanzo

An attempt to obtain the absorption spectrum of californium was unsuccessful because of the small amount of ^{249}Cf available (5 μg); the maximum molar absorptivity predicted for californium is of the order of only 10 or less. However, the spectrum indicated the presence of several absorption peaks at wavelength positions of peaks reported by R. B. Baybarz.[11]

The micro absorption cell used has a path length of 5 cm and a total volume of 150 μl. The measured absorbance of the cell when it contained 1 *M* $HClO_4$ was 1.750. A conical screen attenuator was placed on the reference compartment of the Cary model 14 spectrophotometer to balance against the cell that contained the test solution. The 0-to-1.0-absorbance-unit slide-wire normally provided on the spectrophotometer was replaced with a 0-to-0.1-unit slide-wire. The satisfactory performance of the instrumentation was proved by obtaining successfully, in the manner described, a spectrum of 30 μg of praseodymium contained in 150 μl of 1 *M* $HClO_4$.

4.4.c Infrared Spectrometry

M. M. Murray Gerald Goldberg J. R. Lund

Infrared spectrometry was used to examine or record reference spectra of more than 500 samples of various types. Most studies involved the detection or identification, or both, of impurities in commercial products and in compounds synthesized at ORNL, identification of commercial products, characterization of organic compounds, determination of impurities, or identification of structural changes in both organic and inorganic materials caused by irradiation, chemical degradation, or thermal effects.

Fractions from compounds synthesized at ORNL were examined for structure and purity (Table 4.5). The formation and destruction of the following groups were detected spectrometrically: hydroxyl, carbonyl, carboxyl, olefinic, nitro, nitroso, and nitrate ester. The changes were brought about by irradiation or by thermal or chemical degradation of such materials as 4-*sec*-butyl-2-(α-methylbenzyl)phenol, lubricating oils, uranium carbide, Span 80–Ethomeen–2-ethylhexanol solutions, propane, and graphite-coated reactor fuel.

Commercial products were examined for purity, identification, or composition; they included lubricating oils, liquid scintillators and impurities found therein, surfactants, hydraulic fluids, quaternary amine type column packing, Amberlite LA-2 (liquid ion exchanger), high-temperature

[11]Chemical Technology Division; private communication to D. A. Costanzo, 1965.

Table 4.5. Compounds Synthesized at ORNL That Were Examined by Infrared Spectrometry

m-Aminobenzylamine	Heptadecanone
p-Aminobenzylamine	Heptadodecanoyl bromide
Benzyltrioctylphosphonium bromide	*N*-Heptadecylidene-2-ethylhexylamine
Bicyclic alcohols	Isoleucinol
β-Diketones	*p*-Nitrobenzaldoxime
2,6-Dioximinocyclohexane	Propionic acid
Didodecylnaphthalene	Tetraisobutylphosphonium chloride
Didodecylnaphthylamine	Tetra-*n*-butylphosphonium iodide
2-*n*-Dodecanoylnaphthalene	Tributylsulfonium iodide
2-*n*-Dodecylnaphthalene	Tri-*n*-butylphosphonium bromide
p-Ethylbenzenesulfonic acid	Triisoamyl phosphate
Gold di-(2-ethylhexyl)sulfoxide	Undecylnaphthyl ketone
Heptadecanol	

greases and paint, butadiene-styrene copolymer, *p*-nitrobenzylnitrile, *p*-nitrobenzaldehyde, and bromobenzene.

Gases from the hydrolysis of uranium carbide and from radiation dosimeters were identified. Semiquantitative and quantitative determinations were made of ethylene glycol plus water in lubricating oils, *o*-substituted phenol in transformer oil, and starting materials used in many of the products listed in Table 4.5. The infrared spectra of nine samples of irradiated α-pinene were recorded. Samples of 1-nonyldecylamine, one white and the other yellow, received in separate shipments were found to have essentially identical infrared spectra. Twelve samples of activated alumina (α-$Al_2O_3 \cdot H_2O$) were examined for the presence of sorbed PO_4^{3-}.

Reference spectra of commercially produced biological compounds (Table 4.6) were recorded for future use. The spectrum of the Cu(I) complex of 1,5-cyclooctadiene was also recorded. Also, the spectra of the olefin recovered after chemical decomposition of the complex and of the olefin used to form the complex were compared for possible structural changes.

4.4.d Nuclear Magnetic Resonance Spectrometry

J. R. Lund

A Varian DP-60 (dual-purpose 60-Mc) nuclear magnetic resonance (NMR) spectrometer and a Varian A-60 NMR spectrometer have been available for use by the Analytical Chemistry Division. Since the A-60 instrument is equipped with an integrating circuit, most of the spectra were obtained with it. Integration of a spectrum relates the area under a peak to the number of ^1H nuclei that produced it and thus aids in the interpretation of the spectrum.

The spectra of five phosphonium halides, prepared by the Analytical Chemistry Division for the Chemistry and the Metals and Ceramics Divisions, were studied by NMR spectrometry. They included tetra-*n*-butyl-, tetraisobutyl-, benzyl-tri-*n*-butyl-, and benzyl-tri-*n*-octylphosphonium halides, where the halide was either chloride, bromide, or iodide.

The NMR spectra of the starting materials and of the products from 12 preparations of barium ethylbenzenesulfonates were recorded for the Inorganic and Physical Chemistry Group of the Chemistry Division. Since these salts are not soluble to the extent of at least 10 wt % in ^2H_2O, they were converted to the sodium form by the addition of a slight excess of anhydrous Na_2SO_4 to a suspension of the barium salt in ^2H_2O. The filtrates from the mixtures were then used to record the NMR spectra. It was determined that in each case the sulfonate substitution in ethylbenzene is para-oriented.

The spectra of six lipids and related materials were obtained for the ORAU Medical Division. By use of the 150-μl sample holder, it was pos-

Table 4.6. Biological Materials Examined by Infrared Spectrometry

Adenine	*dl*-3-Indoleacetic acid
N-Acetylhistamine	Kynurine
p-Aminobenzoic acid	Kynurenic acid
p-Cresol	Maleuric acid
Cytosine	1-Methyl-*dl*-histidine
Dithiouridine	*dl*-3-Methoxy-4-hydroxymandelic acid
Gramicidin A and derivatives	Phloretic acid
Hippuric acid	Phenol
Homogentisic acid	Riboflavin
Homovanillic acid	Salicylacetic acid
m-Hydroxybenzoic acid	*l*-Tyrosine
p-Hydroxybenzoic acid	Uracil
5-Hydroxyindole-3-acetic acid	Uric acid
p-Hydroxymandelic acid	Uridine
o-Hydroxyphenylacetic acid	Urocanic acid
p-Hydroxyphenylacetic acid	Vanillic acid
Hypoxanthine	Xanthine

sible to obtain resolvable spectra of 20 to 50% solutions of these compounds in deuterochloroform. Although their molecular weights are of the order of 900, the signal due to the one methine proton of the glycerol part of the molecule is recognizable in the spectrum.

The 32 spectra recorded for members of the Biology Division included those of isomers and mixtures of amyl acetate, soluble ribonucleic acids (sRNA), unsaturated diols, and half-acetate esters of the diols. Gramicidin A, which is a linear *N*-acylated pentadecapeptide ethanolamide, was examined; also, the position of substitution of *m*-iodobenzoic acid in the gramicidin A molecule was studied.

The NMR spectra of a variety of compounds were obtained for the Chemical Technology Division. By use of the 25-μl sample holder, a spectrum was obtained on only 4 mg of methylated aziridine chloride complex. Spectra of eleven oximes were obtained; one of these, the α-hydroxyoxime, 5,8-dimethyl-7-hydroxy-6-dodecanone oxime, was studied at room and elevated temperatures. The spectra of this liquid revealed that it is a highly associated or hydrogen-bonded compound that contains two types of hydroxyl groups and some impurity or additive. The spectra of 4-sec-butyl-2(α-methylbenzyl)phenol (BAMBP) and its cesium complex (1 Cs:5 BAMBP) were compared. The spectra of simple β- and γ-diketones were examined as part of a study of the synthesis of β-diketones whose alpha substituents possess a high degree of steric hindrance.

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

4.5 NEW AND MODIFIED CHEMICAL METHODS

P. F. Thomason

W. D. Shults

4.5.a Determination of Free Acid in Tramex Process Solutions

D. A. Costanzo

A potentiometric method was used satisfactorily to determine free acid[12] in representative Tramex

[12]H. Kubota and D. A. Costanzo, "Reactions in Concentrated Lithium Chloride Solution — Determination of Free Acid and Hydrolyzable Cation," *Anal. Chem.* **36**, 2454 (1964).

process solution. Free acid is determined in the presence of hydrolyzable cations by titration in 11 *M* LiCl with 0.1 *N* LiOH–11 *M* LiCl. From a single titration curve, the total hydrolyzable cations is also estimated.

A potentiometric titration method was used routinely to determine free acid and bound acid in Tramex process extractant solutions that contained Adogen hydrochloride in diethylbenzene and also in Tramex scrub solutions that contained hydroxylamine hydrochloride in 11 *M* LiCl. The titrations are performed in absolute ethyl alcohol with ethanolic sodium hydroxide as the titrant. A glass electrode is used as the indicator electrode in conjunction with a platinum wire, located in the titrant stream, as a reference electrode. Di-2-ethylhexylphosphoric acid and the dibasic acid impurity in decane were also determined in this manner.

4.5.b Separation and Determination of Microgram Amounts of Fluoride

D. C. Canada

Microgram amounts of F^- can be determined by a Zr-SPADNS[13] spectrophotometric procedure; however, NO_3^- interferes. Therefore, the separation of microgram amounts of F^- by a modified micro Willard-Winter method[14] was studied. The NO_3^- distills from the sample aliquot in the presence of Zr, and F^- remains behind as the Zr complex. Phosphoric acid is added to precipitate the Zr and to release F^-, which can then be steam-distilled from the solution.

The method has proved very useful for a variety of samples; for example, F^- was determined in concentrated HCl and in ion exchange resins. Zirconium perchlorate is added to the resin, and the resin is wet-ashed at 150°C with nitric and perchloric acids. The resulting solution is then distilled from phosphoric acid. The precision ($S \cong 5\%$) and accuracy (100% recovery of added F^-) are excellent.

[13]H. W. Wharton, "Isolation and Determination of Microgram Amounts of Fluoride in Materials Containing Calcium and Orthophosphate," *Anal. Chem.* **34**, 1296 (1962).

[14]M. A. Wade and S. S. Yamamura, "Microdetermination of Fluoride Using an Improved Distillation Procedure," *Anal. Chem.* **37**, 1276 (1965).

4.5.c Polarographic Determination of Nitrate in Tramex Process Solutions

D. A. Costanzo

A polarographic method for the determination of NO_3^- in Tramex process solutions was evaluated and adapted for remote operation.[15] This method is based on the catalytic effect that NO_3^- has on the limiting current of the polarographic reduction of U(IV) → U(III) in chloride medium. With 0.2 m*M* UO_2^{2+} in 0.2 *M* KCl–0.01 *M* HCl, the limiting current of NO_3^- is directly proportional to its concentration up to 0.5 m*M*. The limiting current for NO_3^- is measured as the difference between the total current at −1.2 v vs S.C.E. for NO_3^- in the presence of uranium and the limiting current for the U(IV) → U(III) reduction in the absence of NO_3^-. The limiting current for uranium is determined from a blank run. Interferences are PO_4^{3-}, F^-, and large amounts of SO_4^{2-}, which complex or precipitate the uranium.

4.5.d Potentiometric Titration of Technetium(VII)

F. J. Miller

A study of the potentiometric determination of Tc(VII) by use of reducing titrants has shown that this is not a feasible procedure. None of a variety of electrodes tested improved the titration. Solid electrodes such as Pt, Au, and pyrolytic graphite do not give satisfactory results. A stirred Hg-pool electrode is not usable because of some reduction of the Tc(VII) by the Hg itself. Change in pH and the use of various catalytic agents did not improve the titration. The flaw in the titration procedure is evidently the interaction of the test solution with the electrode surface. Scouting experiments indicated that Tc(VII) can be determined more successfully by titration with a solution of As_2O_3 and spectrophotometric detection of the end-point color change.

4.5.e Potentiometric Determination of Plutonium

W. D. Shults J. H. Cooper[16]

Plutonium can be determined volumetrically by oxidation to Pu(VI) with Ag(II), reduction of

[15]I. M. Kolthoff and J. J. Lingane, *Polarography*, vol. II, pp. 536–538, Interscience, New York, 1952.

[16]High-Level Alpha Radiation Laboratories.

Pu(VI) to Pu(IV) with excess Fe(II), and titration of the unreacted Fe(II). Several variations of this procedure have been published, the most promising of which is that of Drummond and Grant.[17] We are evaluating a modified version of their procedure, which will supplement the controlled-potential coulometric procedure now used exclusively for the determination of milligram amounts of plutonium.

4.5.f Extraction–in Situ Spectrophotometric Determination of Uranium

A. F. Roemer W. D. Shults

The hexone extraction–in situ spectrophotometric method for U(VI), reported by Maeck and co-workers,[18] is being evaluated. This simple method offers excellent selectivity, can be used for microgram or milligram quantities of U(VI), and is amenable to remotely controlled operations or to automation (e.g., with the Technicon Auto-Analyzer).

4.5.g Separation and Spectrophotometric Determination of Fluoride

D. A. Costanzo

The pyrohydrolysis method has been used to separate microgram amounts of F^- from HFIR-target-dissolver solutions that contained plutonium, actinides, and fission and corrosion products in a nitric acid medium. In this method, the sample is evaporated to dryness in a nickel or procelain combustion boat. To prevent the loss of F^- during evaporation, either the sample is neutralized with base or aluminum nitrate is added to complex the F^-. After the sample is taken to dryness, an Na_2WO_4-WO_3 flux is added, and the sample is pyrolyzed at 1000°C for 30 min in a quartz combustion tube. The volatile F^- is collected in a dilute solution of NaOH. The isolated F^- is then determined spectrophotometrically by the Zr-SPADNS bleaching method.[13] Large amounts of volatile NO_3^- may accompany the F^- during pyrolysis and constitute an interference. Hydroxylamine in an acid medium is used to destroy the NO_2^-.

4.5.h Miscellaneous Applications of Existing Methods

D. C. Canada D. A. Costanzo J. R. Lund

Several existing methods were adapted for use in the General Analyses Laboratories. An adaptation of the spectrophotometric method of Larsen and Ross[19] was used to determine Ru in "phosphate glasses" and other waste-disposal products. The hydrochlorination method of Fedorov and Linkova[20] was modified for the determination of Al_2O_3 in sintered Al powder. The O_2-evolution method of Seyb and Kleinberg[21] was used to determine KO_2 in K_2O. The potentiometric method of Wagner, Brown, and Peters[22] was used for the quantitative differentiation of primary, secondary, and tertiary amines in amine mixtures. The volumetric method of Critchfield and Johnson[23] was adopted for the determination of primary plus secondary amines in amine mixtures.

In the analytical laboratories of the Transuranium Processing Plant, Tramex process solutions were analyzed for Sn^{2+} content by titration with a standard solution of KIO_3 in an acid medium that contained excess KI.[24]

The dissolution of Al_2O_3 by heating with HCl in a sealed tube was tested to check the feasibility of dissolving similar material that would contain plutonium for the Fluidized-Bed Volatility

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[17] J. L. Drummond and R. A. Grant, "Potentiometric Determination of Plutonium by Argentic Oxidation, Ferrous Reduction, and Dichromate Titration," *Talanta* 13, 477 (1966).

[18] W. J. Maeck, G. L. Booman, M. C. Elliott, and J. E. Rein, "Spectrophotometric Extraction Methods Specific for Uranium," *Anal. Chem.* 31, 1130 (1959).

[19] R. P. Larsen and L. E. Ross, "Spectrophotometric Determination of Ruthenium," *Anal. Chem.* 31, 176 (1959).

[20] A. A. Fedorov and F. V. Linkova, "Determination of Aluminum Oxide in Aluminum Metal by Hydrochlorination," *J. Anal. Chem. USSR* 17, 51 (1962).

[21] E. Seyb, Jr., and J. Kleinberg, "Determination of Superoxide Oxygen," *Anal. Chem.* 23, 115 (1951).

[22] C. D. Wagner, R. H. Brown, and E. D. Peters, "The Analysis of Aliphatic Amine Mixtures; Determination of Tertiary Aliphatic Amines in the Presence of Primary and Secondary Amines and Ammonia," *J. Am. Chem. Soc.* 69, 2609 (1947).

[23] F. E. Critchfield and J. B. Johnson, "Reaction of Carbon Disulfide with Primary and Secondary Aliphatic Amines as an Analytical Tool," *Anal. Chem.* 28, 430 (1956).

[24] H. H. Willard and H. Diehl, *Advanced Quantitative Analysis*, pp. 328–329, Van Nostrand, New York, 1943.

Process experiments. The tests showed that a sample size of about 1 g is the limit for dissolution in the equipment available.

A method was worked out for the Instrumentation and Controls Division to enable them to mix, in the correct proportions, Ciba epoxy resin and its hardener, tetraethylenetetramine.

Samples of oxides of tungsten and of molybdenum resulted from tests of materials used in the Experimental Gas-Cooled Reactor project. Since these oxides resulted from heating the lubricants Bemol (WS_2) and Everlube 811 (MoS_2), it was desired to determine the amount, if any, of "free" sulfur present. The total sulfur content, free and combined, in the heated materials was <1 ppm.

4.6 NEW AND MODIFIED PHYSICAL METHODS

P. F. Thomason

W. D. Shults

4.6.a Density Measurements by the Density-Gradient Technique

D. C. Canada W. R. Laing[25]

The measurement of densities of small single particles is generally done by a time-consuming "sink-float" procedure. The density-gradient method has become a useful analytical tool for rapidly measuring densities of such small particles. Measurement of absolute density to four significant figures is readily made in a gradient column. Density differences of 10^{-7} g/cc are reported in the literature[26] as being detectable. The gradient is formed by partially mixing two miscible liquids in a vertical column and allowing them to reach a steady state. A reservoir at both ends of such a column stabilizes the gradient for several months.

Theoretically, a density gradient would be linear with respect to distance only for an ideal solution under steady-state conditions. For the nonideal solution, the gradient, dC/dx, is nonlinear and is concentration dependent, that is,

$$\frac{dC}{dx} = \frac{A}{D_0\,[1 + f(C)]},$$

where C is the concentration of one of the miscible liquids, x is the distance along the column, A and D_0 are constants (D_0 is the diffusion coefficient of the particular miscible liquid at infinite dilution), and $f(C)$ is some concentration function. Therefore, density standards must be placed in the column as reference points. Standards are generally made from glass and are calibrated by a modified sink-float procedure.[27]

Samples that have been measured in the gradient column include pyrolytic-carbon-coated fuel microspheres, carbon films, single crystals, aqueous droplets, and single particles of alloys. The weights of many of the particles were <100 μg. The major requirements with respect to types of samples are that the samples not dissolve or react chemically with the gradient solutions and that their surfaces be completely wet in the column.

Densities as high as 5.0 g/cc can be measured by this technique. For such high densities, a saturated aqueous solution of thallous formate and thallous malonate, maintained at 85°C, is used. Densities as low as 0.6 g/cc can be measured by use of pentane.

The principles and applications of this density-gradient technique were presented in a paper.[28]

[25] General Analyses Laboratories.

[26] G. Oster and M. Yamamoto, "Density Gradient Techniques," *Chem. Rev.* **63**, 257 (1963).

[27] M. Gordon and I. A. MacNab, "A New Diffusion Gradient Method for Thermal Expansion Studies with Applications to Polystyrene," *Trans. Faraday Soc.* **49**, 31 (1953).

[28] D. C. Canada and W. R. Laing, "Use of a Density Gradient Column to Measure the Density of Microspheres," presented at the American Chemical Society Southeastern Regional Meeting, Louisville, Ky., Oct. 27–29, 1966.

5. Analytical Biochemistry

J. C. White Gerald Goldstein

5.1 MACROMOLECULAR SEPARATIONS PROGRAM

5.1.a Determination of Leucyl- and Phenylalanyl-Transfer Ribonucleic Acids (tRNA)

I. B. Rubin

The aminoacylation of transfer ribonucleic acid (tRNA) by ^{14}C-labeled amino acids was studied. Optimum conditions were established for the quantitative determination of purified leucine- and phenylalanine-accepting tRNA's from *Escherichia coli* B. An analytical procedure was developed for determining picomolar quantities of these two tRNA's; it is based on a filter-paper disk technique and scintillation spectrometry. Relevant experimental variables were investigated; they included pH; type of buffer; incubation time; and concentrations of tRNA, amino acid, adenosine triphosphate, aminoacyl-tRNA synthetase, and divalent-metal-ion cofactors. The optimum pH for the aminoacylation reactions is 8 to 8.5; cacodylate and glycinate buffers are preferred for the formation of leucyl- and phenylalanyl-tRNA, respectively. The ions Mg^{2+} and Mn^{2+} appear to be equally effective as cofactors; sodium chloride and sodium phosphate used in the chromatographic separation of the tRNA's have inhibiting effects. At the pH of optimum formation, the hydrolytic half-lives are 7 min for leucyl-tRNA and 23 to 24 min for phenylalanyl-tRNA.

A paper on this work was presented orally[1] and is now being prepared for submission to *Analytical Biochemistry*.

5.1.b Determination of Tyrosyl-Transfer Ribonucleic Acid (tRNA)

I. B. Rubin

Purified tyrosyl-tRNA has recently been made available for study. An investigation was initiated to establish the optimum conditions for the determination of this compound. The optimum pH range for its formation is 6 to 8; the buffer preference has not yet been established. For crude (unfractionated) tRNA, the optimum pH range is 6 to 9, and there is no significant buffer effect. Various reducing and enzyme-stabilizing agents were tested for their effect on the tyrosine-activating system. None of these enhances significantly the incorporation of tyrosine into its tRNA, and all but thiourea, dithiothreitol, and 2-mercaptoethanol are inhibitory when quantities as small as 10 micromoles are added per assay solution. The ion Mn^{2+}, as compared with Mg^{2+}, seems to enhance aminoacylation; the effectiveness of Ca^{2+} is the same as that of Mg^{2+}. When the incubation time is 20 to 30 min, aminoacylation is independent of Mg^{2+} concentration in the range from 3 to at least 10 micromoles per assay solution. Maximum aminoacylation occurs in the range of adenosine triphosphate (ATP) concentration from about 0.1 to 2 micromoles per assay solution in the presence of both 5 and 10 micromoles of Mg^{2+}; therefore, the Mg^{2+}/ATP mole ratio does not seem to be a controlling factor.

5.1.c Determination of Valyl- and Alanyl-Transfer Ribonucleic Acids (tRNA)

I. B. Rubin

Preliminary studies were begun to establish the optimum conditions for the determination of alanine- and valine-accepting tRNA's in anticipation of having these two compounds in a purified form. The optimum pH range for the alanine system, in crude tRNA, is 5.8 to 7.2, which is a lower and narrower pH range than that for any of the other systems studied so far. The optimum pH range for the valine system is 6 to 9. However, both systems are buffer sensitive; cacodylate is preferred for alanine and Tris for valine. Several new buffers

[1] I. B. Rubin and A. D. Kelmers, "The Determination of Transfer Ribonucleic Acid by Aminoacylation. I. Leucyl- and Phenylalanyl-Transfer Ribonucleic Acid from *E. coli* B," presented at Southeastern Regional Meeting, American Chemical Society, Louisville, Ky., Oct. 27–29, 1966.

having lower pk_a's than cacodylic acid were tested with the alanine system; of them, only 2-(*N*-morpholino)ethanesulfonic acid (MES), in a concentration of 50 micromoles per assay solution, gives results that are equivalent to those obtained with cacodylate buffer at pH 6.6.

5.1.d Double-Labeling Assays

I. B. Rubin Gerald Goldstein

Leucine- and phenylalanine-accepting tRNA's can be determined simultaneously by use of ^{3}H-labeled phenylalanine having a high specific activity and ^{14}C-labeled leucine, both together in the assay solution. Since optimum conditions for the separate determination of each tRNA differ in several important respects, the conditions for their simultaneous determinations must be a compromise. Consequently, this procedure is suitable for qualitative but not quantitative determinations. For the dual-isotope scintillation spectrometry, the discriminator settings must be adjusted to minimize the interference of one isotope with the other; this adjustment decreases the total counts. With the optimum settings, ^{14}C still interferes in the determination of ^{3}H, and a correction amounting to 30% of the ^{14}C counted must be subtracted. On the other hand, ^{3}H interferes in the counting of ^{14}C to the extent of only 0.5%.

5.1.e Automated Analysis of Transfer Ribonucleic Acids (tRNA)

Gerald Goldstein

Several possible methods for tRNA assays that might be automated were considered. It was decided to continue with the paper-disk procedure now in use. AutoAnalyzer components were purchased from Technicon Instruments Corporation that will withdraw a sample, mix it with reagent and enzyme, and incubate the mixture at 37°C for the proper time. Instrumentation is now being developed to remove a known volume of the incubated mixture and to deliver it onto a paper disk. Also, two additional liquid-scintillation counters were purchased; they provide an analysis potential of about 10,000 samples per month. A card-punch adapter was purchased for one of the new counters, and computer programs are being prepared to assist in processing the data.

5.1.f Gas Chromatography of the α-Amino Alcohols

A. D. Horton

A proposed method of analysis of tRNA's is to reduce the aminoacyl esters to amino alcohols and to subsequently determine the alcohols by gas chromatography. The amount of material available for analysis would be too small to obtain a visible recorder trace on the gas chromatograph. However, if the alcohols can be separated, ^{14}C labeling would enable one to detect them as they are eluted from the column.

It was necessary, at first, to determine whether micro, but visible, quantities of amino alcohols can be separated by gas chromatography.

Weinstein and Fenselau[2] reported the separation of glycinol, alaninol, valinol, leucinol, serinol, isoleucinol, methioninol, and phenylalaninol on a 2-m-long column of silicone polymer SE-52 (20 wt % on 60- to 80-mesh Chromosorb W that was treated with hexamethyldisilazane). To obtain retention data for histidinol and tyrosinol, in addition to these compounds, a 9-ft-long by ¼-in.-OD column of SE-52 (20 wt % on acid- and base-washed, hexamethyldisilazane-treated, 60- to 80-mesh Anakrom ABS) was used here. In Table 5.1 the retention data obtained are compared with those of Weinstein and Fenselau.[2] Figure 5.1 is a gas

[2]B. Weinstein and A. H. Fenselau, "Amino Acids and Peptides. II. Gas Chromatography of Amino Alcohols," *J. Chromatog.* 15, 149 (1964).

Table 5.1. Retention Times of α-Amino Alcohols

Alcohol	Retention Time (min)	
	Weinstein and Fenselau[a]	This Laboratory
Glycinol	1.6	2.8
Alaninol	1.9	3.5
Valinol	4.9	7.0
Leucinol	6.6	10.7
Serinol	8.2	13.2
Isoleucinol	9.8	9.0
Methioninol	26.1	18.0
Histidinol		19.1
Tyrosinol		20.7
Phenylalaninol	37.1	26.9

[a]Isothermal at 155°C.

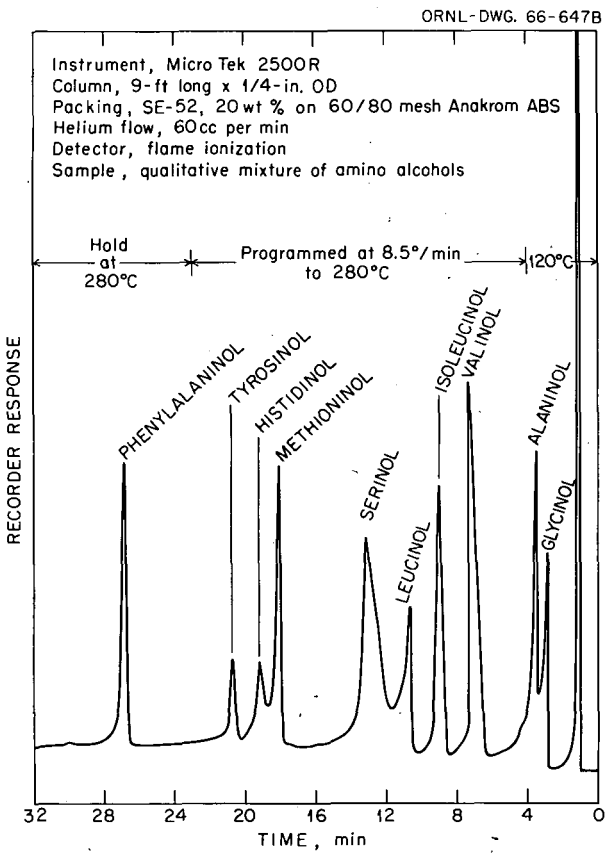

Fig. 5.1. Gas Chromatogram of Some α-Amino Alcohols.

chromatogram of a mixture of some of the α-amino alcohols. The column used here gives better resolution of the α-amino alcohols and faster analysis than were achieved by Weinstein and Fenselau.

5.1.g Determination of Impurities in Transfer Ribonucleic Acids (tRNA)

Gerald Goldstein

Protein. – A modified Folin-Lowry method[3] was found to be satisfactory for determining 20 to 100 μg of protein in a tRNA sample. Compared with a bovine serum albumin used as a standard, each of five batches of tRNA prepared by the Chemical Technology Division had less than 1% protein contamination.

Carbohydrate. – Several procedures for the determination of total carbohydrate with anthrone reagent were tested but were not satisfactory. An enzymatic method for glucose[4] was finally adopted. Glucose oxidase and horseradish peroxidase are used; the pertinent reactions are

$$\text{glucose} + O_2 + H_2O \xrightarrow{\text{glucose oxidase}} \text{gluconic acid} + H_2O_2 ,$$

$$H_2O_2 + \text{reduced chromogen} \xrightarrow{\text{peroxidase}} \text{oxidized chromogen} .$$

The chromogen employed was *o*-dianisidine, the oxidized form of which has an absorption maximum at 436 mμ. Each of three tRNA preparations contained less than 1% glucose contamination.

Quaternary Amines. – The Chemical Technology Division fractionates mixed tRNA on a reversed-phase column that contains Aliquat-204 (dimethyldilauryl ammonium chloride). Bleeding of this quaternary amine from the column was suspected. A method was adapted[5] in which the quaternary-amine cation is paired with the anion of a large dye (bromophenol blue) in basic solution, the complex is extracted with ethylene dichloride, and the absorbance of the dye is measured at 600 mμ. The presence of quaternary amine in some column effluents was confirmed.

Inorganic Phosphate. – An extremely sensitive molybdenum blue spectrophotometric method[6] was adapted to the determination of inorganic phosphate in tRNA. The tRNA is precipitated with trichloroacetic acid, the mixture is centrifuged, and an aliquot of the supernatant liquid is analyzed. As little as 0.025 μg/ml of P can be determined. Eight tRNA preparations were analyzed, two of which were found to be seriously contaminated with inorganic phosphate.

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

[4]H.-U. Bergmeyer (ed.), *Methods of Enzymatic Analysis*, 2d ed., p. 123, Academic, New York, 1965.

[5]M. E. Auerbach, "Germicidal Quaternary Ammonium Salts in Dilute Solutions," *Ind. Eng. Chem. Anal. Ed.* **15**, 492 (1943).

[6]P. S. Chen, Jr., T. Y. Toribara, and H. Warner, "Microdetermination of Phosphorus," *Anal. Chem.* **28**, 1756 (1956).

Deoxyribonucleic Acid. – A modified method[7] for deoxyribonucleic acid (DNA) was tested but is no better than the method currently used.[8]

Water. – Water was determined in several tRNA solids by Karl Fischer titration. Although no standards are available for this material, the results were reproducible and seemed reasonable.

5.1.h Separation of Nucleotides, Nucleosides, and Nucleic Acid Bases by Ligand Exchange Chromatography

Gerald Goldstein K. B. Green[9]

Ligand exchange chromatography is a useful technique for separating compounds that form complexes with metal ions; the principles were discussed by Helfferich.[10] In practice, a cation exchange column is loaded with a complexing metal ion (e.g., Cu^{2+}, Ni^{2+}, or Ag^{+}) that retains the ability to coordinate ligands. Ligands, loaded onto the column, are eluted by a more strongly retained ligand; separations are effected by virtue of the differences among the stabilities of the metal ion–ligand complexes.

To separate nucleic acid components, most of which are bases with free amino groups, a copper-loaded column was used; the elutant was ammonia. After cation exchange resins and column conditions were tested, a column (0.9 by 33 cm) of copper-loaded chelating resin (Chelex 100, 200 to 400 mesh) was prepared. The eluent flow rate was 0.76 ml/min (1.2 ml cm^{-2} min^{-1}) in all cases, and the ultraviolet absorption of the effluent was monitored and recorded. The results are as follows.

Nucleotides. – The nucleotides are anionic under the conditions of these tests (neutral or basic solutions) and are not retained on the column. There appears to be partial separation of 2′- and 3′-phosphate isomers, but this possibility was not explored.

Nucleosides. – The nucleosides having no free amino groups are only weakly retained on the column and can be eluted with water (Table 5.2). Nucleosides that have free amino groups are more strongly retained and are eluted with 1 *N* NH_4OH (Table 5.3). The four major nucleosides (uridine, guanosine, cytidine, and adenosine) can be rapidly and quantitatively separated. Adenosine can be easily separated from nucleotides. This fact suggests that this separation may be useful for end-group analysis.

Nucleic Acid Bases. – With the exception of uracil, which can be eluted with water, the nucleic acid bases are more strongly complexed than the

Table 5.2. Elution with Water of Compounds That Have No Free Amino Groups

Compound	Time of Appearance of Ultraviolet Peak (min)[a]	Volume of Effluent (ml)
Xanthosine	16.5	12.5
Uridine	23.0	17.5
Pseudouridine	23.6	17.9
Thymine riboside	27.3	20.7
Uracil	27.3	20.7
Dihydrouridine	32.4	24.6

[a]Measured from start of effluent flow.

Table 5.3. Elution of Nucleosides with 1 *N* NH_4OH

Nucleoside	Time of Appearance of Ultraviolet Peak (min)[a]	Volume of Effluent (ml)
7-Methylinosine	16.0	12.1
7-Methylguanosine	16.6	12.6
Inosine	19.1	14.5
Guanosine	21.1	16.0
Cytidine	36.2	27.5
Adenosine	55.6	42.2
N^6-Methyladenosine	62.1	47.2
N^6-Dimethyladenosine	91.6	69.6

[a]Measured from start of effluent flow.

[7]K. W. Giles and A. Myers, "An Improved Diphenylamine Method for the Estimation of Deoxyribonucleic Acid," *Nature* **206**, 93 (1965).

[8]K. Burton, "A Study of the Conditions and Mechanism of the Diphenylamine Reaction for the Colorimetric Estimation of Deoxyribonucleic Acid," *Biochem. J.* **62**, 315 (1956).

[9]ORAU Student Trainee from William Jewell College, Liberty, Mo.

[10]F. Helfferich, "Ligand Exchange. I. Equilibria," *J. Am. Chem. Soc.* **84**, 3237 (1962).

nucleosides, and 2 *N* NH_4OH is necessary to elute them (Table 5.4). On the whole, the bases are not well separated.

Instrumentation is now being improved, and additional compounds will be tested as they become available.

Table 5.4. Elution of Bases with 2 *N* NH_4OH

Base	Time of Appearance of Ultraviolet Peak (min)[a]	Volume of Effluent (ml)
Xanthine	45.0	34.2
Adenine	46.1	35.0
5-Hydroxymethylcytosine	53.4	40.6
Cytosine	58.2	44.2
5-Methylcytosine	60.6	46.0

[a]Measured from start of effluent flow.

5.1.i Interaction of Biological Compounds with Fluorescent Metal Chelates

C. A. Horton

Work continued[11] on the effects of various nucleotides, polynucleotides, tRNA, and DNA on the fluorescence of metal-organic adducts and chelates. Various fluorometers and spectrofluorometers were used. These biological compounds have various effects on the fluorescence intensity of the aluminum-morin system under various conditions. The polarization of the fluorescence of this system, both in the absence and presence of biological compounds, was obtained at a single wavelength and over the entire spectral region of fluorescence. The polarization study was based on Laurence's[12] method for determining molecular weight and structure of proteins. The polarization of the fluorescence of the aluminum-morin system by alcohols in concentrations of 2 to 10% did not agree with earlier results reported for this system in absolute ethyl alcohol or glycerol.[13] Figure 5.2 shows the spectrum obtained with polarizing filters located in the path of the fluorescent light

[11]C. A. Horton, "Transfer Ribonucleic Acid (tRNA) Analytical Studies," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1965*, ORNL-3889, p. 32.

[12]D. J. R. Laurence, "A Study of the Adsorption of Dyes on Bovine Serum Albumin by the Method of Polarization of Fluorescence," *Biochem. J.* 51, 168 (1952).

[13]I. Ketskémety, N. Marek, and B. Sárkány, "Relation Between the Absorption and Polarization Spectra of a Fluorescent Morin Compound," *Acta Univ. Szeged., Acta Phys. Chem.* 4, 21 (1958).

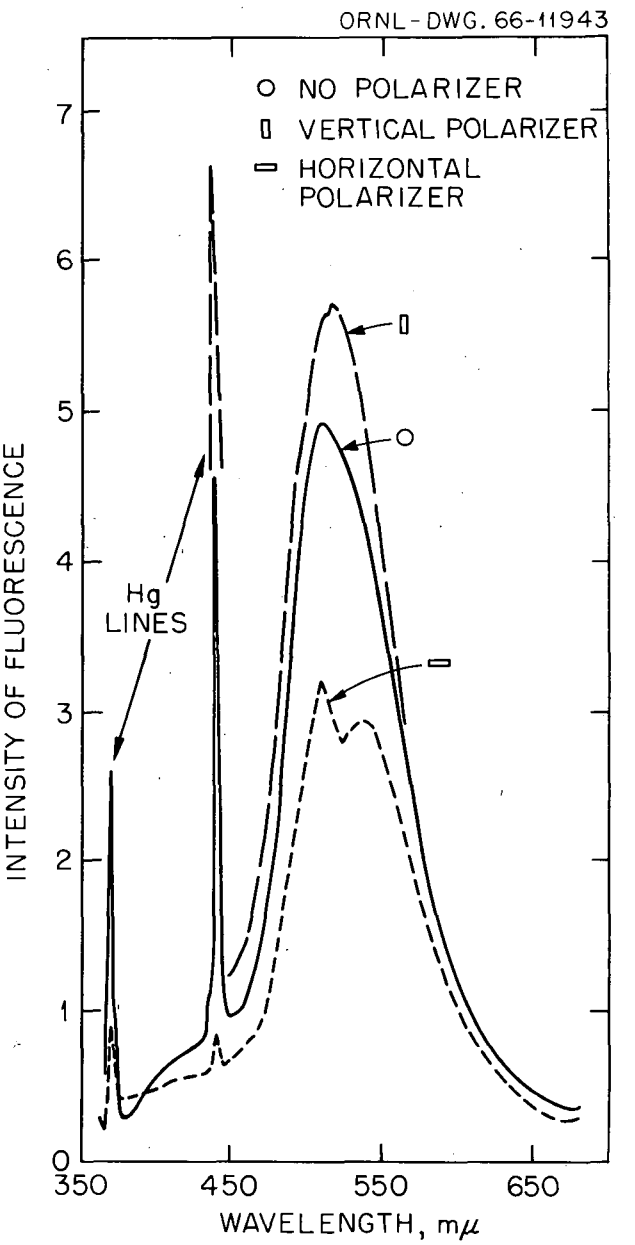

Fig. 5.2. Fluorescence of Aluminum-Morin System With and Without Polarizer.

beam. With a horizontal polarizer, the intensity of fluorescence is diminished by the polarization introduced by the monochromator grating. The spectrum obtained with the horizontal polarizer shows a double maximum of fluorescence intensity not reported before. This effect caused difficulties in measurements made with biological compounds present at the single wavelengths 505, 520, and 540 mμ.

The effect of tRNA on the fluorescence of magnesium-8-quinolinol-5-sulfonate at pH 7.0 in Tris buffer was very slight; a modification of Schachter's method was used to study this effect.[14] With aluminum-8-quinolinol-5-sulfonate at pH 5.0 in acetate buffer, intensities of fluorescence and degree of polarization changed on addition of biological compounds, but not nearly as much as with the aluminum-morin system. With the beryllium-morin system[15] in neutral or slightly alkaline medium, the addition of tRNA had no appreciable effect on the intensity of fluorescence.

The observed effects of biological compounds on the intensity of fluorescence of the aluminum-morin system indicate that this system should be studied further.

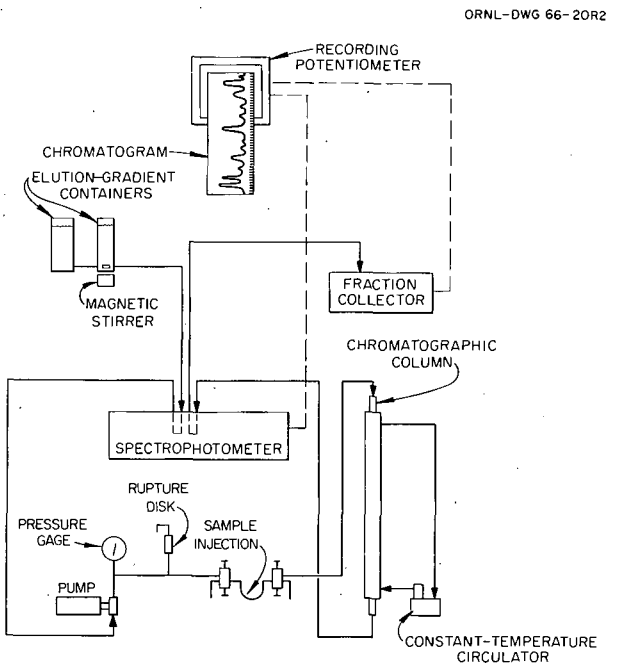

Fig. 5.3. Diagram of Automatic Nucleotide Analyzer.

5.2 BODY FLUIDS ANALYSIS PROGRAM

J. E. Attrill

The feasibility of using the automatic nucleotide analyzer, developed by Anderson,[16] for the analysis of human urine has been studied. Figure 5.3 is a schematic diagram of the instrumentation. It consists of an anion exchange column for separating the components of a specimen, a spectrophotometer for monitoring the column effluent at two wavelengths, and a two-point recorder for plotting chromatograms (time vs concentration of the eluted components) at these wavelengths. A constant-flow Mini-Pump is used to move a gradient of increasing ionic strength, but constant pH, through the system to effect the separation.

Initial analyses of 1-ml portions of a sample of human urine gave excellent resolution of 57 peaks during a 48-hr analysis. Later tests showed that some of these peaks correspond not to one but to several compounds, and experimental work was deemed necessary to optimize the conditions for more complete separations. Further studies by use of other flow rates, other column temperatures, a resin of finer mesh, and other gradient concentrations gave chromatograms that consist of a hundred peaks resolved from a 1-ml fraction of the same urine. The reproducibility of the chromatograms was excellent, but urine specimens had to be kept refrigerated or frozen between runs to prevent bacteriological decay and resulting changes in concentrations of some of the components.

The objectives of the study then became (1) to establish experimental conditions that give maximum separation of components, (2) to identify and characterize the compounds with reference to elution time and absorbance ratios at wavelengths of 260 and 280 mμ, (3) to study methods to decrease analysis time, and (4) to miniaturize the entire system. The automatic nucleotide analyzer possibly may be a valuable analytical tool for clinical analysis.

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[14] D. Schachter, "Fluorimetric Estimation of Magnesium with 8-Hydroxy-5-quinoline Sulfonate," *J. Lab. Clin. Med.* **58**, 495 (1961).

[15] C. W. Sill and C. P. Willis, "Fluorometric Determination of Submicrogram Quantities of Beryllium," *Anal. Chem.* **31**, 598 (1959).

[16] N. G. Anderson, "Analytical Techniques for Cell Fractions. II. A Spectrophotometric Column Monitoring System," *Anal. Biochem.* **4**, 269 (1962).

Because many of the single resolved peaks result from the presence of several compounds, attempts were made to identify the components associated with each single peak. Pure compounds known to exist in milligram quantities in human urine (Table 5.5) were chromatographed under conditions identical with those used for the sample of human urine. Their elution times and absorbance ratios are given in Table 5.6. Since the ratio of the ultraviolet absorbancies at 260 and 280 mμ (A_{280}/A_{260}) is specific for a particular compound, this ratio is an excellent means for rapid tentative identification. The complete ultraviolet and infrared spectra were then used to confirm the identities. About 25 compounds have thus been identified. A 1-in.-diam by 6-ft-long preparation column has been constructed for collecting quantities of the compounds that are difficult to identify in the above manner. Attempts will be made to identify them by other analytical means.

5.3 PYROLYSIS-GAS CHROMATOGRAPHIC STUDIES OF MONONUCLEOSIDES AND MONONUCLEOTIDES

L. P. Turner[17]

A systematic study of the pyrolysis products of some mononucleosides and mononucleotides was begun. Adenosine, guanosine, cytidine, uridine, thymidine, and the sodium salts of 5′-adenosine monophosphate (AMP), 5′-guanosine monophosphate (GMP), 5′-cytidine monophosphate (CMP), and 5′-uridine monophosphate (UMP) were pyrolyzed at 800°C.

Low-molecular-weight products were analyzed by use of a high-activity silica-gel gas chromatographic column. A Carbowax 20M column was used for the medium- and high-molecular-weight products. Both flame-ionization and thermal-conductivity detection were used, with programmed-temperature column conditions. The detection systems were calibrated with known compounds that had been identified previously as products.

The pyrolysis products of the mononucleosides and mononucleotides studied vary little in nature. However, their relative amounts differ markedly from compound to compound, thus providing a means to identify each pyrolyzed structure. The major products identified are: CO, CO_2, CH_4, C_2H_4, C_2H_6, C_2H_2, propylene, ethyl acetylene, acetonitrile, and proprionitrile. The data are now being evaluated quantitatively, and the major medium- and high-molecular-weight products are being identified.

[17]ORAU Research Participant, summer 1966; Assistant Professor, Department of Chemistry, University of Tennessee, Knoxville.

Table 5.5. Compounds Existing in Urine in Larger Quantities[a]

Compound	Concentration	Compound	Concentration	Compound	Concentration
Adenine	1–2 mg/24 hr	Glutamine	100 mg/24 hr	7-Methyluric acid	(Dietary)
Alanine	21–71 mg/24 hr	Glycine	68–199 mg/24 hr	1-Methylxanthine	
β-Alanine	14–23 mg/24 hr	Glycocyamine	21–67 mg/24 hr	7-Methylxanthine	(Depends on coffee and tea consumed)
Allantoin	10–30 mg/24 hr	Guanine	1–2 mg/24 hr		
α-Aminoadiptic acid	8–11 mg/24 hr	Hippuric acid	1000–2500 mg/l	Nicotinamide	0.6–2.0 mg/24 hr
p-Aminobenzoic acid	300–550 mg/24 hr	Histadine	113–320 mg/24 hr	Nicotinamide-6-pyridone	3.6–20 mg/24 hr
o-Aminohippuric acid	17–31 mg/24 hr	Hypoxanthine	6–13 mg/24 hr	Nicotinamide methyl chloride	9 mg/24 hr
β-Aminoisobutyric acid	4–180 mg/24 hr	m-Hydroxybenzoic acid	10–16 mg/l	Nicotinic acid	0.4–2.0 mg/24 hr
Anserine	5–7 mg/24 hr	m-Hydroxyhippuric acid	2–150 mg/l	Oxalic acid	20–40 mg/l
Arginine	25–81 mg/24 hr	5-Hydroxyindoleacetic acid	2–14 mg/l	α-Oxoglutaric acid	13–60 mg/l
Ascorbic acid	5–55 mg/24 hr	3-Hydroxykynurine	1–6 mg/24 hr	Pantothenic acid	1.5–7 mg/24 hr
Asparagine	34–99 mg/24 hr	8-Hydroxy-7-methyl guanine	1–2 mg/24 hr	Phenol	7–13 mg/l
Aspartic acid	59–230 mg/24 hr	o-Hydroxyphenylacetic acid	1.0–400 mg/l	Phenylacetylglutamine	250–500 mg/24 hr
Carnitine	28–56 mg/24 hr	p-Hydroxyphenylacetic acid	0.85 mg/l	Phenylalanine	9–31 mg/24 hr
Carnosine	2–3 mg/24 hr	p-Hydroxyphenylhydracrylic acid	2–150 mg/l	Pyridoxine	0.06–1.2 mg/24 hr
Cholesterol	0.3–1.5 mg/l	Indican	40–160 mg/l	Pyruvic acid	2.5–60 mg/l
Choline	5–9 mg/l	Inositol	8–144 mg/l	Riboflavin	0.5–1.25 mg/24 hr
Citric acid	200–1200 mg/l	Isoleusine	12–28 mg/24 hr	Serine	27–73 mg/24 hr
Creatine	0–800 mg/24 hr	Kynurenic acid	13–19 mg/24 hr	Taurine	86–294 mg/24 hr
Creatinine	1.1–1.7 mg/24 hr	Kynurine	9–17 mg/24 hr	Threonine	15–53 mg/24 hr
p-Cresol	40–117 mg/l	Lactic acid	73 mg/l	Tryptophan	8–25 mg/24 hr
Cystine	10 mg/24 hr	Leucine	9–24 mg/24 hr	Tyrosine	15–49 mg/24 hr
Dehydroascorbic acid	5 mg/24 hr	Lysine	7–48 mg/24 hr	Urea	15–35 g/24 hr
Dimethylsulfone	5–6 mg/24 hr	N-Methylguanine	0.4–0.6 mg/24 hr	Uric acid	6–12 mg/kg/24 hr
1,7-Dimethylxanthine	(Dietary)	1-Methylguanine	10% of 7-Methylguanine	Valine	10 mg/24 hr
Ergothionine	1–10 mg/24 hr	7-Methylguanine	5–8 mg/24 hr	Vanillic acid	8–12 mg/l
Flavins	0.8–1.2 mg/l	1-Methylhistadine	47–384 mg/24 hr	Xanthine	5–9 mg/24 hr
Folic acid	2–6 mg/24 hr	3-Methylhistadine	50 mg/24 hr	Xanthanurenic acid	27–51 mg/24 hr
Glucuronic acid	100–1325 mg/l	1-Methylhypoxanthine	0.2–0.7 mg/24 hr	Xanthanurenic acid-8-methylester	1 mg/24 hr
Glutamic acid	100–500 mg/24 hr	7-Methylnicotinamide	3–20 mg/24 hr		
		1-Methyluric acid	(Dietary)		

[a]Information taken from C. Long (ed.), *Biochemists' Handbook*, pp. 918–36, Van Nostrand, Princeton, N.J., 1961.

Table 5.6. Chromatographic and Ultraviolet-Spectral-Absorption Characteristics of 20 Compounds Usually Present in Urine

Compound[a]	Elution Time (hr)	A_{280}/A_{260}
Noradrenline	0.1	3.9
Hypoxanthine	1.2	0.10
Xanthine	1.8	0.63
Urocanic acid	3.6	0.54
Uric acid	5.2	2.8
Kynurine	5.4	2.3
Maleuric acid	8.3	0.16
3-Methoxy-4-hydroxymandelic acid	13.5	3.7
p-Aminobenzoic acid	14.0	1.5
Hippuric acid	14.1	0.24
p-Hydroxymandelic acid	16.5	1.6
Homovanillic acid	17.0	3.8
p-Cresol	20.0	2.8
p-Hydroxyphenylacetic acid	23.0	2.2
3-Indoleacetic acid	23.5	1.6
m-Hydroxyphenylacetic acid	24.0	1.8
Vanillic acid	26.0	0.47
Kynurenic acid	30.0	0.28
Salicylacetic acid	32.0	3.0
5-Hydroxyindoleacetic acid	40.0	1.5

[a]The usual concentrations of these compounds in urine are given in Table 5.5.

6. X-Ray and Spectrochemical Analyses

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

M. T. Kelley

Cyrus Feldman

6.1.a X-Ray Analysis

H. W. Dunn

X-Ray Absorption-Edge Analysis. – A code for processing x-ray absorption-edge data on both the CDC 1604A and the IBM 360 computers has been tested and found to be successful. All elements heavier than Mn (except Hf, Ta, and W) can be determined in solution by this method.

Electron Probe Code. – A code, originally written by J. D. Brown,[1] for the determination of local chemical composition from the x-ray emission data produced by an electron-probe analyzer was modified and adapted to the IBM 360 computer. Table 6.1 shows the averaged results of the analyses of six 1-μ^2 areas of a specimen of Haynes Stellite 25. The accuracy is considered to be

[1]J. D. Brown, *A Computer Program for Electron Probe Microanalysis*, U. S. Bureau of Mines Report, RI-6648 (1965).

Table 6.1. Results of Electron-Probe Analysis of Haynes Stellite 25

Component	Amount (wt %)	
	Nominal	Found[a]
Co	50	50.8
Cr	20	20.7
Fe	1	3.3
Mn	1.5	0.3
Ni	10	10.7
Si	0.7	0.5
W	15	13.6

[a]Average of results from six 1-μ^2 areas.

satisfactory in all cases except those of the minor elements Fe and Mn. The causes for these discrepancies are being sought.

Electron-Probe Analysis of Tissue. – Specimens of kidney and bone tissues were studied with the electron probe. The localization of uranium was observed in the first case, and the distribution of Ca and P was photographed in the second.

6.1.b Atomic Absorption

Cyrus Feldman N. Marion Ferguson

Determination of Cobalt. – The atomic absorption of solutions of cobalt in dilute nitric acid was investigated by use of a 60-cm-long end-fed absorption tube, hollow cathode, and the detection equipment described previously.[2] When relatively rapid sample feeding and a lean fuel mixture (O_2, 15 lb/in.^{2}; H_2, 1.5 lb/in.^{2}) were used, a detection limit for Co of ~0.01 μg/ml was attained. This limit was lowered to 0.003 μg/ml by scale expansion.

6.1.c Semiquantitative Spectrographic Analysis (Visual Interpolation)

Anna M. Yoakum Cyrus Feldman

Encouraging preliminary results obtained by the Kroonen-Vader Li_2CO_3-base method[3] suggested further testing of the method. Twelve NBS standards that included ferrous and nonferrous alloys, clays, and refractories were analyzed by this method. Standard films, with minor lines identified, were used for interpolation. The results were classified on the basis of the ratio of the concentration found to the true concentration or the reciprocal, the larger value always being the numerator. Of the values obtained, 75% were accurate to within a factor of 1.5, 91% to within a factor of 2, and 99% to within a factor of 3. The method was extended to include rare-earth and platinum-group metals. The relative standard deviation of intensity ratios determined from the spectrograms by densitometry is 7%. A test of a universal semiquantitative method based on the use of a powdered germanium-metal matrix showed better sensitivity than the Li_2CO_3 method for volatile easily reduced metals (e.g., Zn, Cd, Hg, Sb, Tl) but did not show the expected freedom from selective volatility. It would probably be advantageous to develop this approach further as a high-sensitivity method for such elements.

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

A. E. Cameron

J. A. Carter

6.2.a Determination of Technetium in Chromium-Technetium Alloys

J. A. Carter

To determine Tc in Cr-Tc alloys, a Tc channel was added to the Paschen Direct Reader. A calibration curve was established by use of the Tc 2610.00-A line; the Sr 4077.71-A line was the internal standard. Standard solutions were prepared from Tc metal. With the rotating-disk technique, the limit of detection for Tc is about 5 μg/ml. A calibration curve for the determination of Tc in the presence of Cr was obtained that has a theoretical slope. For the Solid State Division, Cr-Tc alloys of Tc content in the range from 1 to 15 wt % have been analyzed.

[2]S. R. Koirtyohann and C. Feldman, "Atomic Absorption Spectroscopy Using Long Absorption Path Lengths and a Demountable Hollow Cathode Lamp," pp. 180–189 in *Developments in Applied Spectroscopy*, vol. 3, ed. by J. E. Forrette and E. Lanterman, Plenum, New York, 1964.

[3]Zell Combs, C. A. Pritchard, and Anna M. Yoakum, "Semiquantitative Spectrographic Analysis (Visual Interpolation)," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1965*, ORNL-3889, pp. 38–39.

7. Mass Spectrometry

A. E. Cameron

7.1 SPARK-SOURCE MASS SPECTROMETRY

J. R. Sites J. A. Carter

The Associated Electrical Industries, Ltd. (AEI) MS-7 spark-source mass spectrometer[1] was used to expose 440 photographic plates from 49 types of samples. Very pure samples of Fe, Ni, and Cu were analyzed, and the results were compared with those for NBS standards. A number of sets of sections cut from Nb rods that had been multipass zone-refined were analyzed for several specific trace impurities; Fig. 7.1 shows the variation in Zr content. The following alloys were analyzed: Cr-Tc, Cr-Ta, Al-Mg-Li, Pb-Th, Bi-Th, U-Th, Zr-Nb, and Ce-Mg; the compounds analyzed were: KCl, LiCl, NaCl, $MnSO_4$, BN, Al_2O_3, and CeO_2. A set of Au, Ag, Pt, Pd, and Ir samples were run.

[1] J. R. Sites and J. A. Carter, "Spark-Source Mass Spectrometry," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1965*, ORNL-3889, p. 39.

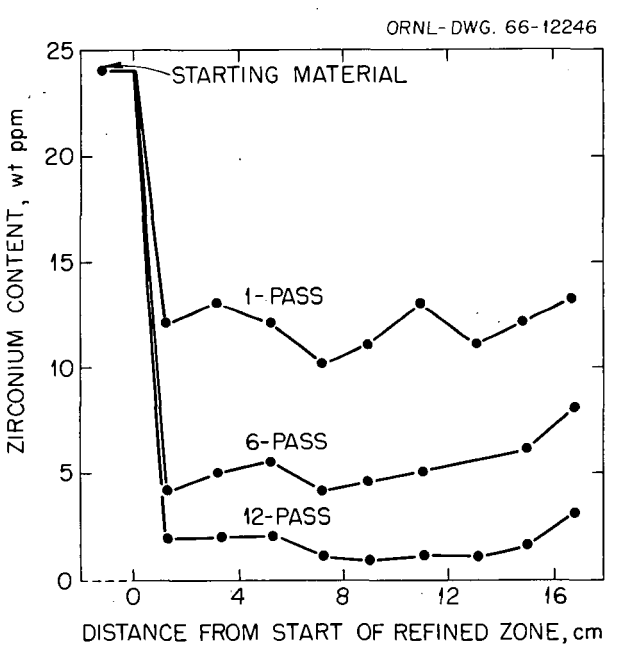


Fig. 7.1. Zirconium Content of Segments of Zone-Refined Niobium Rods.

Boron in reactor-construction metals is being determined with the MS-7 spectrometer for the Metals and Ceramics Division. To provide a quantitative determination of B in stainless steel, Hastelloy, Inconel, and other metals, a calibration curve such as that shown in Fig. 7.2 is used. The slope of the curve is about 1, and the curve is usable for the range of B concentration from <1 to 1000 ppm. National Bureau of Standards samples were used to prepare the curve and to monitor its usability with different emulsions.

The semiquantitative analysis of solutions can be achieved rapidly by evaporating a solution on a metal substrate such as Nb or Au and by using Sr as an internal standard. Sponge Au electrodes used as substrates afford even excitation of the sample, because the surface area is very large. The limit of detection by this technique is better than 0.1 ng of element per electrode. Typical raw-recovery results are summarized in Table 7.1. This technique has been applied to the analysis of rare earths, seawater, paints, solutions of U metal, and solutions of rare earths and rare-earth elements.

The program to calculate impurity concentrations was converted from use of the ORNL CDC 1604 computer to use of the IBM 360/75 computer. A variation of the Churchill two-line method for plotting an emulsion calibration curve was added to the program. Instead of the plot being started at one end of the intensity-vs-transmittance curve where the data may be poor, it is started at the center (50% transmittance) and is completed toward 100% transmittance and then toward 0% transmittance. Thus, the most frequently used center sections of several curves can be compared easily, and empirical emulsion characteristics are seen at a glance.

7.2 TRANSURANIUM MASS SPECTROMETRY

R. E. Eby

The double-stage mass spectrometer was used as a mass separator to obtain a batch of ^{251}Cf

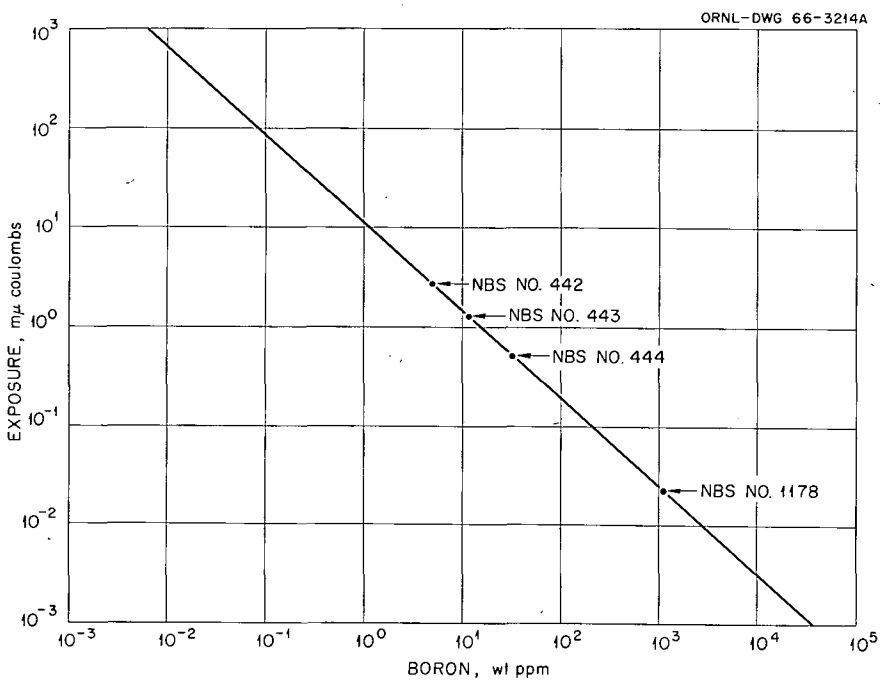


Fig. 7.2. Typical Colibration Curve for the Determination of Boron in Steels with the MS-7 Mass Spectrometer (^{11}B at 40% Transmittance).

Table 7.1. Results of Analyses with the MS-7 Mass Spectrometer[a]

Matrix	Substrate	Concentration of Element Added (wt ppm)	Element Found (wt ppm)				
			Al	Cr	Fe	Mn	Ni
U	Nb	100	100	110	110	150	78
Solution	Nb	10	9.1	9.1	12	10	7.8
Solution	Au	1	1.4	0.9	0.6	0.6	0.5

[a]Sr as internal standard.

for A. Chetham-Strode.[2] The amount of ^{251}Cf collected was $\sim 2.0 \times 10^{10}$ atoms; it contained <30 ppm ^{252}Cf impurity. In this separation $\sim 1/2500$ of the original ^{251}Cf atoms were collected. R. Silva[3] is using the batch to determine the alpha and gamma spectra and various other properties of ^{251}Cf.

[2]Deceased; formerly with Chemistry Division.

[3]Chemistry Division.

7.3 SURFACE IONIZATION STUDIES

G. R. Hertel

7.3.a Effect of Oxygen Contamination on the Surface Ionization of Uranium Tetrafluoride

The positive ions produced when a beam of UF_4 molecules strikes a hot filament of W or Re include U^+, UO^+, and UO_2^+. In a clean well-pumped system (pressure $< \sim 10^{-8}$ torr) with a well-aged properly baked-out filament, U^+ is the only de-

tectable product ion. The temperature dependence of the ion intensity of U^+ obeys the Saha-Langmuir equation from the highest temperature measured down to ~2300°K or below. However, a trace of O_2 markedly affects the U^+ emission; it causes the ion intensity to decrease more rapidly with decrease in temperature than is predicted. This effect is accompanied by the appearances of UO^+ and UO_2^+ ions over limited temperature ranges. The greater the O_2 contamination, as measured by the intensities of the oxide beams, the greater is the effect on the U^+ emission. The ion intensity of UO_2^+ reaches a maximum at ~1800°K; the maximum ion intensity of UO^+ occurs at ~2400°K.

The mean residence time, τ, of the U^+ ion has been determined as a function of temperature over the range from ~2100 to ~2850°K. The evaporation energy (E_i) of U^+ ions was calculated for the case of no detectable O_2 contamination to be 7.50 ±0.09 ev; the calculation was made from the Frenkel equation,

$$\frac{1}{\tau} = W(E_i) = \omega_0(E_i) \exp\left[E_i/(kT)\right] , \quad (7.1)$$

in which W is the probability of desorption of ions from the ionizer, ω_0 is a frequency associated with the bond between the adion and the surface, k is the Boltzmann constant, and T is the temperature of the ionizing surface in °K. Data over the entire temperature range closely obey the equation. In the case of the "worst" O_2 contamination, the data obey Eq. 7.1 only at high temperatures (> ~2400°K), but E_i is 6.27 ± 0.08 ev. At temperatures below ~2400°K, the $1/\tau$ values for U^+ remain almost constant at ~1 sec^{-1} and are essentially equal to the $1/\tau$ values of the UO^+ ions. For neither UO^+ nor UO_2^+ can the $1/\tau$ be described by Eq. 7.1.

These findings explain why the measured ion intensities fail to obey the Saha-Langmuir equation at temperatures below ~2300°K. In a clean O_2-free environment, molecules of UF_4 strike the hot ionizer and either dissociate immediately into atoms, a certain number of U atoms sticking to the surface (the fate of the fluorine is not known), or stick to the surface as molecules, which undergo decomposition and release U^+ ions. In either case, after τ, the U^+ ions leave the surface, the energy required for evaporation being 7.52 ev. When O_2 is present, the U, once on the surface, reacts rapidly with the O_2. The U^+ ion emission is then probably governed by a series of competing reactions, each with its own temperature dependence, which cause a complex τ-vs-T relationship.

Generally, the results are the same for both W and Re ionizers. There are some differences in actual values; τ's are about six times longer when Re is used. Also, E_i determined from the τ for Re was slightly lower (~6.8 ev). This work was done with polycrystalline W and Re surfaces. The nominal value of the work function for polycrystalline materials (e.g., 4.58 ev for W) could not be applied to the data with any precision to determine the ionization potential of U.

7.3.b. Ionization Potential Measurements by Surface Ionization

The first ionization potential of U has been measured by means of a surface-ionization comparison technique.[4] An advantage of this procedure is that calculation of the unknown ionization potential is based on the known ionization potential of a comparison element instead of on the work function of the ionizing surface – a quantity influenced by many variables that are often uncontrolled or unknown. The ion intensity of U^+ as a function of temperature was measured by use of beams of UF_4 molecules and of U atoms and was compared with the ion intensities of Pb^+ and Ag^+ determined under the same conditions with beams of PbF_2, $PbCl_2$, and PbO and of AgBr, respectively. The various Pb compounds were used to validate the assumptions[4] that the molecules dissociate completely on the surface of the hot ionizer and that the data are not influenced by the anion. The behaviors of the different Pb compounds were identical. The compound AgBr was included to check on the experimental procedure by comparison of the ionization potentials of Pb and Ag, values for both of which are well known. The difference between them was measured to be 0.156 ± 0.014 ev, which value agrees well with the value 0.159 ev obtained from the known ionization potentials. The ionization potential of U was determined to be 6.26 ± 0.02 ev. Two other surface-ionization values are available for comparison. The value 6.08 ± 0.08 ev[4] was determined with Li as the

[4] I. N. Bakulina and N. I. Ionov, "Determination of the Ionization Potential of Uranium by a Surface Ionization Method," *Soviet Phys. JETP (English Transl.)* **36**(9), 709 (1959).

comparison element by the same technique as that used here. The value 6.25 ± 0.02 ev[5] is based on the value 4.58 ev taken as the work function of W.

From the behavior of the U^+ emission curve, O_2 contamination on the ionizer filament was apparent. The ions Pb^+ and Ag^+ do not exhibit the same sensitivity to O_2 as does U^+. For Pb^+ and Ag^+, the plot of logarithm of ion intensity (I) vs $1/T$ yields straight lines over the entire temperature range studied, 2100 to 2900°K. The plot for U^+ is straight only at temperatures above ~2700°K.

The ionization potentials of the lanthanide elements are now being measured with this technique.

[5]J. R. Weming, *Thermal Ionization at Hot Metal Surfaces* (Ph.D. thesis), UCRL-8455 (September 1958).

8. Optical and Electron Microscopy

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

8.1 ELECTRON MICROSCOPY OF RADIOACTIVE MATERIALS

The facility for the electron microscopy of radioactive materials, located in Building 3019, is now classified as a type B radiochemical laboratory,[1] with the capability of handling isotopes of very high radioactivity. This classification was achieved as a result of the development of proper preparation procedures and the acquisition of additional equipment for isolating and handling radioactive materials. Neutron-irradiated (10^{20} *nvt*) enriched ^{235}U and PuO_2 of particle size less than 100 A were examined successfully. Surfaces of metallographically polished samples of highly radioactive solids were replicated by means of remotely controlled techniques. Procedures are being developed to increase the efficiency and the resolution of micrographs. The technique for encapsulating radioactive particulates in plastic for examination in the electron microscope was described in a paper.[2]

[1]C. E. Guthrie, E. E. Beauchamp, L. T. Corbin, T. J. Burnett, and T. A. Arehart, *Operating Guide for Radiochemical Laboratories at Various Activity Levels*, ORNL-TM-626 (July 22, 1963).

[2]H. W. Wright and T. E. Willmarth, "Electron Microscopy of Radioactive Powders," presented at 24th Annual Meeting, Electron Microscopy Society of America, San Francisco, Aug. 22–25, 1966.

8.2 NUCLEAR SAFETY PROGRAM

Electron microscopy continued of particulates obtained from experiments designed to simulate fallout from experimental or industrial reactors. This work included the size characterization of radioactive particulate aerosols collected in a fibrous-filter analyzer system,[3] the assessment of various methods for collecting particulates generated in fuel burnup, and the development of a method for using Gelman or Millipore plastic-membrane filters to collect aerosol particulates for examination in the electron microscope.

8.3 ELECTRON MICROSCOPY OF BIOLOGICAL MATERIALS

The Biology Division was assisted in their studies of the configuration of *Escherichia coli* polysomes. The fine structure of ribosomes, revealed in the electron microscope, varies somewhat from specimen to specimen, depending on the procedures used to stain, dry, and shadow.

[3]M. D. Silverman, J. Truitt, W. E. Browning, L. F. Franzen, and R. E. Adams, "Characterization of Radioactive Particulate Aerosols by the Fibrous Filter Analyzer," presented at the 9th AEC Air Cleaning Conference, Boston, Mass., Sept. 12–16, 1966.

8.4 MICROSPHERE DEVELOPMENT PROGRAM

8.4.a Electron Microscopy of Hydrous Uranium Oxide Sols

Electron micrographs and selected-area electron diffraction patterns were obtained for hydrous uranium oxide precipitated from solutions of uranous nitrate and of uranous nitrate and uranous formate; the precipitates were then aged and heated to form sols. Thus, it is possible to characterize UO_2 particles as they are converted from a precipitate to a sol, which is converted to microspheres for use in fuel elements.[4]

8.4.b Examination of Lanthanide Hydroxide Sols by Electron Microscopy and Diffraction

In support of a program to produce microspheres of americium and curium for use as targets in the High-Flux Isotope Reactor (HFIR), numerous samples of the hydroxide sols of Er, Eu, Nd, Pr, and Sm were examined by electron microscopy and by selected-area transmission electron diffraction to determine the physical changes produced by aging.[5]

Figure 8.1 shows the effect of aging on $Pr(OH)_3$. Electron diffraction data are given in Table 8.1; no lattice spacings for $Pr(OH)_3$ have been reported previously in the literature.

The crystal form of the particulates formed during the aging of the lanthanide hydroxides (Fig. 8.2) indicates that the submicro crystals join to form polycrystalline sheets, which tend to roll up into tubelike scrolls on dehydration. This behavior is similar to that of halloysite, a mineral clay.

Further electron microscopy studies are proposed to determine for $Eu(OH)_3$ the effect of solubility on crystal nucleation and growth and the exact time of crystal nucleation.

[4]C. J. Hardy, *Examination of Hydrous Uranium Dioxide Precipitates and Sols by Electron Microscopy, Electron Diffraction, and Spectrophotometry*, ORNL-3963 (August 1966).

[5]T. E. Willmarth, "The Examination of Praseodymium and Europium Hydroxide Sols by Electron Microscopy and Electron Diffraction," presented at the American Meeting of the British Royal Microscopical Society, Chicago, Ill., Aug. 15–19, 1966.

Table 8.1. Electron Diffraction Data for Praseodymium Hydroxide, $Pr(OH)_3$

d(A)	I/I_1 [a]	hkl
5.559	M	100
3.193	M+	110
3.077	VS	101
2.765	W+	200
2.447	W+	111
2.216	S–	201
2.088	VW	210
1.844	S	300
1.832	M+	022
b		211
1.767	M	102
1.601	M	220
b		112
1.536	W	310
b		202
1.413	W+	311
b		212
1.307	W+	302
b	+	320
1.209	W+	410, 222

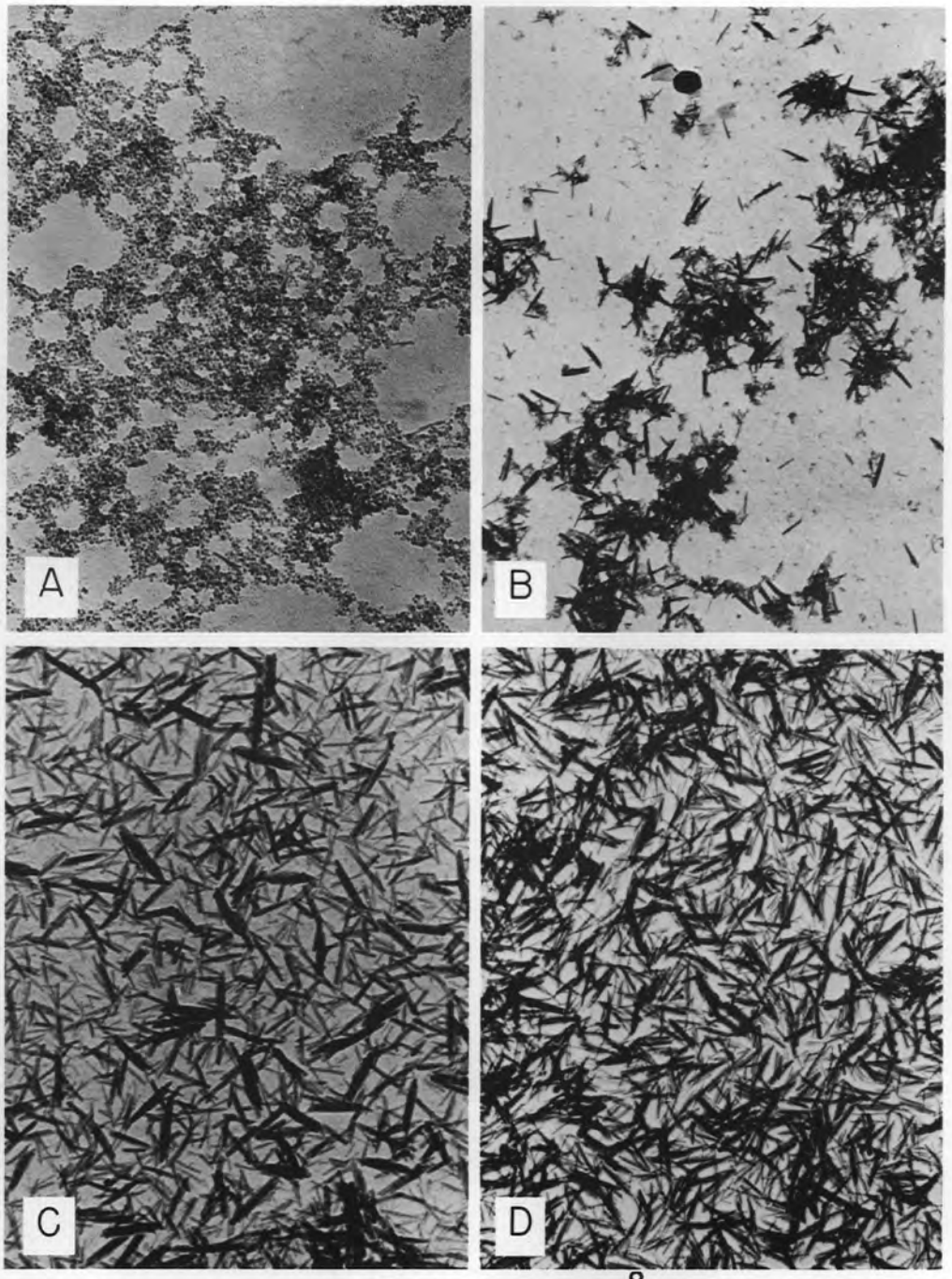
[a]Preferred orientation will cause variance from x-ray data.

[b]Missing or unresolved lines.

8.4.c Examination of Praseodymium and Europium Sol-Gel and Oxide Microspheres by Electron Microscopy

Replicas of fractured and polished surfaces of praseodymium oxide and europium spheres and replicas and microtomed sections of praseodymium and europium sol-gel beads were used to study the external and internal physical structure of the microspheres in relation to their hardness and density. Figure 8.3 shows the external surface of a europium sol-gel microsphere and the external surface of the microsphere after calcination to the oxide, Eu_2O_3.

PHOTO 83060



SCALE ⊢ = 600 Å

Fig. 8.1. Electron Micrographs of Praseodymium Sols, $Pr(OH)_3$, That Show the Effects of Aging. (*A*) freshly precipitated, aged 18 min; (*B*) precipitate washed, aged 80 min; (*C*) precipitate washed, aged 140 min; (*D*) precipitate washed, aged 6 days.

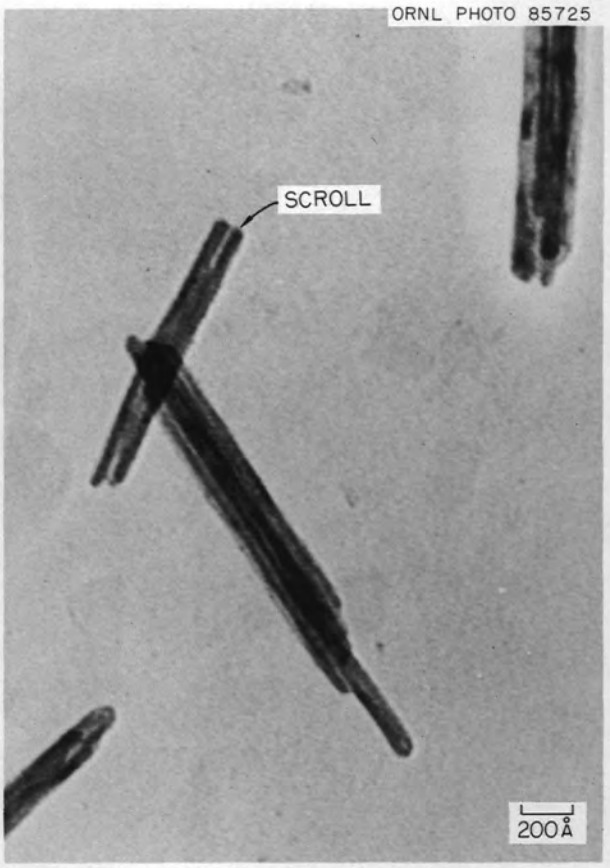


Fig. 8.2. Appearance of Crystals in Europium Sols, $Eu(OH)_3$, After Extended Aging. 658,000×. Reduced 50%.

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

In addition to the work on specific programs, research assistance was given to the Chemistry, Chemical Technology, Metals and Ceramics, Physics, Reactor Chemistry, Reactor, and Thermonuclear Divisions on research problems that required optical and electron microscopy and electron diffraction. Some of the materials studied were Al-Hg amalgams; graphite that contained UC_2 particles; ZnO aerosols; ThO_2; atomized and ball-milled aluminum powders; iron oxides; vaporized indium films; commercial carbon blacks; U_3O_8, UO_2, and stainless steel aerosols; vaporized tryptophane and tyrosine; chemically polished titanium; Al_2O_3; nickel heater wire; Teflon filters; glass capillaries; $Zr(OH)_4$; ion exchange resins; and Ni-Cr aerosols.

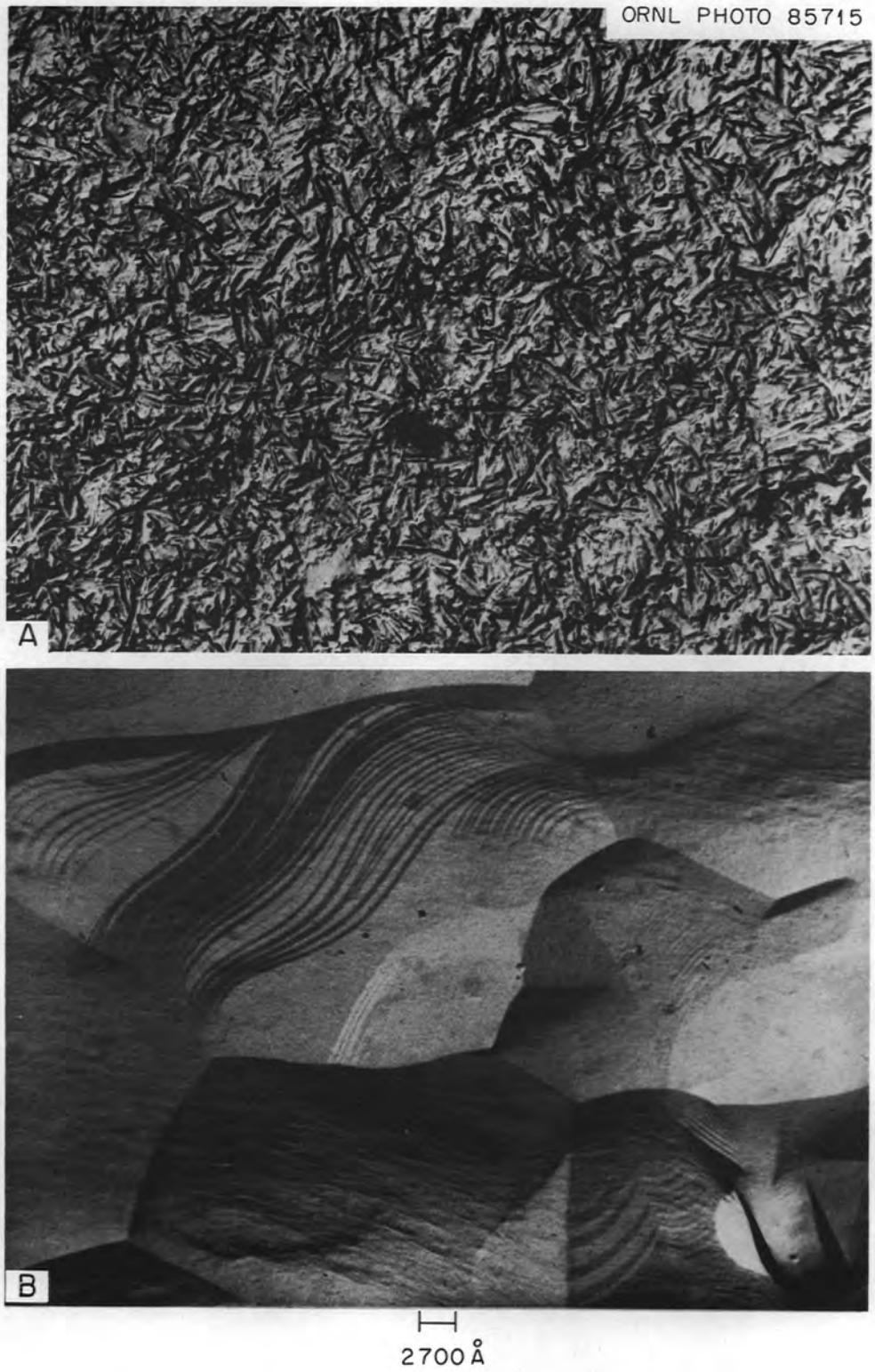


Fig. 8.3. External Surfaces of Europium Sol-Gel, $Eu(OH)_3$, Microsphere (*A*) and of Europium Oxide, Eu_2O_3, Microsphere (*B*).

9. Nuclear and Radiochemical Analyses

J. C. White W. S. Lyon H. H. Ross

9.1 NUCLEAR PROPERTIES OF RADIONUCLIDES

9.1.a Decay Scheme of ^{186}Re

W. S. Lyon J. F. Emery

The decay scheme of 90-hr ^{186}Re was investigated by irradiating enriched ^{185}Re to produce high-specific-activity ^{186}Re. The small amount of 17-hr ^{188}Re produced was allowed to decay for several days. Then, measurements were made by 4π beta-gamma coincidence counting and by NaI(Tl) and Ge(Li) gamma-ray spectrometry with four different solid-state detectors of size from 0.1 to 40 cc.

Since ^{186}Re decays both by K-electron capture and by beta emission followed by the emission of converted gamma rays, K x rays of both W and Os are produced. The high resolution of the 6-cc Ge(Li) detector is demonstrated in Fig. 9.1, which shows that both K_α and K_β groups from each element are clearly resolved. Figure 9.2 presents NaI(Tl) data for an ^{186}Re source over the same energy range; the improvement obtained by use of the solid-state detector is dramatic. In addition, gamma-ray photopeaks from the 123- and 137-kev gamma rays are shown.

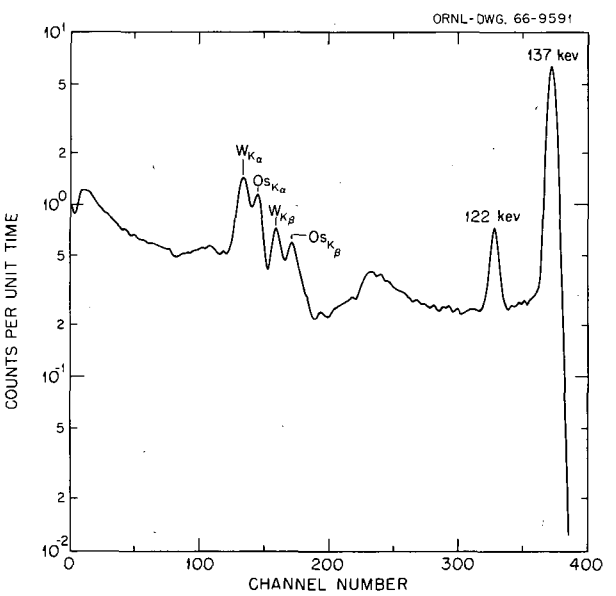


Fig. 9.1. Gamma-Ray Spectrum (0 to 150 kev) of ^{186}Re Obtained with a 6-cc Ge(Li)-Crystal Detector.

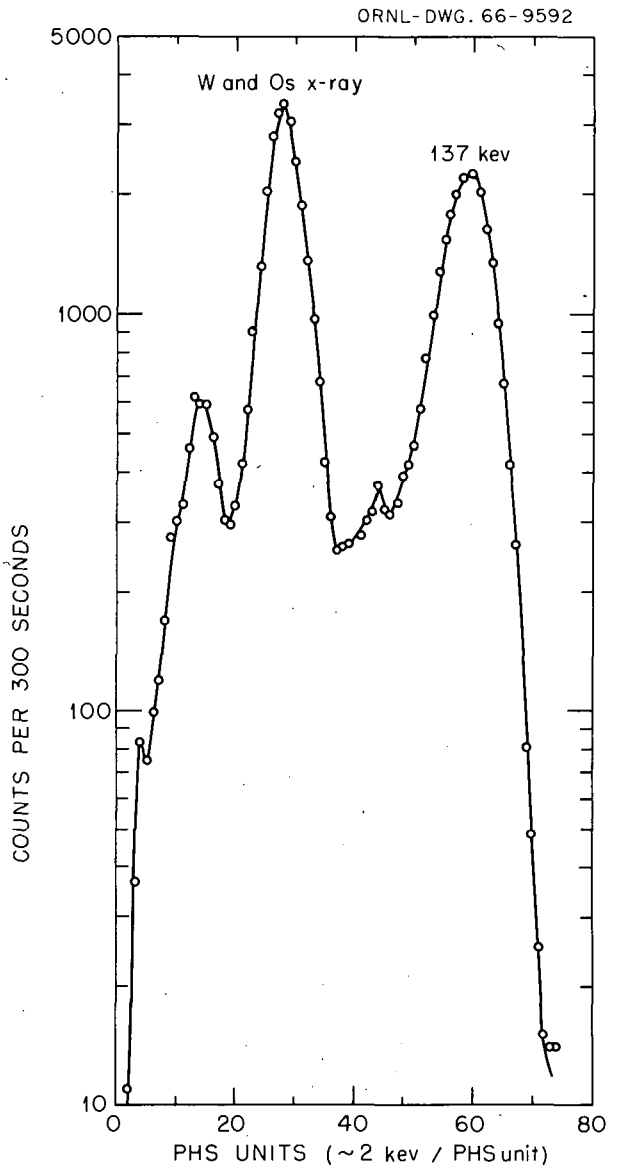


Fig. 9.2. Gamma-Ray Spectrum (0 to 150 kev) of ^{186}Re Obtained with a 3- by 3-in. NaI(Tl)-Crystal Gamma-Ray Spectrometer.

Two very weak gamma-ray transitions at 0.630 and 0.767 Mev exist. These are shown in Fig. 9.3, which was obtained from a 3000-sec count on the 6-cc Ge(Li) detector followed by subtraction of a 3000-sec background. The corresponding gamma-ray branchings are about 0.029 and 0.032%, respectively.

The decay scheme of ^{186}Re that is based on this work is given in Fig. 9.4; a paper on it is being prepared for publication.

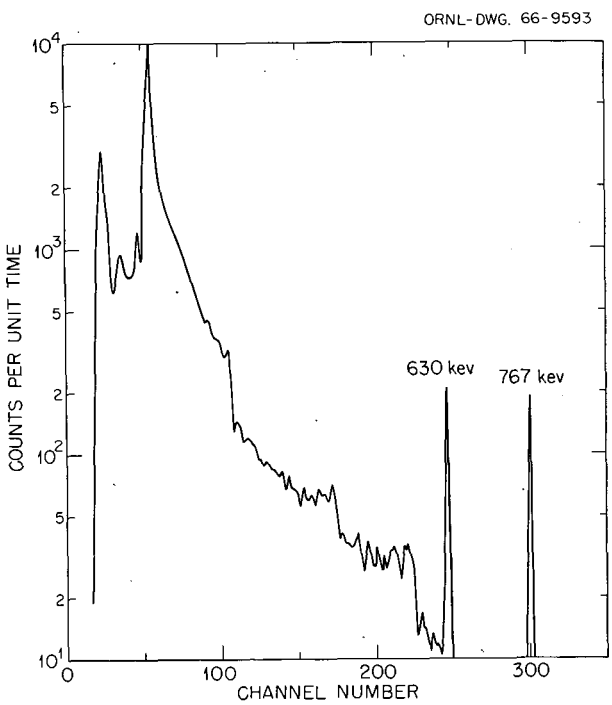


Fig. 9.3. Gamma-Ray Spectrum (0 to 800 kev) of ^{186}Re Obtained with a 6-cc Ge(Li)-Crystal Detector, Showing 0.630- and 0.767-Mev Gamma-Ray Photopeaks.

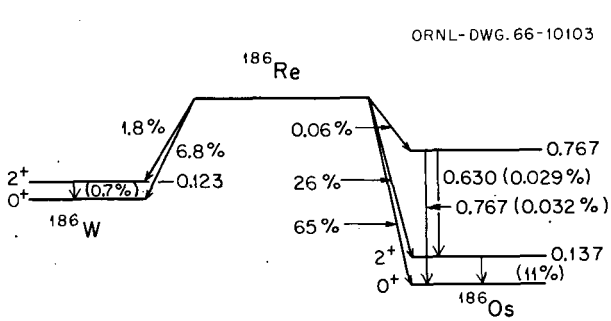


Fig. 9.4. Decay Scheme of ^{186}Re.

9.1.b Gamma Branchings and Fission Yield of ^{105}Ru

W. S. Lyon T. H. Handley
J. F. Emery

The nuclide 4.4-hr ^{105}Ru is one of several short-lived fission products used to measure the number of fissions in ^{235}U fuel burnup. After radiochemical separation of the Ru, one measures the ^{105}Ru radioactivity by NaI(Tl) gamma spectrometry. To determine ^{235}U fissions, a gamma branching of ~48% for the 726-kev gamma ray[1] and a fission yield of 0.9%[2] have been used. Because the number of fissions calculated from the ^{105}Ru is always about a factor of 2 higher than that calculated from other fission products, it seemed possible that the gamma branching value, ~48%, is in error. To check the gamma branching, a few milligrams of ^{104}Ru enriched to >99% was irradiated in the ORR for ~15 min. Ruthenium was separated chemically to isolate ^{105}Ru from possible contaminants, and aliquots of the resulting Ru solution were measured on both NaI(Tl) and Ge(Li) spectrometers. Figure 9.5 is a typical spectrum obtained with the 6-cc Ge(Li) crystal detector. Integration of the gamma-ray photopeaks and multiplication of the integrated area by the appropriate efficiency factor yielded the absolute gamma disintegration rate per aliquot. After all the 4.4-hr ^{105}Ru had decayed to the 36-hr ^{105}Rh daughter, aliquots of the solution were counted by 4π beta-gamma coincidence counting, and the absolute disintegration rate of ^{105}Rh was found. The original activity of the ^{105}Ru parent was then calculated, and the absolute gamma branchings per disintegration were obtained for ^{105}Ru; similar gamma measurements were made for ^{105}Rh. These values are given in Table 9.1. Several new gamma rays have been found, and the region from 660 to 800 kev was resolved. Instead of the previously used value for the 726-kev branching (~48%) being low, it appears to be too high; therefore, experiments are under way to determine the fission yield of ^{105}Ru.

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[1] K. Way, "Nuclear Data Sheets," Natl. Acad. Sci.–Natl. Res. Council, Washington, D.C.

[2] J. O. Blomeke and Mary F. Todd, *Uranium-235 Fission-Product Production as a Function of Thermal Neutron Flux, Irradiation Time, and Decay Time*, ORNL-2127, part 1, vol. 1, p. 30 (Aug. 19, 1957).

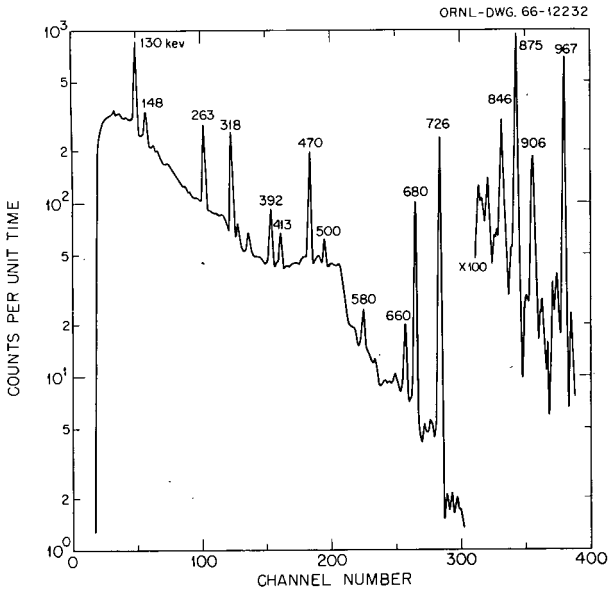

Fig. 9.5. Gamma-Ray Spectrum of ^{105}Ru Obtained with a Ge(Li)-Crystal Gamma-Ray Spectrometer.

9.1.c Gamma-Ray Branching Ratios for ^{116}In, ^{141}Ce, and ^{147}Nd

J. S. Eldridge Peter Crowther[3]
W. S. Lyon

For radiochemical isotopic analysis and absolute radioactivity determinations by gamma-ray spectrometry, one must know the gamma-ray branching ratios of the nuclides. Gamma-ray branchings in ^{116}In, ^{141}Ce, and ^{147}Nd have been measured, and a paper on this subject has been published.[4]

9.1.d Nuclear Properties of Radionuclides

H. B. Hupf[5] J. S. Eldridge
J. E. Beaver[5]

^{123}I. – Two additional bombardments of ^{123}Te were made to study a method of producing this nuclide; yields of ^{123}I and of other iodine isotopes that accompany the production were very similar to those of previous bombardments.[6] Experimental work on this project was completed and has been

Table 9.1. Gamma-Ray Branchings in ^{105}Ru and ^{105}Rh Obtained with a 6-cc Ge(Li) Crystal

$E\gamma$ (kev)	Number of Gammas per Disintegration[a]
	^{105}Ru
130	0.053 ± 0.005
148	0.020 ± 0.004
262	0.051 ± 0.005
316	0.084 ± 0.004
350	0.016 ± 0.004
394	0.038 ± 0.005
414	0.023 ± 0.005
471	0.158 ± 0.010
490	0.021 ± 0.004
501	0.029 ± 0.005
580	0.011 ± 0.003
660	0.020 ± 0.022
680	0.123 ± 0.006
726	0.368 ± 0.010
846	0.0060 ± 0.0015
875	0.0250 ± 0.001
906	0.004 ± 0.002
967	0.017 ± 0.001
1018	0.0028 ± 0.0006
1252	0.00024 ± 0.00008
1322	0.0015 ± 0.0001
1378	0.0004 ± 0.0001
	^{105}Rh
213	0.0040
280	0.0041
306	0.0629
319	0.225
[306 + 319 (NaI)]	(0.24)
443	0.00066
498	0.0011

[a]For ^{105}Ru, average of three values.

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

[4]J. S. Eldridge, P. Crowther, and W. S. Lyon, "Gamma-Ray Branching Ratios for In^{116}, Nd^{147}, and Ce^{141}," *Nucleonics* 24(3), 62 (1966).

[5]Isotopes Division.

[6]J. S. Eldridge, "^{123}I," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1965*, ORNL-3889, pp. 48–49.

described in an oral presentation.[7] A paper on the production and properties of ^{123}I will be submitted for publication.

9.1.e Thermal-Neutron Cross Sections and Resonance Integrals of ^{139}La and ^{140}La

H. A. O'Brien, Jr.[5] J. S. Eldridge
R. E. Druschel[8] J. Halperin[8]

The thermal-neutron activation cross sections and resonance integrals of ^{139}La and ^{140}La were measured by use of a cadmium-filter technique. By the irradiation of samples of ^{139}La in a nuclear reactor and the determination of the ^{140}La and ^{141}Ce produced, a thermal cross section of 2.7 ± 0.3 barns and a resonance integral of 69 ± 4 barns were measured for ^{140}La. Similarly, the values 9.2_5 ± 0.5 barns for the 2200-m/sec cross section and 11.2 ± 0.6 barns for the resonance integral of ^{139}La were measured.

A paper that describes this work was submitted for publication.[9]

9.1.f Reactor Production of ^{141}Ce from Lanthanum and Praseodymium

H. A. O'Brien, Jr[5] J. S. Eldridge

Experiments were concluded that showed the feasibility of producing ^{141}Ce free of the contaminants ^{139}Ce and ^{144}Ce by the neutron irradiation of lanthanum and praseodymium targets. The production of ^{141}Ce from ^{139}La was shown to depend mainly on the resonance neutron flux. The half-life of ^{141}Ce was measured to be 32.38 ± 0.02 days.

An abstract of this work was published.[10] A paper that describes the complete experimental details will be published.[11]

9.1.g Production of 70-day ^{188}W and Development of a 17-hr ^{188}Re Radioisotope Generator

R. E. Lewis[5] J. S. Eldridge

The parameters for the production of 70-day ^{188}W by double neutron capture in ^{186}W targets and the development of an ^{188}Re generator were investigated. Half-life measurements and gamma-ray branchings are being studied to develop suitable radioassay procedures for the generator system.

To produce ^{188}W, targets of enriched ^{186}W were irradiated in several reactor positions. The production rate of ^{188}W was greater in reactor positions having a high epithermal neutron flux than in positions with a well-thermalized neutron flux. Production of hundred-millicurie quantities of ^{188}W was demonstrated by irradiating ^{186}W in neutron fluxes $>1 \times 10^{15}$ neutrons cm^{-2} sec^{-1}.

An ^{188}Re generator was prepared by sorbing ^{188}W as tungstate on the chloride form of Bio-Rad HZO-1, a hydrous zirconium oxide ion exchanger. The ^{188}Re, as perrhenate, was eluted from the column by methyl ethyl ketone (95 v/v%)–0.01 *M* HCl (5 v/v%); the yield of ^{188}Re was 85 to 90%. The ^{188}W contamination of the ^{188}Re product is $\sim 1 \times 10^{-4}$%. The long half-life of ^{188}W is a distinct advantage over that of some other generators, such as the 67-hr ^{99}Mo-^{99m}Tc.

Results of these investigations will be presented orally[12] and will be published.

9.1.h Nuclear Spectroscopy of Neutron-Deficient Radionuclides

T. H. Handley B. Harmatz[13]

Radionuclides of Hafnium and of the Rare-Earth Elements. – A spectrographic survey was made

[7] H. B. Hupf, J. S. Eldridge, and J. E. Beaver, "Production and Characterization of Iodine-123 for Medical Investigators," presented at Central Chapter, Society of Nuclear Medicine, Iowa City, Nov. 7, 1965.

[8] Chemistry Division.

[9] H. A. O'Brien, Jr., J. S. Eldridge, R. E. Druschel, and J. Halperin, "The Thermal Neutron Cross Section and Resonance Integrals of ^{139}La and ^{140}La," submitted for publication in the *Journal of Inorganic and Nuclear Chemistry.*

[10] H. A. O'Brien, Jr., and J. S. Eldridge, "Reactor Production of Carrier-Free Cerium-141 from Lanthanum and Praseodymium," *Trans. Am. Nucl. Soc.* 9(1), 101 (1966).

[11] H. A. O'Brien, Jr., and J. S. Eldridge, "Reactor Production of Cerium-141 from Lanthanum and Praseodymium," submitted for publication in *Nucleonics.*

[12] R. E. Lewis and J. S. Eldridge, "Production of 70-day Tungsten-188 and Development of a 17-hour Rhenium-188 Radioisotope Generator," to be presented at Annual Meeting of the Southeastern Chapter, Society of Nuclear Medicine, Durham, N.C., Nov. 3–5, 1966.

[13] Electronuclear Division.

of transition conversions that occur in lutetium isotopes of mass in the interval $168 \leqq A \leqq 175$. Partial decay schemes were deduced from energy sums, multipolarities, and intensities of the transitions. The proposed excited states are described by use of the Mottelson and Nilsson predictions. The amount of experimental information appears to be sufficient to justify an analysis for systematic properties of intrinsic states and of the associated rotational bands. Previous assignments in related nuclei are incorporated in the energy-level systematics. More complete conversion-electron measurements permit extension of nuclear-level structures of ^{149}Eu, ^{151}Eu, ^{157}Gd, and ^{173}Yb. For the decay of ^{133}La → ^{133}Ba, an energy-level diagram is proposed that accounts for most of the radiation intensity. A paper on this subject was published.[14]

Radionuclides of Osmium and of Rhenium. – Osmium radionuclides in the mass interval $180 \leqq A \leqq 185$ were produced in the ORIC by the (α, xn) reaction on enriched tungsten isotopes. The osmium radionuclides are separated from the tungsten target by distillation and are plated onto 10-mil platinum wires for studies with conversion-electron spectrographs. Partial decay schemes are deduced from energy sums, multipolarities, and intensities of the transitions. This work is a continuation of the systematic studies of neutron-deficient radionuclides.

9.1.i Alpha-Particle and Gamma-Ray Spectral Studies of ^{149}Gd and ^{149}Eu

T. H. Handley I. R. Williams[8]
K. S. Toth[13]

The alpha-particle and photon spectra of 9.5-day ^{149}Gd and the photon spectrum of its daughter, 106-day ^{149}Eu, were studied with an Si(Au) surface-barrier detector and with Ge(Li) and NaI(Tl) gamma-ray spectrometers. Alpha particles from ^{149}Gd were found to have an energy of 3.01 ± 0.01 Mev; the alpha-to-electron-conversion (α/E.C.) branching ratio was found to be $(4.0 \pm 1.2) \times 10^{-6}$. The transition energies, in kev, and the relative intensities (in parentheses) of gamma rays from ^{149}Gd were found to be: 150 (240), 216 (0.8), 253 (1.3), 261 (4.2), 273 (12.5), 299 (118), 347 (100), 461 (2.5), 497 (6.3), 517 (10.5), 535 (12.4), 646 (4.5), 666 (3.6), 750 (30), 790 (27), 812 (0.4), 934 + 939 (11.5), and 949 (3.2). For ^{149}Eu, the measured photon energies, in kev, and relative intensities were: 255 (12), 277 (100), 328 (118), 350 (9), 506 (14), and 529 (13); *K*-shell internal-conversion coefficients were calculated by intercomparison of the photon-intensity data with the conversion-electron-intensity data reported by other workers. The results of this work have been published.[15]

9.1.j Half-Life Measurements

S. A. Reynolds E. I. Wyatt

The measurement[16–18] has continued of the half-lives of radionuclides of importance in the programs of the Isotopes and Chemical Technology Divisions. The decay of beta or gamma radioactivity of particular nuclides is followed by means of an appropriate counter or ionization chamber. The decay of four sources of ^{137}Cs has been followed for seven years; the current tentative value for the half-life of ^{137}Cs is 30.12 ± 0.21 years, which supersedes 30.2 ± 0.3 years, the value reported last year.[16] (Uncertainties are standard deviations.) Figure 9.6 shows the variation of the apparent half-life as a function of the duration of measurement. This plot illustrates how it would be possible to report an erroneous value, for example, 28.8 ± 0.6 at 3.5 years, by not realizing the existence of a systematic error that is much larger than the apparent precision. Of value in avoiding mistakes are such plots as this one, comparison of a new value with previous values carefully determined, and use of sophisticated computer programs. Half-life values reported previously[17,18] have been critically reviewed. Analysis of the earlier data for ^{147}Pm,

[14] B. Harmatz and T. H. Handley, "Nuclear Spectroscopy of Neutron-Deficient Hafnium and Rare Earth Activities," *Nucl. Phys.* **81**, 481 (1966).

[15] I. R. Williams, K. S. Toth, and T. H. Handley, "Alpha-Particle and Gamma-Ray Spectral Studies of ^{149}Gd and ^{149}Eu," *Nucl. Phys.* **84**, 609 (1966).

[16] S. A. Reynolds, "Half-Life Measurements," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1965,* ORNL-3889, pp. 57–58.

[17] H. W. Wright, E. I. Wyatt, S. A. Reynolds, W. S. Lyon, and T. H. Handley, "Half-Lives of Radionuclides – I," *Nucl. Sci. Eng.* **2**(4), 427 (1957).

[18] E. I. Wyatt, S. A. Reynolds, T. H. Handley, W. S. Lyon, and H. A. Parker, "Half-Lives of Radionuclides – II," *Nucl. Sci. Eng.* **11**(1), 74 (1961).

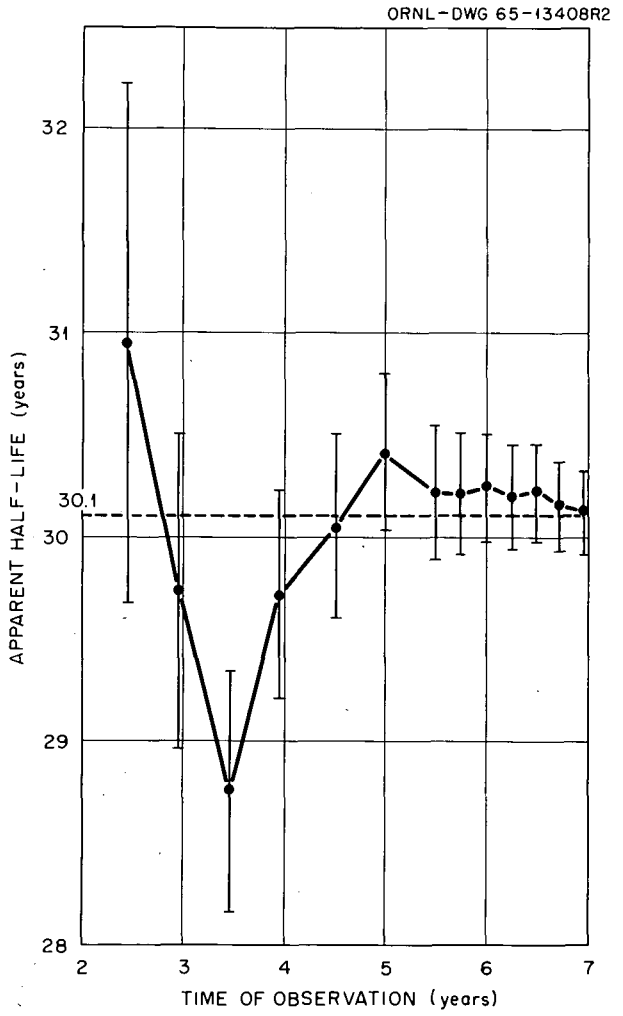

Fig. 9.6. Variation of Apparent Half-Life of ^{137}Cs with Duration of Observation.

which indicated a half-life of 2.50 years,[18] by weighted and unweighted least-squares computer programs showed the probable presence of a contaminant of half-life 200 to 400 days. Reanalysis of the data on this assumption gave a value for the half-life of ^{147}Pm of 2.62 ± 0.01 years. Newly determined half-life values for other radionuclides include: ^{144}Ce, 285.4 ± 0.9 days; ^{131}I, 8.070 ± 0.009 days; ^{47}Ca, 4.530 ± 0.004 days; and ^{67}Cu, 61.88 ± 0.14 hr. These and other findings are reported in a paper submitted for publication.[19]

[19]J. F. Emery, S. A. Reynolds, and E. I. Wyatt, "Half-Lives of Radionuclides — III," submitted for publication in *Nuclear Science and Engineering*.

9.2. COOPERATIVE ISOTOPES PROGRAM

9.2.a Technical Assistance

S. A. Reynolds

The joint Isotopes Division–Analytical Chemistry Division program[20] to characterize and analyze isotopes has continued. Technical assistance has included answering local and outside inquiries, liaison with respect to analytical methods and practices (and occasional participation in developing such methods), measurements of gamma and neutron flux, preparation of articles,[21,22] reviewing of papers and reports, and other activities, some of which are described in the following sections.

9.2.b Quality Control

S. A. Reynolds

The rather general topic of quality control encompasses analytical methods for assay and purity (chemical and radiochemical) of isotope products, as well as computations of data such as specific activities of fission products. Radioactivity standards are frequently purchased from the National Bureau of Standards; the Radiochemical Centre at Amersham, England; the IAEA; and commercial suppliers located in the United States. These standards serve a triple purpose: to confirm calibrations made locally, to evaluate the quality of the radioactivity standards themselves, and to determine the characteristics of the standards with which the users of isotopes calibrate their equipment. It is sometimes necessary to carry on interlaboratory checks to improve measurements by producers and users; the work on ^{197}Hg is a good example (see Sect. 9.3.a). All seemingly valid complaints about assay or purity are investigated

[20]S. A. Reynolds, "Reviews and Technical Assistance," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1965*, ORNL-3889, p. 51.

[21]W. S. Lyon, S. A. Reynolds, and E. I. Wyatt, "Methods for Assay of Radioisotopes," *Nucleonics* **24**(8), 116 (1966).

[22]S. A. Reynolds, "Radioisotope Characteristics, Measurements, and Standards," *Isotopes Radiation Technol.* **4**(1) (in press).

and usually are found to be unjustified, since often they result from errors made by the users or from failures to consider the specifications listed in the latest *Isotopes Catalog*. When impurities interfere with the use of a product even though the product is within specifications, investigation is made, and the product is improved, if possible. Thus, radionuclides induced in quartz of two qualities used for ampuls to contain target materials during irradiation were identified and measured; the use of the quartz of poorer quality was discontinued. Other quality-control work is described in the section "^{14}C Products," just below.

9.2.c Properties of Radioisotopes

S. A. Reynolds

In analyzing radioisotopes, in assessing their usefulness for particular applications, and in planning their production and handling, it is necessary to know such properties as half-lives and the types, energies, and intensities of the emitted radiations. Much of this information can be obtained from the literature, and useful compilations and recommendations of selected values are available, notably from the ORNL Nuclear Data Group. The problem of making selections in cases when no adopted or "standard" value is available has been discussed.[22] A revision[23] of a table of selected half-lives and computed specific radioactivities of 354 well-known radionuclides was prepared. Sometimes the need arises for experimental determination of properties, particularly half-lives (Sect. 9.2.a) and gamma percentages. Examples of these needs are afforded by recent work on gamma percentages for ^{147}Nd and ^{141}Ce,[4] on the finding of 13.8% 0.3-Mev gamma emission from ^{194}Ir,[24] and on the determination of the reactor-neutron capture cross section of ^{46}Ca as 0.77 barn based on typical ^{47}Ca-production runs.[25]

[23]G. Goldstein and S. A. Reynolds, "Specific Activities and Half-Lives of Common Radionuclides," *Nuclear Data* A1, 435 (1966).

[24]J. F. Emery, unpublished data, 1966.

[25]S. A. Reynolds, unpublished data, 1966.

9.2.d ^{14}C Products

S. A. Reynolds

Carbon-14 is available from ORNL as the product $Ba^{14}CO_3$. Various chemical and radiochemical tests have led to the following conclusions about the quality of the product. The results of mass spectrometric and radiochemical measurements of the abundance of ^{14}C generally agree very well. Contaminant radioactivity is not greater than 0.001%. Spectrographic, flame spectrophotometric, and x-ray examinations indicated no significant amounts of nonradioactive impurities. At least 99.999% of the ^{14}C is volatile from dilute HCl at room temperature, presumably as $^{14}CO_2$.

An allegation by a user that the ORNL product was not usable for preparing ^{14}C-labeled propionic acid led to the performance of such a preparation by H. L. Holsopple[26] (Sect. 11.1.e). The purity of the propionic-^{14}C acid so prepared was confirmed by adding propionic acid carrier to an aliquot of an aqueous solution of the ^{14}C-labeled acid and determining, by ^{14}C measurement and by titration with base, the percentage of ether-extractable material. The results obtained by means of the two techniques agreed within experimental error. Further, diethyl ketone carrier was added to a neutralized aliquot; repeated ether extraction indicated that $<0.1\%$ of the ^{14}C was in the form of diethyl ketone or triethyl carbinol, possible byproducts. V. F. Raaen[8] reported the successful synthesis of ^{14}C-labeled *p*-toluic acid by a similar procedure.

9.2.e ^{131}I Products

S. A. Reynolds J. F. Emery

The concentrations of ^{127}I and ^{129}I in an aged ^{131}I product were determined. Iodine-131 remained in microcurie amount, which is sufficient to serve as tracer for iodine. The ratio of ^{127}I to ^{129}I was determined by mass spectrometry, and the concentration of ^{129}I was measured by neutron activation. In Table 9.2, analytical results are compared with values calculated from fission yields. The specific radioactivity of ^{131}I as a function of decay time is given below:

[26]Methods Development Group.

Decay Time (weeks)	Specific Radioactivity[a] (curies/g)
	$\times 10^4$
0[b]	8.7
1	6.8
2	4.8

[a]Specific radioactivity of pure ^{131}I, 12.4×10^4 curies/g.

[b]Not feasible (^{133}I, etc.).

Table 9.2. Results of Analysis of an ^{131}I Product

Nuclide	Calculated	Found
^{127}I, μg per curie of ^{131}I	1.1	1.7
^{129}I, μg per curie of ^{131}I	6.2	4.4
^{131}I, at. %	52	56
curie per gram of total I	6.5×10^4	7.0×10^4

Details are given in a paper submitted for publication.[27] In the course of this work the isomer 9-min ^{130m}I (ref. 28) was discovered independently.[29]

9.2.f ^3H-Labeled Luminous Paints

T. H. Handley R. G. Niemeyer[5]

The widespread use of tritiated luminous paints has raised the question as to whether or not ^3H is released by the paints. Any released ^3H may be a health hazard under certain environmental conditions. Samples of commercially supplied tritiated organic polymers used in luminous paints were tested for release of ^3H. During the drying period following application of the paint, release of ^3H occurred and decreased only slightly with time. Loss of ^3H by the dried paint was roughly exponential with respect to temperature for certain paints; for others, the loss was stepwise. The ^3H was released chiefly as ^3H_2O. A long-range study of ^3H release by aged dried paints is in progress. A paper on this subject has been written for publication.

9.3 MEASUREMENT OF RADIOACTIVITY

9.3.a Standardization of ^{197}Hg

J. S. Eldridge

Standardization problems associated with the medically important nuclide ^{197}Hg were investigated. An interlaboratory comparison of the techniques for standardizing ^{197}Hg was conducted with two ^{197}Hg preparations, one made from enriched ^{196}Hg and the other from natural mercury. Participants included standardization laboratories and pharmaceutical suppliers in the United States and Canada.

Gamma-ray spectra of ^{197}Hg products made from natural mercury and from several enrichments of ^{196}Hg up to 40% were studied in relation to assay problems. Lithium-drifted germanium detectors were compared with sodium iodide detectors as possible assay devices for this nuclide.

Samples of ^{197}Hg were produced by three methods to obtain pure products for half-life studies. Gold foils were bombarded with alpha particles to produce 2.8-hr ^{197}Tl by the reaction $^{197}Au(\alpha,4n)^{197}Tl$. Subsequent decay of the ^{197}Tl yielded a carrier-free preparation of the ground-state isomer, ^{197}Hg. Preparations rich in the 24-hr isomer, ^{197m}Hg, were made by the reaction $^{197}Au(p,n)^{197}Hg$. The third production method involved the neutron irradiation of mercury targets in the neutron flux of a reactor.

A single-crystal sum-coincidence method based on K x-ray–77-kev gamma-ray coincidences was evaluated as an absolute standardization technique and was compared with a method based on absolute gamma-ray spectrometry.

A paper that described this work was presented at the IAEA "Symposium on the Standardization of Radionuclides";[30] it will be published in the proceedings of the symposium.

[27]S. A. Reynolds and F. N. Case, "Isotopic Abundance of Iodine-131 in Fission-Produced Iodine," submitted for publication in the *International Journal of Applied Radiation and Isotopes*.

[28]D. D. Wilkey and J. E. Willard, "Isomer of ^{130}I," *J. Chem. Phys.* **44**, 970 (1966).

[29]C. E. Bemis, Jr., J. F. Emery, and N. K. Aras, "The Decay of 12.8-hr Iodine-130*g* and 8.8-min Iodine-130," *Chem. Div. Ann. Progr. Rept. May 20, 1966*, ORNL-3994, p. 17.

[30]J. S. Eldridge, "Standardization of Mercury-197," Paper SM-79/19 presented at the IAEA Symposium on Standardization of Radionuclides, Vienna, Oct. 10–14, 1966.

9.3.b Methods for Assay of Radioisotopes

W. S. Lyon S. A. Reynolds
E. I. Wyatt

Methods in use within the Analytical Chemistry Division for assay of most radioisotopes either presently or formerly produced were discussed in a published article.[21] A tabulation of the methods used for each radioisotope was also included.

9.3.c Solid-State Gamma-Ray Detectors

J. F. Emery W. S. Lyon

A lithium-drifted germanium, Ge(Li), gamma-ray detector system was assembled to study the usefulness of this type of detector in activation analysis. The system consists of a 40-cc-active-volume Ge(Li) detector (purchased from Princeton Gamma Tech, Princeton, New Jersey), a Tennelec model TC-130 low-noise preamplifier having four specially selected field-effect transistors (FET's) for high-capacitance detectors, a Tennelec model TC-200 amplifier, and a Tennelec model TC-250 biased amplifier.

This combination of electronic components and detector should give a system that has high resolution and is drift free for long periods of time. However, adsorption of water vapor on the cable connector at the detector causes a gradual increase in the leakage current and correspondingly poor resolution. The system can be returned to normal simply by evaporating the moisture from the connector in a vacuum. For this operation the bias voltage applied to the detector is turned off, and the detector is disconnected. Large changes in the bias voltage will probably damage the FET's in the preamplifier.

Gamma-ray detectors having high efficiency are required for ultimate sensitivity in activation analysis. In Fig. 9.7, the photopeak efficiencies at 9.3-cm geometry vs gamma-ray energies of three Ge(Li) detectors – a 6-cc planar, a 6-cc coaxial, and a 40-cc coaxial – are compared with the same type of data taken with the usual 3- by 3-in. NaI(Tl) detector used in activation analysis. Because of the relatively thick (20-mil) aluminum can that covered these detectors and an apparent dead layer (possibly 1 mm) of germanium at the front of the detector, the efficiency of these detectors decreases sharply at low energies. Figure 9.8 shows the photopeak efficiency for a 6-cc Ge(Li) detector in the 60- to 170-kev region. The sharp decrease in efficiency between 60 and 80 kev is very unsatisfactory. Experiments are planned to determine the cause of this decrease and, if possible, to reduce it.

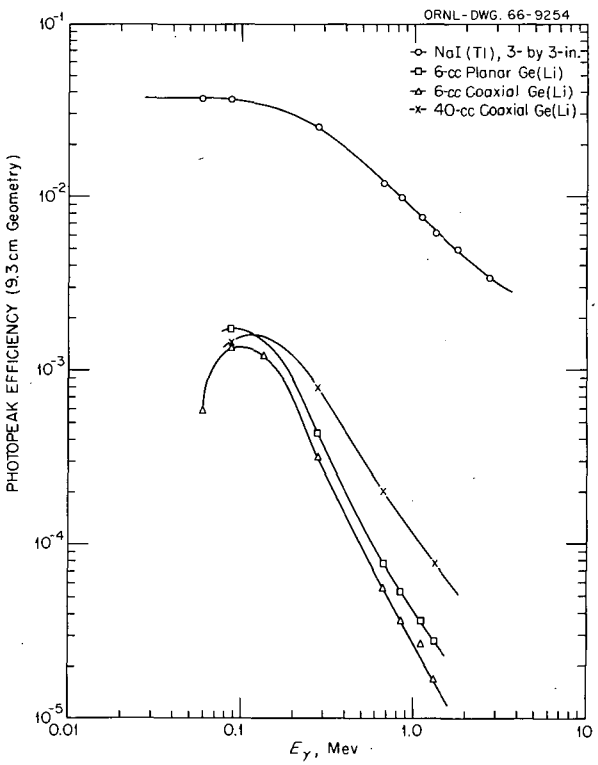

Fig. 9.7. Photopeak Efficiency at 9.3-cm Geometry vs Gamma-Ray Energy of Ge(Li) Detectors of Various Sizes and of a 3- by 3-in. NaI(Tl) Detector.

Figure 9.9 shows spectra of ^{203}Hg obtained with two Ge(Li) detectors, each adjusted for best resolution; for comparison, a spectrum of ^{203}Hg obtained with a very good NaI(Tl) detector is also included. In Fig. 9.10, spectra of ^{60}Co obtained at 9.3-cm geometry are compared with a spectrum of ^{60}Co determined with an NaI(Tl) detector.

9.3.d Alpha Spectrometry

T. H. Handley

Studies are in progress to determine optimum conditions for operating the solid-state silicon

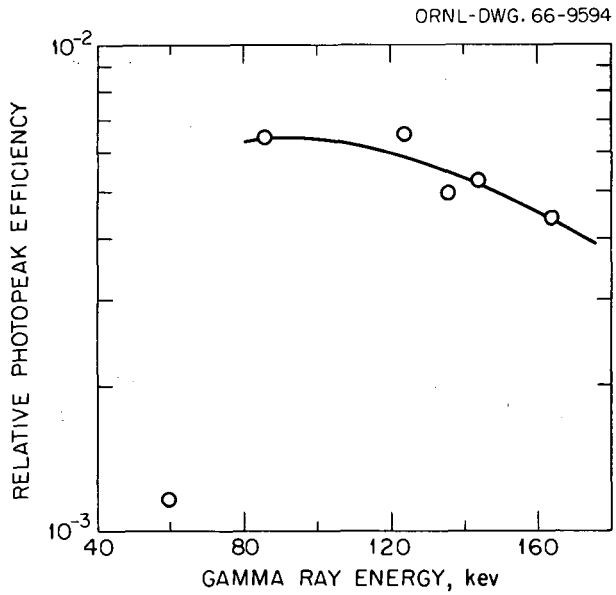

Fig. 9.8. Photopeak Efficiency at ~1-cm Geometry for a 6-cc Ge(Li) Detector.

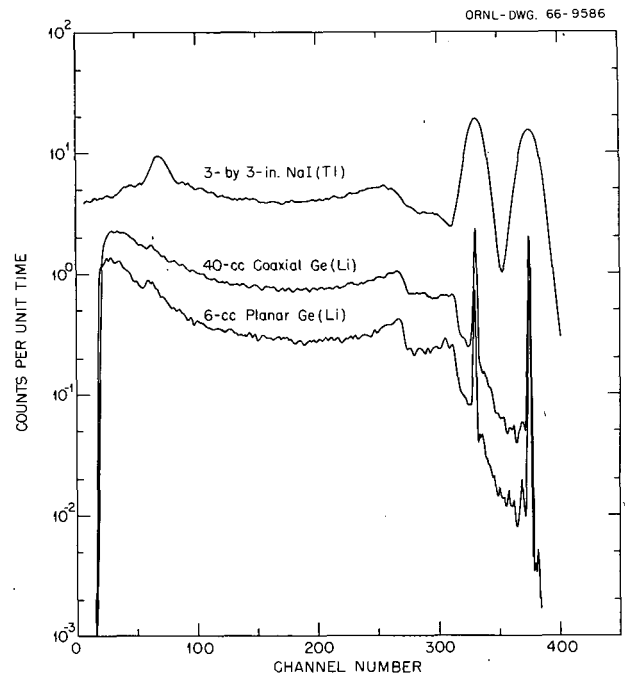

Fig. 9.10. Gamma-Ray Spectra of ^{60}Co Obtained with Various Detectors.

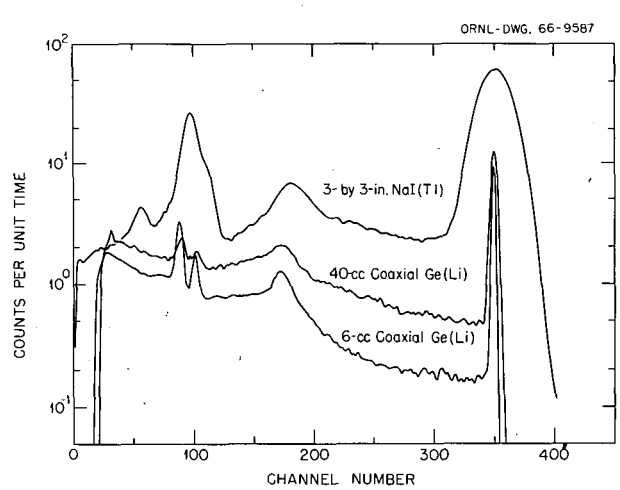

Fig. 9.9. Gamma-Ray Spectra of ^{203}Hg Obtained with Various Detectors.

alpha spectrometers now in use in the analytical service laboratories. Improvement of resolution and reduction of low-energy scatter are desired. It has been determined that a collimator slightly smaller than the detector and placed next to the detector always improves resolution and decreases scatter. For a given source, increase in geometry almost always improves resolution and reduces low-energy scatter. However, because the count rate is reduced, longer counting times or more radioactive sources are required to attain equivalent statistics. Electrodeposited sources always give better resolution and less low-energy scatter than evaporated sources.

9.3.e Thick-Target Yields from Reactions of ^{3}He Particles with Boron, Nitrogen, and Sodium

R. L. Hahn Enzo Ricci

To extend our investigation of the reactions of ^{3}He particles with low-Z elements,[31] thick

[31] R. L. Hahn and E. Ricci, "Interactions of ^{3}He Particles with ^{9}Be, ^{12}C, ^{16}O, and ^{19}F," *Phys. Rev.* **146**(3), 650 (1966).

targets (i.e., targets thicker than the range of the ^{3}He ions) of boron, nitrogen (as Be_3N_2), and sodium (as NaI) were irradiated at the ORNL tandem Van de Graaff accelerator with ^{3}He particles of energies in the range 6 to 18 Mev. The absolute disintegration rates of the different nuclides produced in the irradiations were determined by gamma-ray spectrometry, and the resulting reaction yields, expressed in disintegrations per minute per number of incident ^{3}He ions per minute, were then plotted vs ^{3}He-particle energy. As an example, the yield curves for the reactions of ^{3}He particles with boron are shown in Fig. 9.11. Values for reaction cross sections were determined from these curves with the aid of the computer program THIKSIG (Sect. 9.5.d).

Analysis of the observed (^{3}He,d) reactions on ^{10}B and ^{14}N and the (^{3}He,α) reaction on ^{14}N is being performed with the distorted-wave theory of direct nuclear reactions.[32] Preliminary calculations indicate that the direct-interaction mechanism is successful in accounting for the reaction cross sections observed in this work.

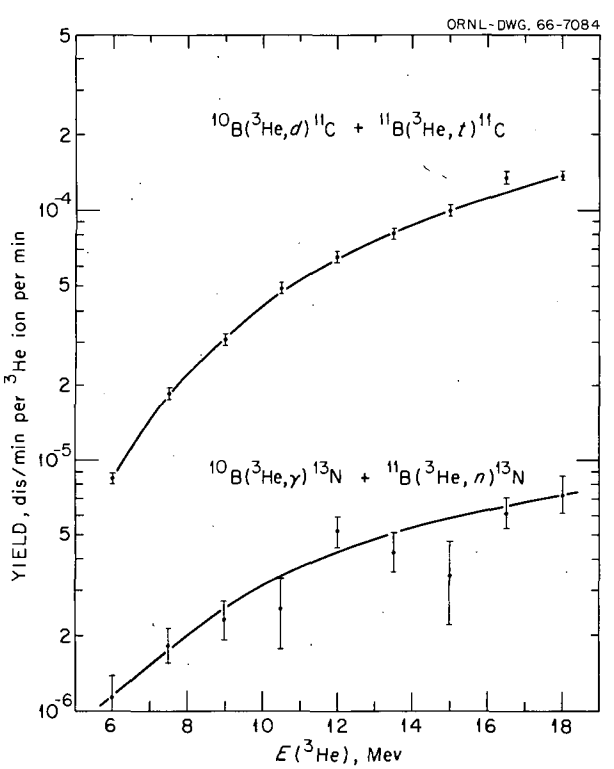

Fig. 9.11. Yields for the Reactions of ^{3}He Particles with ^{10}B and ^{11}B.

9.3.f Angular Distributions of Recoil Nuclei from the ^{12}C(^{3}He,α)^{11}C and ^{12}C(^{3}He,d)^{13}N Reactions

R. L. Hahn

Comparison of excitation functions calculated from the distorted-wave theory of direct nuclear reactions[32] with measured values for the reactions of ^{3}He particles with several low-Z elements has led to the conclusion that the observed (^{3}He,α) and (^{3}He,d) reactions[31] proceed, to a large extent, by way of direct processes (Sects. 9.3.e and 9.7.b). However, to make this comparison, it is necessary to integrate the calculated angular distributions (variation of cross section with angle) for the various nuclear states of interest so as to obtain excitation functions that are similar to those measured. The calculated angular distributions exhibit considerable structure; several maxima and minima occur in the angular interval from 0 to 180°. Contrastingly, the measured excitation functions show little or no structure; therefore, in the comparison of the theoretical and experimental excitation functions, many of the fine details of the calculated angular distributions are lost in the integration step that leads to the calculated excitation functions.

To see some of this detail in our experiments, we have begun a series of measurements of the ^{12}C(^{3}He,α)^{11}C and ^{12}C(^{3}He,d)^{13}N reactions with 25-Mev ^{3}He ions at the Oak Ridge Isochronous Cyclotron. By use of extremely thin (<40 μg/cm^2) ^{12}C targets, we have been able to ensure that essentially all the ^{11}C and ^{13}N nuclei produced in these reactions recoil out of the target foil. The recoiling nuclei are stopped in Al catchers placed at known angles to the target.

From the experiments performed to date, it is clear that there are several peaks in the recoil angular distribution. When the experiments are completed, the data will be analyzed in detail in terms of the direct-reaction theory.

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

9.4 RADIOCHEMICAL STUDIES

9.4.a New Separation Method for Americium Based on the Solvent-Extraction Behavior of Americium(V)

J. R. Stokely F. L. Moore

Pertinent experimental variables for the oxidation and stabilization of Am(V) have been examined and optimized to provide the first highly selective separation method for americium based on solvent extraction. In this new method Am(III) is oxidized to Am(VI) with ammonium persulfate in dilute nitric acid. The resulting solution is cooled in ice, the pH is adjusted to between 4.8 and 5.2 with an acetate buffer, and potential interferences such as the actinide and lanthanide elements are extracted into a 0.5 *M* solution of 2-thenoyltrifluoroacetone in xylene. During the extraction, Am(VI) is probably reduced to Am(V), which does not extract. Of the original amounts present, over 95% of the Am and less than 0.5% of the Cm, Eu, or U remain in the aqueous phase; thus, separation factors greater than 10^4 are provided for some important interferences. The limiting interference is Pu, which apparently behaves much like Am in this system. The interference due to Pu is readily eliminated by a preliminary lanthanum fluoride precipitation. More information on this procedure and other pertinent findings of this investigation will be reported in detail in papers to be published in the near future.

9.4.b Evaluation of Methods for the Determination of Americium

J. R. Stokely

The radiochemical determination of ^{241}Am or ^{243}Am in process and research samples is often complicated by the presence of relatively large amounts of ^{242}Cm and ^{244}Cm. When the ratio of Cm to Am alpha radioactivity is greater than about 10^3, direct measurement by alpha spectrometry is not applicable, and a chemical separation is necessary before Am can be determined accurately. The coprecipitation method of Moore[33] has been used extensively at ORNL to eliminate the interference of Cm. Recently, other methods have been proposed for the determination of Am; it was the purpose of this work to evaluate these new methods.

The calcium fluoride column technique described by Holcomb[34] was tested on a series of samples in which the alpha radioactivity from ^{241}Am amounted to 0.01 to 10% of that from ^{244}Cm. The separation of Am from Cm by this method was highly efficient; the Cm radioactivity in the separated Am was reduced by a factor of 10^5 from its original value. However, serious problems were encountered in the use of the column in a glove box. Adjustment of the flow rate to the prescribed value was tedious and required a stable vacuum system. Also, preparing and cleaning the columns in the glove box was a source of cross contamination; a new column was required for each sample. For these reasons the calcium fluoride column technique is not recommended for the routine determination of Am.

The ^{239}Np method for the determination of ^{243}Am (ref. 35) was tested and modified for use at ORNL. This method involves separation of the ^{239}Np daughter of ^{243}Am from Am, Cm, and fission products. The gamma radioactivity of the separated ^{239}Np is determined and is compared with the radioactivity of the ^{239}Np separated from an ^{243}Am standard. Experiments showed that interference from ^{242}Cm and ^{244}Cm is insignificant as long as the ratio of Cm to ^{243}Am alpha radioactivity is less than 10^6. The precision of the method is good; relative standard deviations of 3 to 5% were attained for three series of ^{243}Am synthetic samples that contained $(1-5) \times 10^4$ alpha counts per minute of ^{243}Am. This method is currently being used for the routine determination of ^{243}Am in samples that do not contain large amounts of fission products.

[33] F. L. Moore, "Separation of Americium from Other Elements: Application to the Purification and Radiochemical Determination of Americium," *Anal. Chem.* 35, 715 (1963).

[34] H. Holcomb, "Separation of Americium from Curium with Calcium Fluoride," *Anal. Chem.* 36, 2329 (1964).

[35] C. J. Banick, "Determination of Americium-243 by Separation and Counting of Neptunium-239 Daughter," *Anal. Chem.* 37, 434 (1965).

9.4.c Improved Method for the Isolation of Trivalent Actinide-Lanthanide Elements from Nitrate Solutions

F. L. Moore

An improved method was developed for the recovery and purification of trivalent actinide-lanthanide elements from nitrate solutions. The method is based on the extraction of their anionic nitrate complexes from aqueous solutions of dilute nitric acid–aluminum nitrate with a quaternary amine, Aliquat 336-S-NO_3, dissolved in xylene or diethylbenzene. The order of decreasing extractability is Ce > Am > Pm > Cf > Bk > Cm > Eu > Tm > Y. Excellent decontamination is achieved from associated corrosion products and many fission products. The method has several advantages and potential applications; these are discussed in a recent publication.[36]

9.4.d Selective Liquid-Liquid Extraction of Berkelium(IV) with 2-Thenoyltrifluoroacetone–Xylene: Application to the Purification and Radiochemical Determination of Berkelium

F. L. Moore

A new, simple, rapid method for the radiochemical purification and determination of Bk was developed. It is based on the liquid-liquid extraction of Bk(IV) with 0.5 *M* 2-thenoyltrifluoroacetone–xylene. The high stability of the Bk(IV) chelate provides marked selectivity in extractions from aqueous solutions of nitric, sulfuric, or hydrochloric acid. Sodium dichromate is an efficient oxidant for Bk(III) tracer under the conditions required for optimum chelation and extraction of Bk(IV). Nitric acid solution (10 *N*) readily strips the Bk from the organic phase. Excellent separation of Bk is effected from many elements, including the alkali metals, alkaline-earth metals, trivalent lanthanides, Ru, Zr, Nb, U, Np, Pu, Am, Cm, Cf, Fe, Ni, Al, and Ag. This purification method has several useful analytical and process applications, which are discussed in an article[37] accepted for publication.

[36]F. L. Moore, "Improved Extraction Method for Isolation of Trivalent Actinide-Lanthanide Elements from Nitrate Solutions," *Anal. Chem.* 38, 510 (1966).

9.4.e Fast Separation of ^{95}Nb from ^{95}Zr in Commercial Radioisotope Solutions

Aart Jurriaanse[38] F. L. Moore

A new, fast, cheap liquid-liquid extraction method was developed for the quantitative separation of carrier-free ^{95}Nb from carrier-free ^{95}Zr tracers present in commercial radioisotope solutions. The method is based on the highly selective extraction of ^{95}Nb tracer by diisobutylcarbinol from dilute aqueous solutions of sulfuric acid–hydrofluoric acid. The ^{95}Nb tracer can be stripped easily into a wide variety of aqueous reagents for tracer work. Radiochemical purities >99.9% for ^{95}Nb and 99.8% for ^{95}Zr are readily achieved. Details are discussed in a recent publication.[39]

9.4.f Liquid-Liquid Extraction of Metal Ions from Aqueous Solutions of Organic Acids with High-Molecular-Weight Amines

F. L. Moore

New liquid-liquid extraction systems for the trivalent actinide and lanthanide elements were developed. High-molecular-weight amines are excellent extractants for removing anionic complexes of these elements from aqueous solutions of most organic acids. The liquid-liquid extraction behavior was determined for systems that contained one of various primary, secondary, tertiary, or quaternary amines and one of the following acids: citric, tartaric, oxalic, ethylenedinitrilotetraacetic (EDTA), α-hydroxyisobutyric, and acetic. The high extractability of the anionic EDTA complexes of trivalent actinide, lanthanide, and other metal ions from both acid and alkaline solution is of considerable practical importance. In the actinide-lanthanide series the order of decreasing extractability is (Am, Cm, Cf) > Eu > Sm > Pm > Ce > Y > Tm. The systems have several

[37]F. L. Moore, "Selective Liquid-Liquid Extraction of Bk(IV) with 2-Thenoyltrifluoroacetone–Xylene: Application to the Purification and Radiochemical Determination of Berkelium," accepted for publication in *Analytical Chemistry*.

[38]Alien Guest from the South African Atomic Energy Board, Pelindaba, Pretoria, South Africa.

[39]A. Jurriaanse and F. L. Moore, "Fast Separation of Niobium-95 from Zirconium-95 in Commercial Radioisotope Solutions," *Anal. Chem.* 38, 964 (1966).

potential applications in separations, which are discussed in a recent publication.[40]

9.4.g Analysis of Radioactive Wastes

S. A. Reynolds

Some characteristics of liquid wastes discharged to disposal trenches were determined. Analyses were done to measure the concentrations of insoluble solids, the distribution of radionuclides between solids and solution, and the chemical and radiochemical composition of the solids. Radiochemical findings are given in Table 9.3. Measured solids concentrations ranged from 0.13 to 0.7 mg/ml. Samples of the solids were analyzed qualitatively by emission spectrography; the principal elements found were Si and Al, with lesser amounts of Ca, Fe, Mg, Mn, and other metals. These results indicate a mixture of materials. Samples were also submitted for x-ray diffraction; these results also indicated a mixture of compounds, considerable amorphous material, or both. Thus, the hope of elucidating the chemical nature of the material was not completely realized, although complex silicates of Al and Ca are surely present.

9.4.h Adsorption of Trace Elements on Human Hair and Elution Therefrom

L. C. Bate

The study of the adsorption of trace elements on human hair[41] and their elution from it was completed. A paper on the work was published.[42]

9.5 APPLICATION OF COMPUTERS

9.5.a Secondary Standardizations by Use of Digital-Computer Techniques to Normalize Multiple Gamma-Ray Spectra for Direct Efficiency Determinations

J. S. Eldridge

A new technique for standardizing gamma-ray-emitting nuclides has been developed. It is based on the use of gamma-ray spectral data in digital format from absolutely standardized samples of radionuclides. A system for collecting a library of gamma-ray spectra from such absolutely standardized samples was perfected in conjunction with a least-squares gamma-spectral resolution program. A pooling program was devised to combine six gamma-ray spectra from multiple sample mounts from the single master solution. The program was written in FORTRAN-FAP for the IBM 7090 computer; it corrects for background, decay, and gain shift and then converts the spectral data from counts per second per channel to counts per disintegration per channel by use of the disintegration rate of the standard sample as an input parameter. The spectral data are treated statistically with an analysis of variance to achieve high accuracy in the library of spectra.

[40] F. L. Moore, "Liquid-Liquid Extraction of Metal Ions from Aqueous Solutions of Organic Acids with High-Molecular-Weight Amines. The Trivalent Actinide-Lanthanide Elements," *Anal. Chem.* 37, 1235 (1965).

[41] L. C. Bate, "Adsorption of Trace Elements by Hair," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1964*, ORNL-3750, pp. 56–57.

[42] L. C. Bate, "Adsorption and Elution of Trace Elements on Human Hair," *Intern. J. Appl. Radiation Isotopes* 17, 417 (1966).

Table 9.3. Results of Analyses of Radioactive Wastes

Radionuclide	Concentration of Component		Percent of Total Radionuclide in Solution Phase
	In Solution Phase (dis min^{-1} ml^{-1})	In Solid Phase (dis min^{-1} mg^{-1})	
^{137}Cs	$(0.5 \text{ to } 3) \times 10^7$	$(1 \text{ to } 7) \times 10^5$	97–100
^{90}Sr	$(0.4 \text{ to } 4) \times 10^6$	$(1 \text{ to } 9) \times 10^6$	34–64
^{144}Ce	$(8 \text{ to } 10) \times 10^4$	$(2 \text{ to } 6) \times 10^6$	9
^{106}Ru	$(0.6 \text{ to } 1.7) \times 10^4$	$(5 \text{ to } 6) \times 10^3$	77–100
^{60}Co	$(0.3 \text{ to } 6) \times 10^4$	$(1 \text{ to } 11) \times 10^4$	30–80

Because the gamma-ray spectra from the pooling program consist of channel-by-channel data in units of counts per disintegration, these response curves give channel-by-channel values for the efficiency of detection of the given gamma-ray spectrometer system. By combining these individual channel efficiencies for some region of the gamma-ray spectrum that is not affected greatly by external processes (e.g., a prominent photopeak), it is possible to assign directly an efficiency factor for the detection of each of the library radionuclides.

An assay system was developed that uses the digital data in the form of punched paper tape. The paper-tape standard can be read into the memory of a multichannel analyzer, where a direct comparison can be made with the unknown sample. The system is independent of detector resolution as long as the region chosen for the comparison of standard and unknown encompasses an entire photopeak.

A library of standard spectra in the described format has been collected for 42 radionuclides by use of a 3- by 3-in. NaI(Tl) detector system and a 10-cm source-to-detector distance. For most nuclides tested, the error of assay by use of the paper-tape system has been less than ±3%.

A paper that described this work was presented at the IAEA "Symposium on the Standardization of Radionuclides"[43] and will be published in the proceedings of that symposium.

9.5.b Gamma-Ray Spectrometry by Use of Computer Library of Spectra

J. S. Eldridge A. A. Brooks[44]

To complement many of the existing techniques of gamma-ray spectral analysis, we have made a collection of gamma-ray response functions for 42 radionuclides measured with a 3- by 3-in. NaI(Tl) spectrometer. Use of digital data from the collection is the basis of a new assay system. In this technique, data from the standard-response functions can be inserted into an analyzer memory from punched paper tape (one of the options for recall of the standard spectra), and selected regions of the standard spectra can be compared with the same region from an unknown sample. Then, by use of a simple normalizing factor, an absolute disintegration rate can be assigned to the unknown sample, and the sample can be identified. In this study, two computer programs were developed for combining multiple spectra and resolving the spectra of mixtures of gamma-emitting nuclides by the method of linear least squares. The paper-tape library gives suitable assay standards for both qualitative and quantitative gamma-ray spectrometry. A paper that describes this work was published.[45]

9.5.c Program AREA LSQ

Enzo Ricci J. R. Stockton[46]

The programs NETAREA[47] and CLSQ[48] were coupled to simplify the handling of nuclear counting data. The new code, AREA LSQ, can operate separately with either of the original programs and can also use them in sequence; in fact, any number of different CLSQ treatments can be performed automatically after one NETAREA run.

9.5.d Program THIKSIG

R. L. Hahn

The yield of a given radionuclide, produced by the irradiation of a thick target with a beam of charged particles, is related to the reaction cross section by

$$Y = n \int_{E_f}^{E_i} [\sigma(E)/S(E)]\mathrm{d}E \ , \qquad (9.1)$$

[43]J. S. Eldridge, "Secondary Standardizations Using Digital Computer Techniques to Normalize Multiple Gamma-Ray Spectra for Direct Efficiency Determinations," Paper SM-79/34 presented at the IAEA Symposium on Standardization of Radionuclides, Vienna, Oct. 10–14, 1966.

[44]Central Data Processing Facility, ORGDP.

[45]J. S. Eldridge and A. A. Brooks, "Gamma-Ray Spectrometry Using Computer Library of Spectra," *Nucleonics* 24(4), 54 (1966).

[46]Mathematics Division.

[47]R. L. Hahn and E. Ricci, "Program NETAREA," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1965*, ORNL-3889, p. 62.

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

where Y is the yield of radioactive product, expressed in disintegrations per minute per number of incident charged particles per minute, n is the number of nuclei per gram of target, $\sigma(E)$ is the reaction cross section at energy E, $S(E)$ is the stopping power of the target material for the particles at energy E, E_i is the initial kinetic energy of the particle beam, and E_f is the lowest energy at which the reaction of interest can occur in the target. To obtain values of the cross section, one usually differentiates the curve of reaction yield vs energy. That is, from Eq. (9.1),

$$\sigma(\overline{E}) \approx \left(\frac{\Delta Y}{\Delta E}\right)\left[\frac{S(\overline{E})}{n}\right], \qquad (9.2)$$

where $\Delta Y/\Delta E$ is the change in yield, ΔY, associated with the energy change, ΔE, and $\overline{E}$ is the average value of the energy within interval ΔE.

Program THIKSIG was written to carry out the numerical differentiation of the reaction-yield curve. Data on the variation of yield with energy serve as input for the code. From tables of the stopping powers of various elements, values of $S(\overline{E})$ are calculated (for targets that are either elements or compounds); $\sigma(\overline{E})$ is then computed by means of Eq. (9.2).

9.6 APPLICATION OF RADIOISOTOPES IN ANALYTICAL CHEMISTRY

9.6.a Precision Photometric Analysis by Use of a Beta-Excited Isotopic Light Source

H. H. Ross

Early in 1966, the Analytical Chemistry Division introduced a new technique for making high-precision photometric measurements.[49] This new method utilizes the unusual spectral and emission properties of a beta-excited light source combined with pulse-height-measuring equipment. The advantages of the technique include high stability, controlled sensitivity, simplicity, small power requirements, direct digital output, and straightforward error analysis.

The disclosure of the method[49] includes a complete discussion of the theory of operation and instrumental requirements; examples of analyses of several colorimetric systems are presented. Initially, measurements were made with modified nuclear counting equipment. Now, a small, solid-state, laboratory instrument specially designed for photometric analysis has been completed.

The electronic portion of the instrument consists of eight subassemblies: (1) linear amplifier, (2) single-channel analyzer, (3) high-speed scaler, (4) precision timer, (5) high-voltage supply, (6) low-voltage supply, (7) charge-sensitive preamplifier, and (8) phototube and divider network. Items 1 through 6 were placed on a single chassis that is the main electronic portion of the photometer; the preamplifier, phototube, and divider network are located in the measuring head of the instrument. Most of these components are commercially available as separate modules.

The measuring head (Fig. 9.12) of the instrument is arranged in three sections. One end section houses the phototube, divider network, and preamplifier. The components are mounted axially and lie in a horizontal plane. The other end of the head contains the isotopic light source, which is so mounted as to minimize the chance of breakage. The emitting face of the source is in line axially with the sensitive end face of the phototube.

The center section of the measuring head holds the samples. Standard 1.0-cm absorption cells are used; four cells can be accommodated simultaneously. A filter holder and one optical colli-

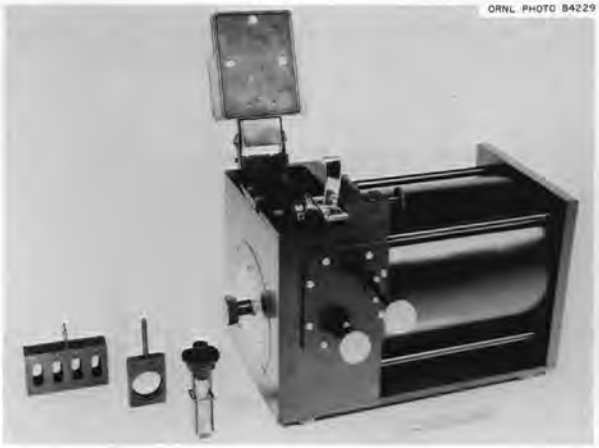

Fig. 9.12. Measuring Head of the Radioisotopic-Light-Source Photometer.

[49] H. H. Ross, "New Concept in Precision Photometric Analysis Using a Radioisotopic Light Source," *Anal. Chem.* 38, 414 (1966).

mator are also located in the center section; another collimator is in the detector compartment. A simple system of interlocks and shutters is incorporated in the housing to minimize the chance of exposing the phototube to room illumination. The entire assembly is constructed of aluminum and is anodized black.

A liquid-scintillator light source, similar to the one used initially, was prepared for use in the present instrument. This light source has characteristics that are essentially identical with those of the one previously described[49] except that the emission rate is greater by a factor of about 30.

The completed instrument is now undergoing final checkout and calibration.

9.6.b Development of Radiation-Stimulated Light Sources

H. H. Ross

A program to study the emission characteristics of phosphors and the design of radiation-stimulated light sources was started. The investigation was limited to phosphors that emit in the wavelength region 600 to 800 mμ. A special high-sensitivity instrument was designed and fabricated both to excite the phosphors and to spectrally analyze and integrate the fluorescent output. The instrument consists of an excitation head (high-intensity, short-wave, ultraviolet- or beta-emitting isotopic source), a Bausch and Lomb 0.5-m grating monochromator blazed at 700 mμ, a bi-alkali photomultiplier detector with S-20 surface characteristics, and the associated electronics to automatically drive the monochromator and to record the output spectrum. By use of a 2865°K tungsten source, the completed system was calibrated for equal response over the desired spectral range.

Over 100 phosphors were screened by use of ultraviolet excitation. Those phosphors that exhibit high fluorescence in the desired spectral region were selected for further study. Also, the fluorescence-conversion efficiencies of the selected phosphors under the influence of beta excitation from a ^{147}Pm source were studied. Table 9.4 shows the emission intensities of some selected phosphors excited by the beta radiation from 80 mc of ^{147}Pm.

Practical radiation-stimulated light sources are now being designed.

Table 9.4. Emission Intensities of Phosphors Excited by ^{147}Pm

Phosphor	Absolute Emission		Wavelength at Maximum Intensity (mμ)
	amp · mμ × 10^6	Relative	
CdS:Ag	2.1 ± 0.06	1	710
ZnCdS:Ag	1.7 ± 0.04	0.81	680
YVO_4:Eu	1.6 + 0.05	0.76	620
YVO_4:Eu, Dy	1.6 ± 0.05	0.76	620
Y_2O_3:Eu	1.0 ± 0.03	0.48	610
Lu_2O_3:Eu	0.97 ± 0.03	0.46	610
$Zn_3(PO_4)_2$:Mn	0.85 ± 0.03	0.40	640
$Cd(BO_2)_2$	0.71 ± 0.02	0.34	620
$CaSiO_2$	0.34 ± 0.01	0.16	610
$LaVO_4$:Eu	0.25 ± 0.01	0.12	620
CdS:Tm, Cu	0.19 ± 0.01	0.09	800
$(MgO)_x(As_2O_5)_y$:Mn	0.079 ± 0.004	0.04	660

9.6.c Analytical Chemistry of Water Environments

W. J. Ross

The demand to upgrade and expand the analytical chemistry of aqueous ecological systems is increasing. A program was initiated to define the current deficiencies and to develop techniques and methods for solving specific analytical problems in water chemistry. Most areas of basic investigation and economic application of water environments could be aided by improved analytical technology. However, our program has been designed to solve those problems brought to us by colleagues engaged in biological, ecological, and engineering work on water and also by members of several marine laboratories who know of our interest in this field. Consequently, the work to date has been consultation, as well as experimentation, in respect to these cooperative efforts.

Determination of Oxygen. – The classical Winkler method for determining dissolved O_2 in water was modified to use a radio-release technique. A radioisotope of I_2 is added to the usual NaI reagent, and the radioactive I_2 subsequently formed through oxidation by O_2 is extracted into chloroform and is measured radiometrically. This method is a sensitive and precise means of determining the O_2 content and its rate of change in stagnant or anoxic water; as little as 0.5 μg of O_2 can be determined.

In anticipation of the need to monitor O_2 in effluents from high-efficiency deaerators, the effects of oxidizing and reducing components on the results of O_2 determinations by the Winkler method were measured.

Determination of Submicro Amounts of Phosphate. – The prominence of PO_4^{3-}, SO_4^{2-}, NO_3^-, and NO_2^- as nutrients in the development of all life in an aqueous ecological system has provided a need for ultrasensitive methods for determining these ions. A similar need exists in cytological studies to determine trace amounts of P. Therefore, an effort is being made to increase the sensitivity for determining P and to develop a method for concentrating, in situ, submicro amounts of PO_4^{3-} from natural reservoirs of saline and fresh water. To achieve the first goal, activation analysis techniques are being tested whereby a stable compound is formed stoichiometrically between PO_4^{3-} and an element that can be measured more sensitively than P after the compound has been irradiated. Significant increase in sensitivity is achieved from the formation and neutron activation of heteropoly acids of PO_4^{3-}, V, and Mo followed by measurement of either of the gamma-emitting isotopes ^{52}V and ^{101}Mo. However, to be effective, the heteropoly acid must be isolated completely from the reagents that contain V or Mo. Several oxygenated solvents (diethyl ether, octanol-1, butyl acetate) and mixtures of solvents will quantitatively extract 10^{-9} g of P as phosphomolybdic acid, $H_3PMo_{12}O_{40}$, but the extraction of phosphovanadomolybdic acid is only partially complete with the reagents tried so far. This investigation is being continued in an effort to extract $H_3PO_4 \cdot HVO_3 \cdot 16MoO_3$ and also to find a solvent with essentially no capacity for the reagents that contain V or Mo.

Concurrently, efforts are being made to concentrate PO_4^{3-} from both static and dynamic systems of large volumes of seawater and of fresh water and to determine P at the same time, if possible. Several systems based on isotopic exchange of free PO_4^{3-} ions with those associated with a highly insoluble phosphate salt have been partially successful for small volumes of water but are not applicable to the removal of PO_4^{3-} from large volumes because of the increase in the solubility of such salts as $(Zr)_3(PO_4)_4$, $MgNH_4PO_4$, and $FePO_4$. Application of ligand exchange chromatography (e.g., cation exchange columns loaded with Fe^{3+}) failed for similar reasons. However, PO_4^{3-} ions are readily adsorbed on Al_2O_3 columns, and this material is being investigated further for use both as a chromatographic bed and as a filler for a moving "collector."

Tracers for the Movement of Water and Sediments. – Although both activation analysis techniques and radiotracers have been used to trace the flow of liquids and solids, the chemical and physical properties of the elements of interest have not been studied systematically. As part of a continuing investigation of the chemistry of submicro and micro amounts of ions in solution, the chemical stability and adsorptive characteristics of several elements are being studied in solutions and in mixtures whose compositions approximate those found in fresh water and in coastal localities. The behavior of tracer-level and microgram amounts of Cd^{2+}, Co^{2+}, Cu^{2+}, Ni^{2+}, and Mn^{2+}, with and without the protection

of complexing agents, is currently under study to determine when and how losses of these elements from solutions occur and how such losses can be prevented until the system under study has been characterized.

Micro Elemental Analysis of Saline Water. – The determinations of several elements have been investigated relative to specific problems in oceanographic research. A referee method was developed for determining Mo in seawater. The Mo is isolated by extraction with tri-*n*-octylphosphine oxide and is determined by activation analysis. A substoichiometric isotope-dilution technique was investigated for determining Co in seawater but was discarded because it is too tedious and unreliable. The degree to which the rare-earth elements can be separated from brine and from each other by extraction into di-(2-ethylhexyl)phosphoric acid was established on both tracer and microgram levels. During the development of a separation scheme for Th, Pa, and U to be used in a geochronological study, a comprehensive investigation was made of the extractability of Pa into cyclohexane solutions of tri-*n*-octylphosphine oxide (TOPO) from systems that contained mineral acids and salts.

9.6.d Neutron Absorption Measurements

J. E. Strain

The technique of neutron absorption measurements by use of isotopic neutron sources[50] has been revised to measure the total absorption cross section of large samples. Because it is possible with this technique to nondestructively measure the absorption cross section with a standard deviation of ±1 ppm boron equivalent, this measurement will be used to rapidly check the purity of a variety of materials. The samples presently being analyzed are Al_2O_3 and ThO_2. The apparatus uses a 2-curie ^{241}Am-Be neutron source in a paraffin moderator. The 10-liter sample is held in an aluminum container that fits around a 2.5- by 30-cm $^{10}BF_3$ neutron counter. The measurement requires only 10 min, exclusive of the time required for sample loading.

[50]J. E. Strain and W. S. Lyon, "The Use of Isotopic Neutron Sources for Chemical Analysis," p. 245, in *Radiochemical Methods of Analysis*, vol. 1, IAEA, Vienna, 1965.

9.6.e Review Article, "Nucleonics," for Analytical Chemistry

W. S. Lyon Enzo Ricci
H. H. Ross

A review[51] of the advances in the entire field of nucleonics during the past two years was written for the Annual Reviews issue of *Analytical Chemistry*.

9.7 ACTIVATION ANALYSIS

9.7.a Sensitivities of Activation Analysis with 18-Mev ^{3}He Particles for 15 Light Elements

Enzo Ricci R. L. Hahn

The thick-target irradiations performed at the ORNL tandem Van de Graaff accelerator[52] gave sensitivity values of activation analysis with 18-Mev ^{3}He particles for Be, B, C, N, O, F, Na, Mg, Al, Si, P, S, Cl, K, and Ca. Figure 9.13 shows the determined instrumental sensitivities in histogram form.

Absolute sensitivities were calculated from the instrumental values; all the results are tabulated and discussed in detail in a forthcoming article.[53] It is clear that the sensitivities are all high; in particular, those for Be, B, C, N, O, F, and S range from 12 to 112 counts/min per ppb. Indeed, this group of elements cannot be determined easily in very low concentrations by other techniques. Thus, ^{3}He activation opens the way for the development of highly sensitive techniques for a group of elements that now present analytical problems.

9.7.b Interactions of ^{3}He Particles with ^{9}Be, ^{12}C, ^{16}O, and ^{19}F

R. L. Hahn Enzo Ricci

The excitation functions determined during the irradiations at the 5.5-Mev Van de Graaff accel-

[51]W. S. Lyon, E. Ricci, and H. H. Ross, "Nucleonics," *Anal. Chem.* 38, 251R (1966).

[52]E. Ricci and R. L. Hahn, "^{3}He Activation Analysis," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1965*, ORNL-3889, pp. 64–65.

[53]E. Ricci and R. L. Hahn, "Sensitivities for Activation Analysis of 15 Light Elements with 18-Mev ^{3}He Particles," submitted for publication in *Analytical Chemistry*.

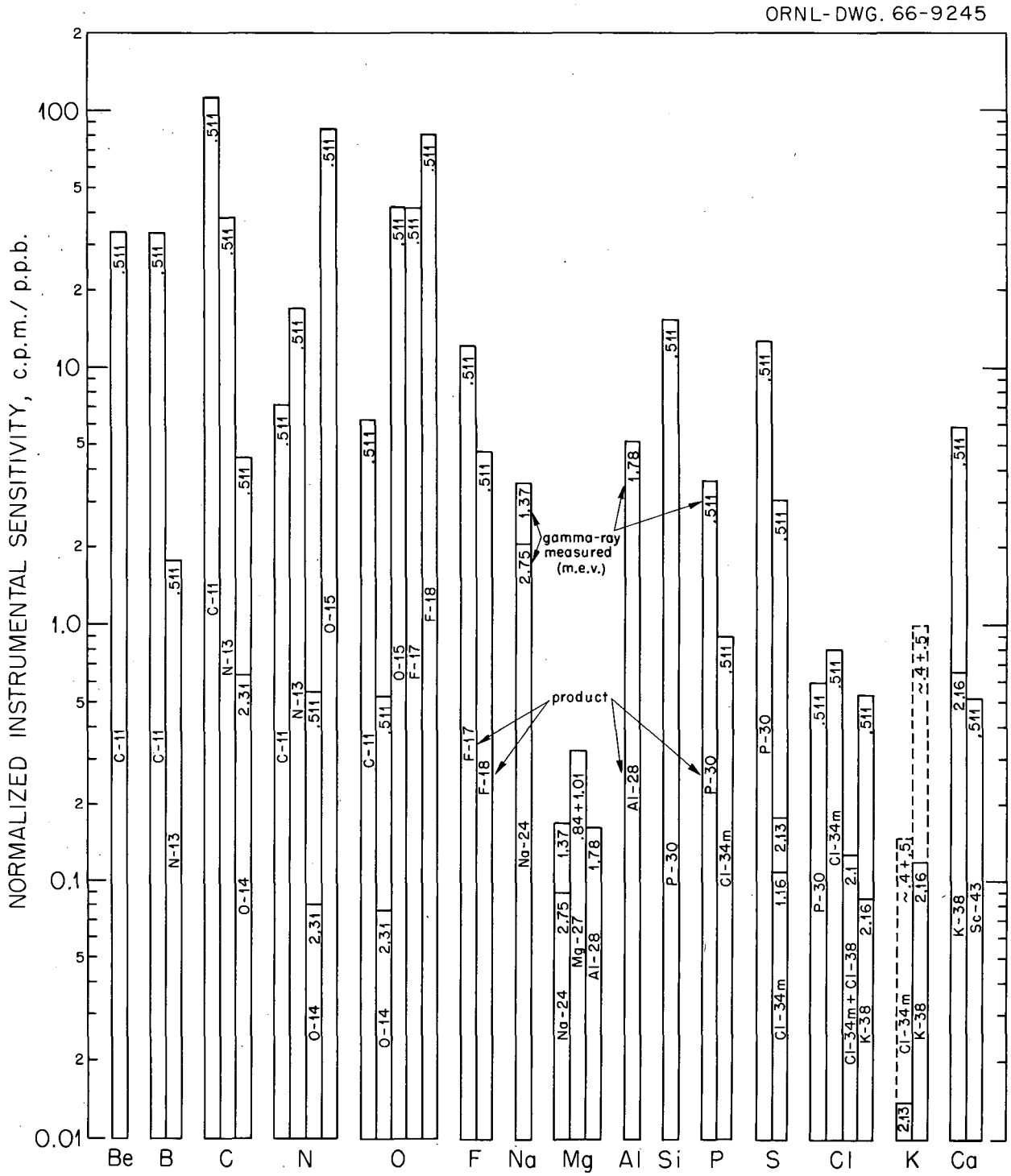

Fig. 9.13. Instrumental Sensitivities of Activation Analysis for Light Elements with 18-Mev ^{3}He Particles. Irradiation: duration, 1 half-life; beam, ^{3}He (100-μa current). Geometry: detector, 3- by 3-in. NaI(Tl); height, 10 cm (including 1.3-cm-thick Lucite absorber); samples between copper disks thick enough to stop all emitted positrons. Sensitivity values normalized to a ^{3}He range of 54.4 mg/cm^2.

erator[54] were compared with results obtained by other authors and found to be in reasonable agreement. In particular, the fine structure of the reaction $^{12}C(^3He,n)^{14}O$ was confirmed in these experiments.[55] An integral excitation function, calculated from the differential data of Towle and Macefield[56] for the $^9Be(^3He,n)^{11}C$ reaction, agreed with the integral data obtained in this work. The distorted-wave theory of direct reactions[32] was used to compute excitation functions for comparison with integral data for $(^3He,\alpha)$ reactions on ^{12}C, ^{16}O, and ^{19}F and for the $(^3He,d)$ reaction on ^{12}C. The results of the comparison of the values calculated from theory and those obtained experimentally indicate that a direct mechanism is operative in all the cases studied except the $^{12}C(^3He,\alpha)^{11}C$ reaction, which appears to proceed to a large extent by way of a compound-nucleus reaction. Figure 9.14 illustrates the agreement between the excitation function for the reaction $^{16}O(^3He,\alpha)^{15}O$, determined in this work, and the curve computed from the direct-reaction theory. The integral data indicate that the primary mechanisms operative in the reactions $(^3He,\alpha)$ on ^{16}O and ^{19}F and in the reaction $(^3He,d)$ on ^{12}C are direct pickup and stripping, respectively. This work is described in detail in a published paper.[31]

[54] R. L. Hahn and E. Ricci, "Excitation Functions for ^{3}He-Induced Nuclear Reactions," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1965*, ORNL-3889, p. 65.

[55] D. R. Osgood, J. R. Patterson, and E. W. Titterton, "The Excitation Function for the Reaction $C^{12}(He^3,n)O^{14}$ Between Threshold and 11.45 MeV," *Nucl. Phys.* **60**, 503 (1964).

[56] J. H. Towle and B. E. F. Macefield, "A Study of the $Be^9(He^3,n)C^{11}$ Reaction," *Nucl. Phys.* **66**, 65 (1965).

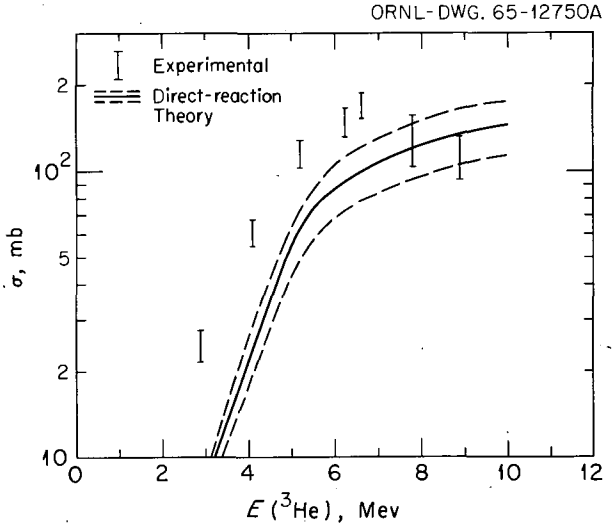

Fig. 9.14. Comparison of the Experimental Excitation Function for the Reaction $^{16}O(^3He,\alpha)^{15}O$ with the Results of the Distorted-Wave Theory of Direct Reactions.

9.8 14-Mev NEUTRON GENERATOR STUDIES

9.8.a Neutron Generator Modification

J. E. Strain

The neutron generator obtained from the Texas Nuclear Corporation was modified to change the beam current from 1.0 to 2.5 ma and to use a target of larger diameter. These modifications should make available a neutron flux of almost 10^{10} neutrons cm^{-2} sec^{-1} and thus increase the usefulness of the generator in activation analysis and in the study of fast-neutron reaction products.

9.8.b 14-Mev Neutron Activation Analysis for Oxygen in Alkali Metals

J. E. Strain

Construction of the facility designed for the determination of O_2 in alkali and refractory metals by 14-Mev neutron activation analysis is nearing completion. A Kaman neutron generator was installed, and the adequacy of its neutron shielding was evaluated. All the other equipment to be included in the facility has been assembled but not yet installed. Figure 9.15 is a conceptual drawing of the completed facility.

9.8.c Determination of ^2H in H_2O by Fast-Neutron Activation

J. E. Strain

A novel method was developed to determine ^2H in pure water by 14-Mev neutron activation analysis. Bombardment of the water sample with fast neutrons produces in the sample deuterons having energies ≤ 7 Mev. These deuterons interact with ^{16}O to produce ^{17}F ($t_{1/2}$ = 66 sec) according to the reaction $^{16}O(d,n)^{17}F$. The amount of ^{17}F

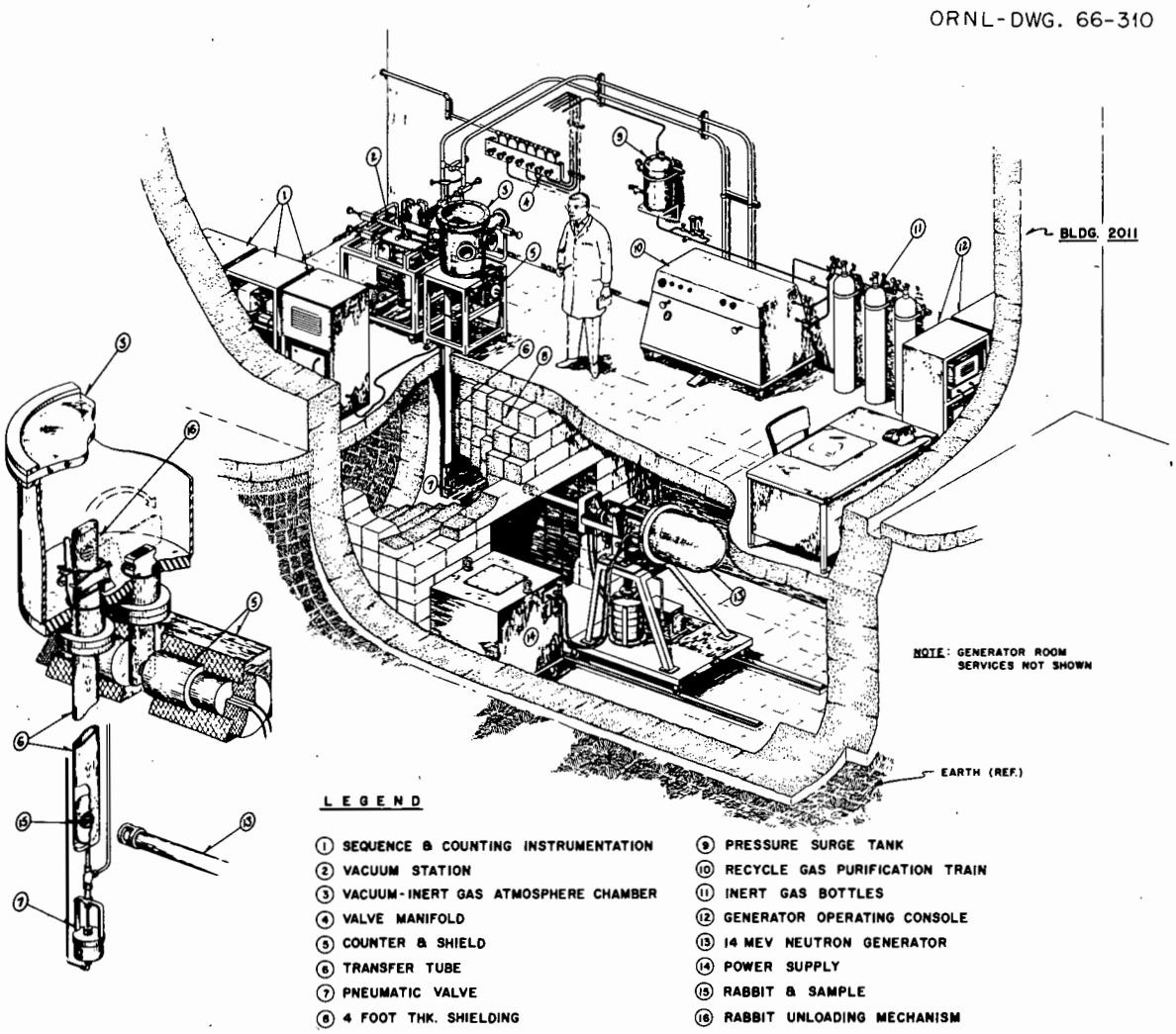

Fig. 9.15. 14-Mev Neutron Activation Analysis Facility.

produced is proportional to the amount of ^{2}H in the sample. The analysis is specific for ^{2}H in pure water and requires only 30 sec of irradiation time and 5 min of counting. The sensitivity is such that ^{2}H can be determined in the concentration range from 0.1 to 99% in a 1-g sample with a relative standard deviation not greater than 10%.

9.8.d 14-Mev Neutron Activation Analysis

J. E. Strain

The 14-Mev neutron generator has been used to solve several analytical problems. Determinations have been made of ^{16}O in refractory metals and in $Mg^{18}O$, aluminum in VF_2, and atom ratios in NaO_2, VF_2, and $Mg^{18}O$. The tracers ^{18}F, ^{44}K, and ^{75}Ge were produced for use in radiochemical separation studies.

9.8.e Monitoring of Leached Fuel Elements with a Neutron Generator

J. E. Strain W. J. Ross

The development of a leached fuel element monitor[57] is essentially complete. The final version

[57] W. J. Ross, J. E. Strain, and J. W. Landry, "Monitoring of Leached Fuel Elements with a Neutron Generator," *Trans. Am. Nucl. Soc.* 9(1), 107 (1966).

of the instrument (Fig. 9.16) is designed to detect the residual U in 8-in.-diam by 5-ft-high dissolver baskets. Radiation fluxes $\leqq 4000$ r/hr from the radioactive cladding do not interfere with the detection of as little as 6 mg of ^{235}U in a basket of leach hulls. A complete basket can be analyzed in about 10 min; thus, the cladding from a complete reactor core could be examined in just over 1 hr. Plans are under way to construct a complete operating prototype of the monitor within a hot cell so that the process can be demonstrated for interested industrial and governmental agencies.

9.8.f Influence of Lucite Surroundings on the Yield from 14-Mev Neutron Reactions

Enzo Ricci

An increase in the ratio R of the end-of-bombardment specific activity of ^{28}Al with respect to that of ^{62}Cu, $R(^{28}Al/^{62}Cu)$, and also of ^{27}Mg with respect to ^{62}Cu, $R(^{27}Mg/^{62}Cu)$, had been expected, and was observed,[58,59] when samples of Si, Al, and Cu were irradiated in a Lucite enclosure rather than as bare metals. Because this effect may have great importance in the measurement of the 14-Mev neutron flux in "rabbit" facilities, a systematic study was made to develop a method to standardize such flux measurements. Samples of SiO_2 and of Cu, sandwiched between slabs of Lucite of various thicknesses, were irradiated for 2.5 min. The faces of the slabs were always parallel to the tritium target; thus, one Lucite slab was interposed between the sample and the neutron generator, and an identical slab was attached to the back of the sample.

In Fig. 9.17 the ratio $R(^{28}Al/^{62}Cu)$ is plotted vs the slab thickness. The curve shows that $R(^{28}Al/^{62}Cu)$ increases sharply (19.5%) as the Lucite thickness increases from 0 to 3.0 cm. Indeed, for the first centimeter (the most important region) the increase is about 8.2%, although scatter of the data is also noticeable. More work will be done on this region, since the plastic used to fabricate most plastic rabbit facilities is less than ~ 1 cm thick.

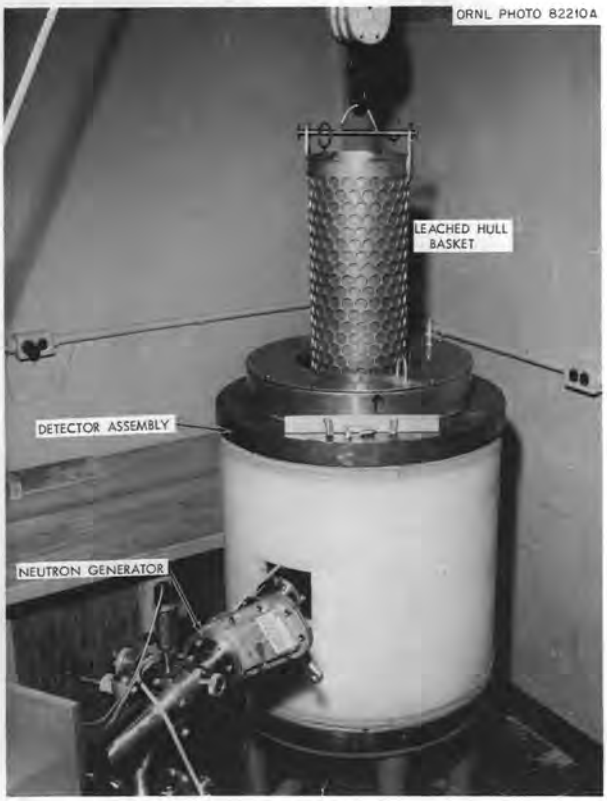

Fig. 9.16. Chop-Leached Fuel Monitor with Dissolver Basket in Place.

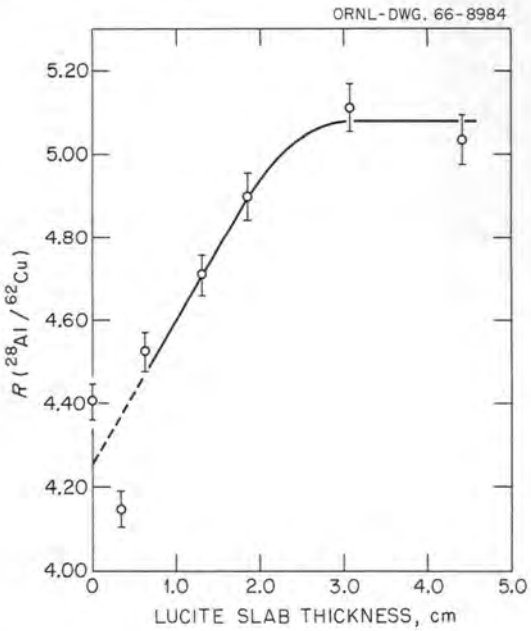

Fig. 9.17. $R(^{28}Al/^{62}Cu)$ vs Thickness of Lucite Surrounding the Target During the Irradiation.

[58] E. Ricci, "Output Spectrum from 14-MeV Neutron Generators. Rapid Estimation and Influence in Cross-Section Measurements and Activation Analysis," *J. Inorg. Nucl. Chem.* 27, 41 (1965).

[59] E. Ricci, "Standardization of Neutron Flux Measurements," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1965*, ORNL-3889, p. 69.

10. Inorganic Preparations

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

Most of the regular programs of last year were continued without significant change. Fused salts, prepared for the High Temperature and Structural Chemistry Group of the Chemistry Division, included anhydrous halides of the rare-earth metals, alkali metals, and alkaline-earth metals and large amounts of KCl and LiCl-KCl eutectic used as solvents. Other continued programs were for the Solid State Division. The purification of KCl for the Research and Development of Pure Materials Group was on a routine basis, with some emphasis placed on reducing specific impurities such as lead. For the Neutron Diffraction Studies Group, alloys of rare-earth metals with each other and with other selected metals were prepared and heat treated as in the previous year. The series of alkali-metal superoxides in preparation for the last two years for the Neutron Diffraction Studies Group culminated with CsO_2. The difficult problem, existing since last year, of producing near 100% NaO_2 was solved after many attempts by different ways. Sodium metal was very gradually oxidized in a flow system with a variable mixture of nitrogen and oxygen that ended with pure oxygen at 100°C. The resulting material was transferred to an autoclave and was gradually heated to 300°C over a period of several days under oxygen at 2000 psi. The product, although chemically pure, contained about 10% of an unknown structural form of NaO_2 besides the regular cubic form. For another section of the same group, a series of spinels was prepared of the form $Fe_xB_{(3-x)}O_4$, where B is Cr, Ti, or Li and x ranges from 2 to 2.75. The syntheses were solid-state reactions at 1000°C in vacuum between the finely divided, mixed, and compacted oxides that contained only the stoichiometric amount of oxygen. For the Advanced Engineering and Development Group, 150 g of ^{6}Li salicylate was prepared by the method of Ross and Yerick.[1]

In a new program initiated during the year for the Spin Resonance Group of the Solid State Division, a series of glasses were prepared from the oxides of phosphorus, arsenic, and germanium. The P_2O_5 glasses were made by sealing P_2O_5, contained in a platinum boat, in an evacuated quartz tube, heating the tube to 800°C, and slowly cooling the melt. To remove bubbles, the colorless transparent oxide was sealed into a new evacuated quartz tube and was reheated; to smooth the surface, the oxide was heated again in a tube that contained argon. Alkali-metal phosphate glasses were also prepared by heating P_2O_5 with amounts of Li_2CO_3, Na_2CO_3, and K_2CO_3 that ranged from 0.3 to 1.6 moles of the alkali-metal carbonate per mole of P_2O_5. Each mixture was melted in a platinum crucible, and the melt was poured onto a copper plate; a second plate was pressed down quickly on the molten mass. These glasses were also colorless and transparent. Attempts were made to prepare glasses from As_2O_3 and As_2O_5 in a similar manner. Some were transparent but orange; others made with low concentrations of alkali-metal carbonates were milky. With GeO_2, a series of glasses was made that consisted of the pure oxide and of mixtures of the oxide with 0.003, 0.03, and 0.3 mole of alkali-metal carbonate per mole of GeO_2. The pure GeO_2 glass and the glasses with 0.003 mole of alkali-metal carbonate were heated in platinum boats at 1300°C in vacuum for ½ hr to remove all bubbles from the viscous melt and then were heated at 1420°C in pure oxygen for 2 hr to replace some oxygen that was removed in the vacuum treatment. These glasses were cooled in air; they were transparent and colorless. With the larger concentrations of alkali-metal carbonate, no vacuum treatment was necessary to remove bubbles; these glasses were heated in air at 1420°C for ½ hr but had to be quenched in liquid nitrogen to prevent crystallization. In connection with the same program, $AlAsO_3$[2] and $AlPO_4$[2] were prepared. The $AlPO_4$ was obtained in the pure state only by growing crystals from a solution of impure $AlPO_4$ dissolved in 6 M H_3PO_4.

[1] H. H. Ross and R. E. Yerick, "A New Liquid Scintillator for Thermal Neutron Detection," *Nucl. Sci. Eng.* 20, 23 (1964).

[2] G. Brauer, ed. (R. F. Riley, translation ed.), *Handbook of Preparative Inorganic Chemistry*, vol. 1, 2d ed., p. 831, Academic, New York, 1963.

The solution was contained in a sealed tube in an autoclave under a pressure of 2000 psi, and the temperature was gradually increased to 300°C. Attempts are under way to grow a single crystal of $AlPO_4$, since its structures duplicate the three different structures of quartz at approximately the same temperatures.[3]

In work for the Metals and Ceramics Division, $TiCl_3$,[4] $TeCl_4$,[5] and anhydrous NiI_2[6] were prepared for the Ion Spectroscopy Group. Pure anhydrous NiI_2 could not be made by described methods. It was obtained by the gradual dehydration[7] of $NiI_2 \cdot 6H_2O$ in a current of HI gas up to a temperature of 400°C. For the Fundamental Research in X-Ray Diffraction Group of the X-Ray Laboratory, 100 g of a high-purity solid solution of 25 mole % AgCl in anhydrous NaCl was made. Portions of this material were doped with anhydrous $CaCl_2$ in concentrations from 0 to 100 ppm in 10-ppm differences.

For the Physics Division, the problem arose of preparing a solid solution of 3 mole % $FeBr_3$ in $CrBr_3$ for the Low Temperature, Nuclear, and Solid State Physics Group. The compounds cannot be melted together, because $CrBr_3$ sublimes and also attacks quartz at elevated temperatures. In addition, $FeBr_3$ begins to lose Br_2 at 59°C.[8] The problem was solved by heating $FeBr_2$, $CrBr_3$, and excess Br_2 in a sealed quartz tube held in an autoclave at 400°C under a pressure of 2000 psi. At this temperature the Br_2 pressure is about 140 atm. By acting as solvent and oxidizer, the Br_2 forms the solid solution and converts the $FeBr_2$ to $FeBr_3$. In other work for the Physics Division, 30 g of anhydrous $^7LiClO_3$ was made for the High Voltage Experimental Program by pouring a solution of $KClO_3$ onto a column of Dowex 50W-X8 resin and receiving the emerging $HClO_3$ in a solution of ^7LiOH. Also provided was 50 g of $Cu(ClO_3)_2 \cdot 6H_2O$ made by metathesis from solutions of $CuSO_4$ and of $Ba(ClO_3)_2$.

For the Physical Chemistry of Molten Salts Group of the Reactor Chemistry Division, 10 g of $SmCl_2$ was made by reducing $SmCl_3$ with samarium metal at temperatures in the range from 500 to 900°C. About the same amount of $EuCl_3$ was reduced to $EuCl_2$ with hydrogen at 600°C.

In an extension of work done last year for the Special Electromagnetic Separations of Heavy Elements Group of the Isotopes Division, two portions of the previously prepared 20 mole % solid solution of $^{235}UCl_3$ in $LaCl_3$ were diluted to 2 mole %.

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[3] W. R. Beck, "Crystallographic Inversions of the Aluminum Orthophosphate Polymorphs and Their Relation to Those of Silica," *J. Am. Ceram. Soc.* **32**, 147 (1949).

[4] G. Brauer, ed. (R. F. Riley, translation ed.), *Handbook of Preparative Inorganic Chemistry*, vol. 1, 2d ed., p. 442, Academic, New York, 1963.

[5] G. Brauer, ed. (P. G. Stecher, translation ed.), *Handbook of Preparative Inorganic Chemistry*, vol. 2, 2d ed., p. 1187, Academic, New York, 1963.

[6] G. Brauer, ed. (P. G. Stecher, translation ed.), *Handbook of Preparative Inorganic Chemistry*, vol. 2, 2d ed., p. 1547, Academic, New York, 1963.

[7] P. Lumme and J. Peltonen, "Thermogravimetric Studies VII. Thermal Stability and Kinetics of Decomposition of Hydrated Nickel(II) Chloride, Bromide, Iodide, Nitrate, and Sulphate," *Suomen Kemistilehti* **37**(B), 162 (1964).

[8] N. W. Gregory and B. A. Thackrey, "An Equilibrium Study of the Thermal Dissociation of Ferric Bromide," *J. Am. Chem. Soc.* **72**, 3176 (1950).

11. Organic Preparations

P. F. Thomason J. C. White H. L. Holsopple

A number of organic compounds not readily available from commercial sources were custom synthesized for research use at K-25 and in the Chemistry, Chemical Technology, Isotopes, and Metals and Ceramics Divisions. In addition, several organic compounds were purified for research members of the Analytical Chemistry, Health Physics, Neutron Physics, and Reactor Chemistry Divisions.

11.1 COMPOUNDS SYNTHESIZED

11.1.a Quaternary Phosphonium Halides

H. L. Holsopple

The three quaternary phosphonium compounds benzyltributylphosphonium bromide, benzyltrioctylphosphonium bromide, and tetra-*n*-butylphosphonium iodide were prepared for the Metals and Ceramics Division according to the reactions

$$(C_4H_9)_3P + C_6H_5CH_2Br \longrightarrow [(C_4H_9)_3PCH_2C_6H_5]Br ,$$

$$(C_8H_{17})_3P + C_6H_5CH_2Br \longrightarrow [(C_8H_{17})_3PCH_2C_6H_5]Br ,$$

$$(C_4H_9)_3P + C_4H_9I \longrightarrow [(C_4H_9)_4P]I .$$

Results of elemental analysis were in good agreement with calculated values (Table 11.1) based on the formulas given above. The details of these preparations together with the NMR and infrared spectra of these compounds are given elsewhere.[1]

11.1.b *m*-Aminobenzylamine

H. L. Holsopple W. J. Ross[2]

m-Aminobenzylamine (>600 g) was prepared for the Physics Group of the K-25 Technical Division. *m*-Nitrobenzaldehyde was caused to react with

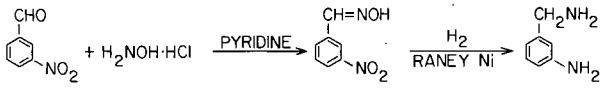
[1]H. L. Holsopple, *Syntheses of Three Quaternary Phosphonium Halides*, ORNL-TM-1572 (July 12, 1966).
[2]Nuclear and Radiochemical Analyses Group.

Table 11.1. Compositions of Three Quaternary Phosphonium Halides

Component	Weight Percent					
	In A[a]		In B[b]		In C[c]	
	Theoretical	Found	Theoretical	Found	Theoretical	Found
Carbon	61.1	61.1	68.7	69.0	49.7	49.4
Hydrogen	9.2	10.0	10.8	10.9	9.4	9.5
Phosphorus	8.3	7.6	5.7	5.6	8.0	7.4
Bromine	21.4	21.2	14.8	14.3		
Iodine					32.8	32.8

[a]A = benzyltributylphosphonium bromide, $C_{19}H_{34}PBr$.
[b]B = benzyltrioctylphosphonium bromide, $C_{31}H_{58}PBr$.
[c]C = tetra-*n*-butylphosphonium iodide, $C_{16}H_{36}PI$.

hydroxylamine hydrochloride and pyridine in ethyl alcohol to form *m*-nitrobenzaldoxime. The oxime was then hydrogenated in methanol at 400 to 500 psi and 80 to 100°C for 4 hr over Raney nickel. The reactions are



Elemental analysis of the product, a clear oil that crystallized on being cooled (mp, 39 to 40°C), gave the following results (in weight percent):

Component	Theoretical[a]	Found
Carbon	68.8	68.6
Hydrogen	8.25	8.40
Nitrogen	22.9	22.4

[a] $C_7H_{10}N_2$.

11.1.c Tri-*n*-butylsulfonium Iodide

H. L. Holsopple

Fundamental studies by the Chemistry Division required the use of a quantity of pure tri-*n*-butylsulfonium iodide. The compound was prepared by the interaction of *n*-butyl iodide and di-*n*-butyl sulfide in nitromethane according to the reaction

$$C_4H_9I + (C_4H_9)_2S \longrightarrow (C_4H_9)_3SI \ .$$

Batches of the reactants were sealed in glass ampuls for eight weeks at moderate temperatures. The theoretical and actual compositions (in weight percent) of the resulting compound are as follows:

Component	Theoretical[a]	Found
Carbon	43.6	43.3
Hydrogen	8.24	8.55
Sulfur	9.71	9.41
Iodide	38.4	38.8

[a] $C_{12}H_{27}SI$.

The details of this synthesis, together with the infrared spectrum of tri-*n*-butylsulfonium iodide, are given in a formal report.[3]

11.1.d Alkyl Phosphates

H. L. Holsopple

Two esters, triisoamyl phosphate (200 g) and tri-*n*-amyl phosphate (110 g), were synthesized and purified for use in Chemical Development B section of the Chemical Technology Division. The compounds were prepared by causing the appropriate amyl alcohol to react with phosphorus oxychloride and pyridine in benzene according to the reaction

$$3C_5H_{11}OH + POCl_3 + 3C_5H_5N \longrightarrow 3C_5H_5NHCl + (C_5H_{11})_3PO_4 \ .$$

The products were purified by fractional distillation, and their purities were established from infrared spectra and elemental analysis. The details of the preparations are given elsewhere.[4]

11.1.e ^{14}C-Labeled Propionic Acid

H. L. Holsopple

As a result of a customer's allegation (Sect. 9.2.b), the Isotopes Division requested the preparation of a quantity of ^{14}C-labeled propionic acid from their $Ba^{14}CO_3$ product. The preparation consisted in generating $^{14}CO_2$ from the $Ba^{14}CO_3$ by means of perchloric acid. The $^{14}CO_2$ was then caused to react with a Grignard reagent prepared from ethyl bromide,

$$CH_3CH_2MgBr + {}^{14}CO_2 \longrightarrow CH_3CH_2{}^{14}COOH \ .$$

Very little (<0.05%) of the original radioactivity was ether extractable from a basic solution of the residue from the reaction. Of the total radioactivity, <0.35% remained in the perchloric acid after the generation of $^{14}CO_2$. From the 2.0 millimoles of $Ba^{14}CO_3$ (116 mc) used, the yield of ^{14}C-labeled propionic acid was 45%, which is satisfactory.

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[3] H. L. Holsopple, *Synthesis of Tri-n-butylsulfonium Iodide*, ORNL-TM-1607 (Aug. 22, 1966).

[4] H. L. Holsopple, *Synthesis of Tri-Isoamyl Phosphate*, ORNL-TM-1487 (Apr. 13, 1966).

11.1.f 1,3-Ethylpentyl-4-ethyloctyl Bromide

H. L. Holsopple

1,3-Ethylpentyl-4-ethyloctanol (200 g) was brominated for Chemical Development C section of the Chemical Technology Division by use of hydrobromic acid in the presence of sulfuric acid. The product was fractionally distilled in a centrifugal molecular still (type CMS-5, Consolidated Vacuum Corporation).

Attempts to brominate the C_{17} alcohol with phosphorus tribromide were unsuccessful. Infrared analysis showed that instead of the expected reaction,

$$3ROH + PBr_3 \longrightarrow 3RBr + (HO)_3P ,$$

another reaction occurred by which a dialkyl phosphite and an alkyl halide were formed:

$$3ROH + PBr_3 \longrightarrow (RO)_2POH + RBr + 2HBr .$$

11.1.g Barium *p*-Ethylbenzenesulfonate

H. L. Holsopple

p-Ethylbenzenesulfonic acid (~200 g) was prepared by the sulfonation of ethylbenzene with chlorosulfonic acid. The barium *p*-ethylbenzenesulfonate was formed by the addition of an equivalent quantity of barium carbonate. Infrared and NMR spectra showed no indication of ortho substitution; hence the product was considered to be essentially 100% 1,4-isomer. Results of elemental analysis of two batches of barium *p*-ethylbenzenesulfonate (in weight percent) are as follows:

Component	Theoretical[a]	Found	
		Batch 1	Batch 2
Carbon	37.8	38.8	37.0
Hydrogen	3.57	3.65	3.64
Sulfur	12.6	12.4	12.6
Barium	27.1	26.8	27.3

[a] $C_{16}H_{18}S_2O_6Ba$.

11.1.h Reports on Organic Compounds Synthesized

H. L. Holsopple

In addition to reports referred to in the preceding sections, four reports[5–8] were prepared that present details of organic preparations described briefly in previous Analytical Chemistry Division annual progress reports.

11.2 ORGANIC PURIFICATIONS

H. L. Holsopple

For the Radiation Physics Group of the Health Physics Division, the following compounds were purified by suitable procedures (sublimation, crystallization, or distillation): benzene, bromobenzene, *o*-bromotoluene, *o*-chlorobenzene, o-chlorotoluene, *m*-dichlorobenzene, *p*-dichlorobenzene, hexafluorobenzene, naphthalene, nitrobenzene, and phenanthrene.

In addition, a quantity of 1,1,2,2-tetrabromoethane was fractionally distilled for use in density-gradient studies (Sect. 4.6.a).

For the Neutron Physics Division, moisture was removed from 4 gal of mineral oil by agitating molten sodium through the oil.

By boiling-point measurements, the purities of two samples of dimethyl sulfoxide were estimated for the Reactor Chemistry Division.

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[5] H. L. Holsopple, *Synthesis of 3,9-Diethyltridecyl-6-amine*, ORNL-TM-1314 (Nov. 5, 1965).

[6] H. L. Holsopple, *Synthesis of α,α′-Dipiperidyl*, ORNL-TM-1470 (Mar. 30, 1966).

[7] H. L. Holsopple, *Synthesis of Methylmethanesulfonate*, ORNL-TM-1486 (Apr. 5, 1966).

[8] H. L. Holsopple, *Synthesis of 2,6-Dioximinocyclohexanone*, ORNL-TM-1510 (Apr. 28, 1966).

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 Divisions																		For Others						
	Scientists	Technicians	Analytical Chemistry	Biology	Chemical Technology	Chemistry	Electronuclear	Health	Health Physics	Inspection Engineering	Instrumentation and Controls	Isotopes	Metals and Ceramics	Neutron Physics	Operations	Physics	Reactor	Reactor Chemistry	Solid State	Thermonuclear	Argonne National Laboratory	EGCR	General Electric	Miscellaneous	TVA	Y-12	Total
Bioanalytical Development	2	3		440	20,830																						21,270
Mass Spectrometry Laboratory	2	4	50		624	70	45		60			7,707	2,010		14	190	1,890	275	809	80				5634		1565	21,023
Optical and Electron Microscopy	3	2	5	49	584	47			29				219			7	5	501	2	41		24		15			1,528
Process Analyses																											
General Analyses Laboratories	11	23	176	26	29,100	1676			350			363	9,715	92	544	18	2,444	4,268	802					560			50,134
High-Level Alpha Radiation Laboratories	9	13			7,729	3			10			503	20	3			29	141						31		6	8,475
High-Radiation-Level Analytical Laboratories	11	12	188		6,802							1,324	487		6,014		2,517	557			162						18,051
Radioisotopes-Radiochemistry Laboratories	10	17	133		12,529	63			1,852	35	45	9,655	373	46	8,612		10,969	11,546	906				1000	33	40		57,837
Spectrochemical Analyses Laboratory (Y-12)	2	1	329		856		37	17,844				11,589	1,110	74			1,105	1,492	1543	148						505	36,632
TRU Mass Spectrometry	1	1	543		364	55						538	375	20			468							12		5	2,380
X-Ray and Spectrochemical Analyses	8	5	348	68	11,817	933			29,701			2,768	10,331	257	271	67	2,307	4,159	1771					1041			65,839
Total	59	81	1772	583	91,235	2847	82	17,844	32,002	35	45	34,447	24,640	492	15,455	282	21,734	22,939	5833	269	162	24	1000	7326	40	2081	283,169

12. Mass Spectrometric Analyses

A. E. Cameron

12.1 MASS SPECTROMETRY SERVICE LABORATORY

J. R. Sites
E. J. Spitzer

The isotopic analysis of any element having more than one stable isotope is routine in the Mass Spectrometry Service Laboratory. Some 7700 results were reported to the Isotopes Division, most of these being for separated stable isotopes.

This laboratory assisted in experiments to determine the neutron-capture cross section of ^{171}Tm. This work required the isotopic analysis of a 0.1-μg mixture of Tm and Yb. Although the mass spectrum contained several isobars, the isotopic patterns of Tm and Yb were separated and their isotopic abundances were determined. Assistance was also given in the determination of the neutron-capture cross section of ^{63}Ni by isotopic analysis of irradiated Ni.

A double mass analysis was done on a sample of KCl that had been irradiated at the Savannah River Plant in what was thought to be a neutron flux of 1×10^{15} neutrons cm^{-2} sec^{-1}. However, from the isotopic abundances of both ^{40}K and ^{36}Cl, the flux was determined to be only 1.6×10^{14} neutrons cm^{-2} sec^{-1}.

Isotopic abundances were determined in a set of samples that possibly contained fission-product Mo from graphite surfaces in the MSRE fuel system.

The two gas mass spectrometers continued to provide supporting analyses for many ORNL projects.

12.2 TRANSURANIUM MASS SPECTROMETRY LABORATORY

R. E. Eby

The Transuranium Mass Spectrometry Laboratory made 63% more analyses than in the previous year. About half of these were for groups making burnup or various other types of studies relative to reactor operations. One-quarter of the analyses were made on isotopes of U and Pu separated by the Isotopes Division. The remaining results were on many different types of samples. Isotopic analyses were made on all the elements from thorium to californium and also on some radioactive lighter elements, for example, europium and strontium.

With the cooperation of the High-Level Alpha Radiation Laboratories (Sect. 14.3) and the High-Radiation-Level Analytical Laboratories, a technique was developed to electrodeposit samples onto short lengths of 5-mil platinum or tungsten wires. These wires, when received by the Transuranium Mass Spectrometry Laboratory, are placed in a rhenium canoe, which is then pinched shut to hold the wire. This technique has the advantages that smaller samples can be used, the possibility of spreading contamination is reduced, the shape of the mass spectrometric peaks is improved, and some impurities cause less problems. About half the samples are now received in this manner.

13. Spectrochemical Analysis Laboratory

A. E. Cameron J. A. Carter

The Spectrochemical Analysis Laboratory reported some 36,600 results on about 2000 samples, which is an increase of 11% in samples and 23% in results. The Isotopes Division supplied about 10% more samples than last year. The stable isotopes of the following 39 elements were analyzed for trace impurities: B, Ba, Br, C, Ca, Cd, Ce, Cl, Cr, Cu, Dy, Eu, Ga, Gd, Ge, Hf, Hg, Fe, K, Li, Lu, Pb, Pd, Mg, Mo, Nd, Ni, S, Se, Si, Sm, Sn, Sr, Ti, Tl, W, Yb, Zn, and Zr. The Health Division continued to supply a large number of environmental air samples. The Reactor Chemistry Division submitted more than 350 samples of Pb and Bi from the experimental program to clean up MSRE salts with molten Pb and Bi; the samples were analyzed for Be, Ce, Eu, La, Li, Nd, Sm, and Th.

Numerous methods and techniques were modified to analyze a variety of alloys and mixtures. For example, modified procedures employing the Paschen Direct Reader were used to determine Al in 5 *M* $LiNO_3$ and Ca in concentrations as low as 5 ppm in doped AgCl-NaCl crystals. Unusual alloy-base materials such as Er, Ho, Nb, Ta, Tb, Sc, V, W, and Zr were analyzed for major and minor constituents.

14. Process Analyses

L. T. Corbin

14.1 HIGH-LEVEL ALPHA RADIATION LABORATORIES

J. H. Cooper

The High-Level Alpha Radiation Laboratories reported 8475 results; about 90% of these were for the Chemical Technology Division. Work continued on the determination of U and Pu in samples from Plutonia Sol-Gel Program. Fired microspheres formed by this process were not only analyzed for major components but the physical properties, surface area, mercury density, and porosity were also measured.

The technique of preparing samples for mass spectrographic analysis was improved. Formerly, an aliquot of a solution of the sample was deposited directly on a tantalum filament and evaporated. Often, undesirable solids were also left on the filament. The present procedure is to electrodeposit the element to be determined (e.g., Am, Cf, Cm, Pu) onto a fine platinum wire from a very dilute solution of nitric acid. The wire is cut into short pieces, and the pieces are placed in a rhenium canoe, which is then crimped shut and inserted in the mass spectrograph (Sect. 12.2).

The High-Level Alpha Radiation Laboratories participated in an interlaboratory-check analysis of 11 standard solutions of plutonium prepared by the New Brunswick Laboratory. For 10 of the 11 samples, the results from our laboratory agreed to within ±0.5% of the true value.

The analytical laboratories of the Transuranium Processing Plant were fully equipped and staffed with 15 trained persons; the laboratories now offer continuous service on shifts.

14.2 GENERAL ANALYSES LABORATORIES

W. R. Laing

Development work has continued on the new vacuum-fusion analyzer. Two persistent problems

were solved. The brass bellows valves downstream from the mercury-diffusion pump leaked after amalgamation of the bellows-solder joints. The brass valves were replaced by stainless steel valves. The glass exit valve of the Toepler pump leaked when exposed to a pressure differential of 1 atm. This valve was replaced with a solenoid-assisted, Teflon, O-ring valve. Results obtained from Nb and Zr samples agree with those obtained with the National Research Corporation model 912 vacuum-fusion analyzer.

The Leco model 589-400 carbon analyzer was modified according to the method of Merkle and Graab.[1] As a result, it is possible to adjust the amplifier gain and thus to achieve a fourfold increase in sensitivity. By adjustment of both the amplifier gain and the attenuation control, carbon was determined in amounts from 1 μg to 5 mg.

For the measurement of the density of individual pyrolytic-carbon-coated microspheres, a density-gradient technique (Sect. 4.6.a) was adapted. These spheres (weight, 0.2 to 2.0 mg; diameter, 0.5 to 1.0 mm) were dropped into a glass column that contained a liquid having a density gradient. The relative positions of the samples and density standards were measured with a cathetometer. Densities can be measured to ± 0.001 g/cc. Also, dual helium densitometers were built for measuring the density of pyrolytic-carbon-coated particles. With them the density of one sample can be measured while a second sample is being evacuated. One person is able to operate both densitometers.

A modification of the ammonium molybdate spectrophotometric method for determining silicon was put into use. It differs in two ways from that previously used, in which the test solution was not heated and the spectrophotometric measurement was made on the aqueous phase. The sample, acid, and ammonium molybdate are heated to accelerate the formation of the silicomolybdate complex. After the complex is extracted into 2-ethylhexanol, it is stripped into water, and the final color is developed in an aqueous system.

A 15,000-psi, hydraulically operated mercury porosimeter was obtained from the American Instrument Company. Except for some breakage of the sight glasses, the instrument has performed satisfactorily in measuring the porosity of a variety of materials. An existing computer program was adapted for use with this instrument.

An ORNL model Q-2792 controlled-potential dc polarograph was used to determine Cd in dilute HCl solutions in the concentration range from 1 to 5 μg/ml. A relative standard deviation of the results for 300 samples was 1%.

Large amounts of mercury are accumulated as waste from coulometers, polarographs, and the Dyna-Caths (mercury-cathode separators). In the past, this mercury had been purified by repeated washings and distillation. Cochran[2] developed a countercurrent liquid-liquid extraction technique that proved useful for purifying radioactively contaminated mercury. This procedure produces a product sufficiently pure for use in coulometric and polarographic analyses. Storage of the purified mercury under argon prevents the formation of an oxide film on its surface.

A biochemistry laboratory for service analyses was put into operation in August 1966. This laboratory analyzes tRNA from the macromolecular separations work of the Chemical Technology and Biology Divisions. A work load of 3000 to 4000 analyses per month is anticipated.

Several other new or modified methods were developed. They are for the determination of rhenium in Re-W alloys; primary, secondary, and tertiary amines in their mixtures; oxalate in solutions that contain HCl; I and S in KBr crystals; I^- and I_2 in charcoal adsorber beds; Al_2O_3 in Al; dissolved H_2 and O_2 in water; and the components of Kennametal bearings.

The General Analyses Laboratories reported 50,134 results.

14.3 HIGH-RADIATION-LEVEL ANALYTICAL LABORATORIES (HRLAL)

C. E. Lamb

Operation of the High-Radiation-Level Analytical Laboratories (HRLAL) progressed, as planned, to a routine activity. Principal support was for the MSRE of the Reactor Division and the Curium Program of the Isotopes and Chemical Technology Divisions. Because the Curium Program and the

[1] E. J. Merkle and J. W. Graab, "Use of Carbon Dioxide to Calibrate Instruments for the Determination of Carbon Below 50 Parts per Million in Metals," *Anal. Chem.* 38, 159 (1966).

[2] D. A. Cochran, "Purification of Mercury by Counter-Current Liquid-Liquid Extraction," HW-84509 (Nov. 16, 1964).

first seven MSRE power runs were completed, the HRLAL staff was reduced by 30%. A glove-box facility was designed for installation in the HRLAL; it is to contain equipment for measuring the physical properties of solid samples from the Chemical Technology Division's Plutonia Sol-Gel Program.

Few problems were encountered in the analysis of radioactive samples following incremental power increases to the full power level of the MSRE.[3] The procedure for the controlled-potential coulometric titration of U was modified to decrease the time required for the reduction of U(VI) and Cu(III) from ~15 to ~5 min and to correct bias in the method. These improvements were achieved by plotting the readout voltage vs the cell current on an *X-Y* recorder and by terminating the titration at 50 instead of 5 μa. Since the titration is only ~99% complete at 50 μa, the remaining readout voltage is obtained by extrapolating an expanded plot of readout voltage vs cell current from 50 to 0 μa. An additional advantage is the elimination of the difficulties involved in establishing the background cell current.

The apparatus developed by the Methods Development Group for measuring the oxide content of 50-g samples of MSRE salt (Sect. 3.1.a) was installed in Cell 3 of the HRLAL. Simulated nonradioactive MSRE salt samples and tin oxide standards were analyzed to check the apparatus and to train persons in its use. The remotely controlled manipulations were performed adequately, and the results were satisfactory. Six 50-g fuel-salt samples were analyzed; their oxide contents were 47, 49, 50, 55, 59, and 66 ppm.

Several MSRE samples were received with a silver and an INOR-8 wire coiled onto the stainless steel cable between the latch and the ladle. The latch, wires, and cable were each separated and prepared for radiochemical analysis.

The quality control program for MSRE analyses, initiated prior to precritical testing, was continued. Limits of error more realistic than those set initially were established for the methods in use.

Malfunctions developed in the equipment used to analyze MSRE samples. They included a broken spring on the Mixer-Mill, a jammed Harvard infusion pump, a broken tubing cutter, a jammed end plug, and a transport container. Most of these difficulties were eliminated by remotely controlled repair or replacement; however, the Mixer-Mill required a 10-min cell entry.

Equipment recently installed in the HRLAL cells includes a quartz combustion tube furnace for carbon determination by burning the sample in oxygen and collecting the effluent gases for the measurement of carbon dioxide, a heat gun for melting waste polyethylene equipment to reduce the volume of solid waste discharged to the burial ground, and a glass mercury-displacement pipetter that uses volumetric pipets to deliver 3 to 15 ml of solution (Fig. 14.1).

Fig. 14.1. In-Cell Macro Pipetter.

[3]R. B. Briggs, "Development and Evaluation of Equipment and Procedures for Analyzing Radioactive MSRE Salt Samples," *MSR Program Semiann. Progr. Rept. Feb. 28, 1966*, ORNL-3936, pp. 165–168.

One of two sealed glove boxes was equipped with an apparatus for releasing gases contained in samples from the Plutonia Sol-Gel Program until the planned glove-box facility is in service. The second glove box was equipped for the dissolution of tantalum-clad samples from the Argonne National Laboratory; the samples are analyzed for Cu, Mg, Pu, and U.

14.4 RADIOISOTOPES-RADIOCHEMISTRY LABORATORIES

E. I. Wyatt

The analytical work performed by the Radioisotopes-Radiochemistry Laboratories the past year covered many programs. The types of analyses were widely diversified. Three persons were engaged solely in neutron-flux determinations and neutron activation analysis. Service irradiation of biological specimens has increased. Radiochemical and radiometric analysis in support of in-pile and out-of-pile meltdown experiments for the Reactor Chemistry and Reactor Divisions have provided most of the work.

Determination of the fission-product distribution in the fused salt, in the graphite moderator, and in other components of the MSRE provided some interesting challenges. To establish the power levels in the reactor, radionuclides having half-lives of less than 3 hr were measured. It was necessary to determine ^{99}Mo, adsorbed on the graphite, after five to seven half-lives. An isotope-dilution procedure was devised for use in hot cells to obtain a sample from the graphite moderator for the mass spectrometric analysis of Mo.

A new radiochemical method for tellurium was developed that uses phenanthroline as a precipitant. It was found that ceric iodate has some unusual properties as an adsorbing medium for various cations. For example, it is useful in separating many fission products from neptunium and cobalt, which are not adsorbed. The ceric iodate work and other recently developed techniques useful for radiochemical separations in hot cells were described in a paper presented by Rickard.[4]

The half-life of ^{133}Ba, reported in 1961 to be 10.7 ± 0.2 years,[5] was reconfirmed after the decay had been followed for a total of nine years. The half-life of ^{124}Sb was redetermined to be 60.3 ± 0.2 days rather than 60.9 days, the value reported previously.[6]

Tellurium-128 enriched to 97% was irradiated with neutrons to prepare ^{129}Te free of other radionuclides. The ^{129}Te was further purified chemically, and its decay was followed for five half-lives on a beta proportional counter with a 30-mg/cm^2 aluminum absorber. A least-squares calculation gives a half-life value of 34.5 ± 0.1 days.

The decay properties of ^{98}Tc and the fission yield of ^{98}Tc were determined by C. F. Goeking, Jr.,[7] in partial fulfillment of the requirements for a Master's degree in chemistry.

14.5 HIGH-RADIATION-LEVEL ANALYTICAL FACILITY (HRLAF)

L. G. Farrar

Analyses of highly radioactive alpha-emitting materials associated with fission products accounted for 40% of the work performed in the High-Radiation-Level Analytical Facility. About half the cell space in the facility was used by the Radioisotopes-Radiochemistry Laboratories; the high-radiation-level spectrochemical analyses work area was expanded into a two-cell operation.

Dissolution and instrumental analyses of curium oxide samples that weighed ~200 mg each were performed without incident. The absolute filters in the off-gas system of the HRLAF were replaced, and the filter frames were gasketed and regrouted to upgrade the filtering efficiency of the system.

Routine inspection revealed acid damage and deterioration of the off-gas header of the main cell for the HRLAF. A temporary coating of urethane foam was applied to prevent loss of containment. Drawings and specifications for

[4] R. R. Rickard, D. R. Mathews, and E. I. Wyatt, "Special Procedures Employed in Radiochemical Analyses in Remote Systems," presented at the Tenth Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Sept. 27–29, 1966.

[5] E. I. Wyatt, S. A. Reynolds, T. H. Handley, W. S. Lyon, and H. A. Parker, "Half-Lives of Radionuclides II," *Nucl. Sci. Eng.* 11(1), 74 (1961).

[6] H. W. Wright, E. I. Wyatt, S. A. Reynolds, W. S. Lyon, and T. H. Handley, "Half-Lives of Radionuclides I," *Nucl. Sci. Eng.* 2(4), 427 (1957).

[7] C. F. Goeking, Jr., *Determination of the Decay Properties of Technetium-98 and Its Independent Yield in Thermal Neutron Induced Fission of Uranium-235* (M.S. thesis), University of Tennessee, August 1966.

replacement of the off-gas duct have been completed.

Containment in the cell-access area was improved by completion of the loading-dock enclosure and by addition of space heaters that do not require outside ventilation.

The procedures for removing solid waste were improved by adding a garbage-disposal unit to grind glass and by melting down plastic containers to smaller volumes. Decontamination problems in the HRLAF were reduced by use of disposable lead shielding containers whenever possible for transfer of dilutions. Manipulators and in-cell equipment are being decontaminated within the work cells by pressure sprays and dip techniques.

Routine hot-cell maintenance has included the repair of 127 manipulators and the decontamination of six work cells. The chain drives of the two in-cell conveyors in the HRLAF were replaced, and minor adjustments were made in the linkages. Excess equipment stored under the cell work pans was removed to the burial ground. A new analytical balance was placed in Cell 7.

14.6 QUALITY CONTROL

G. R. Wilson

From July 1965 through June 1966, only two laboratories participated in the Quality Control Program. The quality level of the General Analyses Laboratories remained about the same. The quality level of the General Hot Analyses Laboratories decreased due to the initiation of the MSRE Quality Control Program in June 1965. In this program, a mixture of the following elements in 0.5 *M* H_2SO_4 are determined: Be, Cr, Cu, Fe, Li, Ni, U, and Zr. The results of the spectrophotometric determination of Ni in low concentration (5 to 8 μg/ml) had a positive bias for three of the four quarters, although the 2*S* values calculated from the results were within the established limits. The results of the amperometric determination of Zr at a concentration level of 1 mg/ml showed a positive bias for two of the four groups of analysts in the General Hot Analyses Laboratories. When these results are omitted from the calculation, the quality level of the remaining control determinations in the General Hot Analyses Laboratories is 93.6%.

Table 14.1 shows the distribution of the control tests by laboratory; Table 14.2 gives the number of control tests and their specific characteristics.

Table 14.1. Distribution by Laboratories of Control Tests for July 1965–June 1966

Laboratory	Number of Control Results		Quality Level (%)[a]	
	Total	Outside Fixed Limits	1965	1966
General Hot Analyses	2016	284	92.2	85.9
General Analyses	2907	80	97.3	97.2
Total	4923	364	96.4	92.6

[a]Control results within prescribed 2*S* limits.

Table 14.2. Distribution by Methods of Control Results for July 1965–June 1966

Type of Method	Constituent	Number of Control Programs		Number of Control Results	
Amperometric	Chromium	1		253	
	Zirconium	1	2	249	502
Colorimetric (spectrophotometric)	Aluminum	1		103	
	Chromium	2		162	
	Iron	3		482	
	Molybdenum	1		141	
	Nickel	3		361	
	Thorium	2		321	
	Uranium	2	14	480	2050
Coulometric	Uranium	3	3	1325	1325
Fluorometric	Uranium	2	2	313	313
Gravimetric	Carbon	2	2	382	382
Photoneutron	Beryllium	1	1	113	113
Potentiometric	Uranium	1	1	15	15
Spectrographic	Lithium	1	1	16	16
Volumetric	Aluminum	1		127	
	Nitrate	1		34	
	Sulfate	1	3	46	207
Total			29		4923

Part C. ORNL Master Analytical Manual

15. ORNL Master Analytical Manual

M. T. Kelley

Helen P. Raaen

15.1 CUMULATIVE INDEXES TO THE *ORNL MASTER ANALYTICAL MANUAL*

The cumulative indexes to the *ORNL Master Analytical Manual* were updated to indicate the changes made in the content of the *Manual* during 1965. The updated indexes are titled "Indexes to the *Oak Ridge National Laboratory Master Analytical Manual* (1953–1965), TID-7015 (Indexes), Revision 3." The document is available from the Clearinghouse for Federal Scientific and Technical Information, National Bureau of Standards, U.S. Department of Commerce, Springfield, Virginia; the price is $1.75. The indexes were issued as a companion document to Supplement 8 of TID-7015.

The computer work required for the preparation of the updated indexes was supervised by Ann K. Haas.[1]

[1]Technical Information Division.

15.2 MAINTENANCE OF THE *ORNL MASTER ANALYTICAL MANUAL*

The eighth supplement to the reprinted form of the *ORNL Master Analytical Manual* was published. It contains the new methods and the revisions issued in 1965. This supplement, designated TID-7015 (Suppl. 8), is available from the Clearinghouse for Federal Scientific and Technical Information, National Bureau of Standards, U.S. Department of Commerce, Springfield, Virginia, at a price of $4.00.

Altogether, 12 new methods were added to the *Manual;* 10 of these were for the purpose of record only. Revisions were made to three methods. Also, the Table of Contents for the *Manual* was updated. (See "Presentation of Research Results.")

A survey was made to determine what methods now contained in the *Manual* are no longer used, what revisions to existing methods are needed, and what new methods are required for current or anticipated analytical work. On the basis of the survey, method-writing assignments were made to persons in the Analytical Chemistry Division.

Part D. Activities Related to Educational Institutions

16. Activities Related to Educational Institutions

Certain activities come into existence when the interests, purposes, and problems of educational institutions and of the Oak Ridge National Laboratory overlap in the area of analytical chemistry. The more formal of these activities include discussions with consultants who are also university faculty members, and thesis-research programs for graduate students. By less formal arrangements, faculty members engage in research in the Division under the ORAU-ORNL Research Participant Program, and students participate in the Summer Student Trainee Programs and in the Loanee Program for special work. Often, a number of the Alien Guests in residency for work in the Division are from foreign educational institutions or from foreign laboratories whose programs intersect those of educational institutions.

The nature and the mutual benefits of these activities are indicated throughout this report. Of the approximately 150 formal presentations of the research of the Division made during the past year, about 10% reflect cooperative ventures with educational institutions. These presentations are enumerated in the "Presentation of Research Results" section, and the work is discussed in the sections of the report indicated.

16.1 CONSULTANTS

The consultants who have worked under subcontract in collaboration with our division are:

J. A. Dean
University of Tennessee

P. H. Emmett
The Johns Hopkins University

H. V. Malmstadt
University of Illinois

Gleb Mamantov
University of Tennessee

G. H. Morrison
Cornell University

M. L. Moss
Institute of Muscle Disease

A. O. Nier
University of Minnesota

L. B. Rogers
Purdue University

W. B. Schaap
Indiana University

H. H. Willard
University of Michigan

16.2 ORAU RESEARCH PARTICIPANTS

Three ORAU Research Participants were with us in the summer of 1966.

L. C. Hall, Professor of Chemistry, Vanderbilt University, worked with *A. E. Cameron* preparatory to the acquisition of a mass spectrograph at Vanderbilt University. Professor Hall plans to use the spectrograph in research on trace analyses in biological systems, water, and semiconductors.

S. E. Manahan, Assistant Professor of Chemistry, University of Missouri, used radioisotope tracer techniques to study the electron exchange between Cu(I) and Cu(II) in acetonitrile (Sect. 1.19). Also, he assisted in evaluating the performance of an electromechanical, mercury-drop-time controller (Sect. 1.6). This work was done in cooperation with members of the staff of the Analytical Instrumentation Group and the Nuclear and Radiochemical Analyses Group.

L. P. Turner III, Assistant Professor of Chemistry, University of Tennessee, investigated the pyrolytic gas chromatography of purines and pyrimidines with *A. S. Meyer* (Sect. 5.3).

16.3 ORNL POSTDOCTORAL PROGRAM

Under the ORNL Postdoctoral Program, **C. A. Burtis, Jr.** (Ph.D. in Biochemistry, Purdue University, 1966) came to our division in October on a two-year assignment, to work in the Bioanalytical Development Group with *Gerald Goldstein.*

16.4 GRADUATE THESIS RESEARCH PROGRAMS

Under three separate programs, three graduate students completed their thesis research in the Analytical Chemistry Division.

16.4.a Educational Assistance – Ph.D. Program

W. D. Shults, of our division, completed the Ph.D. program during 1966 and was awarded the Ph.D. degree in chemistry by Indiana University in June. His thesis is titled "Controlled-Potential Differential DC Polarography" (Sect. 1.1). *M. T. Kelley* was a member of W. D. Shults' Ph.D. Advisory Committee.

16.4.b Oak Ridge Resident Graduate Program of the University of Tennessee

C. F. Goeking, Jr., a member of the Division until September 21,[1] was awarded the M.S. degree in chemistry by the University of Tennessee in August. His thesis research was done partly in the Radioisotopes-Radiochemistry Laboratories under the supervision of *E. I. Wyatt* and partly in the Chemistry Division with G. D. O'Kelley as his Faculty Research Advisor. The title of his thesis is "Determination of the Decay Properties of Technetium-98 and Its Independent Yield in Thermal Neutron Induced Fission of Uranium-235" (Sect. 14.4).

16.4.c Oak Ridge Graduate Fellowship Program

J. R. Stokely, Jr., completed the Ph.D. program during 1966 and was awarded the Ph.D. degree in anayltical chemistry by Clemson University in May. His thesis is "Solvent Extraction and Gas Chromatography of Metal Trifluoroacetylacetonates." *J. C. White* was the Laboratory Advisor to J. R. Stokely, who became a member of our division on February 1.

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[1]Present address: Shell Development Company, Houston, Tex.

16.5 ORNL LOANEES

H. W. Jenkins, a graduate student and assistant in chemistry with *Professor Gleb Mamantov* at the University of Tennessee, is continuing his work toward the M.S. degree in the Research Group with *D. L. Manning*. He is studying the behaviors of reference electrodes in the electroanalytical chemistry of fluoride melts to develop a new and more reliable reference electrode for such use.

F. L. Whiting, a graduate student in the Department of Chemistry with *Professor Gleb Mamantov* at the University of Tennessee, is beginning his doctoral thesis research in the Research Group with *J. C. White* and *J. P. Young*. He is measuring simultaneously the absorbance spectra of U(IV) and U(III) and the electrolytic reduction of the U(IV) to U(III) in alkali-metal fluoride solvents at ~500°C.

16.6 SUMMER STUDENT PROGRAMS

In the summer of 1966, seven students participated in summer student programs.

16.6.a ORAU Student Trainee Program

J. A. Bell, a senior at Presbyterian College, Clinton, South Carolina, worked in the Methods Development Group with *P. F. Thomason* and *W. D. Shults* to evaluate "cation-" and "anion-specific" electrodes (Sect. 4.3).

K. B. Green, a senior at William Jewell College, Liberty, Missouri, worked with *Gerald Goldstein* in the Bioanalytical Development Group on the chromatographic separation of tRNA mixtures (Sect. 5.1.h).

16.6.b ORNL Student Trainee Program

R. L. Caldwell, a senior at the Tennessee Technological University, Cookeville, analyzed materials with the MS-7 mass spectrograph under the direction of *J. R. Sites* of the Mass Spectrometry Service Group and *J. A. Carter* of the Spectrochemical Analysis Group.

16.6.c ORNL Student Technical Assistant Program

J. B. Pressly, a senior at the University of Tennessee, worked with *E. I. Wyatt* in the Radioisotopes-Radiochemistry Laboratories. He used multichannel analyzers to put data on tapes for transfer to the computers.

R. B. Shaffer, a first-year graduate student at the University of Tennessee, worked with *J. E. Attrill* of the Bioanalytical Development Group and also with N. G. Anderson of the Biology Division on the electrophoresis of proteins.

E. S. Wolfe, a first-year graduate student at the University of North Carolina, worked on the development of high-precision methods of dc polarography (Sect. 1.12). This work was done with *D. J. Fisher, R. W. Stelzner,* and *W. L. Belew* in the Analytical Instrumentation Group.

16.6.d ORNL Summer Student Employee Program

Lenora A. Wells, a junior at Furman University, Greenville, South Carolina, did clerical and stenographic work in the offices of the Analytical Chemistry Division under the direction of *H. R. Beatty*.

16.7 ALIEN GUESTS IN RESIDENCY

Four scientists from four countries either were during the year or now are guests in the Analytical Chemistry Division.

Guest	Sponsor	Division Group	See Section
M. M. Al-Kayssi College of Education, Baghdad, Iraq	International Atomic Energy Agency	Nuclear and Radiochemical Analyses Group	
T. M. Florence Australian Atomic Energy Commission Research Establishment, Sutherland, New South Wales	Australian Atomic Energy Commission	Analytical Instrumentation Group	1.8
		Methods Development Group	2.4, 2.5, 4.2.b, 4.2.c
Aart Jurriaanse Atomic Energy Board, Pretoria, Republic of South Africa	South African Atomic Energy Board	Nuclear and Radiochemical Analyses Group	9.4.e
Jun-Lan Wang *(Mrs. Kian-Chu Li)* Institute of Nuclear Science, National Tsing Hua University, Hsinchu, Taiwan, Republic of China	International Atomic Energy Agency	Nuclear and Radiochemical Analyses Group Radioisotopes-Radiochemistry Laboratories	

Presentation of Research Results

Several of the presentations listed below are made jointly with members of other divisions. In these cases the member(s) of the other division(s) is indicated by a single asterisk.

PUBLICATIONS

Books, Theses, Monographs

	AUTHOR(S)	TITLE	PUBLISHER
1	Fisher, D. J.	"Discussion Following Chapter III," p. 67 in *Proceedings of the Robert A. Welch Foundation Conferences on Chemical Research VII. Modern Developments in Analytical Chemistry, November 18–20, 1963, Houston, Texas*	The Robert A. Welch Foundation, Houston, Tex., 1964
2	Fisher, D. J., W. L. Belew, M. T. Kelley	"Recent Developments in D.C. Polarography," pp. 89–134 in *Polarography 1964*, vol. 1 (Proceedings of the Third International Congress, Southampton), ed. by G. J. Hills	Macmillan, London, 1966
3		"A Simple Quasi-Reference Electrode. Applications in Controlled-Potential Polarography and Voltammetry and in Chronopotentiometry," pp. 1043–1059 in *Polarography 1964*, vol. 2 (Proceedings of the Third International Congress, Southampton), ed. by G. J. Hills	Macmillan, London, 1966
4	Goeking, C. F. Jr.	*Determination of the Decay Properties of Technetium-98 and Its Independent Yield in Thermal Neutron Induced Fission of Uranium-235* (M.S. thesis)	University of Tennessee, August 1966
5	Manning, D. L., J. M. Dale, G. Mamantov[1]	"Voltammetry in Molten Fluorides," pp. 1143–1151 in *Polarography 1964*, vol. 2 (Proceedings of the Third International Congress, Southampton), ed. by G. J. Hills	Macmillan, London, 1966

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

6 Raaen, Helen P.	"Instrumentation for Polarography of Glass-Corroding Media," pp. 219–228 in *Analysis Instrumentation 1965* (Proceedings of the Eleventh Annual ISA Analysis Instrumentation Symposium Held May 26–28 at Montreal, Canada)	Plenum, New York, 1966
7 Ricci, E., R. L. Hahn, J. E. Strain, F. F. Dyer	"He^3 Activation Analysis," pp. 200–205 in *Modern Trends in Activation Analysis* (Proceedings, 1965 International Conference)	Activation Analysis Research Laboratory, Texas A and M University, College Station, Tex.
8 Shults, W. D.	"Coulometric Methods," chap. 23 in *Standard Methods of Chemical Analysis*, vol. III, ed. by F. J. Welcher	Van Nostrand, Princeton, N.J., 1966
9	*Controlled-Potential Differential DC Polarography* (Ph.D. thesis)	Indiana University, June 1966
10 Stokely, J. R.	*Solvent Extraction and Gas Chromatography of Metal Trifluoroacetylacetonates* (Ph.D. thesis)	Clemson University, May 1966
11 Strain, J. E.	"Use of Neutron Generators in Activation Analysis," chap. 4 in *Analytical Chemistry* (Progress in Nuclear Energy, Series IX, vol. 4, pt. 3), ed. by H. A. Elion and D. C. Stewart	Pergamon, New York, 1965
12 Wright, R. R.,[2] W. S. Pappas,[2] J. A. Carter, C. W. Weber[2]	"Preparation and Recovery of Cesium Compounds for Density Gradient Solutions," pp. 241–249 in *The Development of Zonal Centrifuges and Ancillary Systems for Tissue Fractionation and Analysis* (National Cancer Institute Monograph 21)	U.S. Dept. of Health, Education, and Welfare, Public Health Service, National Cancer Institute, Bethesda, Md., June 1966

Articles

AUTHOR(S)	TITLE	PUBLISHER
13 Apple, R. F., J. C. White	"Separation and Spectrophotometric Determination of Trace Quantities of Lithium in High-Purity Beryllium and Beryllium Oxide"	*Talanta* 13, 43 (1966)
14 Bate, L. C.	"Adsorption and Elution of Trace Elements on Human Hair"	*Intern. J. Appl. Radiation Isotopes* 17, 417 (1966)
15 Belew, W. L., D. J. Fisher, M. T. Kelley, J. A. Dean[3]	"Determination of Trace Quantities of Uranium by Controlled-Potential DC Polarography in a Tri-*n*-octylphosphine Oxide Extract"	*Microchem. J.* 10, 301 (1966)
16 Butler, C. T.,* J. R. Russell,* R. B. Quincy, Jr., D. E. LaValle	"Growth and Evaluation of High-Purity KCl Crystals"	*J. Chem. Phys.* 45(3), 968 (1966)

[2]Technical Division, ORGDP.

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

17	Christie, W. H., A. E. Cameron	"Reliable Sample Changer for Mass Spectrometer"	*Rev. Sci. Instr.* **37**, 336 (1966)
18	Corbin, L. T.	"High-Radiation-Level Analytical Laboratories" (Laboratory of the Month)	*Anal. Chem.* **37**(13), 83A (1965)
19	Eldridge, J. S.	"Improving the Assay of Mercury-197"	*J. Nucl. Med.* **7**, 339 (1966)
20	Eldridge, J. S., A. A. Brooks*	"Gamma-Ray Spectrometry Using Computer Library of Spectra"	*Nucleonics* **24**(4), 54 (1966)
21	Eldridge, J. S., P. Crowther,[4] W. S. Lyon	"Gamma-Ray Branching Ratios for ^{116}In, ^{147}Nd, and ^{141}Ce"	*Nucleonics* **24**(3), 62 (1966)
22	Florence, T. M.[5]	"Polarography of Nickel in Concentrated Chloride Media"	*Australian J. Chem.* **19**, 1343 (1966)
23	Florence, T. M.,[5] F. J. Miller, H. E. Zittel	"Voltammetric Determination of Aluminum by Oxidation of Its Solochrome Violet RS Complex at the Rotated Pyrolytic Graphite Electrode"	*Anal. Chem.* **38**, 1065 (1966)
24	Galonsky, A.,[6] G. Del Castillo,[7] F. F. Dyer	"Vacuum Chamber with Stable Permeability in a High-Power Accelerator"	*J. Nucl. Energy: Pt. C:* Plasma Physics—Accelerators—Thermonuclear Research **7**, 584 (1965)
25	Goldstein, G.	"Activation Cross Sections for the Reactions of 14.8-MeV Neutrons with ^{99}Tc"	*J. Inorg. Nucl. Chem.* **28**, 676 (1966)
26		"Partial Half-Life for β-Decay of ^{36}Cl"	*J. Inorg. Nucl. Chem.* **28**, 937 (1966)
27	Goldstein, G., J. A. Dean[3]	"Half-Life and Specific Activity of ^{99}Tc"	*J. Inorg. Nucl. Chem.* **28**, 285 (1966)
28		"Rapid Separation of Technetium from Fission Products"	*Radiochim. Acta* **5**, 18 (1966)
29	Goldstein, G., S. A. Reynolds	"Specific Activities and Half-Lives of Common Radionuclides"	*Nucl. Data* **A1**, 435 (1966)
30	Hahn, R. L., E. Ricci	"Interactions of ^{3}He Particles with ^{9}Be, ^{12}C, ^{16}O, and ^{19}F"	*Phys. Rev.* **146**(3), 650 (1966)
31	Harmatz, B.,* T. H. Handley	"Nuclear Spectroscopy of Neutron-Deficient Hafnium and Rare Earth Activities"	*Nucl. Phys.* **81**, 481 (1966)
32	Honaker, C. B.,[8] A. D. Horton	"A Simple Pyrolyzer for Use with Gas Chromatography"	*J. Gas Chromatog.* **3**, 396 (1965)
33	Jurriaanse, A.,[9] F. L. Moore	"Fast Separation of Niobium-95 from Zirconium-95 in Commercial Radioisotope Solutions"	*Anal. Chem.* **38**, 964 (1966)

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

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

[6]Midwestern Universities Research Assoc., Stoughton, Wis.

[7]Michigan State University, East Lansing.

[8]Research Participant, summer 1965; Professor of Chemistry and Chairman, Department of Chemistry, Tennessee Wesleyan College, Athens.

[9]Alien Guest from the South African Atomic Energy Board, Pretoria, Republic of South Africa.

34 Kelley, M. T., W. L. Belew, G. V. Pierce, W. D. Shults, H. C. Jones, D. J. Fisher	"Controlled-Potential Polarography and Coulometry as Micro-Analytical Techniques"	*Microchem. J.* **10**, 315 (1966)
35 Kubota, H.	"Determination of Thallium in Cesium Iodide Scintillators"	*Anal. Chim. Acta* **35**, 534 (1966)
36 LaValle, D. E., R. M. Steele,* W. T. Smith, Jr.[10]	"Rhenium Nitrogen Fluoride and Rhenium Tetrafluoride"	*J. Inorg. Nucl. Chem.* **28**, 260 (1966)
37 Lyon, W. S.	"Radioactivation Analysis as an Analytical Tool"	*Ann. N.Y. Acad. Sci.* **137**, Art. 1, 311 (1966)
38 Lyon, W. S., S. A. Reynolds, E. I. Wyatt	"Methods for Assay of Radioisotopes"	*Nucleonics* **24**(8), 116 (1966)
39 Lyon, W. S., E. Ricci, H. H. Ross	"Nucleonics"	*Anal. Chem.* **38**(5), 251R (1966)
40 Mamantov, G.,[1] D. L. Manning	"Voltammetry and Related Studies of Uranium in Molten Lithium Fluoride–Beryllium Fluoride–Zirconium Fluoride"	*Anal. Chem.* **38**, 1494 (1966)
41 Miller, F. J., H. E. Zittel	"Voltammetry of the Iodine System in Aqueous Medium at the Pyrolytic Graphite Electrode"	*J. Electroanal. Chem.* **11**, 85 (1966)
42 Moore, F. L.	"Improved Extraction Method for Isolation of Trivalent Actinide-Lanthanide Elements from Nitrate Solutions"	*Anal. Chem.* **38**, 510 (1966)
43	"Liquid-Liquid Extraction of Anionic Americium and Europium Complexes of Hydroxyethylethylenediaminetriacetic Acid and Diethylenetriaminepentaacetic Acid"	*Anal. Chem.* **38**, 905 (1966)
44 Myers, W. G.,* A. F. Rupp,* H. B. Hupf,* J. S. Eldridge	"Radioiodine-126"	*J. Nucl. Med.* **7**, 391 (1966)
45 O'Brien, H. A., Jr.,* J. S. Eldridge	"Reactor Production of Carrier-Free Cerium-141 from Lanthanum and Praseodymium"	*Trans. Am. Nucl. Soc.* **9**(1), 101 (1966)
46 Papas, W. S.,[2] S. A. MacIntyre, C. W. Weber[2]	"A Comparative Condensation Pressure Analyzer for Uranium Hexafluoride Purity"	*Anal. Chem.* **38**, 1570 (1966)
47 Raaen, Helen P.	Book review of: *Techniques of Oscillographic Polarography*, R. Kalvoda, American Elsevier, New York, 1965	*Anal. Chem.* **38**(11), 75 A (1966)
48 Ricci, E., R. L. Hahn	"Sensitivities for Helium-3 Activation-Analysis Determination of Light Elements (Z = 4 to 20)"	*Trans. Am. Nucl. Soc.* **9**(1), 104 (1966)

[10]Consultant to Chemistry Division; Professor of Chemistry, University of Tennessee, Knoxville.

49	Ross, H. H.	"New Concept in Precision Photometric Analysis Using a Radioisotopic Light Source"	*Anal. Chem.* **38**, 414 (1966)
50	Ross, W. J., J. E. Strain, J. W. Landry*	"Monitoring of Leached Fuel Elements with a Neutron Generator"	*Trans. Am. Nucl. Soc.* **9**(1), 107 (1966)
51	Sherman, R. L., O. L. Keller, Jr.*	"Modified Debye-Scherrer X-Ray Diffraction Camera for Radioactive Compounds"	*Rev. Sci. Instr.* **37**(2), 240 (1966)
52	Shults, W. D., T. R. Mueller	"Theory of Programmed Current Derivative Chronopotentiometry"	*J. Electroanal. Chem.* **12**, 354 (1966)
53	Walton, J. R., A. E. Cameron	"The Isotopic Composition of Atmospheric Neon"	*Z. Naturforsch.* **21a**, 115 (1966)
54	J. C. White	Book Review of: *Analytical Applications of Ion Exchangers*, J. Inczedy, Pergamon, New York, 1966	*Anal. Chem.* **38**(12), 71A (1966)
55	White, J. C., D. A. Costanzo	"Ninth Conference on Analytical Chemistry in Nuclear Technology"	*Anal. Chem.* **38**(2), 130A (1966)
56	Williams, I. R.,* K. S. Toth,* T. H. Handley	"Alpha-Particle and Gamma-Ray Spectral Studies of ^{149}Gd and ^{149}Eu"	*Nucl. Phys.* **84**, 609 (1966)
57	Young, J. P., G. W. Clark	"Apparatus for the Spectrophotometric Study of Small Crystals"	*Rev. Sci. Instr.* **37**, 234 (1966)

Reports

	AUTHOR(S)	TITLE	REPORT NO. AND DATE
58	Attrill, J. E.	*Progress on the Body Fluids Analysis Program*	May 20, 1966 (unpublished)
59	Barker, G. C.,[11] R. W. Stelzner	"A Square-Wave Polarograph," p. 38 in *Chemistry Division Progress Report for the Period May 1, 1965, to October 31, 1965*	AERE-PR/CHEM-9 (December 1965)
60	Feldman, C.	*A Survey of Sensitivity Limits for the Spectrographic Determination of Trace Impurities in U_3O_8*	ORNL-TM-1590 (Aug. 10, 1966)
61		"The Laser in Metallurgical Spectroscopy," p. 158 in *Proceedings of 19th Metallographic Group Meeting* (held April 20–22, 1965, at the Oak Ridge National Laboratory, Oak Ridge, Tenn.)	ORNL-TM-1161 (February 1966)
62	Goldberg, G.	*The Effects of Trace Impurities of Moisture, Oxygen, Hydrogen, Nitrogen and Carbon Dioxide in Glove Box Atmospheres on the Analysis of the Alkali Metals*	ORNL-TM-1357 (Nov. 10, 1965)
63	Handley, T. H.	*Release of Tritium from Tritium-Labeled Luminous Paints*	ORNL-TM-1478 (June 1966)

[11]Chemistry Division, AERE, Harwell, Didcot, Berkshire, England.

64	Holsopple, H. L.	*Synthesis of α,α′-Dipiperidyl*	ORNL-TM-1470 (Mar. 30, 1966)
65		*Synthesis of Methylmethanesulfonate*	ORNL-TM-1486 (Apr. 5, 1966)
66		*Synthesis of Tri-Isoamyl Phosphate*	ORNL-TM-1487 (Apr. 13, 1966)
67		*Synthesis of 2,6-Dioximinocyclohexanone*	ORNL-TM-1510 (Apr. 28, 1966)
68		*Synthesis of Three Quaternary Phosphonium Halides*	ORNL-TM-1572 (July 12, 1966)
69		*Synthesis of Tri-n-butylsulfonium Iodide*	ORNL-TM-1607 (Aug. 22, 1966)
70	Holsopple, H. L., F. G. Seeley*	*Synthesis of 3,9-Diethyltridecyl-6-amine*	ORNL-TM-1314 (Nov. 5, 1965)
71	Kelley, M. T.	*Statistical Quality Control Report, Analytical Chemistry Division, July Through September, 1965*	Oct. 22, 1965 (unpublished
72		*Statistical Quality Control Report, Analytical Chemistry Division, October Through December, 1965*	Jan. 14, 1966 (unpublished)
73		*Statistical Quality Control Report, Analytical Chemistry Division, January Through March, 1966*	Apr. 12, 1966 (unpublished)
74		*Statistical Quality Control Report, Analytical Chemistry Division, April Through June, 1966*	July 7, 1966 (unpublished)
75		*Statistical Quality Control Report, Analytical Chemistry Division, July Through September, 1966*	Oct. 11, 1966 (unpublished)
76	Raaen, Helen P., ed.	*Analytical Chemistry Division Annual Progress Report for Period Ending November 15, 1965*	ORNL-3889 (January 1966)
77		*Oak Ridge National Laboratory Master Analytical Manual, Supplement 8*	TID-7015, suppl. 8 (March 1966)
78	Raaen, Helen P., Ann K. Haas,* eds.	*Indexes to the Analytical Chemistry Division Annual Progress Reports 1964–1965*	ORNL-3904 (January 1966)
79		*Indexes to the Oak Ridge National Laboratory Master Analytical Manual* (1953–1965)	TID-7015, Indexes, rev. 3 (March 1966)
80	White, J. C.	*Analytical Chemistry Research and Development Group Monthly Summary – December 1965*	Jan. 7, 1966 (unpublished)
81		*Analytical Chemistry Research and Development Group Monthly Summary – January 1966*	Feb. 8, 1966 (unpublished)
82		*Analytical Chemistry Research and Development Group Monthly Summary – February 1966*	Mar. 9, 1966 (unpublished)
83		*Analytical Chemistry Research and Development Group Monthly Summary – March 1966*	Apr. 12, 1966 (unpublished)
84		*Analytical Chemistry Research and Development Group Monthly Summary – April 1966*	May 4, 1966 (unpublished)
85		*Analytical Chemistry Research and Development Group Monthly Summary – May 1966*	June 13, 1966 (unpublished)

86		*Analytical Chemistry Research and Development Group Monthly Summary — June 1966*	July 11, 1966 (unpublished)
87		*Analytical Chemistry Research and Development Group Monthly Summary — July 1966*	July 27, 1966 (unpublished)
88		*Analytical Chemistry Research and Development Group Monthly Summary — August 1966*	Aug. 30, 1966 (unpublished)
89		*Analytical Chemistry Research and Development Group Monthly Summary — September 1966*	Sept. 30, 1966 (unpublished)
90		*Analytical Chemistry Research and Development Group Monthly Summary — October 1966*	Oct. 26, 1966 (unpublished)
91	Willmarth, T. E.	"Electron Optical Study of the Effect of Aging on Carbides in a Niobium-Tungsten-Zirconium Alloy," p. 208 in *Proceedings of 19th Metallographic Group Meeting* (held April 20–22, 1965, at the Oak Ridge National Laboratory, Oak Ridge, Tenn.)	ORNL-TM-1161 (February 1966)
92	Yeatts, L. B., Jr.,* W. T. Rainey, Jr.	*Purification of Zirconium Tetrafluoride*	ORNL-TM-1292 (November 1965)
93	Anonymous	"High-Sensitivity Coulometric Titrator/Q-2564/" in *Engineering Materials List*, TID-4100 (Supplement 30)	CAPE-1196 (November 1965)

New Methods Issued to *ORNL Master Analytical Manual*

	AUTHOR(S)	TITLE	NUMBER(S)	DATE
94	Bate, L. C.	"Particle-Size Distribution in Thorium Oxide, Neutron Activation—Centrifugation Method"	5 10202	4-6-66
95	Robinson, B. J.[12]	"Hot-Cell Dissolutions of Radioactive Nuclear-Reactor Materials"	1 0903 9 00903	9-12-66

Revised Methods Issued to *ORNL Master Analytical Manual*

	AUTHOR(S)	TITLE	NUMBER(S)	DATE
96	Mullins, W. T., G. W. Leddicotte[13]	"Cobalt, Neutron Activation Analysis (Isotopic-Carrier Precipitation) Method"	5 11220	R. 5-15-66
97	Mullins, W. T. G. W. Leddicotte[13]	"Manganese, Neutron Activation Analysis (Isotopic Carrier) Method"	5 11480	R. 5-15-66
98	Mullins, W. T. G. W. Leddicotte[13]	"Zirconium, Neutron Activation Analysis (Isotopic Carrier) Method"	5 11980	R. 5-15-66

[12]Present address: University of Kentucky Medical School, Lexington.
[13]Present address: Research Reactor Facility, University of Missouri, Columbia.

Record-Copy Methods Issued to *ORNL Master Analytical Manual*

	AUTHOR(S)	TITLE	NUMBER(S)	DATE
99	Feldman, C.	"Cesium, Ammonium 12-Molybdophosphate Collection—Flame Photometric Method"	1 211931	6-9-66
100	Kelley, M. T., W. L. Belew, G. V. Pierce, W. D. Shults, H. C. Jones, D. J. Fisher	"Iron, Tri-*n*-Octylphosphine Oxide (TOPO) Extraction—Controlled-Potential Coulometric Titration Microanalytical Method"	1 214121 9 00714121	6-9-66
101	Moore, F. L.	"Separation of Trivalent Actinide Elements from Lanthanide Elements, Tricaprylmethylammonium Thiocyanate Liquid-Liquid Extraction Method"	1 00711 9 00655	6-9-66
102		"Separation of Trivalent Actinide and Trivalent Lanthanide Elements from Other Elements, Tricaprylmethylammonium Nitrate Liquid-Liquid Extraction Method"	1 00712 9 00656	6-9-66
103		"Separation of Trivalent Actinide Elements, Trivalent Lanthanide Elements, and Other Metals from Aqueous Solutions of Organic Acids, Liquid-Liquid Extraction with High-Molecular-Weight Amines Method"	1 00713 9 00657	6-9-66
104	Robinson, B. J.[12]	"Liquidus Temperature and Liquid Density of Mixtures of Fluoride Salts"	1 100033 9 00608	6-9-66
105		"Plutonium, Tri-*n*-octylphosphine Oxide (TOPO) Extraction—Controlled-Potential Coulometric Titration Microanalytical Method"	1 216221 9 00716221	6-9-66
106		"Uranium, Tri-*n*-octylphosphine Oxide (TOPO) Extraction—Controlled-Potential DC Polarographic Method"	1 219227 9 00719227	6-9-66
107	Ross, H. H.	"Wear Rates of Automotive Engine Parts, Thermal Neutron Irradiation—Iron-55 Liquid Scintillation Counting Method"	5 10203	6-9-66
108	Ross, H. H., W. S. Lyon	"Sulfur Dioxide in the Atmosphere, Iodine-131 Radio-Release Method"	2 0916	6-9-66

ORAL PRESENTATIONS

Tenth Conference on Analytical Chemistry in Nuclear Technology

Over 300 scientists, including 24 representatives from 8 foreign countries, attended the Tenth Conference on Analytical Chemistry in Nuclear Technology, which was held on September 27–29, 1966, in Gatlinburg, Tennessee. This was the largest representation from foreign countries to attend any of the conferences in this series. The Conference was again sponsored by the ORNL Analytical Chemistry Division.

Of the 54 papers presented at the meeting, 9 were given by ORNL staff members. The Conference Committee was comprised of 6 ORNL staff members and 3 members from outside ORNL. Members of the committee from ORNL were: M. T. Kelley, Director of the Division; C. D. Susano, Associate Director; and L. J. Brady, L. T. Corbin, H. L. Holsopple, and J. C. White. In addition, Leonard Newman, Brookhaven National Laboratory; R. C. Shank, Idaho Nuclear Corporation; and W. R. Tyson, Savannah River Plant of DuPont, served as members of the Conference Committee.

Concurrent sessions were held on the first two days of the Conference; a single session convened on the third day.

In lieu of published proceedings, tape recordings were made of all the presentations. Reproductions are made available generally to any one who requests the loan of any of these recordings. In addition to the copies at ORNL, copies of the tape recordings have been deposited at the Library of Congress and can be secured on a loan basis simply by contacting that agency.

The Eleventh Conference in this series is tentatively scheduled to be held at the Mountain View Hotel, Gatlinburg, Tennessee, on October 10–12, 1967.

At Meetings of Professional Societies

	AUTHOR(S)	TITLE	PRESENTED AT
109	Apple, R. F.,[14] J. M. Dale, F. L. Whiting,[15] A. S. Meyer, Jr., C. F. Baes*	"Determination of Oxide in Highly Radioactive Fused Fluoride Salts – Hydrofluorination Method"	Winter Meeting, American Chemical Society, Phoenix, Ariz., Jan. 17–21, 1966
110	Bate, L. C.,[14] F. F. Dyer	"Forensic Applications of Trace Elements in Hair"	International Conference on Forensic Activation Analysis, San Diego, Calif., Sept. 19–22, 1966
111	Butler, C. T.,[14*] J. R. Russell,* R. B. Quincy, Jr., D. E. LaValle	"A Method for Purification and Growth of Potassium Chloride Single Crystals"	International Conference on Crystal Growth, Boston, June 20–24, 1966
112	Canada, D. C.,[14] W. R. Laing	"Use of a Density Gradient Column to Measure the Density of Microspheres"	Southeastern Regional American Chemical Society Meeting, Louisville, Ky., Oct. 27–29, 1966
113	Carter, J. A.	"Spark Source Mass Spectrometry"	10th Annual Meeting, Analytical Specialist Group, Union Carbide Corp., Parma Technical Center, Parma, Ohio, May 16–17, 1966
114		"Application of the ORNL Spark-Source Mass Spectrograph to High-Purity Materials Analysis"	5th National Meeting, Society for Applied Spectroscopy, Chicago, June 13–17, 1966
115	Davis, J. H.,[14,16] D. H. Smith	"Isotopic Composition of Lead in Some Galenas from the Southeastern United States"	75th Meeting, Tennessee Academy of Science, Oak Ridge, Tenn., Dec. 10–11, 1965

[14] Speaker.

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

[16] Formerly with the Geologic Branch, Tennessee Valley Authority; present address, American Metals, Inc., Knoxville, Tenn.

116	Eldridge, J. S.	"Improving the Assay of Mercury-197"	13th Annual Meeting, Society of Nuclear Medicine, Philadelphia, June 22–25, 1966
117		"Secondary Standardizations Using Digital Computer Techniques to Normalize Multiple Gamma-Ray Spectra for Direct Efficiency Determinations"	International Atomic Energy Agency's Symposium on the Standardization of Radionuclides, Vienna, Oct. 10–14, 1966
118		"Standardization of Mercury-197"	International Atomic Energy Agency's Symposium on the Standardization of Radionuclides, Vienna, Oct. 10–14, 1966
119	Feldman, C.	"Laser and Electron Microprobe Analysis"	Haynes Stellite Co., Kokomo, Ind., Apr. 25, 1966
120		"Atomic Absorption Spectroscopy"	University of Georgia, Athens, May 9, 1966
121	Florence, T. M.[5]	"Analytical Applications of Alternating-Current and Single-Sweep Polarography"	Analytical Chemistry Group, East Tennessee Section, American Chemical Society, Oak Ridge, Tenn., Oct. 26, 1965
122	Hahn, R. L.	"Nonneutron Activation Analysis"	Advanced Isotope Technology Course, ORAU, Oak Ridge, Tenn., July 29, 1966
123	Hahn, R. L., E. Ricci	"Interactions of ^{3}He Particles with Low-Z Elements"	Joint Meeting of American Physical Society, Canadian Association of Physicists, and Mexican Physical Society, Mexico City, Mex., Aug. 29–31, 1966
124	Heacker, F. K.,[14] C. E. Lamb, L. T. Corbin	"Methods of Remote Analysis"	10th Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Sept. 27–29, 1966
125	Hertel, G. R.,[14] W. S. Koski[17]	"Ion-Molecule Reactions Involving Pentaborane"	152d Meeting, American Chemical Society, New York, Sept. 11–16, 1966
126	Hupf, H. B.,[14*] J. S. Eldridge, J. E. Beaver*	"Production and Characterization of Iodine-123 for Medical Investigators"	Central Chapter, Society of Nuclear Medicine, Iowa City, Iowa, Nov. 7, 1965
127	Jolley, R. L.,[14*] J. S. Wike, E. J. Robinson[12]	"Dissolution and Analyses of Highly Irradiated Plutonia"	10th Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Sept. 27–29, 1966
128	Kelley, M. T.	"Recent Advances in Instrumentation for Flame Photometry"	Winter Meeting, American Chemical Society, Phoenix, Ariz., Jan. 17–21, 1966
129	Kubota, H.	"The Dissolution of Some Inert Metals in Gamma Irradiated Hydrochloric Acid"	152d Meeting, American Chemical Society, New York, Sept. 11–16, 1966
130	Layton, F. L.,[14] W. R. Laing	"Determination of Free Fluoride in Solutions Containing Niobium and Uranium"	2d Southeastern-Southwestern Regional American Chemical Society Meeting, Memphis, Tenn., Dec. 2–4, 1965

[17]Professor of Chemistry and Chairman, Department of Chemistry, The Johns Hopkins University, Baltimore, Md.

131	Lyon, W. S.	"Practice of Activation Analysis"	Neutron Activation Analysis Course, ORAU, Oak Ridge, Tenn., June 7, 1966
132		"Non-Destructive Analysis"	Neutron Activation Analysis Course, ORAU, Oak Ridge, Tenn., June 15, 1966
133		"Absolute Gamma Ray Measurement"	Advanced Isotope Technology Course, ORAU, Oak Ridge, Tenn., July 13, 1966
134	Mamantov, G.,[1,14] D. L. Manning	"Voltammetry and Related Studies of Uranium in Molten $LiF-BeF_2-ZrF_4$"	152d Meeting, American Chemical Society, New York, Sept. 11–16, 1966
135		"Electroanalytical Studies of Uranium(IV) in Molten Fluorides"	10th Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Sept. 27–29, 1966
136	Meyer, H. C.	"Experiences of the Analytical Chemistry Group in the Startup of the Transuranium Process Facility (TRU)"	10th Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Sept. 27–29, 1966
137	O'Brien, H. A., Jr.,[14*] J. S. Eldridge	"Reactor Production of Carrier-Free Cerium-141 from Lanthanum and Praseodymium"	12th Annual Meeting, American Nuclear Society, Denver, Colo., June 20–23, 1966
138	Reynolds, S. A.	"Radiochemical Separation by Solvent Extraction and Ion Exchange"	Neutron Activation Analysis Course, ORAU, Oak Ridge, Tenn., June 9, 1966
139	Ricci, E.,[14] R. L. Hahn	"Sensitivities for ^3He Activation Analysis Determination of Light Elements ($Z = 4$ to 20)"	12th Annual Meeting, American Nuclear Society, Denver, Colo., June 20–23, 1966
140	Rickard, R. R.,[14] D. R. Matthews, E. I. Wyatt	"Special Procedures Employed in Radiochemical Analyses in Remote Systems"	10th Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Sept. 27–29, 1966
141	Ross, H. H.	"Liquid Scintillation Technology"	7th Annual Contractor's Meeting, Division of Isotopes Development, U.S. Atomic Energy Commission, Washington, D.C., Dec. 6–8, 1965
142		"New Concept in Precision Photometric Analyses Using a Radioisotopic Light Source"	17th Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, Pittsburgh, Pa., Feb. 21–25, 1966
143		"Precision Photometric Analysis Using a Beta-Excited Light Source"	National Bureau of Standards Institute for Materials Research – 1st IMR International Symposium, "Trace Characterization – Chemical and Physical," Gaithersburg, Md., Oct. 3–7, 1966
144	Ross, W. J.	"Analytical Chemistry in Oceanography"	7th Annual Contractor's Meeting, Division of Isotopes Development, U.S. Atomic Energy Commission, Washington, D.C., Dec. 6–8, 1965
145	Ross, W. J.,[14] J. E. Strain, J. W. Landry*	"Monitoring of Leached Fuel Elements with a Neutron Generator"	12th Annual Meeting, American Nuclear Society, Denver, June 20–23, 1966
146	Rubin, I. B.,[14] A. D. Kelmers*	"The Determination of Transfer Ribonucleic Acid by Aminoacylation. I. Leucyl- and Phenylalanyl-Transfer Ribonucleic Acid from *E. Coli* B"	Southeastern Regional American Chemical Society Meeting, Louisville, Ky., Oct. 27–29, 1966

147	Shults, W. D.	"Controlled-Potential Differential DC Polarography"	Analytical Group Seminar, Chemistry Dept., Indiana University, Bloomington, Ind., Apr. 4, 1966
148	Sites, J. R.	"Analytical Mass Spectrometry at Oak Ridge National Laboratory"	5th National Meeting, Society for Applied Spectroscopy, Chicago, June 13–17, 1966
149	Strain, J. E.	"Isotopic and Accelerator Neutron Sources" (Invited Lecture)	Workshop in Activation Analysis, Texas Woman's University, Denton, June 13–24, 1966
150	Talbott, C. K.,[14] C. E. Lamb, L. T. Corbin	"Operations in a High-Radiation-Level Analytical Laboratory"	10th Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Sept. 27–29, 1966
151	Wampler, J. M.,[14,18] D. H. Smith, A. E. Cameron	"Isotopic Comparison of Lead from Ivory Coast Tektites and Bosumtwi Crater Materials"	American Geophysical Union, Washington, D.C., Apr. 19–22, 1966
152	White, J. C.	"Analytical Applications of Liquid Ion Exchangers"	152d Meeting, American Chemical Society, New York, Sept. 11–16, 1966
153	Wike, J. S.,[14] C. E. Lamb, L. T. Corbin	"Instrumentation and Equipment in Remote Analyses"	10th Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Sept. 27–29, 1966
154	Willmarth, T. E.	"A Method for Using Plastic Membrane Filters for the Examination of Sub-Micron Particulates by Transmission Electron Microscopy"	3d Biannual Meeting, Southeastern Electron Microscopy Society, Atlanta, Ga., May 13–14, 1966
155		"The Examination of Praseodymium and Europium Hydroxide Sols by Electron Microscopy and Electron Diffraction"	American Meeting of British Royal Microscopical Society, Chicago, Aug. 15–19, 1966
156	Wright, H. W.,[14] T. E. Willmarth	"Electron Microscopy of Radioactive Powders"	24th Annual Meeting, Electron Microscopy Society of America, San Francisco, Aug. 22–25, 1966
157	Yoakum, Anna M.	"Recent Advances in Analytical Emission Spectroscopy"	5th National Meeting, Society for Applied Spectroscopy, Chicago, June 13–17, 1966
158	Young, J. P.	"Effect of Temperature on the Spectra of CrF_2 and FeF_2 as Crystals and in Molten Fluoride Solution"	5th National Meeting, Society for Applied Spectroscopy, Chicago, June 13–17, 1966
159	Zittel, H. E.,[14] T. M. Florence[5]	"A Voltammetric Method for the Determination of Zirconium"	10th Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Sept. 27–29, 1966

[18] Assistant Professor, Earth Sciences Department, Georgia Institute of Technology, Atlanta.

Under the ORNL Traveling Lecture Program

	AUTHOR(S)	TITLE	PRESENTED AT
160	Hahn, R. L.	"Chemical Effects of Nuclear Internal Conversion: Separation of Tellurium Radioisomers"	University of Arkansas, Fayetteville, Feb. 28, 1966
161			University of Missouri, Columbia, Mar. 2, 1966
162			Washington University, St. Louis, Mo., Mar. 3, 1966
163	White, J. C.	"The New Image of Analytical Chemistry in Science"	University of Tennessee, Knoxville, Feb. 3, 1966
164			Xavier University, Cincinnati, Ohio, Feb. 9, 1966
165			Jackson State College, Jackson, Miss., Mar. 29, 1966
166			North Texas State University, Denton, Mar. 31, 1966
167			Texas Woman's University, Denton, Apr. 1, 1966
168			Jersey City State College, Jersey City, N.J., May 3, 1966

PATENTS

	AUTHOR	TITLE	PATENT NO.	DATE ISSUED
169	Moore, F. L.	"Method for Separating Members of Actinide and Lanthanide Groups"	3,276,849	Oct. 4, 1966

Translations

TRANSLATOR	ORIGINAL REFERENCE			TRANSLATION		
	Author (Transliterated)	Source	Language	Language	Translated Title	ORNL-tr Number
1 Kubota, H.	S. Saito and N. Yui	*Nippon Kagaku Zasshi* **79**, 1492–95 (1958)	Japanese	English	"Distribution Equilibria of Alkali Chlorides in the Water–*n*-Butanol System"	1179
2	T. Takeuchi and T. Miwa	*Kogyo Kagaku Zasshi* **69**, 398–401 (1966)	Japanese	English	"The Potentiometric Determination of Primary Aromatic Amines by a Diazo Titration Using a Glassy Carbon Electrode"	1276
3 Miller, F. J.	V. V. Kusmovich	*Physical and Electrochemistry of Molten Salts*, A. V. Gorodskii, ed., pp. 124–48, Ukrainian Academy of Sciences, Kiev (1965)	Russian	English	"Some Relationships in the Polarographic Investigation of Molten Chlorides with the Platinum Electrode"	1197
4	R. Landsberg and R. Nitzsche	*Wiss. Z. Tech. Hochsch. Chem. Leuna-Merseburg* **6**(2), 100–102 (1964)	German	English	"The Anodic Oxidation of Iodide at the Rotating Disc Electrode"	1245
5	L. Astheimer and K. Schwochau	*J. Electroanal. Chem.* **8**, 382–389 (1964)	German	English	"On the Polarography of Technetium. I. D.C. and A.C. Polarographic Studies of Pertechnetate Solutions"	1265

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	Author (Transliterated)	Source	Language	Language	Translated Title	ORNL-tr Number
6	A. Skrivele and S. Sinyakova	*Latvijas PSR Zinatnu Akad. Vestis* **223**(2), 69–76 (1966)	Russian	English	"The Determination of Copper, Zinc, Lead, and Cadmium in Pure Solutions by Amalgamation Polarography with Accumulation (APR)"	1268
7	W. Geissler, R. Nitzsche, and R. Landsberg	*Electrochim. Acta* **11**, 389–400 (1966)	German	English	"The Electrochemical Oxidation of Iodide and Iodine to Hypoiodite at the Graphite Electrode"	1303
8 Raaen, Helen P.	K. Kronenberger and W. Nickels	*Z. Anal. Chem.* **186**, 79–86 (1962)	German	English	"A New Tast Polarograph"	1269
9	K. Kronenberger, H. Strehlow, and A. W. Elbel	*Polarograph. Ber.* **5**, 62–77 (1957)	German	English	"Tast Polarography"	1270
10	A. W. Elbel	*Z. Anal. Chem.* **173**, 70–73 (1960)	German	English	"Tast Polarography"	1271
11	M. von Stackelberg and V. Toome	*Collection Czech. Chem. Commun.* **25**, 2958–2965 (1960)	German	English	"Study of the Detachment Process of the Dropping Electrode I. Geometry of the Process and the Back Pressure"	1272
12	R. Doppelfeld and M. von Stackelberg	*Collection Czech. Chem. Commun.* **25**, 2966–2973 (1960)	German	English	"Study of the Detachment Process of the Dropping Electrode II. Measurement of Current"	1273
13	M. von Stackelberg	*Z. Anal. Chem.* **173**, 90–93 (1960)	German	English	"The Back Pressure of Mercury Drops as a Source of Error in Polarographic Analyses"	1274

Articles Reviewed or Refereed for Periodicals

Reviewer or Referee	Number of Articles Reviewed or Refereed for Indicated Periodical												
	Anal. Chem.	*Anal. Chim. Acta*	*Appl. Spectry.*	*Health Phys.*	*Isotopes Radiation Technol.*	*J. Gas Chromatog.*	*Nucl. Appl.*	*Nucl. Sci. Eng.*	*Spectrochim. Acta*	*Science*	*Talanta*	*U.S. Bur. Mines, Rept. Invest.*	*Total*
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Kubota, H.	1												1
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Mueller, T. R.		1											1
Raaen, Helen P.	1												1
Reynolds, S. A.	4			1	1								6
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Thomason, P. F.	1												1
White, J. C.	3												3
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Indexes

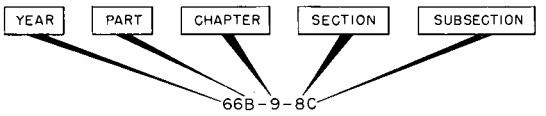
Helen P. Raaen Ann S. Haas[1]

Indexes are a part of the Analytical Chemistry Division annual report for the third time. The numbering system used has made possible the preparation of cumulative indexes to the annual progress reports from the Division. This section of the report contains a key-word index and an author index to this report. The cumulative indexes for the years 1964–1966 are bound separately and are designated ORNL-3904 (Rev. 1).

The Key Word Index is prepared both from the numbered headings that appear within the report and from the titles of the entries in the Presentation of Research Results and in the Translations. To increase the depth of indexing of the work reported, supplementary words were added to some of the headings. These words appear in parentheses, together with the heading, in the key-word index; they do not appear elsewhere in the report.

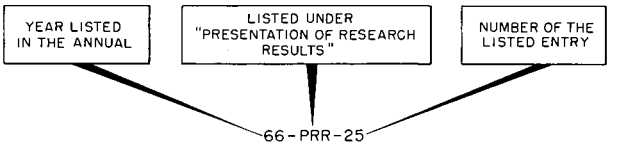
The Author Index is an alphabetical listing of authors, together with number entries that specify the material to which each author contributed. This index includes the authors of material in the body of the report, the authors of the entries listed in the Presentation of Research Results, and the translators of the entries listed in Translations. In the cases of co-authorships, the member of another ORNL division is so indicated by an asterisk which precedes that author's name. If, for any of a number of reasons, an author is not a permanent member of ORNL, the symbol = appears before his name.

An example of the numbering system used to refer to material in the main body of the report is 66B-9-8C, where 66 indicates the year of publication of the annual report, B designates a major part or division of the report, 9 is the chapter number, 8 is a primary section within the chapter, and C is a subsection (designated in the subsection heading with c instead of C); for example,



In the use of the index to locate material in the body of the report, only the last two groups of numbers are needed, because the chapters are numbered consecutively.

For reference to the Presentation of Research Results, the numbering system is of the type 66-PRR-25, where 66 designates the year in which the entry was listed in the annual report, PRR identifies the authored work as being listed in the Presentation of Research Results part of the annual report, and 25 is the number of the entry listed in that part of the report; for example,



In the use of the Index to locate an entry in the Presentation of Research Results part of the report, only the last group of numbers is needed, because the entries are numbered consecutively.

If the entry is in the Translations part of the report, the entry will be identified by the letters TR in the same position as the letters PRR.

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

[1]Technical Information Division.

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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MOORE, F. L. 66-PRR-102
MOORE, F. L. 66-PRR-103
MOORE, F. L. 66-PRR-169

MUELLER, T. R. 66A-01-01
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MUELLER, T. R. 66A-01-09
MUELLER, T. R. 66A-01-10
MUELLER, T. R. 66A-01-13E
MUELLER, T. R. 66A-01-14
MUELLER, T. R. 66A-01-16
MUELLER, T. R. 66-PRR-052

MULLINS, W. T. 66-PRR-096
MULLINS, W. T. 66-PRR-097
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MURRAY, M. M. 66A-04-04C

*MYERS, W. G. 66-PRR-044

N

*NIEMEYER, R. G. 66A-09-02F

O

*OBRIEN, H. A., JR. 66A-09-01E
*OBRIEN, H. A., JR. 66A-09-01F
*OBRIEN, H. A., JR. 66-PRR-045
*OBRIEN, H. A., JR. 66-PRR-137

P

=PAPPAS, W. S. 66-PRR-012
=PAPPAS, W. S. 66-PRR-046

*PEED, W. F. 66A-03-05B

PIERCE, G. V. 66-PRR-034
PIERCE, G. V. 66-PRR-100

Q

QUINCY, R. B. 66A-10
QUINCY, R. B. 66-PRR-016
QUINCY, R. B. 66-PRR-111

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RAAEN, H. P. 66A-01-15
RAAEN, H. P. 66A-04-02A
RAAEN, H. P. 66C-15
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RAAEN, H. P. 66-PRR-047
RAAEN, H. P. 66-PRR-076
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RAAEN, H. P. 66TR-08
RAAEN, H. P. 66TR-09
RAAEN, H. P. 66TR-10
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RAINEY, W. T. 66-PRR-092

REYNOLDS, S. A. 66A-09-01J
REYNOLDS, S. A. 66A-09-02A
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REYNOLDS, S. A. 66A-09-02C
REYNOLDS, S. A. 66A-09-02D
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REYNOLDS, S. A. 66A-09-03B
REYNOLDS, S. A. 66A-09-04G
REYNOLDS, S. A. 66-PRR-029
REYNOLDS, S. A. 66-PRR-038
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RICKARD, R. R. 66-PRR-140

=ROBINSON, B. J. 66-PRR-095
=ROBINSON, B. J. 66-PRR-104
=ROBINSON, B. J. 66-PRR-105
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*RUPP, A. F. 66-PRR-044

*RUSSELL, J. R. 66-PRR-016
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=SCHAAP, W. B. 66A-01-11

*SEELEY, F. G. 66-PRR-070

SHERMAN, R. L. 66-PRR-051

SHULTS, W. D. 66A-01-04
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SMITH, D. H. 66-PRR-151

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STELZNER, R. W. 66A-01-08
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STRAIN, J. E. 66A-09-08C
STRAIN, J. E. 66A-09-08D
STRAIN, J. E. 66A-09-08E
STRAIN, J. E. 66-PRR-007
STRAIN, J. E. 66-PRR-011
STRAIN, J. E. 66-PRR-050
STRAIN, J. E. 66-PRR-145
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TALBOTT, C. K. 66-PRR-150

THOMASON, P. F. 66A-04-02
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*TOTH, K. S. 66A-09-011
*TOTH, K. S. 66-PRR-056

=TURNER, L. P. 66A-05-03

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WALTON, J. R. 66-PRR-053

=WAMPLER, J. M. 66-PRR-151

=WEBER, C. W. 66-PRR-012
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WHITE, J. C. 66A-02
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WHITE, J. C. 66-PRR-013
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WHITE, J. C. 66-PRR-055
WHITE, J. C. 66-PRR-080
WHITE, J. C. 66-PRR-152
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=WHITING, F. L. 66-PRR-109

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WRIGHT, H. W. 66A-08
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WYATT, E. I. 66B-14-04
WYATT, E. I. 66-PRR-038
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*YEATTS, L. B., JR. 66-PRR-092

YOAKUM, A. M. 66A-06-01C
YOAKUM, A. M. 66-PRR-157

YOUNG, J. P. 66A-03-02A
YOUNG, J. P. 66A-03-02C
YOUNG, J. P. 66A-03-03
YOUNG, J. P. 66A-03-04
YOUNG, J. P. 66A-03-04B
YOUNG, J. P. 66-PRR-057
YOUNG, J. P. 66-PRR-158

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ZITTEL, H. E. 66A-02-01
ZITTEL, H. E. 66A-02-02
ZITTEL, H. E. 66A-02-03
ZITTEL, H. E. 66A-02-04
ZITTEL, H. E. 66A-02-05
ZITTEL, H. E. 66A-04-02C
ZITTEL, H. E. 66-PRR-023
ZITTEL, H. E. 66-PRR-041
ZITTEL, H. E. 66-PRR-159

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ADMINISTRATIVE AND CLERICAL	5	9	14
SCIENTISTS	114*		114
ANALYSTS		84	84
SUBTOTAL	119	93	212
OTHERS			
NONCITIZEN GUESTS	2		2
STUDENT GUESTS (UT)	2		2
LOANEES FROM OTHER DIVISIONS	2		2
SUBTOTALS	6		6
GRAND TOTAL	125	93	218

*Includes 4 Loanees out to other Divisions.

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[3]ON LOAN TO CHEMICAL TECHNOLOGY DIVISION
[4]ON LOAN TO ISOTOPES DIVISION
[5]ON LOAN FROM BUDGET DEPARTMENT
[6]ON LOAN FROM THERMONUCLEAR DIVISION
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