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ANALYTICAL CHEMISTRY DIVISION
ANNUAL PROGRESS REPORT
FOR PERIOD ENDING OCTOBER 31, 1967

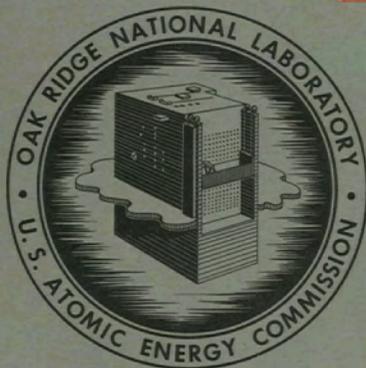
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ANALYTICAL CHEMISTRY DIVISION
ANNUAL PROGRESS REPORT
For Period Ending October 31, 1967

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Helen P. Raen

JANUARY 1968

OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee
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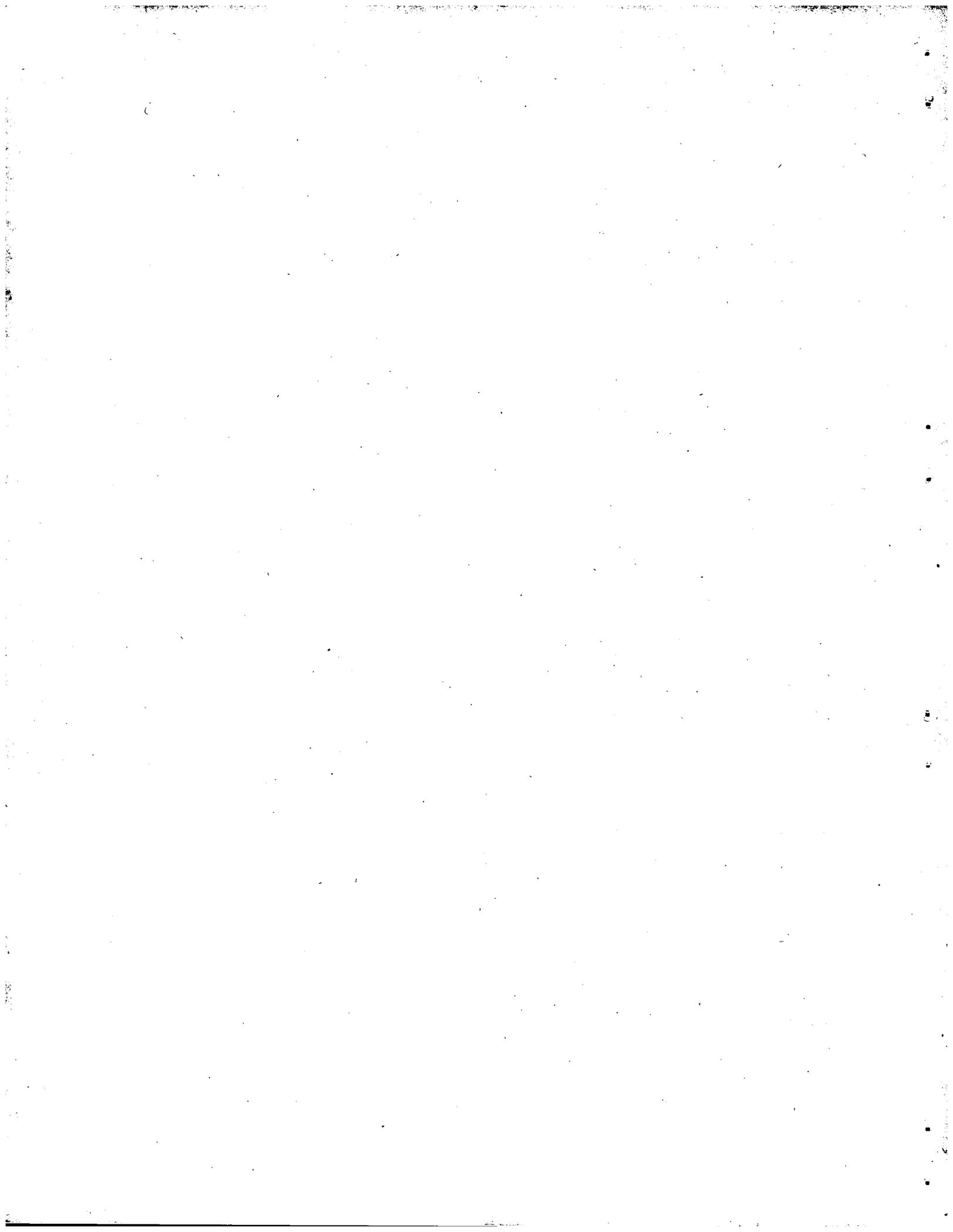
Preface

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 are oriented toward providing the most suitable analytical techniques for the analyses that the Division is called upon to perform. Efforts are being made to provide more rapid analyses by using automated or semiautomated procedures and, where feasible, by using electronic data-acquisition and -processing equipment. With small integrated-circuit units becoming available at reasonable prices, it will be necessary to evaluate the proper

exploitation of such equipment in comparison with larger time-sharing systems and the processing of data by the central computer facilities. Constant attention is given in the service laboratories to assuring a high standard of operation by institution of new or improved methods of analysis and the use of more efficient and refined instrumentation.

The objectives of the major research and development studies in the Division continue to be to provide analyses for the MSRE and for the bioengineering programs. The effort in analytical biochemistry has increased considerably during the last year, and the staff has been strengthened by the acquisition of scientists specifically trained in this field. Although further expansion in analytical biochemistry will probably be at a slower pace, this work will continue to be considered one of the important frontiers in analytical chemistry.



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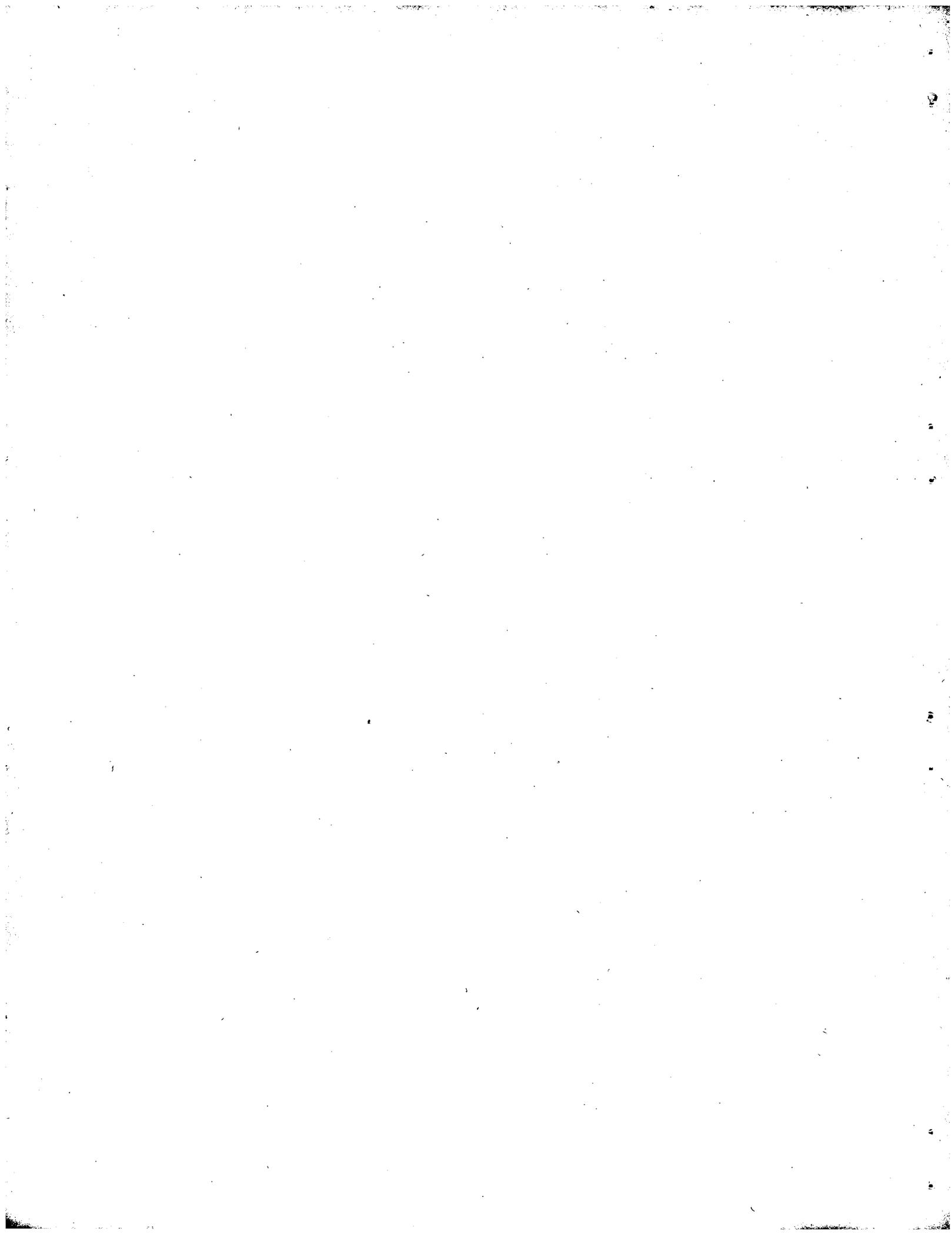
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Summary

PART A. ANALYTICAL RESEARCH

1. Analytical Instrumentation

The prototype of the controlled-potential controlled-current cyclic voltammeter was released to the Reactor Projects Group for obtaining analytical and mechanistic data in molten-salt media. Satisfactory results were obtained, although interference from nearby rf sources caused difficulties. A duplicate of the instrument was constructed in the Department of Chemistry, University of Tennessee; it is performing satisfactorily in room-temperature experiments with aqueous and nonaqueous solvent systems. On the basis of the satisfactory performance of the prototype, a third instrument (ORNL model Q-2943) was constructed by the ORNL Instrumentation and Controls Division for the Reactor Chemistry Division.

A dual set-point voltage comparator (ORNL model Q-2950) was designed. It is needed to calibrate accurately the scan rates used in the controlled-potential controlled-current voltammeter, which range from 0.005 to 500 v/sec. For this application, the comparator controls a timer to measure the time corresponding to a set 2-v scan interval.

A modular, solid-state, controlled-potential coulometric titrator is being designed. The performance of the overall system will be optimized by including design of the titration cell as part of this project.

It has been found that a simple modification to the circuit of the ORNL model Q-2564 high-sensitivity controlled-potential coulometric titrator improves the stability of the instrument and eliminates amplifier limiting that otherwise can occur. It is intended to field modify five titrators now in use in the Division (Analytical Services Laboratories).

Several additions and revisions were made to the circuit of the prototype of the controlled-

potential dc polarograph-voltammeter (ORNL model Q-2792) that improve its performance. These changes have also been incorporated into two of the Q-2792 instruments that are in use in the Division (Analytical Services Laboratories). The performance of the improved instrument is being tested extensively.

An apparatus (ORNL Q-2942) was developed for the precise electromechanical control of the drop time (t) of a polarographic D.M.E., and its performance was evaluated. The t achieved with this apparatus is independent of sample constituents, capillary characteristics, and electrode potential; a short t can be obtained with a low flow rate of mercury and negligible convective disturbance to the current at a D.M.E. The t is substantially more reproducible than that of a good uncontrolled D.M.E. at constant potential. Two additional units have been fabricated and checked out and are being used with two of the Q-2792 polarographs now in service in the Services Laboratories.

A study of the long-term precision attainable in single-cell first-derivative dc polarography is under way. By control of temperature, drop time, mercury head, sparging, and instrument stability, a short-term $S = 0.03\%$ can be achieved, but long-term $S \approx 1\%$. It is planned to investigate the utility of controlling directly the average mercury flow rate as a means to improve the long-term precision of this polarographic method.

An analog time integrator was fabricated. It has been used by the X-Ray and Spectrochemistry Group to calibrate a light-beam splitter that will be used in turn to calibrate photographic emulsions.

A current amplifier was built; it has a calculated maximum error of $\pm 0.1\%$. It is now being used by the Nuclear and Radiochemistry Group.

An automatic output sampler was designed for obtaining samples from the output of a continuous-flow analyzer and for transferring the samples to

filter-paper disks. It is being used satisfactorily by the Analytical Biochemistry Group for the semi-automated determination of transfer ribonucleic acids.

A chapter, "Remote Pipetting," has been prepared for inclusion in Volume 10 of *SERIES IX, ANALYTICAL CHEMISTRY, Progress in Nuclear Energy*. Instruments and apparatus for pipetting, designed specifically for and presently used in fully enclosed shielded hot cells located in this country and in Europe, are described.

The electronics unit of a model VI flame spectrophotometer has been redesigned to improve its performance and reliability; the model VI-A instrument is in use for remotely controlled determinations by the X-Ray and Spectrochemistry Group. The electronics units of two modified model Q-1887 flame spectrophotometers have also been redesigned (model VI-B) and are in use by the X-Ray and Spectrochemistry Group. A complete model VI-C flame spectrophotometer is being designed; it will be used by the Radioisotopes-Radiochemistry Laboratory.

The third and fourth papers of a series of five on controlled-potential differential dc polarography were published; the fifth was accepted for publication. These three papers are on the subjects of comparative and subtractive polarography.

The ultimate sensitivity with which a signal can be measured in the inevitable presence of noise depends on the signal-to-noise ratio, S/N. If the noise that accompanies the signal is random and if a large number of repeated measurements can be made and summed in register with respect to the signal, the S/N of the processed data is increased relative to that of a single measurement in proportion to the square root of the number of measurements. It has been shown that the sensitivity of measurement with a flame photometer is increased substantially by use of a signal-averaging computer to enhance S/N. Several other investigations with a signal-averaging computer have been planned.

2. Effects of Radiation on Analytical Methods

The effects of radiation on various analytical methods and reagents were studied further. New methods or conditions to minimize the deleterious effects of radiation were evaluated.

A voltammetric method for micro amounts of fluoride was developed. It is based on the reaction

of fluoride with the zirconium-alizarin red S complex and the anodic reactions of alizarin red S at the rotating pyrolytic-graphite electrode (R.P.G.E.).

For the separation of fluoride, a rapid distillation usable in hot cells was developed. Fluoride is distilled as fluosilicic acid, which is then measured spectrophotometrically as the alizarin complexone. The chromophore is radiation sensitive; its absorbance decreases 1% per 1000-rad dose of gamma radiation from ^{60}Co .

The uranium thiocyanate complex used in the spectrophotometric determination of uranium was proved to be radiation sensitive. Optimum conditions for using the method under radiation were established.

Relative to the Spray and Absorption Technology Program of the Reactor Division, a study was begun of the effects of radiation on various radiation-safeguard spray solutions. Solutions of thiosulfate were investigated. In this connection, the anodic reactions of thiosulfate at the R.P.G.E. were determined in an effort to establish a highly sensitive, rapid, and selective voltammetric method for following the effect of gamma radiation on thiosulfate. Thiosulfate has anodic waves at $\sim +0.8$ and $\sim +1.1$ v vs S.C.E. whose heights are proportional to thiosulfate concentration. The $+0.8$ -v wave results from the oxidation of thiosulfate to tetrathionate, the $+1.1$ -v wave from oxidation of tetrathionate. These findings indicate that a voltammetric method for thiosulfate is feasible.

It was found possible to use radiolytically generated reagents for homogeneous separations. With chloral hydrate as a source of radiolytically produced chloride and acid, silver was precipitated as the chloride and barium as the sulfate. The use of radiolytic reactions offers a fresh approach to the study of nucleation processes.

In conjunction with Project Salt Vault, studies were made to determine the effects of the radiation from the radioactive wastes on water and other materials present in natural salt (NaCl). Hydrochloric acid was found to be a by-product of the storage of highly radioactive wastes in salt formations. It was shown that the hydrolysis of iron chloride in shale, which is an impurity in natural salt, releases HCl. Irradiation of solid NaCl caused no measurable release of chlorine at elevated temperatures. The release of an unidentified oxidant thought to be an organic peroxide was observed at the demonstration site.

3. Analytical Chemistry for Reactor Projects

The determination of oxide in highly radioactive MSRE fuel samples was continued. Oxide concentrations of several samples from the second ORR molten-salt loop were also determined. The replacement of the moisture-monitor cell was the first major maintenance performed since the oxide equipment was installed in the hot cell. A method was developed for the determination of U^{3+} in radioactive fuel by hydrogen reduction. Oxidized species in molten fuel salt samples are reduced by sparging the samples with hydrogen. The rate of production of HF is a function of the ratio of the oxidized to reduced species in the melt. A computer program was developed to calculate the expected HF yields for any melt composition. The fuel samples run to date do not reflect the beryllium additions made to reduce the reactor fuel, a fact possibly accountable for by an interference stemming from the radiolytic generation of fluorine in the fuel samples. This problem will be investigated further. Also, a method is being developed for the remote measurement of ppm concentrations of HF in helium or hydrogen gas streams. The effects of BF_3 on MSRE pump oil were studied. Measurements were made of increases in hydrocarbon concentrations of an He- BF_3 gas stream after contact with the oil. A thermal-conductivity detector was used to monitor the BF_3 concentration in the test gas stream. Development studies are being made on the design of a gas chromatograph to be used for the continuous determination of sub-ppm and ppm concentrations of permanent-gas impurities and water in the helium blanket gas of the MSRE. This problem of analyzing radioactive gas samples prompted the design and construction of an all-metal six-way pneumatically actuated diaphragm valve. A helium breakdown-voltage detector with a glass body was designed and constructed to permit the observation of the helium discharge. Under optimum conditions this detector has exhibited a minimum detectable limit below 1 ppb of impurity.

Design work was continued on the experimental molten-salt test loop that will be used to evaluate electrometric, spectrophotometric, and transpiration methods for the analysis of flowing molten-salt streams. Measurements of the potentials of the Ni/Ni²⁺ couple in molten fluoride salts have indicated that this couple may be useful as a reference electrode for fluoride melts. Controlled-

potential, voltammetric, and chronopotentiometric studies were carried out on the reduction of U(IV) in molten fluoride salts; a new cyclic voltammeter was used. It was concluded that the U(IV) \rightarrow U(III) reduction in molten LiF-BeF₂-ZrF₄ is a reversible one-electron process but that adsorption phenomena must be taken into account for voltammetric measurements at fast scan rates or for chronopotentiometric measurements at short transition times.

To permit the observation of absorption spectra of highly radioactive materials, it is planned to install a spectrophotometric facility having an extended optical path integral with a hot cell. The basic spectrophotometer and associated equipment have been ordered. Spectrophotometric evidence has shown that 2LiF-BeF₂ is compatible with SiO₂ in a system that contains excess SiF₄ at temperatures up to $\sim 1000^\circ K$. The ability to contain 2LiF-BeF₂ melts in silica cells permitted more precise measurement of the molar absorptivities of U(IV) than had been possible with windowless cells. Studies of the solubility of Cr(III) in 2LiF-BeF₂ at 550°C were also made in silica cells. An investigation of the spectra of U(VI) in molten fluoride salts has been initiated. The simultaneous electrochemical generation and spectrophotometric observation of species in molten fluorides was demonstrated experimentally. The spectra of these species are obtained in the vicinity of the working electrode and should permit the direct identification of unusual and perhaps unstable solute species. The reduction of U(IV) and reoxidation of U(III) has been observed with this system.

In the production of UO₂ microspheres by the Sol-Gel Process, an important step is the removal of carbonaceous impurities by treatment with steam. A system was designed and constructed to continuously sample and determine the hydrocarbon content of the steam from tests on this procedure. The system has been used to monitor the hydrocarbon content of steam from the decarbonation of various UO₂ samples as a function of temperature and time. An in-line method was needed to monitor the concentrations of the reagent reduction products, BrF₃ and Br₂, in the effluent from the Fluidized-Bed Volatility Pilot Plant. Some success was realized with a gas chromatographic technique by use of a 7-ft Alcoa T-60 alumina column with a 10% Kel-F loading. Elution times were determined for BrF₃, BrF₅, Br₂, and UF₆. This study was

terminated when construction of the Fluidized-Bed Volatility Pilot Plant was discontinued.

The design of an analytical system for the in-line analysis of the gases generated by in-pile tests of fuel elements was changed to incorporate a quadrupole residual-gas analyzer. The substitution of the residual-gas analyzer will permit the measurement of more components over greater ranges of concentrations and at an acceptable analysis frequency. Tests of a quadrupole residual-gas analyzer in our laboratories indicate that the instrument will function properly in the proposed application. Therefore, specifications have been written and submitted for bids. It will still be necessary to make a separate determination of CO. A CO infrared analyzer was installed in the gas-sampling hood at the ORR and was calibrated. Actual tests carried out with irradiated gases from an in-pile experiment showed that the instrument and CO measurements are not affected by radiation at the maximum anticipated level. The program was continued on the gas chromatographic determination of volatile organic air pollutants that are most likely to form volatile organic iodides with fission product iodine. The results of the determination of total, unsaturated, and oxygenated hydrocarbons are tabulated for samples taken from the secondary containment of each reactor at ORNL, the Nuclear Safety Pilot Plant, various locations external to research buildings, and the Clark Center Recreation Park. Samples from the CVTR in Columbia, South Carolina, were also analyzed.

4. Special Research and Development Activities

For gas chromatography a new preparative gas chromatograph was acquired. Work with it has consisted exclusively of the purification of organic compounds. The major factor in obtaining ultrapure compounds is the proximity of the elution peaks of the impurities to the peak of the parent compound. A modification of the chromatograph developed at ORNL allows different cut points to be selected on each side of the parent peak and provides the possibility of producing compounds of 99.99% purity. A method was developed for the gas chromatographic determination of ppm concentrations of sulfur hexafluoride in methane. The results from triplicate samples show a deviation from the mean of $\pm 5\%$. A pyrolysis-gas-chromatographic technique was

used to determine the volatile organic substances in a mold-release compound and in aluminum powder that caused difficulty for the Metals and Ceramics Division in extruding aluminum parts by a powder metallurgical process.

Precise procedures were developed for resolving complex mixtures of constituents of tRNA by thin-layer chromatography on PEI-cellulose. The tRNA constituents in alkaline hydrolysates of *E. coli* B and of yeast were isolated and identified. A method was perfected to separate and identify the mushroom toxins α -, β -, and γ -amanitin. Numerous samples of methanol extracts of mushrooms and mushroom pellets were analyzed as a means to determine the efficiency of fermentation processes for the production of the toxins in quantity. The cross-linked dextran Sephadex G-75 Superfine Gel was evaluated for the thin-layer chromatographic separation of proteins according to molecular weight. The few experiments made did not give satisfactory separations.

In electroanalytical studies a vertical-orifice rapid Teflon D.M.E. was shown to be suitable for rapidly obtaining reproducible and theoretically correct polarograms when the solution polarographed contained 0.1 w/v % polyacrylamide to suppress maxima. Data that justify this conclusion were obtained for the $Tl^+ \rightarrow Tl^0$ reference reaction. A 1 M hydroxylamine hydrochloride solution was found to be a suitable supporting medium for the polarographic determination of U(VI) in the presence of Cu(II), because the half-wave potentials are separated by ~ 270 mv. Studies of ion-specific electrodes revealed that an optimum pH range exists for each electrode. Different makes of calcium, divalent, fluoride, and nitrate ion-specific electrodes were evaluated.

A new Beckman IR-12 infrared spectrophotometer was put into operation, and many organic and inorganic materials were examined with it.

A nuclear magnetic resonance spectrometer has been used to elucidate the chemical structure and configuration of a number of organic compounds as a service to several research divisions.

A variety of special work included the polarographic determination of W in W-Re thermocouple wire; study of arsenazo as a chromogenic reagent for plutonium; development of fluorometric methods for phosphate with aluminum-morin and tin-flavonol systems; sealed-tube dissolution of uranium nitride; pyrohydrolysis of fluoride-containing materials by

use of quartz or platinum combustion tubes; determination of periodate, iodate, and iodide in mixtures of the three; density-gradient determination of the density of pyrolytic carbon particles; and study of the autodecomposition of bromate in acid nitrate media. A Cary model 14 spectrophotometer is being fitted with a glove box to permit spectrophotometric studies of highly radioactive alpha-emitting substances. Also, a Leco Nitrox-6 Analyzer was installed in a glove box in the High-Radiation Analytical Facility (HRAF) to determine oxide-to-metal ratios of alpha-emitting metal oxides. The effect of salt content on the absorbance of solutions of tRNA's was studied. Two chapters are being prepared for inclusion in Volume 10 of SERIES IX, ANALYTICAL CHEMISTRY, **Progress in Nuclear Energy**.

5. Analytical Biochemistry

Analytical assistance to the Biology and Chemical Technology Divisions was continued as part of the Macromolecular Separations, Body Fluids Analysis, and Molecular Anatomy Programs. In the analysis of transfer ribonucleic acids (tRNA's), the optimum conditions for the assay of valine- as well as leucine- and phenylalanine-accepting tRNA's were established. Techniques for the isolation of aminoacyl synthetase enzymes were examined, and an improved enzyme preparation of higher purity and specific activity has been prepared. As part of a continuing effort to improve the analysis, various disk pretreatments were tested. The assay procedure has been automated; 40 samples per hour can be analyzed with precision and accuracy not significantly different from those of manual assays. Studies of the separation of nucleic acid components by ligand-exchange chromatography were continued, and this technique is now used routinely to determine terminal nucleoside, base ratio, and molecular weight.

In the Body Fluids Analysis Program, ultraviolet-absorbing components isolated from urine by anion exchange chromatography are being collected for characterization by infrared, nuclear magnetic resonance, and mass spectrometry. Because only a very small quantity of many of these compounds will be available, other techniques such as the use of ^{14}C -labeled compounds and preconcentration by gel-permeation chromatography are also being applied.

In the Molecular Anatomy Program, our main objective is to isolate and characterize antigen activity in virally induced tumors. Gel electrophoresis, both analytical and preparative, is used extensively. A new method, centrophoresis, in which proteins migrate through a density gradient in both electrical and centrifugal fields, is now being used, and antigen activity has been concentrated. The amino acid content of various normal tissues and of tumorous tissue was measured to determine whether significant differences exist. Urine samples from normal persons and from patients with various pathological conditions were also analyzed. At present the results of both these studies are inconclusive.

The bulk of the amino acid analytical work is now concerned with sequence analysis of proteins. The sequence of the β chain of mouse hemoglobin has been partially determined.

6. X-Ray and Optical Spectrochemistry

Computer subroutines were written to calculate and to plot chemical compositions from electron-probe data by use of fundamental physical data stored on tape. The ASTM Committee E-2 "Tentative Recommended Practice in Photographic Photometry" was revised completely. A beam splitter was designed and tested for use in calibrating spectrographic emulsions. To facilitate the chemical collection of impurities in bone ash, phosphate was removed by electro dialysis through anion-permeable membranes; a similar method is being used to prepare ultrapure reagents. Commercial freeze-dried foods are being evaluated as possible standards for the interlaboratory-check analysis of tissue. The limits of sensitivity for the determination of rare-earth elements in LiF-BeF_2 type nuclear fuel and of numerous trace elements in $^{233}\text{UO}_2\text{-ThO}_2$ were extended by combining preconcentration procedures with arc spectrographic analysis.

7. Mass Spectrometry

Work with the MS-7 spark-source mass spectrograph increased 30%. Unusual alloys and metallic samples made from separated stable isotopes were analyzed for trace constituents. The spark-source technique for the analysis of solutions was shown to be satisfactory for determining Nb in ^{231}Pa and

for studying the penetration of irradiated graphite by constituents of MSRE salts.

A single-magnetic-stage mass spectrometer was constructed and put into use for the analysis of organic compounds. A wide variety of samples of mass up to 600 have been analyzed satisfactorily.

The first ionization potentials of the lanthanides were measured by the surface-ionization comparison technique.

A mass spectrometric search was made for neon in minerals and in volcanic glasses. The analyses are important in distinguishing between primordial and radiogenic components and their role in the origin of the atmosphere and its relationship to the origin of the solar system and the genesis of elements.

A high-resolution double-focusing mass spectrometer was assembled and tested. It has a resolution of 4000 and accepts solid and gaseous inorganic samples.

8. Optical and Electron Microscopy

Optical and electron microscopy and diffraction studies were made on a wide variety of materials to give research assistance in support of numerous ORNL and other AEC-sponsored programs concerned with nuclear technology. Four typical studies involving fundamental and applied chemistry, metallurgy, and nuclear safety are discussed briefly.

The examination of highly radioactive materials in the form of prepared particles, corrosion products, and replica-stripped material has continued at the electron microscopy facility in Building 3019. This work is illustrated by an electron micrograph of particulate $^{238}\text{PuO}_2$.

Several new instruments have been installed; among them are a dark-field device for the Philips EM 200 electron microscope and a precision plate developer for electron-microscope plates. A Philips EM 300 electron microscope has been ordered for studying highly radioactive alpha emitters at the Transuranium Processing Plant.

9. Nuclear and Radiochemical Analyses

Gamma branching in ^{95}Zr and cross sections for production of ^{113}Sn and ^{250}Bk by neutron capture were measured. Alpha decay of the new isotope ^{154}Ho was studied, and the standardization of

^{197}Hg was firmed up through interlaboratory comparisons. A film-recording radiation-event monitor for pulsed x and gamma radiation was developed, the use of the beta-excited isotopic-light-source photometer was extended to the ultraviolet region, and a semiconductor x-ray emission spectrometer that has an isotopic x-ray source was investigated. The development of radiation-stimulated light sources continued. Improvements to and computer applications in analytical alpha spectrometry were made.

A number of new facilities have been studied, proposed, or commenced; these include a facility for activation analysis with a ^{252}Cf source, a high-intensity analytical cyclotron, an activation analysis laboratory at the HFIR, target assemblies and pneumatic transfer for photon and fast-neutron activation at the ORELA, possibility of using the 86-in. cyclotron as a neutron source, and a prototype low-level radiation counting laboratory for the Lunar Receiving Laboratory in Houston. Work continued on interactions of ^3He particles with selected low-Z elements, and a method to calculate sensitivities, interferences, and optimum bombardment energies was developed. The recoil technique was evaluated as an aid in activation-analysis nuclide identification. A number of applied activation analysis problems were solved, and a chapter on radioactivation was prepared for a handbook.

A new 14-Mev neutron generator facility for determining oxygen in alkali metals was completed; the older system, however, has continued to be used for some applied problems. Fluorine was determined in fluorspar ores, and a dual-axis sample rotator was developed to assist in this and other problems that require high precision. Development of the leached fuel element monitor was completed.

The Cooperative Isotopes Program continued with technical assistance, quality control, and aid in isotope characterization being supplied. High-energy gamma rays in ^{233}U , ^{239}Pu , and ^{241}Am were studied, the half-lives of a number of radio-nuclides were redetermined, and a radiochemical study was made of the HFIR cooling water. Attempts are under way to use radioisotopic tagging as an aid in locating stolen copper wire.

New separation methods reported include: californium from curium and berkelium from cerium by extraction chromatography, americium by liquid-liquid extraction, berkelium(IV) from cerium(IV) by

anion exchange, and liquid-liquid extraction of niobium with TTA. Pure $^{245,246}\text{Pu}$ was prepared; evidence for photofission of iron was reported. A method to improve liquid-scintillation counting in aqueous systems was developed. Water research was broadened to include assistance in testing of desalination plants, ecological investigations of local watersheds, application of tracers and activation analysis in tracing the flow of rivers and streams, and removal of sodium from activated samples.

In the transuranium research program a laboratory was equipped for special analytical service, a number of lanthanide and actinide β -diketones were prepared, controlled-potential coulometric titration of americium was reported, and the feasibility of determining the formal potential of the Bk(III)-Bk(IV) couple was studied.

10. Inorganic Preparations

Programs of the preceding year were continued. These included the preparation of fused salts, mostly rare-earth-metal halides, for the Chemistry Division; and for the Solid State Division: the preparation of compounds of the spinel (MgAl_2O_4) structure, the preparation of GeO_2 glasses, the production of high-purity KCl, and the heat treatment of rare-earth-metal alloys. In conjunction with this latter work, a project was undertaken to produce 50 g of the isotope ^{160}Gd in the metallic form by the reduction of $^{160}\text{GdF}_3$.

For the Physics Division a series of alloys and compounds that contain isotopes of germanium and of nickel were prepared. Other miscellaneous single preparations were made for various groups.

11. Organic Preparations

The Organic Preparations Laboratory, as in past years, continued to support ORNL research divisions through custom synthesis of organic compounds needed for experimental activities. These compounds included *p*-aminobenzylamine, heptadecylamine, tri-*n*-butylbenzylphosphonium chloride, tetra-*n*-butylphosphonium bromide, α,α' -dipiperidyl, cesium *p*-ethylbenzenesulfonate, and bicyclo-[1.1.0]butane.

Divisions using the services of the Organic Preparations Laboratory were: Analytical Chemistry, Chemistry, Chemical Technology, and Metals and Ceramics, and the K-25 Technical Division.

PART B. SERVICE ANALYSES

12. Mass Spectrometric Analyses

The work of the Mass Spectrometry Service Laboratory increased 25% over last year, an increased number of samples being submitted from the Isotopes and Reactor Chemistry Divisions. Isotopic abundances of samples from irradiated HFIR components were determined. Purity of compressed gases was certified. An electron bombardment source that uses a five-sample wheel was developed and tested for use with the 12-in. 90° mass spectrometer.

The Transuranium Mass Spectrometry Laboratory analyzed 88% more samples than last year, at the same time decreasing the cost per analysis to about half. The isotopic analysis of all elements from thorium through californium was done routinely. Sample types became increasingly diversified.

13. Emission Spectrochemical Analyses

The Spectrochemistry Laboratory analyzed about 13% more samples than last year. Trace impurities were determined in the stable isotopes of 45 elements. Also, a large number of samples of air, water, metals, and unusual alloys were analyzed.

14. Process Analyses

The Process Analyses Laboratories performed 208,523 analyses, a 65% increase over the past year with no increase in personnel. The increases were samples received from the Transuranium Process and Biochemical Programs. Brief statements of the new developments and work in each laboratory follow.

The High-Level Alpha Radiation Laboratory determined californium and einsteinium in solutions from the Transuranium Processing Plant. Fluoride was isolated by pyrohydrolysis and measured spectrophotometrically. Isotopic dilution and mass spectrometry were used to measure extremely low levels of plutonium.

The General Analyses Laboratory completed the development on a new vacuum-fusion gas chromatographic analyzer; the cost of vacuum-fusion analyses was thereby reduced to half. The precision of the determination of tRNA's was improved significantly. New methods were used to

assist the Molten-Salt Reactor and coated-particle-development work. Assays at the ppb level were made in water-pollution studies.

The General Hot-Analyses Laboratory continued to supply analytical support to other ORNL divisions. A glove-box facility was installed and put into service. The MSRE analyses continued with no problems. Some analytical work was done for facilities outside ORNL.

In the Radioisotopes-Radiochemistry Laboratory most of the work was in support of the programs of the Reactor and Reactor Chemistry Divisions. Some analyses are being made on new experimental targets from the 86-in. cyclotron. A 4096-channel analyzer and a flame photometer were ordered.

The South Annex of Building 2026 (HRLAL) was completed and has been occupied.

The number of control samples included in the Statistical Quality Control Program decreased during the past year by 14%; the overall quality of the work improved.

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 1966. The updated indexes are available from the Clearinghouse for Federal Scientific and Technical Information; they are designated TID-7015 (Indexes), Revision 4.

The ninth supplement to the reprinted form of the *Manual* (TID-7015, Suppl. 9) was issued; it includes two new methods and revisions to three methods. Ten additional methods, not included in Suppl. 8, were written for record only. 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

The research program of the Analytical Chemistry Division is indicative of the diversification of the total effort in analytical chemistry at the Oak Ridge National Laboratory.

Investigations in analytical instrumentation, nuclear methods of chemical analysis, electro-analytical chemistry of the molten state, radiation effects on analytical methods, and mass spectrometry are carried out for the Physical Research Division of the Atomic Energy Commission. Studies of applications for radioisotopes are done for the AEC Division of Isotopes Development.

Analytical research and development is also being conducted in several areas of biochemistry and bioengineering for the National Institute of General Medical Sciences of the National Institutes of Health. Programs were continued to provide specific assistance to research divisions and projects of the Oak Ridge National Laboratory, for example, the Molten-Salt Reactor Project and Transuranium Process Program.

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

1. Analytical Instrumentation

D. J. Fisher
R. W. Stelzner

1.1 CONTROLLED-POTENTIAL CONTROLLED-CURRENT CYCLIC VOLTAMMETER

T. R. Mueller H. C. Jones

The prototype of the controlled-potential controlled-current cyclic voltammeter^{1,2} was released for evaluation in voltammetry of molten salts in December 1966. As a result of its initial applications, several design changes were made. An instrument that incorporates some of the design changes was constructed in the Department of Chemistry, University of Tennessee.

¹T. R. Mueller and H. C. Jones, "Controlled-Potential and Controlled-Current Cyclic Voltammeter," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1966*, ORNL-4039, p. 1.

²T. R. Mueller, "Controlled-Potential and Controlled-Current Cyclic Voltammeter," presented at the Analytical Chemistry Instrument Demonstration Conference, An Industrial Cooperation Conference, Oak Ridge National Laboratory, Oak Ridge, Tenn., Oct. 16-17, 1967.

Because of the excellent performance of the prototype, a third instrument (ORNL model Q-2943) was constructed by the Instrumentation and Controls Division for the Reactor Chemistry Division. The maximum current capability of the Q-2943 is 500 ma; its performance specifications are identical with those of the prototype. It was fabricated in the Standard Nuclear Instrument Modules (NIM)³ system (ORNL Q-2800) to facilitate its use as a research instrument, to simplify its duplication through the use of printed circuits, and to provide for plug-in modules to extend its capabilities by the addition of other functions (e.g., current integration and current- or potential-time differentiation).

Because the fast circuitry of the cyclic voltammeter requires wide amplifier bandwidth, considerable difficulty has been experienced in attempts to eliminate the spurious response triggered by

³L. Costrell, "Standard Nuclear Instrument Modules," TID-20893 (Rev.), January 1966.

noise pickup at the cell. Much of the noise was traceable to modulated rf signals produced in adjacent laboratories. Accordingly, data collection was restricted to periods when these noise sources were not in operation. Radio-frequency shielding of the cell has been recommended.

With some cells it is difficult to stabilize the potentiostat. It is hoped that current investigations will reveal whether the cell configuration or external noise pickup is the primary cause of instability.

In spite of these annoyances, the prototype and the second instrument have been used successfully to obtain both analytical and mechanistic data in molten-salt media. The results of these studies will be published elsewhere. The second instrument has also performed satisfactorily in room-temperature experiments made at the University of Tennessee with aqueous and nonaqueous solvent systems.⁴

Although the voltammeter undoubtedly will undergo further minor changes, the basic instrument is a useful research tool. A manuscript that describes the voltammeter is being prepared for publication. A check-out and test procedure that explains the operation of the instrument and indicates the adjustments and calibration required when it is put into use was written. This procedure can also be used in servicing and routinely checking the instrument.

1.2 DUAL SET-POINT VOLTAGE COMPARATOR (ORNL MODEL Q-2950)

T. R. Mueller

Generally when counter timers are dc coupled an error of about $\pm 0.3\%$ may be introduced in the "time interval" mode. The error arises from drift in the gating levels of the "start" and "stop" inputs. Calibration of linear voltage ramps in the controlled-potential controlled-current cyclic voltammeter (Sect. 1.1) required that the timing error be no greater than $\pm 0.05\%$ for rates of rise in the range 0.005 to 500 v/sec.

To achieve accurate timing, a low-drift comparator was developed that has an operational amplifier in the input stage. When operated from a power supply with $\pm 0.01\%$ regulation, this device senses the input voltage with a maximum error of ± 0.1 mv.

Two set points are obtained by electronically switching the reference voltage. When the low-voltage set point is reached, output and trigger pulses are generated. The output pulse starts a timer. The trigger pulse changes the state of a bistable multivibrator to establish the high-voltage set point. When the input voltage reaches the high-voltage set point, output and trigger pulses are again generated. The output pulse stops the timer. The trigger pulse resets the multivibrator and thus reestablishes the low-voltage set point.

The accuracy of the voltages of the set points was ensured in the following way. Direct-current voltages were applied to the input, and the appearances of the output pulses were observed. Voltage ramps with rates of rise between 0.1 and 10 v/min, calibrated by independent absolute methods, were then presented to the comparator. Rates of rise calculated from time intervals obtained with the comparator agreed with the known values to within $\pm 0.01\%$ for a 2-v interval. Measurements on the 500-v/sec ramp in the cyclic voltammeter indicated a relative standard deviation (S) $< 0.1\%$, whereas previous measurements that used the gating circuits in several time-interval meters showed $S > 0.3\%$. Additional characteristics of the comparator are given below.

Input

Set-point interval: 2 ± 0.5 v; can be changed to 20 v without change in other specifications

Set-point drift at 25°C: < 0.1 mv; 10 μ v short term

Rate of rise of ramp: dc to 20,000 v/sec

Output Pulse

Amplitude: -13 v

Rise time (90%): < 0.2 μ sec

Duration: 10 μ sec; can be shortened if required

Power

$+15$ v dc at 90 ma; -15 v dc at 6 ma

⁴G. Mamantov, Consultant; Associate Professor of Chemistry, University of Tennessee, Knoxville; personal communication to T. R. Mueller, Aug. 16, 1967.

1.3 SOLID-STATE CONTROLLED-POTENTIAL COULOMETRIC TITRATOR

H. C. Jones T. R. Mueller

The design of the solid-state controlled-potential coulometric titrator⁵ is being modified to allow the use of modular construction techniques (NIM).³ It is expected that the use of four modules in the construction of this instrument will minimize the downtime required for servicing by permitting direct substitution of modules. The design of cells currently used with our coulometric titrators is being reevaluated to optimize overall performance.

1.4 MODIFICATIONS TO THE HIGH-SENSITIVITY CONTROLLED-POTENTIAL COULOMETRIC TITRATOR (ORNL MODEL Q-2564)

T. R. Mueller H. C. Jones

We reported previously⁶ that the design of the model Q-2564 high-sensitivity controlled-potential coulometric titrator⁷ was being changed to eliminate titration errors which result from overload or instability of the potentiostat and that the performance of the modified instrument was to be evaluated. One instrument was modified. Its performance indicated that a modification less extensive than that originally planned should be adequate. Accordingly, the simple modification was made in a second instrument, which was then tested extensively with electroanalytical cells in use in several of the Analytical Services Laboratories. Both mercury-pool and platinum-gauze working electrodes, as well as cells of different geometries, were used. P. S. Gouge,⁸ R. Burns,⁹ and J. R. Stokely¹⁰ assisted with the testing.

⁵H. C. Jones, "Solid-State Controlled-Potential Coulometric Titrator," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1966*, ORNL-4039, p. 1.

⁶T. R. Mueller and H. C. Jones, "Modifications to the ORNL Model Q-2564 High-Sensitivity Controlled-Potential Coulometric Titrator," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1966*, ORNL-4039, p. 7.

⁷H. C. Jones, W. D. Shults, and J. M. Dale, "High-Sensitivity Controlled-Potential Coulometric Titrator," *Anal. Chem.* **37**, 680 (1965).

⁸General Analyses Laboratory.

⁹General Hot-Analyses Laboratory.

¹⁰Methodology Group.

Both instruments were stable, the titration times were not materially lengthened, and errors due to amplifier limiting were eliminated. The four other instruments of this type in use in the Division will be modified in the simpler way.

1.5 CONTROLLED-POTENTIAL DC POLAROGRAPH-VOLTAMMETER (ORNL MODEL Q-2792)

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

The circuit of the ORNL model Q-2792 controlled-potential dc polarograph-voltammeter^{11,12} was revised to incorporate recently available superior components and to make changes proved desirable by operational experience. The following changes or additions were made in the prototype instrument:

1. The current amplifier was changed from Philbrick Researches, Inc., type SP656 to type SP2BU to decrease flicker noise and recovery time.
2. The potential-control amplifier was changed from type SP656 to type EP45ALU to decrease recovery time after an overload in the potentiostat circuit.
3. The amplifiers in the filter circuits were changed from type EP85AU to type EP25AU to reduce noise level.
4. The operational amplifiers in all derivative and inverter circuits were changed from type P35A to type EP25AU to eliminate tedious trimming steps in the check-out procedure.
5. A potential-correction circuit was added for the input to the X axis of the X-Y recorder to produce polarograms having accurate $E_{1/2}$ values for the regular, first-, and second-derivative modes at all available scan rates. Figure 1.1 illustrates the effect of this correction circuit for first-derivative polarograms.

¹¹W. 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. 4-6.

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

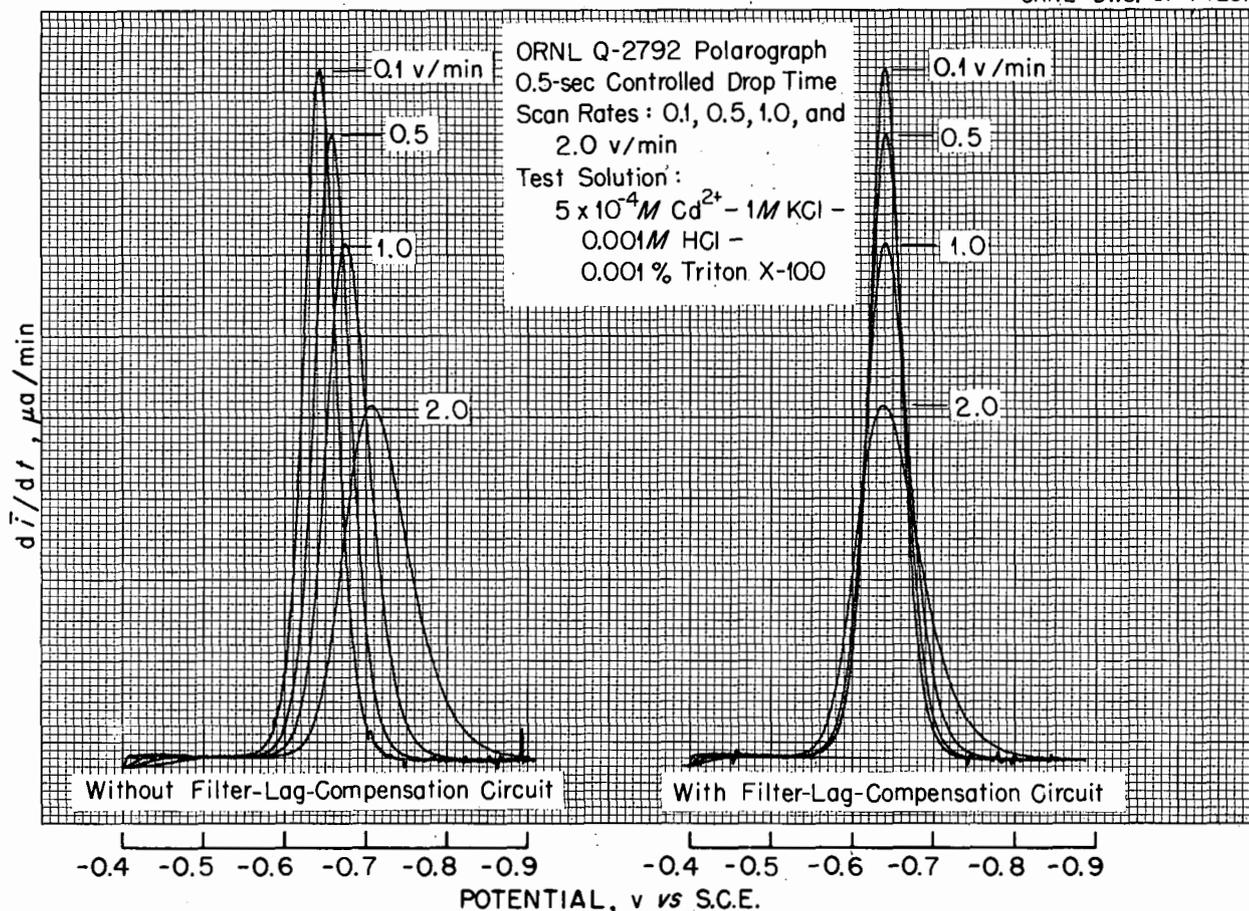


Fig. 1.1. First-Derivative Polarograms of $5 \times 10^{-4} M$ Cd^{2+} With and Without Potential Correction on the X Axis of the Recorder.

6. A diode was placed across the Y input of the X-Y recorder to prevent Y servo-amplifier overload in the recording of a first-derivative polarogram.
7. The capacitors in the first- and second-derivative computing circuits were changed to a type of higher quality (polystyrene) to increase the precision and accuracy of these circuits.
8. Components affecting the frequency response in the first-derivative circuit were returned to their original calculated values, because a superior filtering system has now been installed.¹³
9. The initial-potential control was calibrated for greater convenience in operation.
10. A derivative-offset circuit with a range of 0 to 10 v was added for high-sensitivity polarographic work.
11. Reed relay circuits with high leakage resistance were added to the scan generator to minimize drift.
12. A cell-potential indicator in the form of an external voltmeter was added to assist the operator.
13. Buffer resistors (10 ohms each) were placed in both the X and Y inputs of the recorder to isolate circuit capacitance.

¹³R. W. Stelzner, W. L. Belew, D. J. Fisher, and M. T. Kelley, "Improved Averaging Filters for Polarographic Instrumentation," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1966*, ORNL-4039, p. 3.

Existing drawings were revised to include these changes (ORNL Dwg. Q-2792-R3). Line drawings of both the block diagram (ORNL Dwg. 67-3063A)

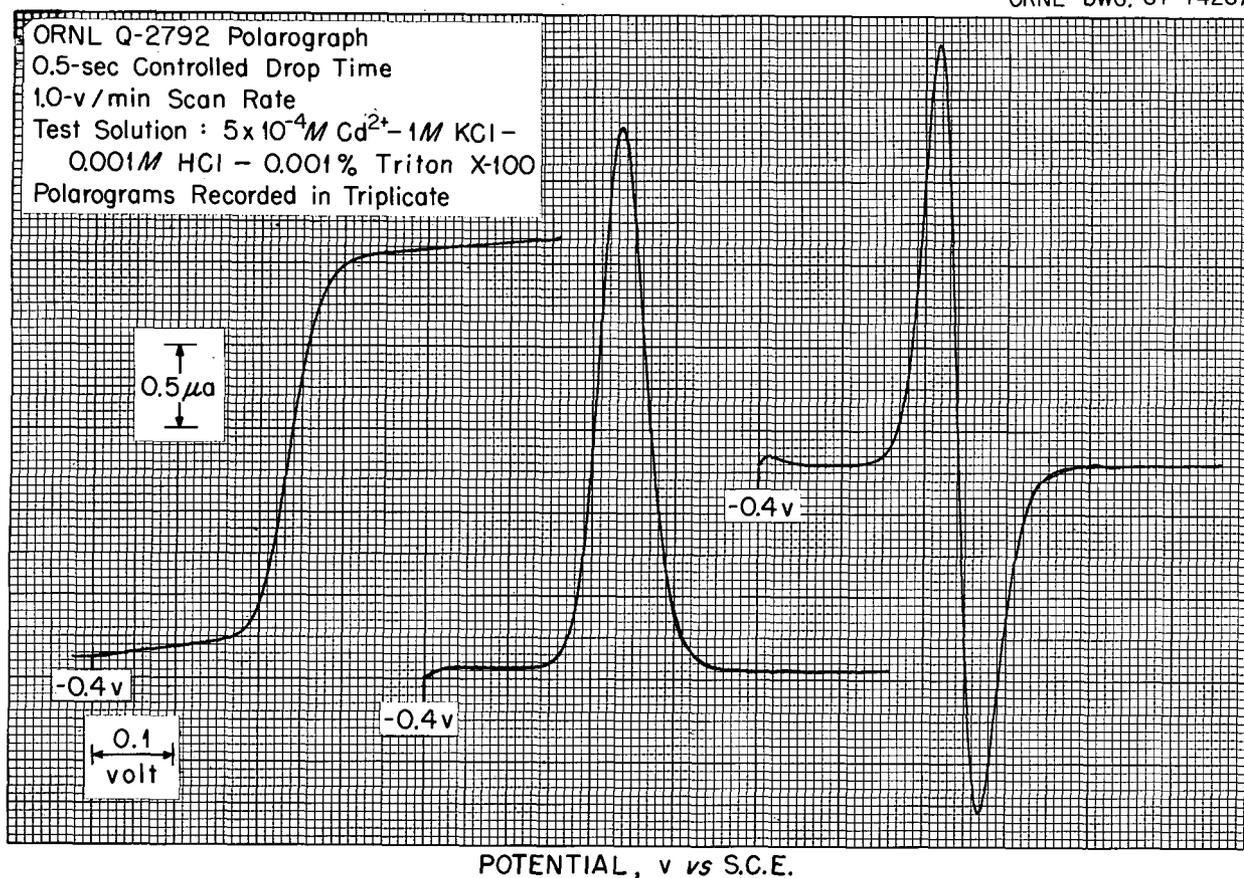


Fig. 1.2. Reproducibility of Three Types of Readout for the ORNL Model Q-2792 Controlled-Potential DC Polarograph-Voltammeter: Regular, First-Derivative, and Second-Derivative.

and of the complete circuit diagram (ORNL Dwg. 68-6567R) were prepared for use in a publication.

Arrangements were made with the ORNL Instrumentation and Controls Division for field modification of the serial Nos. RO-0002 and RO-0003 instruments to bring them up to date with the current Q-2792-R3 drawings.

Extensive testing of the revised prototype instrument to determine whether it adheres to specifications under simulated and actual electrochemical cell conditions has been under way for several months. This study has been facilitated by preliminary design work on a simulated polarographic cell. Figure 1.2 illustrates the reproducibility of the three types of readout obtained with the Q-2792 polarograph-voltammeter: regular, first-derivative, and second-derivative dc polarograms for $5 \times 10^{-4} M$ Cd^{2+} solution. In each case,

three replicate polarograms are superimposed by the X-Y recorder.

The Q-2792 instrument has been described orally;¹⁴⁻¹⁶ also, a journal article about the instrument is being written. In addition several

¹⁴M. T. Kelley, "Electroanalytical Research at Oak Ridge National Laboratory," presented at a seminar at Rudjer Bošković Institute, Zagreb, Yugoslavia, Sept. 11, 1967.

¹⁵W. L. Belew, "A Solid-State, Controlled-Potential DC Polarograph and a Precision Drop-Time Controller," presented at the Eleventh Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Oct. 10-12, 1967.

¹⁶W. L. Belew, "Controlled-Potential DC Polarograph-Voltammeter (Q-2792)," presented at the Analytical Chemistry Instrument Demonstration Conference, An Industrial Cooperation Conference, Oak Ridge National Laboratory, Oak Ridge, Tenn., Oct. 16-17, 1967.

papers have been published¹⁷⁻²⁰ that report work done with this instrument.

1.6 APPARATUS FOR PRECISE CONTROL OF DROP TIME OF A DROPPING-MERCURY ELECTRODE (D.M.E.) (ORNL MODEL Q-2942)

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

An apparatus for the precise electromechanical control of the drop time (t) of a dropping-mercury electrode (D.M.E.) was developed and evaluated.^{21,22} The apparatus simplifies the procedure for using a D.M.E., because the t obtained is not a function of sample constituents, capillary characteristics, or electrode potential. Also, the apparatus permits the selection of a short constant t (a particular advantage for first-derivative dc polarography) at a low flow rate of mercury, which conditions are conducive to diffusion-controlled mass transport.

Figure 1.3 is a photograph of the apparatus. The capillary is held in the clamp on the pivot arm directly above the Teflon cap of the cell. The pivot arm is mounted in such a way that it is free to move only in a horizontal plane with respect to the cell cap. The two relay coils are used to actuate the pivot arm and are positioned to allow 0.004 in. free movement of pivot arm between rub-

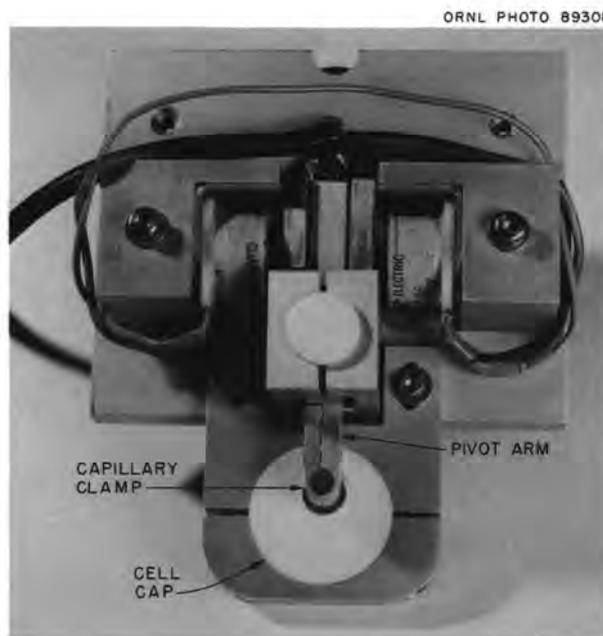


Fig. 1.3. ORNL Model Q-2942 Polarographic Drop-Time Controller.

ber stops. The relay coils are energized alternately to cause movement of the pivot arm from side to side between the stops. In operation, the capillary is suddenly displaced in one direction to shear the mercury drop, held in that position for a fixed period of time, and then displaced in the opposite direction to shear the next drop from the capillary. This method of operation controls the capillary movement at all times and results in the least amount of capillary vibrations and cell-current disturbance. The timing circuit used to fix the drop time is essentially a GE hybrid timing circuit.²³ A unijunction transistor circuit is used as the timing element to activate a flip-flop circuit each time the unijunction transistor fires. Silicon controlled rectifiers are used as switches to control the voltage to the relay coils.

The maximum precision that could be expected from the completed drop-time controller was determined by measuring the reproducibility of the timing circuit. The short-term ≤ 10 min reproduci-

¹⁷H. E. Zittel and F. J. Miller, "Anodic Reactions of the Halides in Dimethyl Sulfoxide at the Pyrolytic Graphite Electrode," *Anal. Chim. Acta* **37**(2), 141 (1967).

¹⁸H. E. Zittel and T. M. Florence, "Voltammetric and Spectrophotometric Study of the Zirconium-Alizarin Red S Complex," *Anal. Chem.* **39**, 320 (1967).

¹⁹H. E. Zittel and T. M. Florence, "Voltammetric Method for Determination of Zirconium," *Anal. Chem.* **39**, 355 (1967).

²⁰F. 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).

²¹W. L. Belew and H. C. Jones, "Apparatus for Precise Control of the Drop Time of the Dropping-Mercury Electrode (D.M.E.) in Polarography," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1966*, ORNL-4039, p. 2.

²²W. L. Belew, "Polarographic Drop Time Controller," presented at the Analytical Chemistry Instrument Demonstration Conference, An Industrial Cooperation Conference, Oak Ridge National Laboratory, Oak Ridge, Tenn., Oct. 16-17, 1967.

²³"Hybrid Square Wave Multivibrator" and "Flip-Flop and DC Latching Relay," *General Electric Silicon Controlled Rectifier Manual*, pp. 52-53 and 96-97, respectively, 2d ed., General Electric Co., New York, 1961.

Table 1.1. Comparison of Drop-Time Precision of Free-Falling vs Controlled-Drop-Time Dropping-Mercury Electrodes

Type of D.M.E. Orifice	Potential (v vs S.C.E.)	Free-Fall		Controlled	
		<i>t</i> (sec)	<i>S</i> (%)	<i>t</i> (sec)	<i>S</i> (%)
Horizontal	0	5.2002	0.25	0.5005	0.007
	-1.0	5.3587	0.33	0.5005	0.005
	-1.5	4.323	0.19	0.5005	0.008
	-2.0	2.6563	0.34	0.5006	0.008
Vertical	0	1.2515	0.27	0.5005	0.008
	-1.0	1.2821	0.18	0.5005	0.007
	-1.5	1.0153	0.46	0.5005	0.010
	-2.0	0.6631	1.40	0.5004	0.033

bility of *t* was <0.01% in all cases. For data collected over a period of 20 days, the *t*'s were reproduced to *S* = 0.03%. Thus, for any one series of polarograms the *t*'s should be reproducible to better than *S* = 0.01%; for extended periods *S* $\hat{=}$ 0.03% can be expected.

To determine whether use of the drop-time controller increases the reproducibility of *t*, individual drop times were measured with a seven-place electronic counter. Measurements were made of the *S* of both the controlled *t* and the free-fall *t* with vertical- and horizontal-orifice capillaries; Table 1.1 gives the data. The data show that use of the drop-time controller significantly increases the reproducibility of *t* and that a constant and precise *t* can be obtained at any potential from 0 to -2.0 v.

Oscilloscopic current-time curves obtained at -0.8 v with 5×10^{-4} M Cd²⁺ in 1 M KCl indicate that no problems caused by stirring or vibration occur when *t* \geq 0.25 sec.

The prototype apparatus has been in use for ten months and has proved to be reliable and precise; it has not required adjustment during this period. Both vertical- and horizontal-orifice capillaries can be used with the device. In the replacement of capillaries no adjustment of the apparatus is required. Mechanical and electrical drawings for the construction of the polarographic drop-time controller have been completed. Two units, designated model Q-2942, were fabricated. These have been put into use in conjunction with two of the Q-2792 polarographs now in the Analytical Services Laboratories.

1.7 INVESTIGATION OF PRECISION ATTAINABLE IN SINGLE-CELL FIRST-DERIVATIVE DC POLAROGRAPHY

W. L. Belew D. J. Fisher
M. T. Kelley R. W. Stelzner
E. S. Wolfe²⁴

Of the various polarographic methods, comparative methods are the most precise but least simple. A simpler high-precision method would be more useful in analyzing samples. Such a method is being developed.

The *S* of wave heights of diffusion-controlled polarograms of replicate solutions obtained in regular dc polarography has been assessed typically as 1 to 2%.^{25,26} An *S* of 0.5% has been obtained for regular and 0.6% for first-derivative dc polarography.²⁷ By comparative, dual-cell, fast-linear-scan polarography, substances in millimolar concentration have been determined with *S* = 0.1%.^{26,28} In the one- and the two-cell compara-

²⁴ORNL Graduate Program Research Associate from University of North Carolina, Chapel Hill.

²⁵J. K. Taylor, "Polarography as an Analytical Tool," *J. Assoc. Offic. Agr. Chemists* **47**, 21 (1964).

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

²⁷D. J. Fisher, W. L. Belew, and M. T. Kelley, "Recent Developments in D.C. Polarography," pp. 89-134 in vol. 1 of *Polarography 1964*, ed. by G. J. Hills, Macmillan, London, 1966.

²⁸H. M. Davis, "Komparative Polarographie hoher Präzision," *Chem. Ingr.-Tech.* **37**, 715 (1965).

tive methods²⁹ (Sect. 1.13.a) with the controlled-potential differential dc polarograph, residual-current corrections to diffusion-current (\bar{i}_d) values were made by subtracting averaged \bar{i}_t values in measuring millimolar concentrations; $S = 0.1\%$ was obtained by both methods. Our one-cell comparative method has the advantage that it is easier to maintain constancy of the characteristics of one capillary electrode and of an electric current than to maintain constancy (or matching) of the characteristics of two capillary electrodes. The slope of the residual current vs E curve is more reproducible than the magnitude of the residual current.²⁷ Hence, it was decided to determine with what precision concentration measurements can be made by single-cell first-derivative dc polarography.

In this investigation, peak heights of derivative polarograms of samples are measured relative to those of polarograms of similar standards in order to obtain concentration values for the samples. Therefore, to perform a single polarographic analysis with $S \leq 0.1\%$, an empirical proportionality calibration factor determined with a standard just prior to measuring polarograms of a sample must have at least this good a precision. Also, the S of factors determined over a period of time long enough to establish a factor and to measure polarograms of a sample must be 0.1% or less. However, we believe that, to readily and reliably determine sample concentrations with $S \leq 0.1\%$, it is necessary to control experimental conditions within limits such that this precision can be obtained for measurements made over a longer term (a reasonable work period) of at least several hours. That is, conditions must be controlled well enough so that measurements of polarograms of replicates of a standard placed in the cell in succession over a period of several hours have $S \leq 0.1\%$. With well-performing polarographic instrumentation, just as the sensitivity with which concentration can be determined depends on the signal-to-noise ratio of the D.M.E. itself²⁷ and is not limited by instrument performance, so the precision with which concentration can be determined depends on the behavior of the D.M.E. itself. The experimental conditions that must be controlled include temperature, average mercury flow rate (\bar{m}), D.M.E. drop time (t), sparging, and

instrument stability. Also, polarographic maxima should be absent.

In work that has been done, temperature was controlled³⁰ to $\pm 0.005^\circ\text{C}$, t was controlled at $\frac{1}{2}$ sec ($S = 0.03\%$) (Sect. 1.6), the mercury head (h) was maintained at a high constant value by manual adjustment, and instrumentation was provided that enables measurement of first-derivative peak heights with high precision. With this degree of control, over a short term (seven peak heights obtained and measured in succession), $S = 0.03\%$. However, over a long term (several hours) $S \sim 1\%$. It was found that maintaining h at a constant high value did not result in controlling \bar{m} to the required precision. The value of \bar{m} also depends on the back pressure, which is a function of the interfacial tension at the surface of the mercury drop. It is planned to investigate the utility of directly controlling \bar{m} as a means for improving the long-term precision of this polarographic method.

1.8 TIME INTEGRATOR ATTACHMENT

H. C. Jones

A self-powered analog time integrator was fabricated that uses a Philbrick Researches, Inc., SP656 chopper-stabilized operational amplifier. The integrator operates from an input voltage source and produces an output voltage that is proportional to the time integral of the input voltage. The input impedance of the integrator is at least 1 megohm. The integrator is intended for operation with input signals that range from about ± 2 mv to ± 20 v with a maximum integral error of about $\pm 0.1\%$. An input attenuator that has five ranges is provided for varying the sensitivity of the integrator. The integrator has been used by the X-Ray and Spectrochemistry Group to calibrate a light-beam splitter that will be used in turn to calibrate photographic emulsions.

1.9 CURRENT AMPLIFIER

H. C. Jones T. R. Mueller

A self-powered current amplifier was fabricated that uses a Philbrick Researches, Inc., P25AU

²⁹W. D. Shults, D. J. Fisher, and W. B. Schaap, "Controlled-Potential Differential DC Polarography. Comparative Polarography," *Anal. Chem.* **39**, 1379 (1967).

³⁰T. R. Mueller, "Arbitrary-Set, Proportional Temperature Controller," *Anal. Chem.* **37**, 172 (1965).

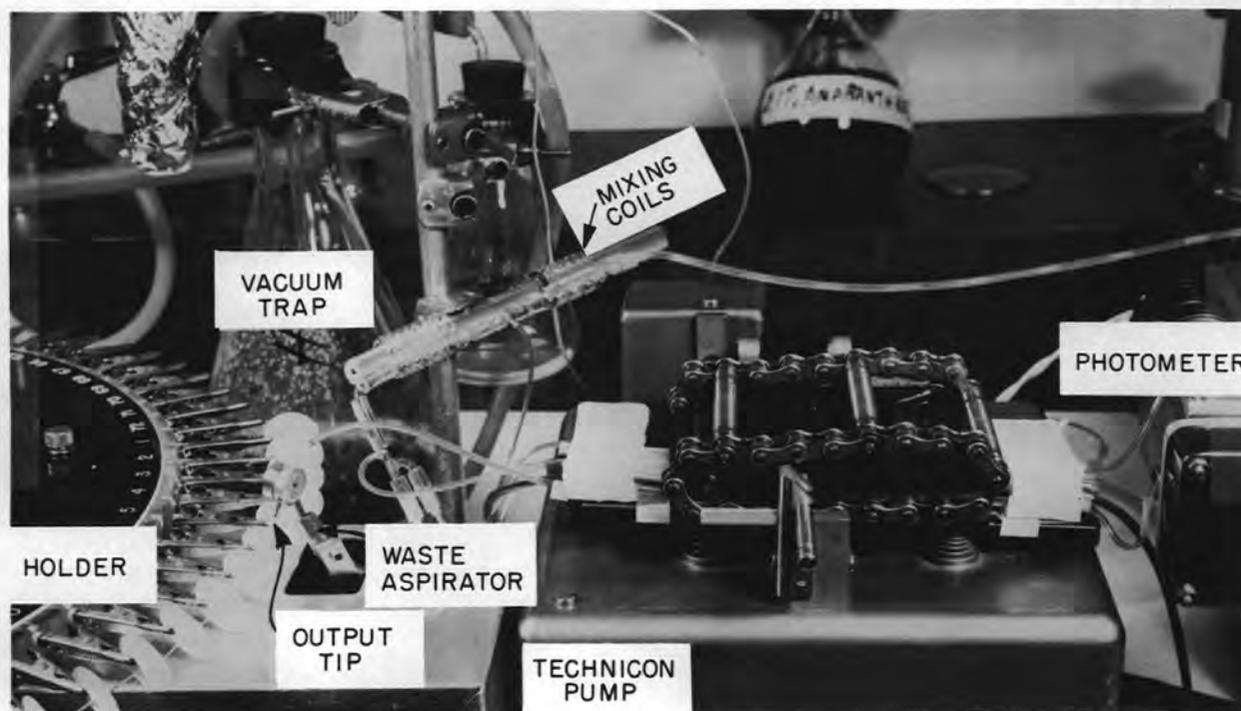


Fig. 1.4. Output Sampler and AutoAnalyzer Pump.

operational amplifier. The current amplifier has variable gain and will produce an output of ± 10 v for currents that range from about $\pm 5 \times 10^{-9}$ to $\pm 670 \times 10^{-9}$ amp with a calculated maximum output error of about $\pm 0.1\%$. Variable zero-offset current up to $\pm 150 \times 10^{-9}$ amp is provided. The amplifier is now being used by the Nuclear and Radiochemistry Group.

1.10 INSTRUMENTATION FOR ANALYTICAL BIOCHEMISTRY

W. L. Maddox

A prototype sampler was completed that is used with the Technicon AutoAnalyzer³¹ in a semi-automated filter-paper-disk technique for determining transfer ribonucleic acids. Late in 1966 the sampler was turned over to the Analytical Biochemistry Group for evaluation. A description of the procedures used and the results of their tests are given in Sect. 5.1.e.

³¹Trademark of Technicon Corp., Ardsley, N.Y. 10502.

Figure 1.4 shows the AutoAnalyzer proportioning pump with the output sampler attached and delivering a sample onto a filter-paper disk. When the output tip is not displaced as shown, it sits in contact with a vacuum aspirator, which continuously removes the liquid as it emerges. To the right in the figure is the flow-cell photometer, which, together with a meter-relay (not shown), paces the sampler's automatic operation. At the left appears the holder on which the filter-paper disks are mounted.

The output sampler is synchronized with the AutoAnalyzer input sampler by use of a dye (Amaranth Red) in the rinsing solution that is picked up between samples; barren zones of the stream at the output are then red, and the sample-rich zones are colorless, or nearly so, although a steady state is never reached. The transmittance of the output stream is observed by the photometer (a Welch model 1 Densichron), whose output is the signal to the meter-relay. An operating point for the meter-relay may be set for either increase or decrease of color in the stream; once the meter-relay contacts have been made, they will hold

until one sampling sequence is completed. The sampler is basically an assemblage of cams and motors which, on command (relay closing in the photometric circuit), moves the output tip into contact with a filter-paper disk for a few seconds, withdraws the tip, moves the disk receiver forward one position, cuts itself off, and waits for the next command.

Papers^{32,33} that describe this apparatus have been presented; another is being submitted for publication.³⁴

1.11 REMOTE PIPETTING

W. L. Maddox

A chapter titled "Remote Pipetting"³⁵ was prepared for inclusion in the forthcoming book Vol. 10, *Remote Analysis of Radioactive Materials*, SERIES IX, ANALYTICAL CHEMISTRY, of *Progress in Nuclear Energy*. The presentation consists mostly of descriptions of instruments and apparatus designed specifically for and presently used in fully enclosed shielded hot cells located in this country and in Europe.

1.12 MODEL VI FLAME SPECTROPHOTOMETERS

R. W. Stelzner M. T. Kelley
H. C. Jones

The electronics unit of a model VI flame spectrophotometer (located in Building 3019) that had been used by the X-Ray and Spectrochem-

³²W. L. Maddox and M. T. Kelley, "An Output Sampler for the Technicon AutoAnalyzer," 21st International Congress of the International Union of Pure and Applied Chemistry, Prague, Czechoslovakia, Sept. 4-10, 1967.

³³G. Goldstein, W. L. Maddox, and I. B. Rubin, "A Semi-Automated Filter Paper Disk Technique for the Determination of Transfer Ribonucleic Acid," 1967 Technicon Symposium on Automation in Analytical Chemistry, New York, Oct. 2-4, 1967.

³⁴W. L. Maddox and M. T. Kelley, "An Automatic Sampler for Obtaining Discrete Samples from the Output of a Continuous-Flow Analyzer," to be submitted to *Chemical Instrumentation*.

³⁵W. L. Maddox, "Remote Pipetting," to be included in vol. 10, *Remote Analysis of Radioactive Materials*, of SERIES IX, ANALYTICAL CHEMISTRY, ed. by H. A. Elion and D. C. Stewart, in *Progress in Nuclear Energy*, Pergamon, New York.

istry Group was redesigned to improve its performance and reliability. The modified high-impedance Brown amplifier system was replaced by a modern solid-state operational amplifier and a solid-state power supply. A multiplier-phototube signal current of 1 na produces a stabilized output voltage of 5 v and a full-scale deflection on the chart recorder. The new photometer (designated model VI-A) is used for the remotely controlled determination of lithium for the MSRE and TRU programs.

Two modified Q-1887 flame spectrophotometers in use in Building 4500S (X-Ray and Spectrochemistry Group) have also been upgraded with solid-state amplifiers and power supplies (model VI-B).

A complete model VI-C flame spectrophotometer (burner, optics, monochromator, and electronics units) is being designed to be installed in Building 3026 to replace an obsolete ORNL model Q-1457 system. The new unit, when fabricated and assembled, will be used by the Radioisotopes-Radiochemistry Laboratory for the analysis of radioactive materials.

1.13 CONTROLLED-POTENTIAL DIFFERENTIAL DC POLAROGRAPHY

W. D. Shults D. J. Fisher
W. B. Schaap³⁶

1.13.a Comparative Polarography

Two papers that describe our work in comparative polarography have been published.^{29,37} The abstract of the first paper,²⁹ which treats of the technique itself, follows:

"Comparative polarography is a differential polarographic technique that involves the measurement of the small difference between two diffusion currents, one due to the electroactive species of interest in the unknown solution and one due to the same electroactive species (present in accurately known concentration) in a similar reference solution. This compar-

³⁶Ph.D. Thesis Adviser; Professor of Chemistry, Indiana University, Bloomington.

³⁷W. D. Shults and W. B. Schaap, "Controlled-Potential Differential DC Polarography. Determinate and Statistical Errors in Comparative Polarography, Theory and Experiment," *Anal. Chem.* 39, 1384 (1967).

tive technique provides analytical results with accuracy and precision of 0.1% when cathode ray differential polarographic instrumentation is used in conjunction with two polarographic cells having synchronized dropping-mercury electrodes. The present paper gives the results of our study of the comparative technique with our controlled-potential differential dc polarograph that does not require the use of synchronized dropping-mercury electrodes. We have evaluated the dual-cell comparative technique and we have developed and evaluated a single-cell comparative technique. Using either of these dc comparative techniques, we obtain analytical results with accuracy and precision of 0.1% under optimum experimental conditions."

The second paper³⁷ discusses the development and experimental verification of the theory of determinate and statistical errors in comparative polarography; the abstract follows:

"Expressions for the determinate error and the statistical error associated with comparative polarographic determinations are derived, and theoretically predicted errors are compared with experimentally observed errors. An equation for the optimum compensation fraction, $f = C_R/C_U$, is also suggested; it may be used as a guide in the design of comparative polarographic procedures."

1.13.b Subtractive Polarography

A paper that describes our work in subtractive polarography has been accepted for publication;³⁸ the abstract follows:

"Subtractive polarography is a differential polarographic technique that allows the measurement of wanted signals in the presence of unwanted signals. The technique requires two polarographic cells, with D.M.E.'s operated at the same potential. The 'unknown' solution is placed in one cell and gives rise to the total current. The solution in the second cell contains those components of the 'unknown' solution that give rise to the unwanted portion of the total signal. Measurement of the difference between the polarographic currents at the electrodes in the two solutions thus yields the wanted portion of the total signal. This technique has

been evaluated with a controlled-potential differential dc polarograph that utilizes dropping-mercury electrodes having uncontrolled drop times, and the results are presented in this paper. The results indicate that subtractive polarography affords improved sensitivity, selectivity, and resolution over that of conventional dc polarography, and an improvement in resolution over that of derivative polarography. An additional improvement in resolution can be achieved by means of derivative subtractive polarography."

1.14 ENHANCEMENT OF FLAME-PHOTOMETRIC SENSITIVITY BY INCREASING SIGNAL-TO-NOISE RATIO (S/N) WITH A SIGNAL-AVERAGING COMPUTER

D. J. Fisher H. C. Jones
R. W. Stelzner

Signals of interest are inevitably accompanied by noise. The signal-to-noise ratio (S/N) establishes the ultimate sensitivity with which S can be measured. A procedure for obtaining an instrument system with which high-sensitivity measurements can be made consists in: (1) designing the analytical procedure and the system components to maximize S and to minimize N and thus to optimize S/N , and (2) processing the system voltage that has an optimized S/N in such a way as to obtain, for final readout, a processed voltage having a higher S/N . If the N that accompanies S is random and if a large number of repeated measurements can be made and summed in register with respect to S , then the S/N of the processed data is increased relative to that of a single measurement in proportion to the square root of the number of measurements. The signal-averaging computer is an instrument designed for this purpose.

If a substantial part of N that accompanies S in flame photometry is random, then the sensitivity of measurement of flame photometry should be increased by use of a signal-averaging computer. The ORNL model VII flame spectrophotometer was designed with beam chopping and with narrow-band tuned amplification to optimize S/N . A trigger circuit has now been built so that it is possible to use this instrument with a signal-averaging computer to determine whether a substantial portion of N , at high measuring sensitivity, can be averaged out by this means. We have demonstrated that

³⁸W. D. Shults, D. J. Fisher, and W. B. Schaap, "Controlled-Potential Differential DC Polarography. V. Subtractive Polarography," accepted for publication in *Chemical Instrumentation*.

the sensitivity of measurement with a well-designed flame spectrophotometer is increased substantially by use of a signal-averaging computer.

A paper is in preparation that describes this work. Several other investigations with a signal-averaging computer have been planned.

2. Effects of Radiation on Analytical Methods

Hisashi Kubota

H. E. Zittel

2.1 VOLTAMMETRIC METHOD FOR FLUORIDE

H. E. Zittel

T. M. Florence¹

Of the procedures used currently to estimate micro amounts of fluoride, most are colorimetric and are affected detrimentally by radiation. Generally an organometallic complex is bleached by formation of a stronger metal-fluoride complex. Fluoride is often determined in this way by its bleaching of the zirconium-alizarin red S (Zr-ARS) complex. Both ARS and Zr-ARS exhibit anodic reactions at the rotating pyrolytic-graphite electrode (R.P.G.E.).² A voltammetric method for micro amounts of fluoride based on the anodic reaction of ARS at the R.P.G.E. was developed; the procedure is simple and rapid. The amount of free ARS liberated by fluoride from a Zr-ARS complex is measured at +0.7 v vs S.C.E. As little as 20 ppb of fluoride can be determined with good accuracy and precision. Of the common ions, only PO_4^{3-} interferes at the levels of concentration studied. An article on this work was accepted for publication.³

2.2 EFFECTS OF GAMMA RADIATION IN THE SPECTROPHOTOMETRIC DETERMINATION OF URANIUM AS THIOCYANATE COMPLEX

H. E. Zittel

Lucy E. Scroggie⁴

Gamma radiation has an adverse effect in the spectrophotometric determination of uranium as the thiocyanate complex in water and in acetone-water

solutions. When stannous chloride is present, the absorbance of uranium thiocyanate decreases with increasing radiation dose until radiolytically formed elemental sulfur appears and causes an apparent increase in the absorbance. In the absence of stannous chloride, the turbidity caused by the sulfur increases the absorbance over the whole dose range studied. These effects are more severe for acetone-water than for water solution.

The experimental results of this study show that the spectrophotometric thiocyanate method for uranium is highly susceptible to radiation-caused error, but that, under optimum conditions, reasonable levels of radiation cause little or no error. The conditions are: (1) low uranium concentration, preferably <10 ppm; (2) water rather than acetone-water medium; (3) presence of stannous chloride as a protective agent; and (4) maximum radiation dose of $\sim 3 \times 10^4$ r in water medium and $\sim 1 \times 10^4$ r in acetone-water medium. Radiation doses above these levels introduce appreciable positive or negative error depending on the dose level.

An article on this work is to be published.⁵

¹Alien Guest from Australian Atomic Energy Commission, Sutherland, New South Wales, Australia.

²H. E. Zittel and T. M. Florence, "Voltammetric and Spectrophotometric Study of the Zirconium-Alizarin Red S Complex," *Anal. Chem.* 39, 320 (1967).

³H. E. Zittel and T. M. Florence, "A Voltammetric Method for Fluoride," *Anal. Chim. Acta* (in press).

⁴Chemicals Division, Union Carbide Corporation, P.O. Box 8361, South Charleston, W.Va.

⁵H. E. Zittel and L. E. Scroggie, "Effects of Gamma Radiation on Spectrophotometric Determination of Uranium as Thiocyanate Complex," *Anal. Chim. Acta* (in press).

2.3 ANODIC REACTIONS OF THIOSULFATE AT THE ROTATING PYROLYTIC-GRAPHITE ELECTRODE (R.P.G.E.)

H. E. Zittel

In the study of the effects of gamma radiation on thiosulfate, a way to follow the radiation damage to dilute solutions of this reagent was required. The available methods such as ultraviolet spectrophotometry and reaction with I_2 are not sufficiently sensitive, rapid, and selective. Since voltammetry as a general method has these features, the anodic behavior of thiosulfate at the rotating pyrolytic-graphite electrode (R.P.G.E.) was determined. Thiosulfate was found to exhibit anodic waves at $\sim +0.8$ v and $\sim +1.1$ v vs S.C.E. in media of pH from 1 to 10. Neither wave is affected significantly by change in pH or in thiosulfate concentration. The wave at $\sim +0.8$ v is attributed to the oxidation of thiosulfate to tetrathionate. The wave at $\sim +1.1$ v is attributed to the oxidation of tetrathionate, because voltammetry of tetrathionate alone gave an anodic wave at $\sim +1.1$ v vs S.C.E. Each wave is highly irreversible, and its height is proportional to thiosulfate concentration. Neither sulfite nor sulfide interferes; therefore, a voltammetric method for thiosulfate that is both rapid and specific seems feasible.

2.4 RADIOLYSIS OF RADIATION-SAFEGUARD SPRAY SOLUTIONS

H. E. Zittel J. R. Lund

The study in progress of the effects of radiation on radiation-safeguard spray solutions is related to the Spray and Absorption Technology Program of the Reactor Division. A variety of proposed safeguard solutions such as aqueous solutions of boric acid with and without added base and acid and alkaline solutions of thiosulfate are being evaluated. Test portions of the solutions are given total doses of 5×10^5 to 5×10^7 r of ^{60}Co gamma radiation. The changes in pH and in iodine equivalence of the irradiated solutions and the amounts of radiolytic H_2 evolved in the samples are measured as functions of total radiation dose. The effects of other variables such as temperature and pressure on the radiolytic behavior of these solutions are now being determined.

2.5 RAPID DISTILLATION SEPARATION OF FLUORIDE

Hisashi Kubota

A rapid distillation separation of fluoride that can be used in hot cells was developed. The separation and the subsequent analytical procedure were planned to simplify fluoride determination in a hot cell and to give accurate and reproducible results.

Fluoride is distilled under controlled conditions as fluosilicic acid. The still head is a Kjeldahl trap covered with asbestos and provided with Nichrome heating wire. The vapor from a 1:1 H_2SO_4 solution transports the fluoride; no external air or vapor stream is used.

The fluoride is determined as the alizarin complexone;⁶ La(III) is used in place of Ce(III). Alizarin tolerates sulfate up to a certain point. As a result, the distillation temperature and distillate volume must be kept within specified ranges to ensure that the sulfate content is acceptable. Sulfate causes $\sim 5\%$ decrease in the absorbance of the chromophore under the conditions used. A single distillation requires about 15 min. When present in the maximum amount that can be contained in 1 ml of solution, silica is the only substance that will interfere. The relative standard deviations (S) of results from several analysts are:

Fluoride (μg)	S (%)
5	12
10	4.7
15	2.2
20	2.1
25	1.3

The procedure can be scaled down to determine 1 to 5 μg of fluoride, a range for which the reproducibility of fluoride determinations is generally poor. The S's of results by the procedure so modified were 14% at 1 μg , 10% at 2.5 μg , and 4% at 5 μg .

The principle of this separation can also be utilized to separate larger amounts of fluoride. For example, 500 μg can be separated in a distilled volume of 20 ml with recovery of $\sim 95\%$.

⁶S. S. Yamamura, M. A. Wade, and J. H. Sikes, "Direct Spectrophotometric Fluoride Determination," *Anal. Chem.* **34**, 1308 (1962).

Since this analysis was designed to be used in a hot cell, the effect of radiation in the course of the spectrophotometric procedure was studied. A 1% decrease in absorbance occurs per 1000 rads of absorbed dose; this rate of bleaching is linear up to doses as large as 50,000 rads, which radiation stability is considered to be sufficient.

2.6 STUDIES RELATED TO PROJECT SALT VAULT

Hisashi Kubota J. R. Lund

Project Salt Vault is a demonstration to show the feasibility of storing highly radioactive solidified wastes in natural salt (NaCl) formations. This demonstration has been under way at an old salt mine in Lyons, Kansas, for several years. Spent fuel elements were canned, placed in holes dug in the mine floor, and allowed to cool there. Since the radioactivity of these fuel elements was somewhat lower than that anticipated for actual disposal material, supplementary heat was supplied.

Tests over the past years indicated that water stored in cavities within the salt can be released by thermal effects and thus create corrosion problems. The salt from the Lyons mine was found to shatter and to release its moisture at a temperature slightly above 250°C.⁷ Other studies have indicated that water also can be released by migration to the warmer side of a cavity.⁸ Recently, the amounts of water involved have been a factor of 2 higher than the water contents previously determined. As a result, the moisture determinations were repeated in tests in which the heating was continued up to 600°C. Apparently, a considerable amount of water is still trapped after the initial large release at 250 to 300°C, and the salt is rid of this water only after it is heated above 500°C. Samples taken along a vertical profile showed various moisture contents, which reflect the differences in deposition conditions at different geologic periods.

⁷H. Kubota, "Thermal Stability of Natural Salt," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1963*, ORNL-3537, p. 46.

⁸R. L. Bradshaw, Health Physics Division, "Report on Joint Studies on Migration of Negative Crystals in Salt," personal communication to Florentino Sanchez, ORAU Student Trainee from St. Edward's University, Austin, Tex.

The demonstration holes were rigged with air lines whose outflows were continually monitored for possible harmful products. Hydrochloric acid appeared unexpectedly. A close study of the environment indicated that shale, present as an impurity in natural salt, could be the culprit. Samples of shale from the Lyons mine were heated in a tube furnace while a stream of moist air was passed over them. The effluent air contained hydrochloric acid produced by hydrolysis of iron chloride. Under the conditions of the measurements, the amount of acid released was 1 to 5 meq/hr per gram of shale. The rate of release is controlled by factors such as temperature, state of subdivision and iron content of the shale, and humidity. Thus, in this mode of disposal the production of a corrosive compound is possible.

Irradiation of solid NaCl gave no measurable release of chlorine, particularly at elevated temperatures.⁹ The effluent air from the demonstration holes was analyzed periodically with an MSA-type chlorine detector to check the validity of the experimental results. No chlorine was detected at the initial stages of the demonstration. When the temperature was raised in compliance with one phase of the experimental plan, the detector became discolored and indicated the release of 0.1 to 1.2 ppm of chlorine. The impurity in the effluent air was concentrated in a cold trap and was analyzed by gas chromatography. The results indicated the absence of chlorine but the presence of an organic compound. This fraction was collected and analyzed by mass spectrometry. Several fragments with mass numbers in the forties were detected. It is believed that this unidentified material was an organic peroxide formed and released from organic impurities in the salt. The threshold temperature for its release is about 180°C.

2.7 APPLICATIONS OF RADIOLYTICALLY GENERATED REAGENTS IN ANALYTICAL CHEMISTRY

Hisashi Kubota

The homogeneous occurrence of radiolytic reactions indicates the possibility of using radiolytically produced reagents for homogeneous separa-

⁹H. Kubota, "Effect of Radiation on Sodium Chloride," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1963*, ORNL-3537, p. 45.

tions. An example of a homogeneously produced reagent is the hydrochloric acid released by the radiolysis of chloral hydrate. Studies were made in which either the chloride or the acid component was used to effect homogeneous precipitations.

Silver was precipitated as the chloride as follows. An aliquot of a standard solution of Ag^+ was placed in a beaker and was made 0.01 M in chloral hydrate and slightly acid with nitric acid. The resulting solution was exposed to gamma radiation from ^{60}Co . A grainy precipitate formed that was dense and easy to filter. The precipitate was of better quality (easier to filter and quantitatively recoverable) when the dose rate was low.

Sulfate was complexed with ethylenediamine-tetraacetic acid (EDTA) in a medium made basic with sodium hydroxide. After the addition of

chloral hydrate, the solution was irradiated. The large doses required to release sufficient acid to precipitate BaSO_4 also decomposed the EDTA. The decomposition products colored the solution and the precipitate a dirty brown. The precipitate attained constant weight at an ignition temperature of 550°C , which is considerably lower than 900°C , the ignition temperature recommended for BaSO_4 precipitated by conventional methods.

Use of radiolytic reactions is a fresh approach to the study of nucleation processes. It is particularly advantageous in that the rate of reagent generation can be readily controlled by control of the dose rate. As the second example given above indicates, this technique is complicated by the possible degradation of some of the solution components, particularly organic compounds.

3. Analytical Chemistry for Reactor Projects

A. S. Meyer
J. M. Dale

3.1 MOLTEN-SALT REACTOR EXPERIMENT

3.1.a Determination of Oxide in MSRE Salts

R. F. Apple J. M. Dale

The results of the determinations of oxide in radioactive MSRE salt samples for the period October 1966 to July 1967 are summarized below:

Sample	Oxide Concentration (ppm)
FP-8-7 (fuel)	44
FP-9-2 (fuel)	44
FP-11-28 (fuel)	58
FP-12-4 (flush)	41
FP-12-18 (fuel)	57

During the last week of December 1966 the moisture-monitor cell in the oxide apparatus became inoperative. Because other experiments were being performed in the same hot cell, the moisture-

monitor cell was not replaced until March 1967. The insensitive cell showed superficial evidence of radiation damage; the potting compound used to seal the tube that contains the spiral electrodes in a stainless steel housing had shrunk and cracked. Measurements of the flow revealed that substantially all the flow was still passing through the electrolysis tube; therefore, the damage to the potting compound could not have caused the cell failure. Electrical-resistance measurements indicated that the failure was caused by either removal or some alteration of the P_2O_5 electrolyte film.

The replacement of the moisture-monitor cell was the first major maintenance required for the hot-cell apparatus since its installation in February 1966. The cell had been in operation for several additional months during laboratory tests of the apparatus. This one-year operation represents a reasonable service life for a moisture-monitor cell, even under normal operating conditions in a nonradioactive environment.

While the hot-cell equipment was inoperable, the oxide development apparatus in Building 4500S was

Table 3.1. Oxide Concentrations of Fuel and Solvent Salt Samples from the Second ORR Molten-Salt Loop

Sample Designation	Condition of Salt as Received	Sample Weight (g)	Oxide Concentration (ppm)
9-29-66	Pellets	10.4	200
10-17-66	Pellets	10.1	220
11-21-66	Crushed	18.8	420
F-194	Crushed	18.4	820
Solvent salt batch No. 2	Fused into ladle	48.4	115
Solvent salt batch No. 17	Fused into ladle	13.1	520

reactivated. Several samples of nonradioactive fuel and solvent salt from the second ORR molten-salt loop were analyzed for oxide; Table 3.1 summarizes the results. Probably the high oxide contents of the third and fourth samples were due in part to contaminants acquired during the brief exposure when the crushed samples were transferred to the hydrofluorinator. The high oxide content of sample No. 17 compared with that of sample No. 2 represents the oxide picked up when the flush salt was circulated in the loop.

Two samples of radioactive fuel (IPSL-19 and IPSL-24) submitted from In-Pile Salt Loop 2 contained 265 and 240 ppm of oxide respectively. Sample IPSL-24 was stored under helium at 200°C from the time of sampling to analysis (~six months).

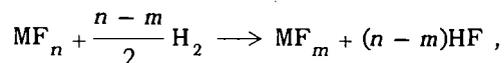
3.1.b Determination of Uranium(III) in Radioactive MSRE Fuel by a Hydrogen-Reduction Method

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

A proposed explanation of the unexpected distribution of certain fission products in the MSRE system is that the fuel had become sufficiently oxidizing to produce significant partial pressures of volatile fission product fluorides such as MoF₆, TeF₆, and RuF₆, which then migrated into the graphite and the blanket gas. Because prevention of the accumulation of fission products in graphite is vitally important to the satisfactory operation of breeder reactors, a

method to determine U³⁺/U⁴⁺ ratios in the radioactive fuel samples was needed.

The possibility that major fractions of the iron and nickel in the fuel are present as colloidal metal particles¹ precluded any adaptation of the hydrogen-evolution method for U³⁺, because these components would also yield hydrogen on acid dissolution. An alternate, suggested by C. F. Baes,² is a transpiration method in which a sample of the molten fuel is sparged with hydrogen to reduce oxidized species according to the reaction



in which MF_n may be UF₅, NiF₂, FeF₂, CrF₂, or UF₄ in order of their observed reduction potentials. The rate of production of HF is a function of the ratio of oxidized to reduced species in the melt.

Some components of the oxide apparatus³ were adapted for the transpiration measurement on radioactive fuel samples. The inlet of the hydrogenator is connected to sources of thoroughly dried hydrogen and helium, and the outlet is connected to a heated manifold fitted with six liquid-nitrogen-cooled NaF traps. This arrangement permits the separate collection of the HF from four successive reduction

¹"Voltammetric Determination of Ionic Iron and Nickel in Molten MSRE Fuel," *MSR Program Semiann. Progr. Rept. Feb. 28, 1966*, ORNL-3936, p. 162.

²Reactor Chemistry Division.

³"Determination of Oxide in MSRE Fuel," *MSR Program Semiann. Progr. Rept. Feb. 28, 1966*, ORNL-3936, p. 154.

steps with hydrogen and from two blank spargings with helium on the same 50-g fuel sample. When the transpiration run is completed, the traps are disconnected from the manifold and removed from the hot cell for desorption and titration of the collected HF.

The results of these titrations are interpreted in terms of U^{3+} concentrations in the samples by comparing the HF yields for the reduction steps with those calculated for hypothetical salt compositions. If U^{4+} is the only reducible species in the melt, a derivation based on a material balance and the hydrogen-reduction equilibrium yields the following rather simple relationship for U^{3+} as a function of hydrogen exposure:

$$U \ln \left(\frac{U}{U - U^{3+}} \right) - U^{3+} = Bt, \quad (3.1)$$

in which

U = concentration of total uranium, mole fractions,

U^{3+} = concentration of U^{3+} , mole fractions,

t = reduction time from "0" U^{3+} concentration, min,

$$B = \frac{KP_{H_2}^{1/2} V_G}{SRT},$$

where

$$K = \frac{U^{3+} P_{HF}}{U^{4+} P_{H_2}^{1/2}}, \text{ atm}^{1/2},$$

P_{H_2} = partial pressure of H_2 , atm,

P_{HF} = partial pressure of HF, atm,

V_G = sparge flow, liters/min,

S = quantity of fuel sample, total moles of all components,

R = gas constant, liter-atm $^{\circ}K^{-1}$ mole $^{-1}$,

T = temperature at which the sparge flow is measured, $^{\circ}K$.

When corrosion products (M_j^{2+}) are present, the relationship assumes a different form as each corrosion-product ion successively undergoes

reduction to the metal:

$$a_n U - U^{3+} + U \ln \left(\frac{U - a_n U}{U - U^{3+}} \right) + 4 \sum_{j=1}^{j=n} \frac{K^2}{K_{M_j}^2} \left(\frac{1}{a_n} - \frac{U}{U^{3+}} \right) = Bt_n, \quad (3.2)$$

in which

$$a_j = \frac{1}{1 + (K_{M_j}/K) (M_j^{2+})^{1/2}},$$

M_j^{2+} = concentration at $t_n = 0$ of the j th corrosion product in order of reduction, mole fraction,

t_n = time from the instant reduction of the n th corrosion product starts to the instant reduction of the (n th + 1) corrosion product starts, min,

$$K_{M_j} = \frac{P_{HF}}{(M_j^{2+})^{1/2} P_{H_2}^{1/2}}.$$

The yield of HF from any reduction step is calculated from the initial and final concentrations of U^{3+} and corrosion products as follows:

yield of HF, micromoles = $\left\{ (U_i^{3+} - U_f^{3+}) \right.$

$$\left. + 2 \sum_{j=1}^{j=n} \left[(M_j^{2+})_i - (M_j^{2+})_f \right] \right\} S \times 10^6. \quad (3.3)$$

The relationship of Eqs. (3.1) and (3.2) is illustrated in Fig. 3.1, in which the yield of HF is plotted as a function of Bt for an oxidized fuel assumed to contain U^{4+} and Fe^{2+} in concentrations approximating the elemental analyses of the MSRE fuel. On a logarithmic plot the HF yield follows a near-linear relationship [Eq. (3.1)] derived for an iron-free melt until a critical U^{3+}/U^{4+} ratio is reached and reduction of Fe^{2+} starts. The reduction of Fe^{2+} then predominates, with the rate of change of U^{3+} decreased in accordance with Eq. (3.2) until essentially all the iron is reduced and the HF yield approaches that derived from U^{4+} reduction alone.

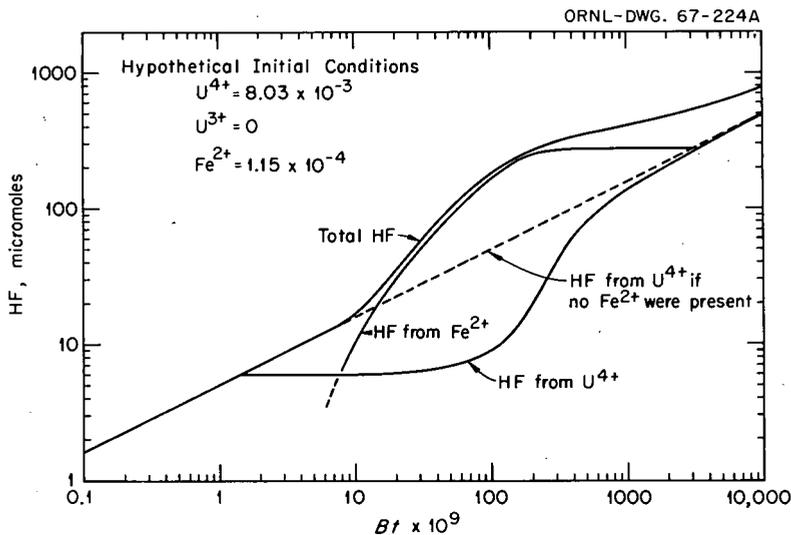
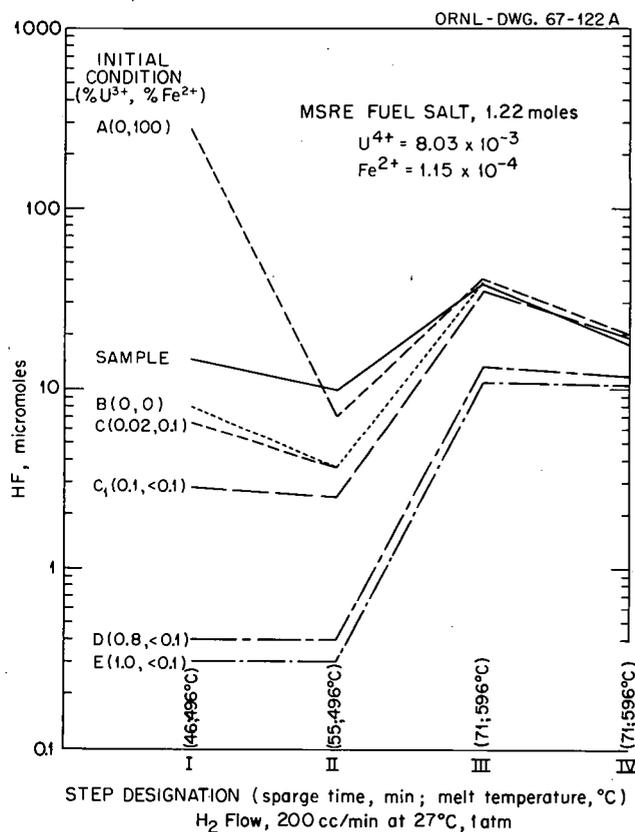


Fig. 3.1. Calculated Yields of HF from the Hydrogenation of a 50-g MSRE Fuel Sample at 1000°K.

Fig. 3.2. Calculated and Observed Yields of HF from MSRE Fuel Sample FP-9-4.

Figure 3.2 is a plot of hydrogen fluoride yields from the first sample (FP-9-4) and from various hypothetical initial fuel compositions. The four reduction steps were selected on the criteria that the first two steps at 496°C would reduce at least 99.9% of the Fe^{2+} and Ni^{2+} without materially affecting the U^{3+} concentration and that the last two steps at 596°C would reduce U^{4+} without reducing Cr^{2+} . The HF yields from the latter reductions thus indicate the "oxidation state" of the fuel. Broken lines connect calculated yield points, and a solid line connects the experimentally measured yields of HF from the sample. The relatively low yields of HF in the first two steps are in agreement with earlier experimental evidence¹ that only small fractions of the iron and nickel were present in the ionic state. The high yields in the last two steps indicate that the fuel was in an oxidizing or near-oxidizing condition. On the basis of the HF yield of step III (Fig. 3.2) and in consideration of estimated experimental errors, a ratio of U^{3+}/U of 0.000 to 0.001 was assigned.

A computer program was developed that permits the calculation of HF yields expected for any



preselected reduction steps on any melt composition. By use of the present fuel composition and the experimental conditions of the transpiration experiment as input data to the program, HF yields were calculated for various initial concentrations

Table 3.2. U^{3+}/U Ratios in MSRE Fuel Salt

Sample No.	Integrated Power (Mwhr)	U^{3+} by Burnup Oxidation (eq)	Be Added (eq)	U^{3+}/U by Calculation ^a	U^{3+}/U by Analysis (%)	
					Step III ^b	Step IV ^b
FP-9-4	10,978	1.87		0.31	0	0.1
FP-10-25	16,450	0.93	3.61	0.58	0.35	0.45
FP-11-5	17,743	0.40		0.54	0.37	0.37
FP-11-13	20,386	0.26	2.59	0.77	0.37	0.42
FP-11-32	25,510	0.88		0.69	0.33	0.34
FP-11-38	27,065	0.26		0.66		
FP-11-49	30,000	0.50	1.86	0.80		
FP-12-6	32,450	0.40		0.76	0.42	0.37
FP-12-11	33,095	0.10	3.95	1.14	0.38	1.2
FP-12-21	35,649	0.50	4.44	1.54	0.39	0.50

^aCalculated by W. R. Grimes.²

^bSee Fig. 3.2.

of U^{3+} . Concentrations of U^{3+} in samples were determined by comparing the experimental with the calculated HF yields. Table 3.2 shows the U^{3+} results obtained from the HF yields of the third and fourth reduction steps of the analyses compared with expected values calculated by W. R. Grimes.² For the calculation of the results it was assumed that: 0.16% of the uranium in the fuel was originally present as U^{3+} ; the increase in chromium concentration from 38 to 65 ppm, which occurred before the first sample was taken, resulted in the reduction of U^{4+} to U^{3+} ; each fission event results in the oxidation of 0.8 atom of U^{3+} ; and there have been no other losses of U^{3+} .

No results are listed for samples FP-11-38 and FP-11-49. Although these samples were run in the normal manner, a total of over 2000 micromoles of HF was evolved for the four hydrogen-reduction steps for each sample compared with about 55 micromoles for the previous runs. Since this increase in HF yield coincided with an increase in radioactivity in the traps used to collect the HF, it appeared likely that the increase in sample radioactivity during the extended period of reactor operation might be responsible. If the induction period for the radiolytic generation of fluorine were shortened due to the increase in radioactivity of the sample, the fluorine evolved during the loading of the sample could react with the inner walls of the Monel hydrogenation vessel. The copper fluoride and nickel fluoride formed would

be reduced subsequently during the hydrogenation steps to produce HF.

This hypothesis appears to be supported by the results of the following experiment. One of the samples that produced the high HF yields was allowed to stand in the hydrogenator at room temperature for about a week. The sample was then subjected to additional hydrogenation steps, and HF was produced in quantities comparable with those obtained in the original runs. After standing several more days at room temperature, the sample was removed from the hydrogenator. Smaller but significant quantities of HF were obtained when the empty hydrogenator was subjected to the high-temperature hydrogenation procedure.

The last three samples (FP-12-6, FP-12-11, and FP-12-21) were taken after a relatively brief period of reactor operation following a lengthy reactor shutdown period. None of these analyses produced the excessively high HF yields that were observed for the previous two samples. These results appear to be further confirmation that the excessive HF yields were caused by a buildup in sample radioactivity with extended reactor operation.

Since the first addition of beryllium to the fuel, all the determinations of U^{3+} not obviously affected by radioactivity have been in the range 0.33 to 0.50% (the one result of sample FP-12-11 could be explained by a leaky valve) and do not reflect the beryllium additions in the periods between the samplings. This discrepancy could be accounted for by the evolution of fluorine in much smaller

quantities than appeared to be the case in samples 11-38 and 11-39. If this is true, the only permanent solution would be to maintain the samples at 200°C during the time of transfer to the hot cell for analysis. However, an apparatus is now being designed that will permit the hydrogenation of synthetic fuel samples under carefully controlled conditions. This experiment should provide a check of the validity of the transpiration method and indicate further whether or not fluorine evolution is a real problem.

Also, a method is being developed for the remote measurement of ppm concentrations of HF in helium and in hydrogen gas streams. The technique is primarily for application to the U^{3+} transpiration experiment but, if successful, should also be applicable to the determination of HF in MSRE off-gas. The method is based on the collection of HF on a small NaF trap that is held at 70°C to prevent the adsorption of water. The collection of HF is followed by its desorption at a higher temperature to give a concentrated pulse of HF that can be measured by thermal conductivity.

A components testing facility has been set up that includes a dilution system to produce HF "standards" of concentration as low as 20 ppm, a thermostated trap with self-resistance heating for fast heat-up, and a thermal-conductivity cell with nickel filaments. Initial tests have revealed that the thermal-conductivity cell now being used is too sensitive to perturbations in the carrier flow and that the last traces of HF are desorbed too slowly from commercial pelletized NaF. The use of thermal-conductivity cells of different geometry and having better flow-control valves is expected to eliminate the first of these problems. The second problem will require further development studies to determine whether the slow desorption rate represents an inherent property of the NaF or is a result of impurities in the NaF. Other trapping materials will also be investigated.

A special Monel valve has been made and will be used in the remote HF-measuring system. The valve incorporates two valving systems in the same metal body and will provide for the simultaneous adsorption and desorption of HF from alternate traps. This arrangement will eliminate any dead legs in the HF trapping system and will permit the measurement of incremental quantities of HF evolved from one hydrogenation step in the U^{3+} transpiration experiment.

One additional step is being taken with regard to the computer program used for data analysis. Because of equilibrium shifts of oxidized and reduced species in the molten fuel salt with temperature changes, the starting U^{3+} concentration in the analysis sample at the temperature of the initial hydrogenation steps will necessarily be different from the U^{3+} concentration in the fuel in the reactor. The computer program is presently being modified to take this difference into account.

3.1.c Analysis of Off-Gas from Tests of Compatibility of MSRE Pump Oil with BF_3

C. M. Boyd

A total-hydrocarbon detector and a thermal-conductivity detector were used to determine hydrocarbons and BF_3 in the gas from tests on the effects of BF_3 on MSRE pump oil (Gulf Spin 35). A gas-sampling manifold permits measurement of increases in hydrocarbon concentration of an He- BF_3 gas stream after contact with the oil. A parallel stream of helium through a separate oil reservoir is a reference. The hydrocarbon analyzer was modified by the addition of a sampling pump, which draws in the gas at near atmospheric pressure, compresses it, and passes it through the analyzer. The portions of the test gas to be analyzed are first passed through a saturated solution of KF to remove the BF_3 and thereby to protect the pump and other components of the analyzer. This solution has a low vapor pressure of water, which prevents the condensation of water in the analyzer. In the first tests, with the BF_3 concentration at the 2000-ppm level and the temperature of the oil at 150°F, the hydrocarbon concentration in the off-gas was less than 50 ppm.

The thermal-conductivity detector was used to monitor the BF_3 concentration in a test gas produced by mixing flows of pure BF_3 and helium. The detector and flow capillary were calibrated by passing a known volume of the gas mixture through the detector and then through a trap that contained a dilute solution of NaOH. The solution was analyzed for boron and fluoride, and the BF_3 concentration of the gas was then calculated. With this detector, changes in BF_3 concentration of ± 10 ppm can be measured.

3.1.d Development of a Gas Chromatograph for Analysis of the MSRE Blanket Gas

C. M. Boyd A. S. Meyer

A gas chromatograph is being developed for the continuous determination of permanent-gas impurities and water in the helium blanket gas of the MSRE. The chromatograph will require two columns to separate the components desired. Tests have shown that a parallel-column system composed of a 5A molecular-sieve column and a Porapak S column should be practical. The H_2 , O_2 , N_2 , CH_4 , and CO are separated on the molecular-sieve column, and the H_2O and CO_2 are separated on the Porapak column. The chromatograph will have separate compartments, each controlled at a different temperature. The sample valve, columns, and detectors may also require different temperatures for optimum operation.

The necessity to determine impurities at sub-ppm and ppm concentrations calls for the use of two types of systems or detectors. A helium-breakdown detector can be operated either in the high- and low-sensitivity modes or in the high-sensitivity mode in conjunction with a thermal-conductivity detector.

The chromatograph that is used to analyze the reactor off-gas will be subjected to radiation from samples whose radioactivity is at the 10-curie/cc level. This condition precludes the use of organic construction materials. An all-metal sampling valve and a detector unaffected by this radiation are necessary. The organic Porapak column cannot be used at this level of radiation; therefore, the determination of H_2O in these samples will not be possible. A silica-gel column will be used to resolve CO_2 .

The problem of radioactive samples and the transmittal of gas samples that contain ppm levels of H_2O from the source to the detector require the use of a heated all-metal sampling valve. Such a valve was designed and constructed; it is similar to the conventional Phillips pneumatically actuated six-way diaphragm valve (Fig. 3.3). The Teflon diaphragm was replaced by a 1-mil-thick Inconel diaphragm, which contacts and seals the redesigned entry orifices (Fig. 3.4). With this metal diaphragm a spacer is required to permit the flow of gas without excessive back pressure. Spacers made from 2-mil-thick gold were needed to give a leak-tight seal between the diaphragm and the valve faces.

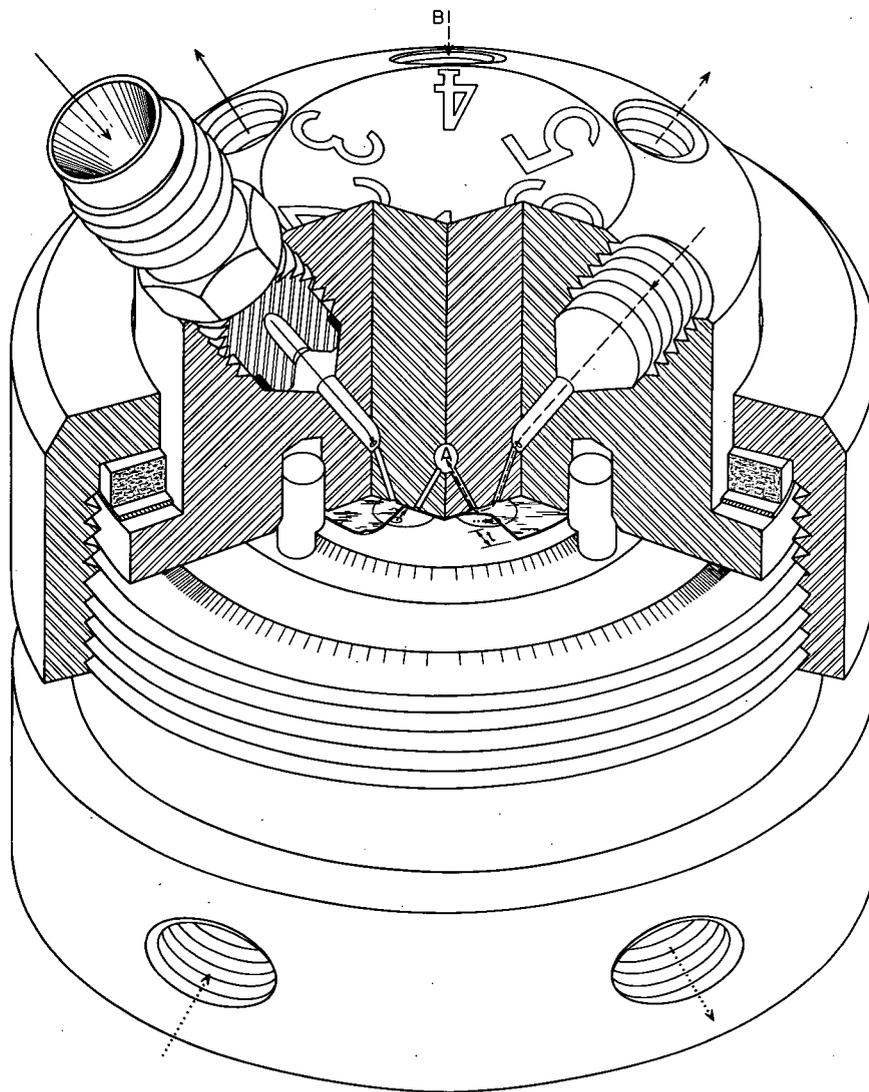
The gold was annealed at 1500°F and, after installation in the valve, was subjected to 32,000-lb pressure. Pressure maintained by the assembly screws was sufficient for retaining this seal.

Helium-ionization-type detectors are sensitive to sub-ppm levels of permanent gases. A helium breakdown-voltage detector was used on the MTR Capsule Test Facility (Test 47-6).⁴ It was not affected by the radiation from the gas samples, possibly because relatively high current levels (microamperes) are used with it. A constant-current power supply is used with this detector. A decrease in breakdown voltage, rather than an increase in ionization current, indicates an increase in the electrical conductivity of the gas. The breakdown voltage of pure helium was about 500 v and was decreased ~50 v by impurity of 1-ppm concentration. The minimum detectable limit is controlled primarily by the noise level but under optimum conditions is <1 ppb.

In previous attempts to improve the stability of the detector discharge, electrodes of various radii were used, the anode and cathode being in a concentric arrangement. In this early model of the detector, which was contained inside a Swagelok tube fitting, it was impossible to observe the helium discharge. A test detector was therefore constructed that has a glass body with Kovar-seal tube connections through which the electrodes are mounted. The tips of these electrodes (Fig. 3.5) are removable, which allows the testing of electrodes of various shapes and spacings. The effects on the helium discharge can be observed through the glass. Tests with this detector indicate that a minimum noise level is obtained with a smooth glow discharge on the anode probe. Maximum sensitivity dictates the use of very pure helium carrier gas, but an excessively high purity also causes sparking or arcing in the helium discharge. The addition of mercury vapor by the presence of a small source of the metal in the tip of the anode stabilized the discharge. The more practical solution of adding a contaminant by a controllable gas flow is being tested. This approach is also a possible method for adding larger amounts of contaminant to allow the detector to operate in the less sensitive mode if it should ever be necessary to determine high levels of impurities in the blanket-gas samples.

⁴"Reactor Off-Gas Analysis," *MSR Program Semiann. Progr. Rept. July 31, 1964*, ORNL-3708, p. 328.

ORNL-DWG. 65-77



- CARRIER
- SAMPLE
- A TO SAMPLING LOOP
- B -----→ FROM SAMPLING LOOP
-→ ACTUATING GAS

Fig. 3.3. Conventional Phillips Valve.

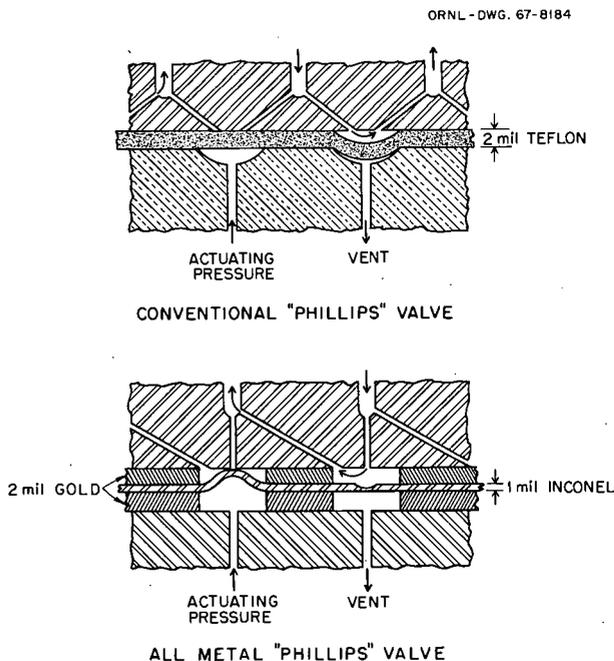


Fig. 3.4. Modified All-Metal Sample Valve.

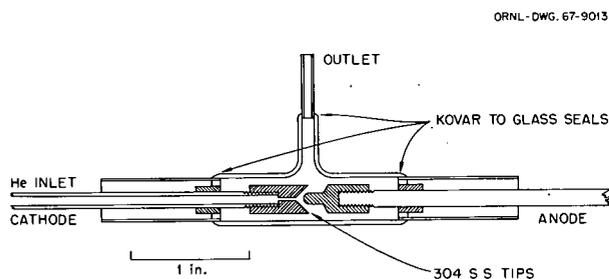


Fig. 3.5. Diagram of Helium Breakdown-Voltage Detector.

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

3.2.a In-Line Test Facility

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

Design work has been continuing with the assistance of J. H. Evans⁵ on the experimental molten-salt test loop that will be used to evaluate electrometric, spectrophotometric, and transpiration

⁵Equipment Design Section, General Engineering Division.

methods for the analysis of flowing molten-salt streams.⁶ The operation of flow equipment such as capillaries, orifices, and freeze valves will also be tested. Figure 3.6 is a schematic flow diagram of the proposed test loop. The first drafts of the engineering drawings have been completed; it is planned to soon start construction of the various components of the system.

3.2.b Electromotive Force (EMF) Measurements on the Nickel-Nickel(II) Couple in Molten Fluorides

H. W. Jenkins⁷ D. L. Manning
Gleb Mamantov⁸

Potentiometric measurements on concentration-type cells were conducted to evaluate the usefulness of the Ni-Ni²⁺ couple as a reference electrode for fluoride melts. The experiments were conducted in a small vacuum dry box (24 in. long, 20 in. deep, 15 in. high) outfitted with a furnace, vacuum and controlled-atmosphere facilities, and a moisture monitor. The melt (~400 ml) is contained in a graphite cell. The inner-electrode compartment consists of a nickel electrode in contact with a melt of fixed Ni²⁺ concentration contained in a thin-walled boron nitride thimble. From the equation

$$\Delta E = \frac{RT}{2.3nF} \log \frac{X_{Ni_1}}{X_{Ni_2}} \quad (3.4)$$

and from plots of ΔE vs $\log X_{Ni_2}$, it was demonstrated that the Ni-Ni²⁺ couple exhibits Nernstian behavior at 500°C. Experiments were conducted in which the Ni²⁺ concentration (X_{Ni_2}) was varied from about 10⁻⁵ to 10⁻³ mole fraction in molten LiF-BeF₂-ZrF₄ (65.6-29.4-5.0 mole %) and also in molten LiF-NaF-KF (46.5-11.5-42.0 mole %). From the standpoint of Nernstian reversibility, therefore,

⁶R. F. Apple, J. M. Dale, A. S. Meyer, and J. P. Young, "Analytical Methods for the In-Line Analysis of Molten Fluoride Salts," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1966*, ORNL-4039, p. 18.

⁷ORAU Graduate Fellow from the University of Tennessee, Knoxville.

⁸Consultant; Associate Professor of Chemistry, University of Tennessee, Knoxville.

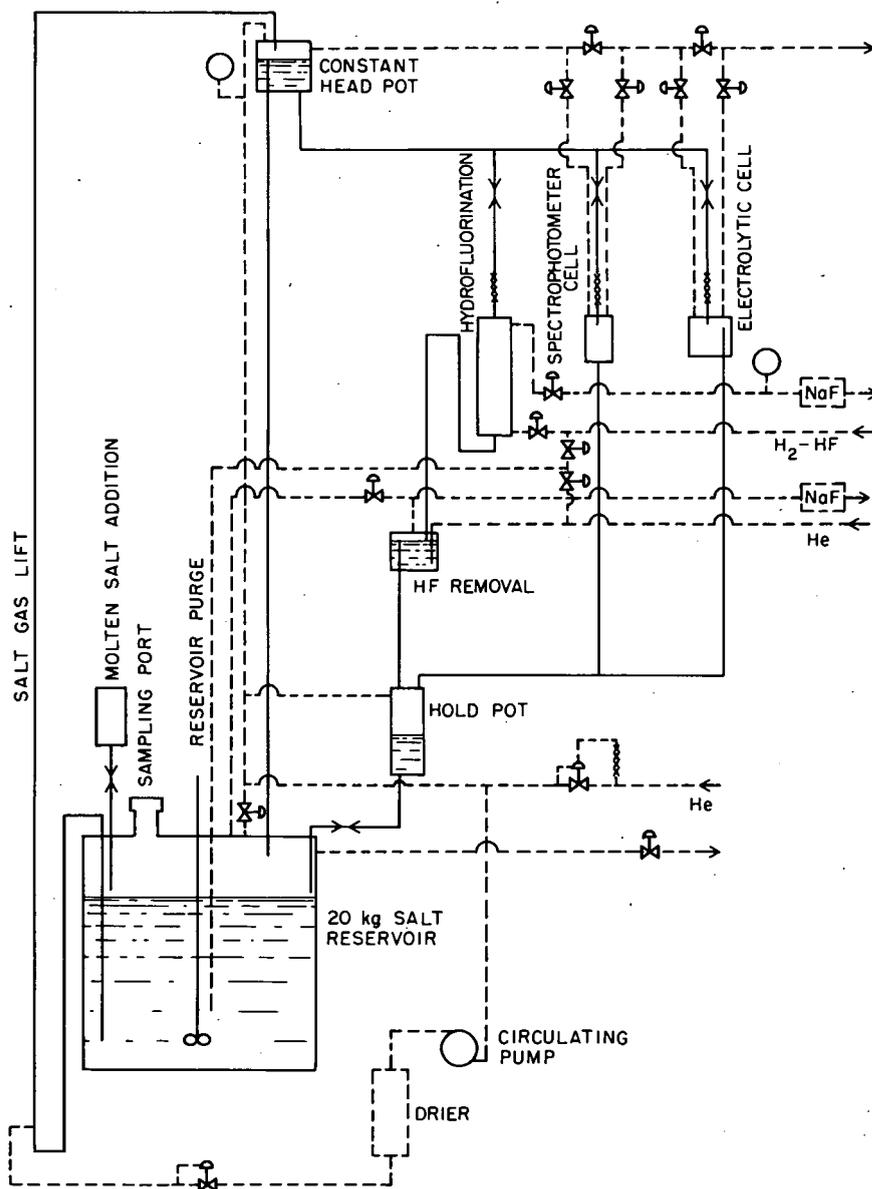


Fig. 3.6. Schematic Flow Diagram of an Experimental Molten-Salt Test Loop.

the Ni-Ni²⁺ couple appears to be a good choice for a reference electrode for molten-fluoride environments.

So far the results of stability studies are encouraging. For a run in molten LiF-NaF-KF, the emf was constant to within ± 2 mv over a two-week period. These studies are continuing, and studies of the stability over longer periods are planned.

3.2.c Electroreduction of Uranium(IV) in Molten LiF-BeF₂-ZrF₄ at Fast Scan Rates and Short Transition Times

D. L. Manning Gleb Mamantov⁸

Controlled-potential voltammetric and chronopotentiometric studies were carried out on the reduction of U(IV) in molten LiF-BeF₂-ZrF₄

(65.6-29.4-5.0 mole %). The controlled-potential controlled-current cyclic voltammeter used was constructed by T. R. Mueller.⁹ In the controlled-potential mode, scan rates from 0.005 to 500 v/sec are available, and cell currents as large as 100 ma can be measured. In the controlled-current mode, currents in the range from a few microamperes to 100 ma can be passed through the cell. The built-in time base permits transition times from 4 msec to 400 sec to be measured. The instrument can also be operated in a potential-step mode for chronoamperometric experiments. Readout of the curves is accomplished with a Tektronix type 549 storage oscilloscope.

In molten $\text{LiF-BeF}_2\text{-ZrF}_4$ at 500°C, the U(IV) → U(III) reduction occurs at about -1.2 v vs a platinum quasi-reference electrode. At scan rates from ~0.02 to ~1 v/sec, the peak current (i_p) obeys the Randles-Sevcik equation, that is, the peak current is proportional to the concentration of U(IV) and also to the square root of the rate of voltage scan ($v^{1/2}$). The diffusion coefficient for U(IV) at 500°C is $\sim 2 \times 10^{-6}$ cm²/sec. At faster scan rates, however, the i_p vs $v^{1/2}$ plots frequently exhibit upward curvature, particularly at platinum and platinum-rhodium indicator electrodes. It appears that this deviation from the Randles-Sevcik equation is caused, in part, by adsorption of U(IV) on the surface of the electrode. Correction for the adsorption was made by plotting $i_p/v^{1/2}$. The slope reflects adsorption effects, and the intercept contains the Faradaic term. The diffusion coefficient calculated from the intercept was in good agreement with the value from the linear i_p vs $v^{1/2}$ plots.

In chronopotentiometry, adsorption of reactant causes the $i_0 \tau^{1/2}$ product, where i_0 = current density, amp/cm², to increase as transition time τ decreases; this effect was observed for U(IV) at short transition times (<100 msec). In general, the potential-time traces were not as well defined as the voltammograms; however, reasonably precise transition times could be established. Approximate corrections for adsorption were made according to the method of Tatwawadi and Bard.¹⁰ The diffusion coefficient

calculated from chronopotentiometric data was in agreement with the voltammetric value.

It is concluded that the U(IV) → U(III) reduction in molten $\text{LiF-BeF}_2\text{-ZrF}_4$ is a reversible one-electron process. However, for the analyses of voltammograms at fast rates of voltage scan or of potential-time curves at short transition times, adsorption phenomena could be pronounced and must be taken into account.

3.2.d Spectrophotometric Studies of Molten-Salt Systems

J. P. Young

Spectrophotometer for Hot-Cell Use. — It is planned to install a spectrophotometer having an extended optical path that is integral with a hot cell. The path will project into and out of the cell so that absorption spectra of extremely radioactive materials can be obtained routinely. The facility will be similar to that proposed for the continuous spectrophotometric determination of U(III) in the circulating fuel salt of a Molten-Salt Breeder Reactor.¹¹ It will be possible to use both windowed and windowless cells. Experience gained with this facility will be applicable to the reactor installation. The arrangement of the components to provide the extended path is being determined. Equipment and engineering funds have been approved for the design, purchase, and installation of all components in the cell facility. The basic spectrophotometer (Cary model 14H) has been ordered.

Spectra of Uranium(III) and Uranium(IV) in Molten Fluoride Salts. — The spectra of U(III) and U(IV) in molten 2LiF-BeF_2 , $\text{LiF-BeF}_2\text{-ZrF}_4$, and LiF-NaF-KF were obtained at temperatures up to 540°C over the wavelength range 200 to 2400 nm. From a comparison of these spectra with those obtained in other molten-salt systems and in aqueous systems, the coordination number of the uranium species in the molten-fluoride media studied is thought to be 8 or 9. It was not possible to obtain chemically a solution of U(III) free of U(IV). Under the experimental conditions studied, U(III) reacts with alkali-metal fluorides

⁹Analytical Instrumentation Group.

¹⁰S. V. Tatwawadi and A. J. Bard, "Chronopotentiometric Measurement of Adsorption. Riboflavin on a Mercury Electrode," *Anal. Chem.* **36**, 2 (1964).

¹¹"Spectrophotometric Studies of Molten-Salt Reactor Fuels," *MSR Program Semiann. Progr. Rept. Aug. 31, 1965*, ORNL-3872, p. 145.

to form small amounts of alkali metals. A paper on this work was published.¹²

Compatibility of Silicon Dioxide with Molten Fluoride Salts. — C. F. Baes, Jr.,¹³ pointed out that, according to the thermodynamics of chemical reactions, molten fluoride salts such as $2\text{LiF}\cdot\text{BeF}_2$ should be compatible with SiO_2 in a static system that contains excess SiF_4 at temperatures up to $\sim 1000^\circ\text{K}$. In collaboration with C. F. Baes, Jr., and C. E. Bamberger,¹³ experimental data were obtained that support this postulate. Such variables as melt composition and temperature affect the equilibrium products. Dissolved O^{2-} and SiF_4 probably are involved in the overall reaction with SiO_2 and can react with components in the melt.

From the constancy of the absorbance of U(IV) at 640 and 1090 nm, the U(IV) concentrations of melts held in SiO_2 containers under SiF_4 pressure at 400 mm remained constant, within experimental error, for at least 48 hr. During longer periods (up to one week) the net absorbance of U(IV) did not change, but the SiO_2 container became etched and thus caused a shift in the absorbance of the blank. Since UO_2 is more insoluble in molten $2\text{LiF}\cdot\text{BeF}_2$ than is BeO , U(IV) was used as an "internal indicator" to measure any increase in O^{2-} concentration. The fact that the U(IV) concentration did not decrease infers that the melt did not react with the container. The cause of the etching of the container is not completely understood but may be the reaction of gaseous or dissolved SiF_4 with SiO_2 to promote devitrification. The ability to contain $\text{LiF}\cdot\text{BeF}_2$ melts in silica cells has permitted the molar absorptivities (ϵ 's) of U(IV) to be calculated more precisely. The ϵ 's of U(IV) in molten $2\text{LiF}\cdot\text{BeF}_2$ at 550°C are reported to be ~ 7 and ~ 14 liters mole⁻¹ cm⁻¹ at 640 and 1090 nm respectively. These values are based on the spectra of melts contained in windowless cells. From measurements in silica cells of known path length, the ϵ of U(IV) is 7.3 ± 0.3 and 14.4 ± 0.5 liters mole⁻¹ cm⁻¹ at 640 and 1090 nm respectively in molten $2\text{LiF}\cdot\text{BeF}_2$ at 560°C . The similarity of the windowless- and windowed-cell data is evident; the improved precision of the latter values is to be expected.

¹²J. P. Young, "Spectra of Uranium(IV) and Uranium(III) in Molten Fluoride Solvents," *Inorg. Chem.* **6**, 1486 (1967).

¹³Reactor Chemistry Division.

Table 3.3. Effect of Temperature on the ϵ 's of U(IV) in Molten $2\text{LiF}\cdot\text{BeF}_2$

Temperature (°C)	ϵ (liters mole ⁻¹ cm ⁻¹)	
	At 640 nm	At 1090 nm
498	8.0	16.4
560	7.3	14.4
650	6.2	13.8
698	5.7	12.9

Also, the effect of temperature on the ϵ of U(IV) at several wavelengths in $2\text{LiF}\cdot\text{BeF}_2$ was studied in silica cells; Table 3.3 gives the results.

Spectral Studies of Chromium(III) in Molten Lithium Fluoride-Beryllium Fluoride. — In further work with C. E. Bamberger,¹³ it was demonstrated spectrophotometrically that the solubility of Cr(III) in $2\text{LiF}\cdot\text{BeF}_2$ at 550°C is relatively high (> 0.4 mole %). The melts were contained in silica cells. The ϵ of Cr(III) at 690 nm was calculated to be 6.6 liters mole⁻¹ cm⁻¹. Peaks also occur at 442 and 302 nm; the ϵ 's of Cr(III) at these wavelengths are ~ 10 liters mole⁻¹ cm⁻¹.

Spectral Studies of Uranium(VI) in Molten Lithium Fluoride-Beryllium Fluoride. — A study of the spectrum of U(VI) in molten fluoride salts was begun in collaboration with G. I. Cathers,¹⁴ who furnished the solute Na_2UF_8 . It should be possible to determine the solubility of U(VI) in such solutions. The container material for molten-salt solutions of U(VI) must be inert to oxidation. Silica is unsatisfactory, because the spectrum of Na_2UF_8 that was dissolved in $2\text{LiF}\cdot\text{BeF}_2$ under SiF_4 overpressure in a silica cell is identical with that of UO_2F_2 dissolved under similar conditions. Apparently, the melt contained enough oxide to cause the formation of UO_2^{2+} . At 550°C the dissolved species exhibited an absorption peak at 419 nm and the foot of a strong absorption peak in the ultraviolet region that had a shoulder at 310 nm. The spectrum generally corresponds very well to that reported for UO_2^{2+} in aqueous HClO_4 .^{15,16}

¹⁴Chemical Technology Division.

¹⁵J. T. Bell and R. E. Biggers, "The Absorption Spectrum of the Uranyl Ion in Perchlorate Media: Part II. The Effects of Hydrolysis on the Resolved Spectral Bands," *J. Mol. Spectry.* **22**, 262 (1967).

¹⁶J. T. Bell and R. E. Biggers, "The Absorption Spectrum of the Uranyl Ion in Perchlorate Media. Part III. Resolution of the Ultraviolet Band Structure," submitted for publication in the *Journal of Molecular Spectroscopy*.

Simultaneous Electrochemical Generation and Spectrophotometric Study of Solutes in Molten Fluoride Salts. — In collaboration with F. L. Whiting¹⁷ and G. Mamantov,⁸ work was begun in which solute species of interest are generated electrochemically in a molten salt while the spectra of the species in the vicinity of the electrode are obtained simultaneously. Such studies will be confined to fluoride melts. The technique should permit the formation and direct identification of unusual and perhaps unstable solute species. The novel feature of this electro-optical study is the ability to observe only the region adjacent to the generating electrode.

To demonstrate the technique, U(III) was generated and observed spectrally in molten LiF-BeF₂-ZrF₄ that contained U(IV). In a modified captive-liquid cell¹⁸ equipped with a three-wire electrode system, a solution of 2 wt % U(IV) in molten LiF-BeF₂-ZrF₄ at 540°C was maintained at a potential sufficient to cause the reduction of U(IV) to U(III) at a working electrode of 0.1-cm² area. About 0.75 microequivalent of U(III) was generated; it was possible to observe spectrophotometrically as little as 0.4 microequivalent of U(III). By spectral observation at 360 nm during intermittent electrolysis, the formation, diffusion, and reoxidation of U(III) could be followed readily. A note on this work was published.¹⁹

Apparatus was designed that permits further studies in either windowless or silica cells in which the light beam is masked to an area slightly larger than the generating electrode. Under the experimental conditions used, large cathodic currents result when SiF₄ at atmospheric pressure is present over the melt. The presence of SiF₄ does not affect anodic voltammograms. The reason for this behavior is not understood. However, since SiF₄ is a product of the reaction of fluoride salts with SiO₂, these results indicate that silica containers are useful for the generation and spectral study of only oxidized solute species.

3.3 FUEL PROCESSING

3.3.a Determination of Hydrocarbon Content of Steam

C. M. Boyd

In the production of UO₂ microspheres by the Sol-Gel Process, an important step is the removal

of carbonaceous impurities by treatment with steam. The Reactor Chemistry Division desired a method for the continuous determination of the hydrocarbon content of the steam from tests on this procedure. A dilution system was designed and constructed for sampling this steam and passing it through a Beckman model 109 total hydrocarbon analyzer. This analyzer, which contains a flame-ionization detector, has a maximum full-scale sensitivity of 1 ± 0.1 ppm. At this sensitivity the output signal was attenuated so that a 2000-ppm CH₄ standard could be used directly for calibration. Since the steam will be at atmospheric pressure, it must be drawn into the analyzer and compressed by a sampling pump. To prevent condensation of the steam, the system of valves and flow capillaries was maintained at 110°C. It was possible to take a steam sample at a known flow and to dilute it 20- or 50-fold with air to obtain a sample that was unsaturated with water vapor at room temperature. Provisions were made for checking this dilution by the addition of a more concentrated CH₄ standard to the steam; tests indicated that a dilution error as small as ±5% was possible. This technique of adding a standard to the steam before dilution permits the calculation of total evolved hydrocarbons rather than hydrocarbon concentration only. The system has been used to monitor the hydrocarbon content of steam from the decarbonation of various UO₂ samples as a function of temperature and time.

3.3.b In-Line Analysis of the Components of Gases from the Fluidized-Bed Volatility Pilot Plant

W. F. Peed²⁰ D. R. Anderson²¹
A. S. Meyer

An interhalogen process had been chosen for the selective volatilization of uranium from plutonium

¹⁷Student Guest from the Department of Chemistry, University of Tennessee, Knoxville.

¹⁸J. P. Young, "Windowless Spectrophotometric Cell for Use with Corrosive Liquids," *Anal. Chem.* **36**, 390 (1964).

¹⁹J. P. Young, G. Mamantov, and F. L. Whiting, "Simultaneous Voltammetric Generation of U(III) and Spectrophotometric Observation of the U(III)-U(IV) System in Molten Lithium Fluoride-Beryllium Fluoride-Zirconium Fluoride," *J. Phys. Chem.* **71**, 782 (1967).

²⁰On loan from Thermonuclear Division.

²¹ORAU Student Trainee from Elizabethtown College, Elizabethtown, Pa.

in the Fluidized-Bed Volatility Process. The uranium, dispersed in a fluidized alumina bed as U_3O_8 , is converted to volatile UF_6 by treatment with BrF_5 at $450^\circ C$. Subsequently the plutonium is volatilized by treatment with fluorine. Since the BrF_5 was to be regenerated and recycled, an in-line method was needed to monitor the concentration of reagent reduction products, BrF_3 and Br_2 , in the effluent from the fluidized-bed reactor. This stream would also be expected to contain CF_4 , SF_6 , HF, COF_2 , reagent contaminants, and volatile fission products such as MoF_6 .

A general survey of the physical properties of these stream components indicated that, except for the spectrophotometric measurement of Br_2 , no direct determinations would be practical without preliminary separations. Therefore, gas chromatography was selected. A Greenbrier laboratory chromatograph was used that had been modified by the installation of a Teflon-plug sampling valve, a Gow-Mac gas-density detector, and a heated flow system to permit the use of Freon-114 (1,2-dichlorotetrafluoroethane) as a carrier. Freon-114 was selected to suppress the signal of the major component, BrF_5 , by virtue of their comparable densities. A system was constructed for the preparation of calibration standards by saturating helium with each of the interhalogen compounds and UF_6 .

Screening tests of column packing materials were run with relatively small columns (7-ft by $\frac{1}{4}$ -in.-OD nickel tubing) to more rapidly obtain results. In all cases Kel-F-10 oil was used as a substrate at loadings from 5 to 50% and at temperatures of $55^\circ C$ and $80^\circ C$. Columns packed with Teflon-6 as support material exhibited excessive tailing, particularly for the UF_6 peaks, apparently as a result of absorption of the solute by the support. It was calculated that large columns (about 20 ft by $\frac{1}{2}$ in.) would be required and that analysis time would exceed 1 hr, a period unacceptable for process analysis.

Since alumina is not attacked rapidly by the halogenating reagent even at pilot-plant temperatures, attempts were made to use alumina as a support material. With Alcoa T-60 alumina, a moderately high-fired material used in the fluidized-bed reactor, the tailing was reduced substantially, and, surprisingly, relatively brief interhalogen pretreatments were required for column conditioning. With a 10% Kel-F loading, which was the most favorable concentration, elution times (min) from a 7-ft column at $80^\circ C$ and a carrier flow of

10 ml/min were: BrF_3 , 6.5; BrF_5 , 9.5; Br_2 , 9.5; and UF_6 , 12.5. No discernible resolution between Br_2 and BrF_5 was obtained under any of the conditions tested. Attempts to use various grades of activated alumina were unsuccessful, because the interhalogens and uranium were not eluted even after repeated treatments with ClF_3 .

Theoretically it is possible to completely suppress the signal from BrF_5 by using a mixed carrier of accurately matching density and thus to permit the desired measurement of Br_2 in the presence of BrF_5 . Mixtures of Freon-114 and Freon-318 (octafluorocyclobutane) would be suitable.

Although complete resolution was not demonstrated in this study, extrapolations indicate that it might be possible to obtain satisfactory analyses within an acceptable period (15 to 20 min) by using 20-ft columns packed with alumina-supported Kel-F oil and operated above $100^\circ C$. This study was terminated when construction of the Fluidized-Bed Volatility Pilot Plant was discontinued.

3.4 NUCLEAR SAFETY

3.4.a Analysis of Purge Gas from In-Pile Tests of Reactor Fuel Elements

W. F. Peed²⁰ R. F. Apple
A. S. Meyer

The design of an analytical system²² for the in-line analysis of the gases generated by in-pile tests of fuel elements has been changed to incorporate a residual-gas analyzer (RGA). The Analytical Chemistry Division is assisting the Reactor Chemistry Division in the design of an RGA system and in the procurement of components. The substances to be determined include H_2 , O_2 , N_2 , H_2O , CO_2 , inert gases, fission products, and other possible components, such as organoiodine compounds, that are produced when fuel-element specimens are purged in-pile with moist inert gases or with dry or moist air. Requirements for frequency of analyses will vary from several per minute for a 5-min meltdown experiment to only occasional analyses during steady-state exposure of graphite-matrix GCR-type elements to an

²²R. F. Apple and A. S. Meyer, "Analysis of Purge Gas from In-Pile Meltdown Experiments," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1966*, ORNL-4039, p. 24.

atmosphere of moist helium. Concentrations will range from low-ppm to percentages.

In this application the RGA will replace the originally planned combination of different analytical transducers, each of which responds continuously to a single component of the sample. The substitution of the RGA will permit the measurement of more components over greater ranges of concentrations and at an acceptable analysis frequency. It will be necessary to make a separate determination of CO with a process infrared analyzer (Beckman model 315), because the parent peaks of CO and N₂ are not resolved in mass spectra.

The infrared analyzer has been installed in the gas-sampling hood at the ORR. Absorption cells and detector units have been calibrated to provide full-scale responses in the range 250 ppm to 25% CO. A gas-sampling technique was developed to transfer samples from a simulated reactor containment vessel at reduced pressure to the analyzer, and then to compress the sample to atmospheric pressure for absorption measurements. This technique was used to determine gas mixing rates for in-pile meltdown experiments. When CO was injected into air at 100°C to simulate an air burnup test, the concentrations were within 1% of those calculated for instantaneous mixing, even when the injection rate was reduced to one-half the designed flow. Similar results were obtained for injections into unheated air. When injections were made into unheated argon, however, the injected gas stratified with the upper layer enriched in CO. Mixing occurred within a 5-min interval after one of the heaters had been turned on. Actual tests carried out with irradiated gases from an in-pile experiment showed that the instrument is not affected by radiation. The manifold in the lead-shielded gas-sampling hood at the ORR was modified so that the absorption cell of the analyzer can be evacuated and connected either to the reactor containment vessel or to an evacuable 2-liter container. With this arrangement a calibrating mixture of radioactive vessel gas and CO was prepared and transferred to the infrared cell. A comparison of these results with those previously obtained for CO in nonradioactive gas showed that the measurement of CO is not affected by radiation at the maximum anticipated level.

The analyzer has also been used throughout two GCR-type experiments of about one-week duration

each to record CO concentrations in the range 1000 to 2000 ppm.

To adapt the RGA to the continuous monitoring of the purge gas, a small fraction of the effluent stream will be bled at about 1 to 10 torrs into an intermediate-pressure system located in a shielded hood at the ORR. By way of a capillary, a continuous sample will be withdrawn from the intermediate-pressure system and injected into the ionizer section of the RGA, which is to be located in an oven outside the hood. Since the pressure in the analyzer will be of the order of 10⁻⁶ torr, no hazardous quantity of radioactive gases will be present outside the shielded area. The vacuum system, which includes a mechanical pump for the intermediate-pressure region, a differential ion pump for the RGA, and several isolation valves, will be completely contained within the sampling hood.

Criteria considered in the selection of the components of an RGA system included compactness of the vacuum system with fail-safe interlocks to prevent the buildup of activity during a power failure and an RGA of adequate sensitivity, resolution, and stability.

A quadrupole RGA was borrowed from the Ultek Division of the Perkin-Elmer Corporation and was tested in our laboratories on an approximate mock-up of the proposed gas-inlet system. Although it was not possible to obtain sensitivities below 100 ppm because of an inadequate pumping system and severe contamination of the instrument (a demonstrator), the equipment proved remarkably stable and reproducible. Also, the sensitivity improved throughout the limited period of these tests as steps were taken to reduce the background from adsorbed gases. It appears that the RGA will function in this application within the specified 1-ppm sensitivity. It is anticipated that by use of a picoammeter and a two-pen recorder, both of which are available commercially, the operation of the RGA can be centralized in the control room rather than operated remotely on the reactor balcony. By suitable choice of electrometer output and recorder sensitivity, a dynamic range in recording of more than 1000 can be achieved without changing the sensitivity of the picoammeter.

Specifications for the components of this system have, therefore, been written and submitted for bids.

3.4.b Gas Chromatographic Determination of Volatile Air Pollutants

A. D. Horton A. S. Meyer

In the vicinity of ORNL, samples of air were collected²³ from the secondary containment of each reactor, the Nuclear Safety Pilot Plant, various locations external to research buildings, and the Clark Center Recreation Park.

Samples of air and of charcoal were received from the blower inlet and the primary containment of the CVTR in Columbia, South Carolina. These samples were taken by employees of the Phillips Petroleum Company, who followed ORNL analytical chemistry procedures and used equipment that was developed, fabricated, and pretested by this laboratory. The cooperative program with Phillips is expected to continue at an accelerated pace during the coming year. Several reactor-containment systems in different localities are to be sampled.

Analysis of the volatile hydrocarbons in these samples by gas chromatography gives the concentration of certain classes of organic compounds,

namely unsaturated and/or oxygenated compounds that are most likely to form volatile organic iodides with fission product iodine. These volatile iodides are very difficult to remove from reactor-containment air.

Volatile organic compounds from 2.5 liters of air were trapped,²³ and the contents of the trap were swept onto a 9-ft by 1/4-in. column that contained 20 wt % silicone 550 on 60- to 80-mesh Anakrom ABS. Total hydrocarbons were determined by use of the flame-ionization detector. Unsaturated hydrocarbons were determined on the same column by subtracting from the total hydrocarbons the hydrocarbons that passed through a mercuric perchlorate olefin absorber.²⁴ Oxygenated hydrocarbons were determined, also on the silicone 550 column, by subtracting the hydrocarbons that

²³A. D. Horton, "Determination of Organic Pollutants in Air," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1966*, ORNL-4039, p. 24.

²⁴R. L. Martin, "Determination of Hydrocarbon Types in Gasoline by Gas Chromatography," *Anal. Chem.* **34**, 896 (1962).

Table 3.4. Results of Gas Chromatographic Determination of Volatile Organic Air Pollutants

Location of Sample	Hydrocarbon Concentration as CH ₄ Equivalent ^a (vol ppm)		
	Total	Total Unsaturated	Total Oxygenated
Outside entrance to W corridor, Bldg. 4500S	2.782	2.548	<i>b</i>
E loading dock, Bldg. 4500S	1.760	1.613	<i>b</i>
NSPP	10.690	10.380	9.650
ORR	7.500	7.140	7.400
MSRE	5.500	5.150	2.900
BSR	1.247	1.185	0.749
HFIR	0.330	0.230	0.100
HPRR	0.213	0.146	0.067
Clark Center Recreation Park	0.188	0.139	0.087
CVTR, Columbia, S.C.			
Primary containment	54.300	47.900	37.300
Blower inlet	22.900	2.900	<i>c</i>

^aMinimum detectable, 1 ppm.

^bProcedure not available when this sample was taken.

^cInsufficient sample.

Table 3.5. Identifiable Volatile Organic Air Pollutants

Pollutant ^a	Pollutant Concentration as CH ₄ Equivalent (vol ppb)										
	W	E Loading	NSPP	ORR	MSRE	BSR	HFIR	HPRR ^b	Clark Center Recreation Park	CVTR	
	Corridor, Bldg. 4500S	Dock, Bldg. 4500S								Primary Containment	Blower Inlet
Methane ^c	230	210	47	20	42	56	39	45	36	1	9
Ethane	21	11	3	2	4	7	2	1	1	14	
Ethylene	1	70	3	3	2	3	2	<1	<1	14	2
Propane	<1	1	1	1	4	3	2	1	1	9	6
Propylene	7	12		<1		1				140	
Isobutane			1				1				2
<i>n</i> -Butane	3	7	3	7	4	1	17	1	1		9
Butene-1		8									9
Isobutylene + <i>cis</i> - butene-2										210	
Isopentane			4	7	2	1	14		1		3
<i>n</i> -Pentane	4		1	1	2	2	6		<1	110	2
2-Ethylbutene-1				3							
Pentene-1	5	90	7				12		4		
Pentene-2 (<i>cis</i> and <i>trans</i>)	62	190									
<i>n</i> -Hexane	18	100	16	6	3	3	23	2		2900	3
Hexene-1	160	66									
<i>n</i> -Heptane	14	50	2	10		8				3000	770
Total	~526	815	88	~61	63	85	118	~51	~46	6398	815

^aCompounds listed in order of elution from the column.

^bNo personnel in building – reactor had not operated for three weeks.

^cMethane is not completely trapped by liquid nitrogen.

Table 3.6. Total Hydrocarbons in Charcoal Traps of the CVTR

C _n Paraffins of Corresponding Boiling Range	Hydrocarbon Content (wt %)						Blower Inlet ^a Single Filter
	Primary Containment May Pack						
	Run 1 ^a			Run 2 ^b			
	Top	Middle	Bottom	Top	Middle	Bottom	
<C ₆	0.18	0.04	0.11	0.90	0.17	0.14	0.10
C ₇	0.85	0.45	0.02	0.14	<0.16	1.00	0.56
C ₈	0.72	0.05	0.002	0.32	0.42	2.04	0.81
C ₉	0.81	<0.001	<0.001	0.70	3.41	0.77	0.67
C ₁₀	0.73	0.02	0.04	1.66	0.67	0.02	0.51
C ₁₁	0.17	0.005		4.09	0.05	0.01	0.10
C ₁₂	0.03	0.002		1.41			0.02
>C ₁₂	0.02	0.001		0.34	0.73	0.02	0.02
Total	3.51	0.57	0.17	9.56	7.61	4.00	2.79

^aTrap on stream 100 hr; rate of flow, 60 ft³/hr.

^bTrap on stream 1500 hr; rate of flow 60 ft³/hr.

passed through a 1% solution of NaHSO₃²⁵ from total hydrocarbons. Identifiable hydrocarbons were determined by use of an 8-ft by 1/4-in. column that contained 3 wt % squalane on 30- to 60-mesh medium-activity silica gel.

Hydrocarbons were expelled from a charcoal sample by heating it to 400°C in the Honaker-Horton pyrolyzer²⁶ and sweeping the volatile components onto the silicone 550 column. Total hydrocarbons in each of the boiling ranges that correspond to the boiling points of the normal paraffins were determined.

Table 3.4 lists the results of the determinations of total, unsaturated, and oxygenated hydrocarbons; identifiable hydrocarbons are listed in Table 3.5. Results for the volatile organic compounds in the charcoal traps are shown in Table 3.6.

²⁵C. F. Ellis, R. F. Kendall, and B. H. Eccleston, "Identification of Some Oxygenates in Automobile Exhausts by Combined Gas Liquid Chromatography and Infrared Techniques," *Anal. Chem.* **37**, 511 (1965).

²⁶C. B. Honaker and A. D. Horton, "A Simple Pyrolyzer for Use with Gas Chromatography," *J. Gas Chromatog.* **3**, 396 (1965).

The extent of contamination of air and of charcoal by volatile organic compounds depends on the environment; the amount will vary from day to day or even from hour to hour. A major factor is the number of internal-combustion engines operating in the vicinity. Open cans of solvent; fresh paint; the density of human population; the number and intensity of heat sources adjacent to volatile or easily decomposed organic compounds; the use of pumps, compressors, or other oil-filled equipment; tobacco smoke – all these and many unnamed sources contribute to the contamination of the containment air. The newer buildings, in general, are less contaminated than the older ones. In the case of outdoor samples, the concentration of contaminants is directly proportional to the density of automobiles operating in the immediate vicinity of the sampling point. Of the samples taken, that from the Clark Center Recreation Park contained organic contaminants in lowest concentration. This sample was taken as a control and was collected at the extreme west end of the park in the winter season.

4. Special Research and Development Activities

P. F. Thomason
W. D. Shults

A. S. Meyer
J. M. Dale

4.1 CHROMATOGRAPHY

4.1.a Gas Chromatography

Preparative Gas Chromatography (A. D. Horton).

— A preparative gas chromatograph manufactured by Varian-Aerograph has been in almost continuous operation since February 1967. The work with it has consisted exclusively of the purification of organic compounds. Benzene, *n*-pentane, *n*-hexane, *n*-heptane, dichloromethane, carbon tetrachloride, chloroform, 2-ethylnaphthalene, and aniline have been purified to >99.99 mole %. Lesser purity (mole %) was achieved for 1-chloropropane (99.96), 1-chloronaphthalene (99.97), *n*-hexylnaphthalene (99.60), and 1,2-dimethylnaphthalene (99.00). The purity of the reagents submitted varied from about 30 to 99.95 mole %; it was always enhanced after the chromatography.

The major factor in obtaining ultrapure compounds is not the original purity of the compound but the proximity of the elution peaks of impurities to that of the parent compound. Figure 4.1 illustrates both the typical problems that make it difficult to collect ultrapure compounds and a partial remedy for some of these problems. Ac-

cording to the manufacturer's specifications, the turntable that positions the sample-collecting bottles under the exit tip of the column is actuated by a micro switch attached to the recorder scale. The micro switch is actuated by the pointer attached to the pen carriage. The downscale contact with the switch returns the bottle to a standby position. Thus, as shown in Fig. 4.1 A, the "cut" must be made in the same position on both the leading and trailing sides of the peak.

A two-switch arrangement (Fig. 4.1 B) was developed at ORNL. It allows different cut points to be selected on each side of the peak or two cuts on either side of the peak. This arrangement has resulted in an improvement of several percent in the purity achieved for some compounds and makes possible the purification of compounds to 99.99% purity (Fig. 4.1 C). Under the worst conditions, only ~10% of the compound injected into the chromatograph will be collected as pure compound.

Gas Chromatographic Determination of Sulfur Hexafluoride in Methane (A. D. Horton). — The size of the sulfur hexafluoride molecule is such that it cannot pass into a 5-A molecular sieve.

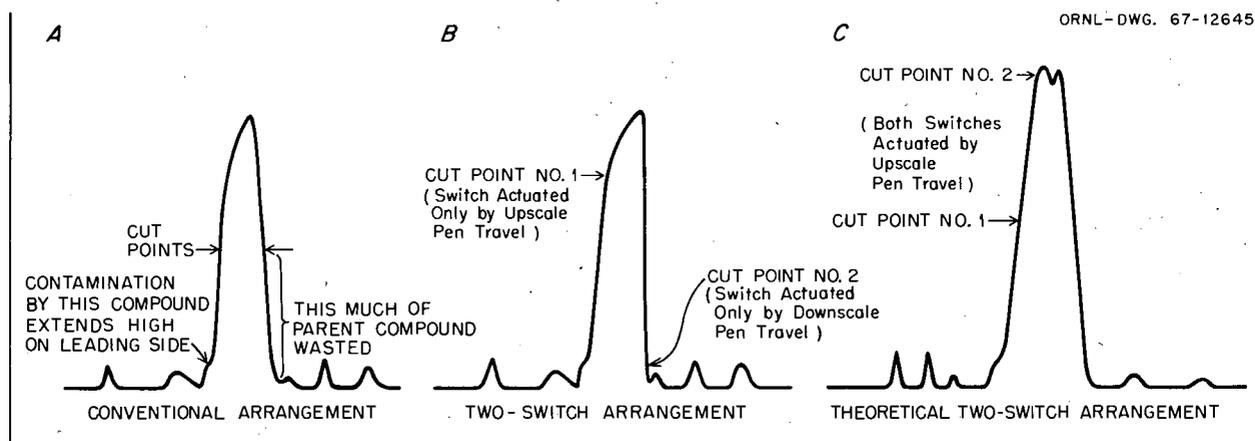


Fig. 4.1. Preparative Chromatogram of 1,2-Dimethylnaphthalene (A and B) and Theoretical Chromatogram (C) That Illustrate the Advantages of the Two-Switch Arrangement for Fraction Collection.

Therefore, it evolves from the gas chromatographic column before hydrogen. This property makes possible the determination of as little as 5 ppm of SF₆ in large quantities of air, methane, or other gases except neon. The thermal-conductivity detector was used to analyze three different samples of SF₆-CH₄ mixtures for D. R. Nelson.¹ The samples were analyzed in triplicate with a deviation from the mean of $\pm 5\%$; their SF₆ contents were 14.4, 17.0, and 20.6 ppm.

Application of Gas Chromatography to Problems in Powder Metallurgy (A. D. Horton). – M. M. Martin² reported difficulty in extruding aluminum

parts by powder metallurgical processes, because gases freed from the mold-release compound or from the aluminum powder itself caused the extrusion to swell. Samples of the aluminum extrusion powder and of a mold-release compound (Fisk No. 604) were pyrolyzed in the Honaker-Horton pyrolyzer³ at the temperature used in the extrusion process. Organic volatiles evolved from a silica gel-squalane column were determined by use of the hydrogen flame-ionization detector; Table 4.1 gives the results. These materials are still being used in the extrusion process, but the technique has been altered to prevent occlusion of the gases by the aluminum powder.

Table 4.1. Volatile Organic Compounds Obtained from the Pyrolysis of Fisk No. 604 Mold Release and S-51 Aluminum Powder

Compound ^a	Amount Obtained (wt ppm)	
	Fisk No. 604 (436°C) ^b	S-51 Aluminum (600°C) ^b
Methane		250
Ethane	2	160
Ethylene	2	460
Propane		80
Propylene	19	
Isobutane		3
<i>n</i> -Butane	3	300
Butene-1	89	
Isobutylene + <i>cis</i> -butene-2	215	2
Isopentane	11	2
<i>n</i> -Pentane	44	130
Pentene-1	111	10
2-Methylbutene-1		30
<i>n</i> -Hexane	85	50
Hexene-1	246	60
Hexene-2	96	10
Hexene-3	80	
3-Methylpentene-1		50
2-Ethylbutene-1	296	
<i>n</i> -Heptane	152	
<i>n</i> -Octane + heptene-1	217	140
Heptene-2		5
Total	1668	1742

^aCompounds listed in order of elution from the column.

^bPyrolysis temperature.

4.1.b Thin-Layer Chromatography

Thin-Layer Chromatography of Constituents of Transfer Ribonucleic Acid (tRNA) (Helen P. Raaen, Frances E. Kraus⁴). – Procedures were developed for resolving complex mixtures of constituents of tRNA by thin-layer chromatography on freshly prepared 500- μ -thick layers of polyethyleneimine (PEI)-cellulose MN-300. The procedures are adaptations of those described by the Randeraths.⁵⁻⁷ The effectiveness of the separations was demonstrated by isolating (with three exceptions) the individual components of a mixture that contained 10 nucleobases, 10 nucleosides, and 12 nucleotides. On one chromatofilm, nucleobases and nucleosides are resolved by two-dimensional development, first with water and then with *n*-butanol-methanol-water-NH₄OH_{conc} (60:20:20:1, v/v); on another, nucleotides are separated by sequential develop-

¹Health Physics Division.

²Metals and Ceramics Division.

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

⁴ORAU Student Trainee from Nazareth College, Nazareth, Ky.

⁵E. Randerath and K. Randerath, "Resolution of Complex Nucleotide Mixtures by Two-dimensional Anion-exchange Thin-layer Chromatography," *J. Chromatog.* 16, 126 (1964).

⁶K. Randerath, "Two-dimensional Separation of Nucleic Acid Bases on Cellulose Layers," *Nature* 205, 908 (1965).

⁷K. Randerath and E. Randerath, "Ion-exchange Thin-layer Chromatography. XV. Preparation, Properties and Applications of Paper-like PEI-Cellulose Sheets," *J. Chromatog.* 22, 110 (1966).

ment in the first direction with 0.2, 1.0, and 1.6 M LiCl, washing with methanol, and then sequential development in the second direction with 0.5, 2.0, and 4 M HCOOH-HCOONa buffer of pH 3.4. The work included the evaluation of several developing solvents and the comparison of freshly prepared adsorbent layers of cellulose MN-300 and of PEI-cellulose MN-300 with commercially available layers of the same types.

The method was used to isolate and identify the tRNA constituents in the alkaline hydrolysates of *E. coli* B and of yeast and in an eluate fraction from an *E. coli* B solution separated on a Sephadex-100 column. A journal article has been prepared to be submitted for publication. Also, the details of the separation procedures are recorded as a method⁸ for inclusion in the *ORNL Master Analytical Manual*.

Thin-Layer Chromatography of Amanita Toxins (Helen P. Raaen). — Amanita toxins are polypeptides that contain unusual amino acids. The crystalline structure of these high-molecular-weight compounds is of interest to C. K. Johnson of the Chemistry Division. These deadly toxins are present in certain species of mushrooms, for example, *Amanita phalloides* and *Galerina marginata*. Sullivan, Brady, and Tyler⁹ have published a method to separate α - and β -amanitin by thin-layer chromatography on silica gel adsorbent and to identify them by the characteristic violet color of their complexes with *trans*-cinnamaldehyde on exposure to HCl vapor. The method was modified somewhat to give sharper bands and better resolution of the amanitins. The improvement was achieved by the use of shaped layers of adsorbent and overrun of the development. It was found that freshly prepared chromatoplates give much more satisfactory results than do three commercially available plates. Some 130 samples were analyzed by the improved method. The results were used to determine the effectiveness of the fermentation runs being made by the Biology Division to prepare the pure crystalline amanitin toxins in quantity. The improved thin-layer chromatographic

method was written for inclusion in the *ORNL Master Analytical Manual*.¹⁰

Thin-Layer Chromatography of Proteins on Sephadex G-75 Superfine Gel (Frances E. Kraus,⁴ Helen P. Raaen). — Limited experiments were made to evaluate thin layers of Sephadex gel for the chromatographic separation of proteins according to molecular weight. Eleven proteins (molecular-weight range, 12,400 to 69,000) were chromatographed individually and as a mixture on 500- μ -thick layers of Sephadex G-75 Superfine. The procedure of Johansson and Rymo¹¹ was followed; the developer was 0.02 M Na₃PO₄–0.2 M NaCl of pH 7.0. Satisfactory resolution of the protein mixture was not achieved under the experimental conditions used.

4.2 ELECTROANALYTICAL CHEMISTRY

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

Helen P. Raaen

Evaluation of Vertical-Orifice Rapid Teflon D.M.E.'s for Obtaining Fundamental Polarographic Data. — In the continued evaluation of vertical-orifice rapid Teflon D.M.E.'s,¹² a D.M.E. was used that consists of a 72- μ -diam Teflon segment and a 70- μ -diam, 98-mm-long glass-capillary segment. The reference reaction studied was $Tl^+ \rightarrow Tl^0$ in 0.1 M KCl–1 mM HCl–0.1 w/v % polyacrylamide. The results show that 0.1 w/v % polyacrylamide adequately suppresses the maxima that occur on polarograms obtained with this D.M.E. When polyacrylamide is used, the D.M.E. is suitable for rapidly obtaining reproducible and theoretically correct polarograms. The data for the $Tl^+ \rightarrow Tl^0$ reference reaction that justify this conclusion are:

1. The average of five measured $E_{1/2}$ values (–0.458 to –0.460 v) is –0.459 v vs S.C.E.; the accepted value is –0.460 v.

⁸Helen P. Raaen, "Separation of Complex Mixtures of Nucleic Acid Bases, Nucleosides, and Nucleotides by Two-Dimensional Thin-Layer Chromatography on Polyethyleneimine-Cellulose," Method No. 1 00703 (10-1-67), *ORNL-Master Analytical Manual*; TID-7015, suppl. 10 (to be published).

⁹G. Sullivan, L. R. Brady, and V. E. Tyler, Jr., "Identification of α - and β -Amanitin by Thin-Layer Chromatography," *J. Pharm. Sci.* 54(6), 921 (1965).

¹⁰Helen P. Raaen, "Separation of Amanita Toxins by Thin-Layer Chromatography on Silica-Gel G Chromatoplates," Method No. 1 00702 (8-23-67), *ORNL Master Analytical Manual*; a method for record only.

¹¹B. G. Johansson and L. Rymo, "Thin-Layer Gel Filtration," *Acta Chem. Scand.* 16(8), 2067 (1962).

¹²Helen P. Raaen, "Evaluation of Vertical-Orifice, Rapid Teflon D.M.E.'s for Obtaining Fundamental Polarographic Data," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1966*, ORNL-4039, p. 28.

2. The known electron change for the reaction ($n = 1$) is obtained both from measurement of peak width of the first-derivative peak recorded at the slow scan rate 0.3 v/min (0.95) and from agreement between the measured value for the slope of the log $[\bar{i}/(\bar{i}_d - \bar{i})]$ vs potential plot (0.061) and the theoretical value (0.059).
3. The known reversibility of the reaction is shown by the facts that the value for log $[\bar{i}/(\bar{i}_d - \bar{i})]$ is zero at the measured $E_{1/2}$; the theoretical value is obtained for the slope of the log $[\bar{i}/(\bar{i}_d - \bar{i})]$ vs potential plot, and the average-current first-derivative peak recorded at 0.3 v/min is symmetrical.
4. The value for $\bar{i}_d/h^{1/2}$ remains constant (0.591 to 0.596 $\mu\text{a}/\text{cm}^{1/2}$) over a wide range of h (69 to 118 cm).
5. The value for I_d remains constant [3.03 to 3.15 μa (millimoles/liter) $^{-1}$ ($\text{mg}^{2/3}/\text{sec}^{1/2}$) $^{-1}$] over a wide range of h (69 to 118 cm).
6. Both the $[\text{Ti}^+]$ vs \bar{i}_d and the $[\text{Ti}^+]$ vs $(d\bar{i}_d/dt)_{\text{max}}$ relationships are linear up to $[\text{Ti}^+] = 1 \text{ mM}$; data were not recorded at higher $[\text{Ti}^+]$.

A journal article that describes this work is being prepared for publication.

4.2.b Controlled-Potential Coulometric Determination of Uranium(VI) in the Presence of Copper(II)

Karen E. Pashman¹³ W. D. Shults

Samples that contain U(VI) and Cu(II) are presently analyzed coulometrically by an indirect procedure. Both Cu(II) and U(VI) are reduced coulometrically; then copper is reoxidized, and U(VI) is determined by the difference in amount of charge in the reduction and reoxidation electrolyses.¹⁴ Ideally, uranium should be determined by a direct procedure. We studied a number of media, polarographically and coulometrically, in an attempt to find conditions that allow the direct determination of U(VI) in the presence of Cu(II). Citrate, acetate, and ascorbic acid media of

various concentrations and pH's were unsuitable, but hydroxylamine sulfate proved to be a promising supporting electrolyte. In 1 M $(\text{NH}_2\text{OH})_2 \cdot \text{H}_2\text{SO}_4$ the half-wave potentials of Cu(II) and U(VI) differ by ~ 270 mv; therefore, Cu(II) can be reduced (-0.05 v vs S.C.E.), and then the U(VI) can be reduced (at -0.30 v vs S.C.E.). This medium should be useful also for determining U(VI) in the presence of other ions that are reduced chemically by hydroxylamine, for example, Cr(VI), Fe(III), Pu(IV), and Pu(VI). Results are comparable with those obtained when U(VI) is determined directly in sulfuric acid medium.

4.2.c Evaluation of the Performance of Ion-Selective Electrodes

W. D. Shults A. F. Roemer, Jr.¹⁵
Susan S. Potterton¹⁶

Our studies of the performance of various ion-selective electrodes have continued. In addition to the electrodes mentioned in the last annual report,¹⁷ we have tested the Corning and the Orion calcium activity electrodes and the Orion divalent ion, fluoride activity, and nitrate activity electrodes. We have purchased an Orion sulfide electrode for future evaluation. In general, all these electrodes respond in a Nernstian fashion to activities of their particular ions in the pIon range 1 to 4. More dilute solutions can be measured with suitable calibration, but the electrode response curve is not linear and the response itself is rather sluggish. Invariably, there is an optimum pH range for each electrode.

The fluoride and nitrate electrodes have been tested more thoroughly than the others. Such factors as working range, accuracy, precision, response time, memory effects, interferences, effect of temperature, type of reference electrode, and mode of operation were studied. These electrodes can be used to measure activities at the 10^{-1} M level with an error of $\pm 1\%$ and relative standard deviation of $\sim 1\%$; at 10^{-5} M the error

¹⁵Retired.

¹⁶ORAU Student Trainee from Randolph-Macon Woman's College, Lynchburg, Va.

¹⁷P. F. Thomason, W. D. Shults, and J. A. Bell, "Studies of Specific-Ion Electrodes," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1966*, ORNL-4039, p. 31.

¹³ORAU Student Trainee from McMurray College, Abilene, Tex.

¹⁴W. D. Shults and P. F. Thomason, "Controlled-Potential Coulometric Determination of Copper and Uranium," *Anal. Chem.* 31, 492 (1959).

is about $\pm 5\%$ and the relative standard deviation is $\sim 3\%$. Their response times and memory effects are similar to those of a glass pH electrode. Sleeve-type reference electrodes are preferable to fiber-tipped electrodes. The optimum mode of operation is the "two-standard" technique in which the instrument is calibrated with standards of composition similar to that of the unknown but concentrations both greater and less than that of the unknown. Hence, measurements can be made with about the same facility as a conventional pH measurement, and results are comparable with those obtained by colorimetric procedures. The major disadvantage of the nitrate electrode is the necessity to reassemble it every two to three weeks; it incorporates a liquid ion exchange type of membrane. A manuscript that describes our experience with the nitrate electrode has been prepared for publication.¹⁸

4.3 SPECTROMETRY

4.3.a Infrared Spectrometry

M. M. Murray

The acquisition of a Beckman IR-12 spectrophotometer has increased the capabilities of the infrared laboratory. The new instrument has an extended range of 200 to 4000 cm^{-1} (50 to 2.5 μ); that of the previous instrument was 600 to 4000 cm^{-1} (16.6 to 2.5 μ). It is a four-grating instrument that uses long-pass filters to eliminate subordinate orders and has the high resolution that is characteristic of grating instruments.

The instrument is equipped with an ordinate-scale expander and a tracking-accuracy control (T.A.C.). The scale expander permits the expansion of any absorbing band of 5% transmittance or greater to full scale. The T.A.C. has two functions for use in increasing definition. Whenever a signal is generated, the first automatically narrows the slits, and the second automatically reduces the scanning rate. Both functions are triggered by any signal of magnitude greater than that of the background noise.

Other attachments and ancillary equipment obtained with the instrument include a purging ap-

paratus for supplying the instrument with dry air, thus eliminating interference from atmospheric water vapor and possible damage to the CsI thermocouple window; a micro die capable of making 1- by 5-mm alkali halide pellets; a 12-mm-diam die for making alkali halide disks of that diameter; a hydraulic press for use with the dies; a mirror-type beam condenser for examining micro samples in the long-wavelength end of the spectral range; an attenuated total reflectance unit for examining infrared-opaque liquids, films, or semisolids; minimum- and micro-volume gas cells; polyethylene cells for liquids; and a programmed pyrolyzer provided with a cell for trapping gaseous pyrolysis products and with a condenser attachment for trapping liquid pyrolysis products. All this equipment along with that already existent in the laboratory provides considerable versatility and permits the examination of nearly all types of samples capable of being examined by infrared methods.

A wide variety of organic and inorganic samples was examined by infrared spectrometry. 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 reaction, or thermal effects.

Fractions from compounds synthesized at ORNL were examined for structure and purity (Table 4.2). Materials produced by irradiation, chemical reaction, or other processes were identified; a summary of the results follows. Irradiation of tall

Table 4.2. Compounds Synthesized at ORNL That Were Examined by Infrared Spectroscopy

<i>p</i> -Aminobenzylamine	Bicyclobutane
Didodecyl-naphthalene	α ,- α' -Dipiperidyl
Heptyldecylamine	Phenylneotridecanoate
Didodecyl-naphthalenesulfonic acid	LiFe_5O_8 (ordered)
Hydrobromide of diethyl-guanidinodiacetate	LiFe_5O_8 (disordered)
<i>p</i> -Ethylbenzenesulfonic acid	F_2O
Bromo-3-chlorocyclobutane	

¹⁸Susan S. Potterton and W. D. Shults, "An Evaluation of the Nitrate-Selective Electrode," *Anal. Letters* 1, in press (1967).

oil (a by-product of paper production) produces CO, CO₂, H₂S, and C₂H₄; treatment of aqueous KOH with F₂ produces F₂O; and carburization of sol-gel fuels produces a mixture of polynuclear hydrocarbons as a by-product. Commercial products were examined for impurities, water, radiation effects, or signs of oxidation. Spectra were obtained for Itrans 1-6 (proprietary name for infrared window materials), powdered and fused silicas, phenol-aldehyde resins, MgO, lubricating oils, pentaphenyl-trimethyltrisiloxane, NaBF₄, hydrolyzed esters, powdered aluminum, and Teflon. Biological materials for which infrared spectra were taken included RNA, water-soluble fractions of humic acid, nucleic acids, hyatomelanin acid, humic acid, fulvic acid, and fractions from a gel-filtration column. Other analyzed materials that are in use or produced at ORNL included MoF₆, SiF₄, residue from the ignition of graphite-coated sol-gel fuel, and a solution from an Orion calcium-ion electrode.

Future work includes a study, in cooperation with L. C. Brown,¹⁹ of the production of ClO₄⁻ from ClO₃⁻ by irradiation and the possible production of SiF₄Cl₂ in the solid state.

4.3.b Nuclear Magnetic Resonance Spectrometry

J. R. Lund

Nuclear magnetic resonance spectrometry is a convenient nondestructive way to elucidate chemical structure and configuration — especially of organic compounds, to determine quantitatively components of some mixtures, and to estimate the ratios of protons in liquid or soluble organic compounds that absorb in different regions of the spectrum. For recording spectra, the Varian A-60 and DP-60 spectrometers are available to the Analytical Chemistry Division, which also offers the service of spectra interpretation. The A-60 operates in the range +1000 to -2000 cps from an arbitrary zero where most organic resonances occur. It has an integrator circuit for counting protons and a homonuclear spin decoupler to help simplify complex spectra. The DP-60 is usable over a considerably wider range and has provision for observing fluorine resonances.

Over 160 spectra were recorded of a variety of compounds that resulted from studies in progress in many groups. Two groups of the Reactor Chemistry Division used gas chromatography to isolate products from organic compounds that had been exposed as mixtures to radiation. As part of the Organic Synthesis with Isotopic Sources Program, the products obtained from irradiation of a mixture of tetrahydrofuran and cyclohexene were studied. Although the spectra of all these fractions indicated incomplete separation of the reaction products, it was possible to determine the ratios of the types of protons (aliphatic, olefinic, aromatic) present in each fraction. From an analysis of the spectra, together with molecular-weight, elemental, and infrared analyses, it was possible to postulate likely configurations for the products. The Effects of Radiation on Materials Group isolated 15 fractions from an irradiated mixture of naphthalene and *n*-hexane; these fractions were subjected to an intensive spectroscopic study. Although only 1 to 5 mg of material was available, the spectrum was recorded in a 25- μ l sample holder, and the sample was recovered and passed on for use in obtaining the ultraviolet, infrared, and mass spectra of these products. Interpretation and correlation of these spectra revealed that ethyl-, *n*-propyl-, isopropyl-, *sec*-butyl-, *n*-hexyl-, and four different branched-chain hexylnaphthalenes had been isolated. These alkyl isomers were positioned alpha and/or beta on the naphthalene ring; in addition three partially saturated compounds, 1,1-, 1,2-, and 2,2-dihexyldihydronaphthalene were identified.

Spectra recorded for members of the Biology Division include those of the optical and position isomers of glutathione, a naturally occurring tripeptide that seems to arrest the effects of irradiation in mice. The synthesis of these isomers requires a 22-step procedure involving the "blocking" and "unblocking" of reactive groups (-NH₂, -SH, -COOH) in order to produce the desired compound. The spectrum of several glutathione products compared favorably with the spectra of the purest commercially available glutathione; however, spectra of the isoglutathione products showed that significant quantities of impurity remained in the preparations. In connection with the study of gramicidin A (a linear *N*-acylated pentadecapeptide ethanolamide), spectra were recorded of crude and refined gramicidin A, *m*-iodobenzoylgramicidin A, tryptophan, and desformylgramicidin A-HI salt.

¹⁹Chemistry Division.

Of particular interest in these studies was the fate of the terminal protons (alcoholic or formyl) in the gramicidin A molecule on substitution of the other groups.

The products from the steps of the synthesis of didodecyl-naphthalenesulfonic acid by members of the Chemical Technology Division were examined. The spectra were used to confirm that 2-lauroyl-naphthalene, 2-dodecyl-naphthalene, 2-dodecyl-6-lauroyl-naphthalene, and 2,6-didodecyl-naphthalene had been prepared. The orientations of the substituents in the final product, 2,6-didodecyl-naphthalene-1-sulfonic acid, were deduced from the spectra.

The spectra of numerous materials were recorded for members of various groups for the purposes of identification or comparison. Among these were phenolic resins, tall oil, a silicon pump oil, dioctyl phthalate, glycerolthioether, and a variety of saturated aliphatic hydrocarbons.

4.4 NEW AND MODIFIED CHEMICAL METHODS

4.4.a Precise Determination of Oxygen in Metals and Mixed Metal Oxides

Gerald Goldberg

A rapid direct method was needed to determine oxygen at various concentrations in metals and in mixed metal oxides. From experience the inert-gas-fusion separation method was selected in combination with a gas chromatographic estimation of the evolved carbon monoxide for increased precision and accuracy. In the inert-gas-fusion method, the sample is normally dissolved in a molten-metal bath contained in an induction-heated graphite crucible. The oxygen of the oxide, in contact with the carbon, is reduced to carbon monoxide. The carbon monoxide is then transferred with an inert carrier gas to some suitable analytical apparatus. The selection was further prompted by the fact that the apparatus can be adapted to the handling of alpha-emitting samples within a glove box as is done at the Savannah River Laboratory.²⁰

²⁰P. E. Doherty, *Precise Determination of Oxygen in Refractive Oxides by Inert Gas Fusion—Gas Chromatography*, DP-1115 (July 1967).

For our work a Leco induction furnace was used in conjunction with a Leco Nitrox-6 Analyzer (Laboratory Equipment Corporation, St. Joseph, Michigan). With the Nitrox-6 Analyzer the carbon monoxide and nitrogen are stored in a cold trap until evolution is complete. The gases are then separated chromatographically and are passed through a thermal-conductivity detector. The detector output is fed to an integrator, which translates the volume of gas into total counts. These counts are plotted on a calibration curve. An analysis for both oxygen and nitrogen can be completed in about 15 min. Samples that contain 5 μ g to 5 mg of oxygen and less nitrogen can be analyzed by use of the attenuator on the Nitrox unit. Calibration curves are plotted for each attenuation.

For alpha-emitting samples, the furnace section was removed from the rf generator and was mounted in a metal glove box of special design. The box is partitioned to protect an analytical balance from the heat radiated by the fusion system. A door in the partition facilitates the transfer of samples between compartments. The leads from the work coil to the power supply pass through bulkhead fittings located in a Teflon window at the back of the glove box. The cooling-water- and gas-transfer lines also connect to the furnace through this window. A sensing device was installed in the box that will automatically cut off the water and power supplies if a rupture develops in the water line within the box.

Series of blanks and oxygen standards were analyzed prior to installation of the furnace in the glove box. Leco tin capsules and standards were used. The crucibles were baked at 2450°C. The samples were analyzed at 2000°C, at which temperature the blanks were most reproducible. Some 70 blanks and standards were run by use of the same crucible with no difficulty. Samples of NBS U_3O_8 and ThO_2 were also analyzed for oxygen with an error no greater than $\pm 1\%$. The accuracy remained the same for the standards after the furnace was installed in the glove box.

An attempt to use acetanilide as a standard for nitrogen was unsuccessful. However, an average deviation of $< 1\%$ between integrator counts was found from the ignition of samples of uranium nitride. The accuracy of these nitrogen results will be established after standard curves are developed with Leco nitrogen standards.

4.4.b Analysis of Tungsten-Rhenium Thermocouple Wire

P. F. Thomason

The W:Re ratio in a W-Re alloy determines the characteristics of its use as a thermocouple material. Dissolution of this alloy by Na_2O_2 fusion in nickel crucibles is not feasible, because the crucible also dissolves and thus contaminates the melt. It was found that boiling 30% H_2O_2 attacks the W-Re wire. On evaporation of the mixture to dryness in a Pyrex beaker after several additions of 30% H_2O_2 , a residue of tungsten oxides and rhenium oxides remains. The residue is very soluble in ~20% KOH solution. The resulting solution can be diluted to a given volume with 2 M KOH. Aliquots of this solution can be diluted with concentrated H_3PO_4 and then diluted further to a known volume with distilled water to give a solution that contains tungsten in proper concentration for polarographic determination.

The most suitable supporting medium for the polarographic determination of tungsten in W-Re thermocouple wire appears to be 42 vol % H_3PO_4 (~4.5 M) as proposed by Meites.²¹ The $E_{1/2}$ of the W(VI) → W(V) reduction in this medium is -0.59 v vs S.C.E. The i_d vs C curve is linear for W concentrations from 18 to 180 $\mu\text{g}/\text{ml}$ ($i_d = 0.08$ to $0.80 \mu\text{a}$ for $t = 5$ sec). The W- H_3PO_4 solution is stable for at least 4 hr.

Rhenium was determined spectrophotometrically by the α -furildioxime method.

4.4.c Spectrophotometric Determination of Plutonium

J. C. Guyon²² W. D. Shults

The reaction between arsenazo (2-*o*-arsenophenylazo-1,8-dihydroxy-3,6-naphthalenedisulfonic acid) and plutonium has been used to determine Pu(IV) in weakly acid media. Waterbury²³

²¹L. Meites, ed., *Handbook of Analytical Chemistry*, pp. 5-87, McGraw-Hill, New York, 1963.

²²ORAU Research Participant; Associate Professor of Chemistry, University of Missouri, Columbia.

²³G. R. Waterbury and J. Dahlby, unpublished data cited in C. F. Metz and G. R. Waterbury, "The Transuranium Actinide Elements," p. 309 in vol. 9 of *Treatise on Analytical Chemistry, Part II*, ed. by I. M. Kolthoff and P. J. Elving, Interscience, New York, 1962.

has mentioned that Pu(III) and Pu(VI) also react with the reagent in less acid media. An investigation was undertaken to adapt the system to the determination of total plutonium and to consider the possibility of developing a method for the determination of the concentrations of the plutonium ions in their individual oxidation states in the presence of one another.

The arsenazo-Pu(IV) reaction was shown to be capable of measuring total plutonium as Pu(IV) in the range 0.5 to 10 ppm. The system is sensitive ($\epsilon_{575} = 25,900$) and fairly selective. Conditions somewhat different from those of Ockenden²⁴ were found to be optimal. The 1:1 complex forms rapidly in acid solution and is stable for several hours.

Preliminary studies of the complexes of Pu(III) and Pu(VI) with arsenazo indicate that procedures for oxidation-state determinations may be feasible; however, further work will be required.

The results of these investigations are being reported in a technical memorandum.²⁵

4.4.d Fluorometric Determination of Phosphate

J. C. Guyon²² W. D. Shults

The approach used in this research involved the premise that, if a metal ion forms a fluorescent chelate and also forms a complex ion with phosphate, the phosphate should inhibit the formation of the fluorescent species. The intensity of the fluorescence should decrease with increasing phosphate concentration, thus making the determination of phosphate possible. The aluminum-morin²⁶ system was shown to be satisfactory for the determination of 20 ppb to 1 ppm phosphate and the tin-flavonol²⁷ system for measuring 40 to 200 ppm phosphate. For each system the effects of time, interferences, and concentrations

²⁴D. W. Ockenden, *Complexes of Plutonium(IV) with Phenyl-Arsenic Acid Derivatives*, IGO-RW-2 (Feb. 1, 1956).

²⁵J. C. Guyon and W. D. Shults, *Spectrophotometric Determination of Total Plutonium: Observations on the Reactions Between Arsenazo and Pu(III), Pu(IV), and Pu(VI)*, ORNL-TM report in preparation.

²⁶H. H. Willard and C. A. Horton, "Fluorometric Determinations of Traces of Fluoride," *Anal. Chem.* **24**, 862 (1952).

²⁷C. F. Coyle and C. E. White, "Fluorometric Determination of Tin with Flavonol," *Anal. Chem.* **29**, 1486 (1957).

of various reagents were studied, and a recommended analytical procedure was prepared. The methods have been used to determine trace quantities of phosphate in water. The results of this work were presented orally;²⁸ a paper is being prepared for open-literature publication.

4.4.e Sealed-Tube Dissolutions

D. C. Canada

A sealed-tube technique²⁹ has proved useful for dissolving certain substances that are difficultly soluble or are being analyzed for trace impurities or for a constituent that is evolved on dissolution. By this technique, nitrogen in UN_x was determined accurately. Also, high-purity crystalline MgO was analyzed for trace impurities. These materials were dissolved at 130°C without the use of an overpressure on the sealed tubes.

4.4.f Separation and Determination of Fluoride

D. A. Costanzo

The pyrohydrolysis method for the separation of microgram amounts of fluoride from TRU dissolver solutions was evaluated.^{30,31} In this method a test portion of the sample is taken to dryness in a combustion boat (Pt, Ni, or porcelain). To prevent loss of fluoride during the evaporation, the sample is neutralized with base and the fluoride is complexed with aluminum nitrate. The flux (Na_2WO_4 - WO_3 or U_3O_8) is then added, and the sample is pyrolyzed at 1000°C for 30 min in a combustion tube (quartz, nickel, or platinum). The volatile fluoride is collected in a dilute solution of NaOH.

²⁸J. C. Guyon and W. D. Shults, "The Fluorometric Determination of the Phosphate Ion," presented at the Eleventh Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Oct. 10-12, 1967.

²⁹C. F. Metz and G. R. Waterbury, *Sealed-Tube Dissolution Method with Applications to Plutonium-Containing Materials*, LA-3554 (Nov. 30, 1966).

³⁰D. A. Costanzo, "Separation and Spectrophotometric Determination of Fluoride," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1966*, ORNL-4039, p. 36.

³¹J. H. Edgerton, "Fluoride in Mixtures of Fluoride Salts, Micro Pyrohydrolysis Method," Method Nos. 1 212890 and 9 00712890 (9-20-56), *ORNL Master Analytical Manual*; TID-7015, sects. 1 and 9.

The isolated fluoride is then determined spectrophotometrically by the Zr-SPADNS bleaching method.³² Satisfactory separation of fluoride (0 to 30 μ g) was obtained by use of a quartz- or platinum-lined nickel combustion tube. Very poor recovery of fluoride was obtained with the nickel combustion tube and nickel boats because of the reaction of fluoride with nickel and the retention of fluoride on the nickel surfaces.

For use in remotely controlled operations a platinum-lined nickel tube, platinum combustion boats, and pyrohydrolyzer apparatus were designed and fabricated. As a flux, U_3O_8 was selected rather than the Na_2WO_4 - WO_3 mixture because of the ease with which the spent flux can be handled and removed from the combustion boats.

4.4.g Determination of Periodate, Iodate, and Iodide in Mixtures of the Three Species

D. C. Canada

In connection with the Fluidized-Bed Volatility Process periodate, iodate, and iodide were determined in mixtures of the three species. In the process the passage of fluorine gas through a KOH-KI solution oxidizes the I^- to IO_3^- and IO_4^- . The IO_4^- is titrated with arsenious acid at pH \sim 8 in bicarbonate buffer solution; IO_3^- plus IO_4^- are titrated in acid solution with a standard solution of sodium thiosulfate. Total iodine is found by reducing IO_3^- and IO_4^- to I^- with solid sodium arsenite and titrating the resultant solution with a standard solution of silver nitrate.

4.4.h Spectrophotometric Determination of Molybdenum and Tungsten

P. F. Thomason

A spectrophotometric method for the determination of molybdenum was required in the analytical service work for the Transuranium Processing

³²H. W. Wharton, "Isolation and Determination of Microgram Amounts of Fluoride in Materials Containing Calcium and Orthophosphate," *Anal. Chem.* **34**, 1296 (1962).

Plant. The method of Nelson and Waterbury,³³ in which dithiol is the chromogenic reagent for both molybdenum and tungsten, was evaluated.

The method is based on the fact that the Mo(VI)-dithiol complex is extractable into pentyl acetate from 5 to 6 M HCl, whereas the W(V)-dithiol complex is extractable from 9 to 11 M HCl. The absorbance of the molybdenum complex in pentyl acetate is measured at 685 nm, the Beer's law relationship is obeyed, and the molar absorptivity (ϵ) is 24,000. The absorbance of the tungsten complex in pentyl acetate is measured at 640 nm, Beer's law is obeyed, and ϵ is 23,200. A résumé of the method and its evaluation was forwarded to J. H. Cooper³⁴ for possible use in the analysis of TRU process solutions.

4.5 NEW AND MODIFIED PHYSICAL METHODS

4.5.a Density Gradient Applications

D. C. Canada

The density-gradient technique³⁵ has been used in several applications, one of which is the study of the effect of radiation on the density of pyrolytic carbon particles. Minute differences in density (± 0.0002 g/cc) that are effects of radiation can be measured by comparing two particles before and after irradiation.

4.6 OTHER ACTIVITIES

4.6.a Autodecomposition of Bromate in Acid Nitrate Media

D. A. Costanzo

For the Chemical Technology Division, studies were undertaken of the autodecomposition of bromate in solutions under conditions representative of those to be encountered in the proposed BERLEX

³³G. B. Nelson and G. R. Waterbury, "Spectrophotometric Determination of Microgram Quantities of Molybdenum and Tungsten in Plutonium Using Dithiol," *Analytical Chemistry in Nuclear Reactor Technology, Fifth Conference, Gatlinburg, Tenn., Oct. 10-12, 1961*, TID-7629, pp. 62-68.

³⁴High-Level Alpha Radiation Laboratory.

³⁵D. C. Canada and W. R. Laing, "Use of a Density Gradient Column to Measure the Density of Microspheres," *Anal. Chem.* 39, 691 (1967).

phase of transuranium element processing. Berkelium(III) is oxidized to Bk(IV) with bromate in 6 to 8 M KNO₃. The Bk(IV) is separated from the trivalent actinide elements by selective extraction into a decane solution of di-2-ethylhexylphosphoric acid. If the extraction is not done within a few hours after the bromate reagent is prepared, autodecomposition of bromate occurs, and the berkelium is not separated.

The effect of temperature on the rate of decomposition of bromate in the reagent 0.2 M KBrO₃-2 M LiNO₃-8 M HNO₃ was studied; the results follow:

Temperature (°C)	Half-Time (hr) ^a
25	10
35	3
50	0.6

^aThe time at which the concentration of bromate is 50% of the initial concentration.

Also, the effect of nitric acid concentration on the rate of decomposition of bromate in the reagent 0.2 M KBrO₃-2 M LiNO₃ was determined; the results are:

HNO ₃ Concentration (M)	Half-Time (hr)
4	100
5	26.5
6	10.5
8	3

Two experiments were carried out to determine the effect of elemental bromine on the rate of decomposition of bromate and to follow the concentration of hydrogen ion. For the reagent 0.2 M KBrO₃-8 M HNO₃-0.035 M Br₂, 50% of the bromate has been consumed after 17.5 hr compared with 27.5 hr for the reagent 0.2 M KBrO₃-8 M HNO₃ (initial concentration of Br₂ equal to zero). In the latter experiments the concentration of hydrogen ions was observed to remain constant throughout the course of the decomposition reaction.

The data indicate the following: (1) For the reagent 0.2 M KBrO₃-2 M LiNO₃-8 M HNO₃, the activation energy for the decomposition of bromate is 15.6 kcal/mole; (2) for each 10°C rise in temperature, the rate of decomposition of bromate increases by a factor of 3.3; (3) for the reagent

0.2 M KBrO_3 –2 M LiNO_3 , the decomposition half-time for bromate is inversely dependent on the 4.3 power of the nitric acid concentration; and (4) the autodecomposition reaction is catalyzed by hydrogen ion and elemental bromine.

It is thought that the decomposition of bromate in strong nitric acid solution is initiated by the formation of oxides of nitrogen, which are produced by thermal or photochemical decomposition of nitric acid.

A method was developed to determine bromate in solutions that contain elemental bromine. In this method, the elemental bromine is destroyed by reaction with ammonium bicarbonate. The bromate is then determined by iodometric titration.

4.6.b Glove Box for Absorption Spectrophotometry of Alpha-Emitting Materials

D. A. Costanzo

A glove box and absorption-cell compartment were designed³⁶ for use with the Cary model 14 spectrophotometer; they will permit measurements on highly radioactive alpha-emitting solutions. The unique feature of the design is that the absorption cell which contains the radioactive sample solution is positioned in a metal compartment provided with quartz windows and is attached to the glove box by a polyurethane bellows (Fig. 4.2). Complete containment is maintained within the cell compartment and glove box.



Fig. 4.2. Glove Box for Absorption Spectrophotometry of Alpha-Emitting Materials.

The cell compartment is lowered and positioned within the cell compartment of the spectrophotometer.

4.6.c Salt Effect in the Measurement of Optical Density of Transfer Ribonucleic Acids

D. C. Canada

Purification of certain transfer ribonucleic acids (tRNA's) requires fractionation on ion exchange resin columns. The concentration of tRNA in the effluent is determined from the optical density of the solution at 260 nm. Variation in the salt concentration of the solutions appeared to cause corresponding variation in optical density. This possibility was studied with phenylalanine-accepting tRNA.

A solution of relatively pure phenylalanine-accepting tRNA of 11.30 ODU³⁷ was passed through P-II Bio-Gel to remove salts. The effluent solution was used to make standard additions to process solutions that contained salts in the expected concentrations. The results indicate that, for the solutions studied, optical density measurements are suitable for monitoring the fractionation process.

4.6.d Preparation of Chapters for Progress in Nuclear Energy

Two chapters are being contributed for inclusion in Vol. 10, *Remote Analysis of Radioactive Materials*, of SERIES IX, ANALYTICAL CHEMISTRY, in *Progress in Nuclear Energy*.

Application of Electrochemical Techniques to Remote Analysis (W. D. Shults). – A chapter titled "The Application of Electrochemical Techniques to Remote Analysis" was prepared for inclusion in Volume 10.³⁸

³⁶ORNL Drawings Nos. EL-001-D, EL-002-D, and EL-003-D.

³⁷One optical density unit (ODU) is the total absorbance of 1.0 ml of solution of optical density 1.00 measured at 260 nm in a cell of 1-cm light path with distilled water as the solvent; $\text{ODU} = \text{OD} \times \text{volume}$.

³⁸W. D. Shults, "The Application of Electrochemical Techniques to Remote Analysis," to be included in vol. 10, *Remote Analysis of Radioactive Materials*, of SERIES IX, ANALYTICAL CHEMISTRY, ed. by H. A. Elion and D. C. Stewart, in *Progress in Nuclear Energy*, Pergamon Press, New York.

Remote-Controlled Titrimetry (P. F. Thomason).
 – A chapter titled "Remote-Controlled Titrimetry"
 is being prepared for inclusion in Volume 10.³⁹

³⁹P. F. Thomason, "Remote-Controlled Titrimetry,"
 to be included in vol. 10, *Remote Analysis of Radio-
 active Materials*, of SERIES IX, ANALYTICAL CHEM-
 ISTRY, ed. by H. A. Elion and D. C. Stewart, in *Prog-
 ress in Nuclear Energy*, Pergamon Press, New York.

5. Analytical Biochemistry

Gerald Goldstein

5.1 MACROMOLECULAR SEPARATIONS PROGRAM

5.1.a Determination of Leucine- and Phenyl- alanine-Accepting Transfer Ribonucleic Acids (tRNA^{leu} and tRNA^{phe})

I. B. Rubin A. D. Kelmers¹
 Gerald Goldstein

The optimum conditions for the quantitative de-
 terminations of picomole amounts of leucine- and
 phenylalanine-accepting tRNA's were established.
 The effects of various factors on these determina-
 tions are discussed in a published paper.²

5.1.b Determination of Valine-Accepting Transfer Ribonucleic Acid (tRNA^{val})

D. W. Hatcher

Partially purified valine-accepting transfer ribo-
 nucleic acid was recently made available for study,
 and the optimum conditions for its determination
 were established. The general techniques used in
 this type study and the results of a preliminary
 study on this particular tRNA have been described
 by Rubin.^{3,4}

¹Chemical Technology Division.

²I. B. Rubin, A. D. Kelmers, and G. Goldstein, "The
 Determination of Transfer Ribonucleic Acid by Amino-
 acylation. I. Leucine and Phenylalanine Transfer
 Ribonucleic Acid from *E. coli* B," *Anal. Biochem.* **20**,
 533 (1967).

³I. B. Rubin, "Studies on the Phenylalanyl Transfer
 Ribonucleic Acid (tRNA) System," *Anal. Chem. Div.*
Ann. Progr. Rept. Nov. 15, 1965, ORNL-3889, p. 33.

⁴I. B. Rubin, "Determination of Valyl- and Alanyl-
 Transfer Ribonucleic Acids (tRNA)," *Anal. Chem. Div.*
Ann. Progr. Rept. Oct. 31, 1966, ORNL-4039, p. 38.

The following conditions were found to be
 necessary for charging of tRNA with valine:

pH	7.5 ± 0.5
Buffer	0.1 M Bicine (<i>N,N</i> -bis[2-hydroxyethyl]glycine)
ATP	0.0005 ± 0.0002 M
K ⁺	0.002 to 0.02 M
Mg ²⁺	> 0.0025 M (0.25 M has no adverse effect)
ATP:K ⁺ :Mg ²⁺	1:4:10 mole ratio (for maximum charging)
Na ⁺	< 0.02 M
Valine- ¹⁴ C	7.5 × 10 ⁻⁹ mole per ODU ⁵ of tRNA

The charging is complete within 10 min at 37°C,
 5 × 10⁻¹⁰ mole of valine being incorporated per
 ODU of partially purified valine-tRNA.⁵ The order
 of addition of reagents has no effect on charging.

Under these conditions and a 20-min incubation
 time, a threefold increase in charging is realized
 over that obtained under the conditions recommended
 in the preliminary study.⁴

5.1.c Preparation of Aminoacyl-Transfer Ribonucleic Acid (tRNA) Synthetases from *E. coli* B

I. B. Rubin

Several procedures for the preparation of solu-
 tions of mixed aminoacyl-tRNA synthetases were

⁵One optical density unit (ODU) is the total absorb-
 ance of 1.0 ml of solution of optical density 1.00 meas-
 ured at 260 nm in a cell of 1-cm light path with dis-
 tilled water as the solvent; ODU = OD × volume.

Table 5.1. Comparison of Specific Activities of Aminoacyl-tRNA Synthetase Enzyme

Amino Acid	Specific Activity (units ^a per milligram of protein)		
	Peak I	Peak II	Enz L-11 ^b
Arg	0.3	7.5	0.7
His	<0.01	0.4	0.05
Ile	3.5	0.2	0.2
Leu	0.01	3.2	0.3
Lys	<0.01	0.8	0.2
Met	2.1	<0.01	<0.01
Phe	2.0	0.1	1.6
Ser	<0.01	1.2	0.3
Tyr	2.1	0.2	0.2
Val	0.1	3.8	0.4

^aOne unit = 1 nanomole of tRNA charged per minute at 37°C.

^bEnzyme used by General Analyses Laboratory.

studied quantitatively to establish a standard method of processing *E. coli* B cells to obtain these enzymes. The enzymes are critical factors in the determination of specific tRNA's.⁶ A combination of the procedures described by Kelmers, Novelli, and Stulberg⁷ and by Muench and Berg,⁸ after modification, gave the most consistent product. The resulting procedure is, in brief, as follows. The lysate from fresh *E. coli* B cells is centrifuged, and the supernatant liquid is diluted to a protein concentration of 10 mg/ml. The nucleic acids are then separated by precipitation with streptomycin sulfate. The supernatant liquid from the precipitation is dialyzed, and the protein is precipitated from it with ammonium sulfate (75% of saturation). The precipitate is dissolved and is chromatographed on a DEAE-cellulose column. A stepwise elution with a phosphate buffer yields two distinct protein fractions. These final products contain less than 1% of the original nucleic acids;

⁶J. C. White, C. A. Horton, and I. B. Rubin, "Analytical Biochemistry," *Anal. Chem. Div. Ann. Progr. Rept.* Nov. 15, 1964, ORNL-3750, p. 29.

⁷A. D. Kelmers, G. D. Novelli, and M. P. Stulberg, "Separation of Transfer Ribonucleic Acids by Reverse Phase Chromatography," *J. Biol. Chem.* 240, 3979 (1965).

⁸K. H. Muench and P. Berg, "Preparation of Aminoacyl Ribonucleic Acid Synthetases from *Escherichia coli*," in *Procedures in Nucleic Acid Research*, ed. by G. L. Cantoni and D. R. Davies, pp. 375-83, Harper and Row, New York, 1966.

however, apparently some ribonuclease does accompany the synthetases. Synthetase activities are divided between the two protein fractions. Table 5.1 shows the distribution of the enzymes between the fractions corresponding to the two elution peaks and a comparison of their specific activities with that of an enzyme preparation currently in routine use. Specific activities for the other amino acids are too low for their quantitative measurement.

5.1.d Miscellaneous Studies on the Analysis of Transfer Ribonucleic Acid (tRNA)

I. B. Rubin

Studies to evaluate the various steps of the procedure for the assay of tRNA have continued.⁶ Pretreatment of the paper disks by soaking them in trichloroacetic acid (TCA) led to apparently higher count rates, but this increase was found to be due to precipitation of the aminoacylated tRNA on the surface of the paper disk instead of distribution within the disk. Pretreatment of the paper disks with the respective "cold" amino acid had no effect. Drying time of the paper disk, once the aliquot had dried for 30 sec, had no effect on the results whether the disk was dried for as long as 30 min with a hot-air blower or 90 min at ambient temperature. Reduction of the volume of the assay solution from 0.5 to 0.25 ml had no deleterious effects and allowed a 50% saving of ¹⁴C-labeled amino acids. Exposure of the scintillation vials and fluid to room fluorescent light for 5 hr did not change the average background count rate or the precision of this count rate, whereas repeated usage of the vials and scintillator, up to five times, increased the background count rate by only several counts per minute and did not change the precision of this count rate. A number of washing procedures, which varied from the eight-step procedure recommended by Bollum⁹ to a three-step procedure, were tested and compared with the usual washing procedure; they did not change the results. Methanol or isopropanol can be substituted for ethanol, and 3 vol % hydrochloric or

⁹F. J. Bollum, "Filter Paper Disk Techniques for Assaying Radioactive Macromolecules," in *Procedures in Nucleic Acid Research*, ed. by G. L. Cantoni and D. R. Davies, pp. 296-300, Harper and Row, New York, 1966.

perchloric acid can be substituted for TCA. However, blank disks processed along with series of sample disks were found to have picked up an average of 25 counts/min above background. These tests on various aspects of the assay procedure for tRNA will continue.

5.1.e Automated Determination of Transfer Ribonucleic Acids (tRNA's)

Gerald Goldstein W. L. Maddox
I. B. Rubin

The filter-paper-disk procedure for the determination of tRNA² was automated successfully. Technicon AutoAnalyzer components were purchased that withdraw a sample, mix it with reagent and enzyme, and incubate the mixture at 37°C. The flowing stream then passes through a debubbler and a flow-cell colorimeter and back through the proportioning pump to a stream sampler. A diagram of the output mechanism is shown in Fig. 5.1. Details of the stream-sampling mechanism are given in Sect. 1.10. A dye is dissolved in the sampler wash water; the appearance of the dye at the colorimeter activates the output sampler, which delivers a fixed volume of solution to the filter-paper disk.

Correlation of the concentration of ¹⁴C-labeled aminoacylated tRNA in the stream with the transmittance reading from the flow cell (Fig. 5.2) shows that, in the last 20-sec period before the appearance of the dye, the amount of aminoacylated tRNA delivered to the disk is reproducible and that the count rate is almost the same as that of a similar volume taken in manual assays. In routine use, the automated method compares favorably both in precision and accuracy with the manual method (Table 5.2).

A paper that described this instrument was presented.¹⁰

¹⁰G. Goldstein, W. L. Maddox, and I. B. Rubin, "A Semi-Automated Filter Paper Disk Technique for the Determination of Transfer Ribonucleic Acid," presented at the Technicon Symposium, *Automation in Analytical Chemistry*, New York, Oct. 2-4, 1967.

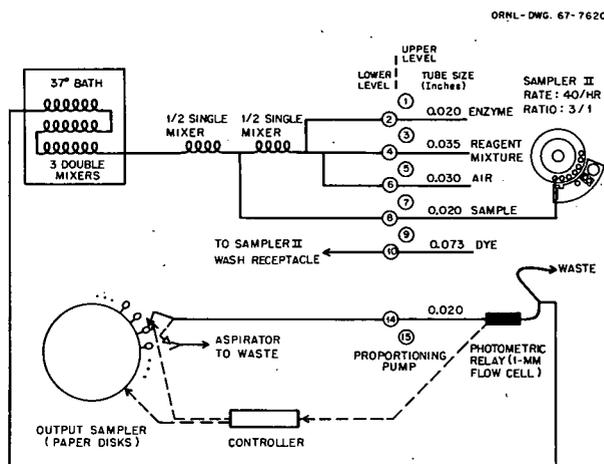


Fig. 5.1. Flow and Schematic Diagram of Output Mechanism for Automated tRNA Assays.

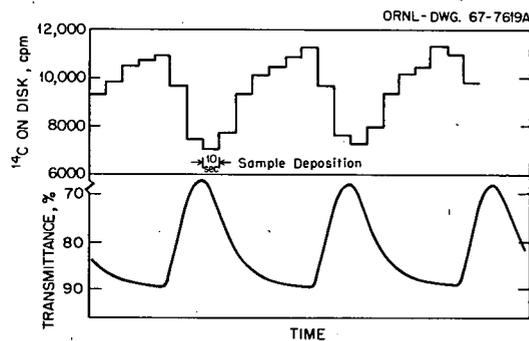


Fig. 5.2. Correlation of Photometer Reading with Concentration of Aminoacylated tRNA.

Table 5.2. Comparison of AutoAnalyzer with Manual-Assay Results

Amino Acid Charged	Number of AutoAnalyzer Replicates ^a	Results			
		Counts per Minute		Standard Deviation (%)	
		AutoAnalyzer	Manual	AutoAnalyzer	Manual
Phe	46	708	757	6	4
Tyr	64	547	481	5	4
Val	50	958	1138	6	5
Leu	46	1468	1633	5	4

^aNumber of replicates for manual assay = 3.

5.1.f Ligand-Exchange Chromatography of Nucleotides, Nucleosides, and Nucleic Acid Bases

Gerald Goldstein

Ligand-exchange chromatography with a copper-loaded chelating resin (Chelex 100) is a useful technique for the rapid separation of nucleic acid components. Nucleotides are not retained on the column and are eluted with water. Slightly basic nucleosides and bases are retained only weakly and can be resolved by elution with water; the more basic nucleosides can be separated by elution with 1 M NH₄OH. Nucleic acid bases are retained more strongly than nucleosides but can be eluted and separated with 2.5 M NH₄OH. A paper that describes this work has been published.¹¹

5.1.g Terminal Nucleoside Assay of Ribonucleic Acid by Ligand-Exchange Chromatography

C. A. Burtis Gerald Goldstein

The recent development at the Oak Ridge National Laboratory of methods for the separation of individual tRNA's by reversed-phase chromatography has necessitated a rapid micro method for the determination of the purity of tRNA. Alkaline hydrolysis of tRNA yields a mixture of 2'- and 3'-nucleotides of the internal bases and a nucleoside derived solely from the 5'-linked terminus. Enzymatic dephosphorylation prior to alkaline hydrolysis allows the assay of nucleosides having primary phos-

phate residues in the 2'- or 3'-position of the terminal nucleoside.

To quantitatively separate the nucleosides from the nucleotides in an alkaline hydrolysate of tRNA, ligand-exchange chromatography was employed (Sect. 5.1.f). An aliquot of the neutralized tRNA hydrolysate was eluted from a copper-loaded chelating column (0.9 by 50 cm) with 1 M NH₄OH. The column effluent was monitored at 260 nm. A log-to-linear converter, which permits absorbance to be recorded linearly, and a scale expander were added¹² so that at maximum sensitivity a full-scale deflection on the recorder corresponded to 0.10 ODU.⁵ The nucleoside-peak area was integrated, and nucleoside concentration was calculated from calibration factors derived from the analysis of standard solutions of the nucleoside. The resultant end-group values were then compared with the biological assay (aminoacylation) as a measure of purity (Table 5.3). To demonstrate the feasibility of assaying for nucleosides other than adenosine, homopolymers of RNA were assayed for terminal nucleosides after prior enzymatic removal of terminal phosphate (Table 5.4).

The versatility of the method was further demonstrated by end-group assay of commercial yeast tRNA, which is known to have terminal nucleosides other than adenosine (Table 5.5).

¹¹G. Goldstein, "Ligand-Exchange Chromatography of Nucleotides, Nucleosides, and Nucleic Acid Bases," *Anal. Biochem.* **20**, 477 (1967).

¹²J. T. Hutton, "Multipoint Recorder Programmable Range Converter," *Instrumentation and Controls Div. Ann. Progr. Rept. Sept. 1, 1966*, ORNL-4091, p. 110.

Table 5.3. Purity Estimation of tRNA

tRNA Source	Adenosine	Aminoacylation		Purity ^a (%)
	End Group (picomoles/ODU ⁵)	(picomoles/ODU ⁵)		
Partially purified methionine	990 (±2%)	Methionine	820 (±5%)	83
		All others	140 (±5%)	
Partially purified phenylalanine	1330 (±2%)	Phenylalanine	880 (±5%)	66
		All others	150 (±5%)	
Purified leucine	1270 (±2%)	Leucine	1200 (±5%)	95
		All others	60 (±5%)	
Purified phenylalanine	1350 (±3%)	Phenylalanine	1340 (±5%)	99
		All others	<10	

^aCalculated by dividing aminoacylation by terminal adenosine.

Table 5.4. End-Group Assay of Homopolymers of Ribonucleic Acid

Homopolymer	Number of Determinations	Amount with Indicated Type of 5'-Linked Terminus (picomoles/ODU)	
		---p ^N _p	---p ^N
Polyadenylic acid	4	780 (±4%)	0
Polycytidylic acid	4	1670 (±5%)	0
Polyguanylic acid	3	850 (±5%)	0
Polyuridylic acid	3	790 (±5%)	0

Table 5.5. End-Group Assay of Yeast tRNA

Nucleoside	End Groups ^a (picomoles/ODU)	
	---p ^N _p	---p ^N
Adenosine	580 (±4%)	170 (±5%)
Cytidine	270 (±4%)	930 (±5%)
Total end groups	1950 (±4%)	

^aIn each case three determinations were made.

Routine adenosine assays can be done in 60 to 70 min on a 50- by 0.9-cm column; cytidine, guanosine, and uridine assays in about 120 min on a 100- by 0.9-cm column. The method is sensitive, since the terminal nucleoside content of a tRNA sample of 10 ODU (equivalent to 3 to 4 μg of adenosine) may be assayed with a relative standard error of ±3 to ±6%.

A paper on this work will be presented orally,¹³ and a manuscript was submitted for publication.¹⁴

5.1.h Base-Ratio Determination of RNA by Ligand-Exchange Chromatography

C. A. Burtis

Earlier methods for base-ratio determination of nucleic acids have involved alkaline hydrolysis

¹³C. A. Burtis and G. Goldstein, "Assay of 3'-Terminal Nucleosides of Transfer Ribonucleic Acids by Ligand-Exchange Chromatography," presented at Southeastern Regional Meeting, American Chemical Society, Atlanta, Ga., Nov. 1-3, 1967.

¹⁴C. A. Burtis and G. Goldstein, "Terminal Nucleoside Assay of Ribonucleic Acid by Ligand-Exchange Chromatography," submitted to *Analytical Biochemistry*.

followed by separation of the resultant nucleotides by chromatography or electrophoresis and then determination of nucleotide concentration by ultraviolet spectrophotometry. Disadvantages of these methods include possible degradation of the *N*-bases by the alkaline hydrolysis and the long time required for chromatographic separation. A method has been developed whereby RNA is hydrolyzed to 5'-nucleotides by venom phosphodiesterase and the 5'-nucleotides are subsequently dephosphorylated to nucleosides by alkaline phosphatase. The resulting nucleosides are separated and quantitatively determined by ligand-exchange chromatography as described for end-group assay (Sect. 5.1.g). Samples of RNA were analyzed in duplicate, and the results were compared with values derived from a widely used cation exchange chromatographic method¹⁵ for base composition (Table 5.6).

To further compare the two methods, homopolymers of RNA were analyzed by each method, and the results were compared with the results of phosphate analyses after enzymatic dephosphorylation (Table 5.7). The results of the phosphate analyses agree with the base compositions determined by ligand-exchange chromatography, but the ion exchange method gave high results for uridylic acid and low results for adenylic and cytidylic acids. The ligand-exchange method is advantageous in that the chromatographic time is relatively fast (1 to 2 hr) and the enzymatic hydrolysis reaction is relatively mild. In addition very little sample is required (0.05 to 1.0 mg), and ±2 to ±3% relative standard error may be expected.

¹⁵S. Katz and D. C. Comb, "A New Method for the Determination of the Base Composition of Ribonucleic Acid," *J. Biol. Chem.* 238, 3065 (1963).

Table 5.6. Base Composition of tRNA

tRNA Source	Separation Method	Mole Percent ^a			
		Adenosine	Cytidine	Guanosine	Uridine
Yeast	Ion exchange	16.7	21.5	32.7	29.1
	Ligand exchange	19.5	24.0	33.9	22.7
<i>E. coli</i> B	Ion exchange	17.0	26.2	32.9	23.9
	Ligand exchange	18.4	28.8	33.9	18.9
Partially purified tRNA ^{Phe}	Ion exchange	16.6	26.1	32.2	25.0
	Ligand exchange	20.4	27.4	33.9	18.5
Purified tRNA ^{Phe}	Ion exchange	16.3	26.3	32.2	25.3
	Ligand exchange	21.4	29.5	31.7	17.4

^aEach sample was analyzed in duplicate by each method.

Table 5.7. Base and Phosphate Composition of RNA Homopolymers

RNA Homopolymer	Composition (micromoles/ODU)		
	Base		Phosphate
	Ion Exchange	Ligand Exchange	
Polyadenylic acid, (AMP) _n	0.098	0.104	0.103
Polycytidylic acid, (CMP) _n	0.162	0.185	0.179
Polyuridylic acid, (UMP) _n	0.124	0.103	0.104

5.1.i Determination of Molecular Weights of RNA Homopolymers by Ligand-Exchange Chromatography

C. A. Burtis

For molecular-weight determination of RNA homopolymers the terminal nucleosides and the base composition of each homopolymer were determined by ligand-exchange chromatography. Homopolymers of RNA were purchased from Miles Laboratories, Elkhart, Indiana. Since the homopolymers terminate in 3'-phosphates, the phosphates were enzymatically removed before end-group assay. The number of nucleosides per homopolymer molecule was then calculated by dividing the quantity of nucleosides

per ODU by the quantity of terminal nucleosides per ODU. The molecular weight of the homopolymer can then be calculated by multiplying the number of nucleotides per chain by the molecular weight of the polymer's monomeric nucleotide. Table 5.8 gives the results.

The molecular weights determined for polyadenylic and polycytidylic acids agree well with those determined by spectrometric measurements. The observed error with polyuridylic acid was not unexpected, since end-group assay, when the terminal nucleoside is uridine, requires a rechromatography step, which would introduce an error into the procedure. It is hoped that the method will prove applicable to natural RNA.

Table 5.8. Results of Determination of Molecular Weight of RNA Homopolymers by Ligand-Exchange Chromatography

Polyribonucleic Acid	Terminal End Groups (picomoles/ODU)	Nucleoside Concentration (picomoles/ODU)	Nucleosides per Polymer Molecule	Molecular Weight
Polyadenylic acid, (AMP) _n				
Ligand exchange	7.8 × 10 ²	9.89 × 10 ⁴	127	48,500
Miles			120-150	48,000 ^a
Polycytidylic acid; (CMP) _n				
Ligand exchange	1.77 × 10 ³	2.02 × 10 ⁵	113	36,500
Miles			120-150	34,200 ^a
Polyuridylic acid, (UMP) _n				
Ligand exchange	7.5 × 10 ²	9.89 × 10 ⁴	131	42,500
Miles			120-150	35,400 ^a

^aMolecular weight determined from ultraviolet spectra and extinction coefficients supplied by Miles Laboratories, Elkhart, Ind.

5.2 BODY FLUIDS ANALYSIS PROGRAM

5.2.a Automatic High-Resolution Analysis of Urine for Ultraviolet-Absorbing Constituents

C. D. Scott¹ J. E. Attrill
N. G. Anderson¹⁶

A high-pressure high-resolution nucleotide analyzer was adapted for the separation of the ultraviolet-absorbing constituents of urine. More than 100 chromatographic peaks were resolved. Significant differences were detected among the chromatograms of the urine of normal, leukemic, and schizophrenic subjects. A report of this work has been published.¹⁷

5.2.b Fractionation of Urine by Gel-Permeation Chromatography

C. A. Burtis

A 2.5- by 100-cm jacketed Sephadex column with upward-flow adapters was packed with Sephadex G-10 and was equilibrated with water at 37°C. A

¹⁶Biology Division.

¹⁷C. D. Scott, J. E. Attrill, and N. G. Anderson, "Automatic, High-Resolution Analysis of Urine for Its Ultraviolet-Absorbing Constituents," *Proc. Soc. Exptl. Biol. Med.* 125, 181 (1967).

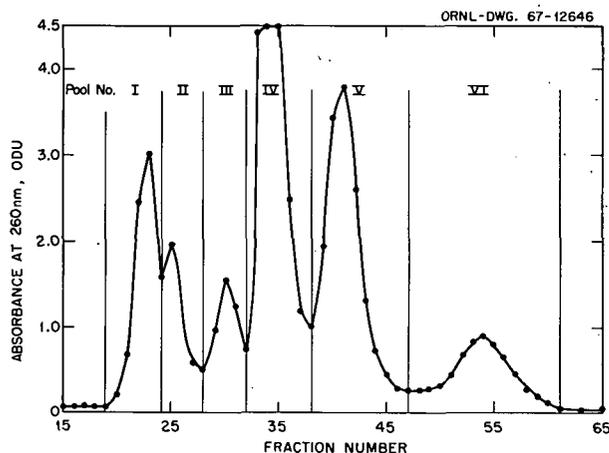


Fig. 5.3. Chromatogram That Shows the Separation of Urinary Components by Gel-Permeation Chromatography.

1-ml urine sample that had been sterilized by membrane filtration was placed on the column by way of a Chromatronix sample valve. The sample was then eluted with water (1.92 ml/min), and the column effluent was monitored at 260 nm by a spectrophotometer and recorder. Effluent fractions of 7.5 ml were collected for later assay. Six ultraviolet absorbancy peaks were observed (Fig. 5.3) and were analyzed for various components; Table 5.9 gives the results.

Table 5.9. Components Separated from Urine by Gel-Permeation Chromatography

Peak No.	Components Found
I	Ninhydrin-positive
II	Inorganic phosphate, Na ⁺ , K ⁺
III	Ninhydrin-positive, Na ⁺ , K ⁺
IV	Ninhydrin-positive, uric acid, pentoses, hexoses, chlorides, Na ⁺ , K ⁺
V	Ninhydrin-positive, urea
VI	Possibly <i>N</i> bases (low 280/260 ratio)

Tests for phenols and alcohols were negative because of the insensitivity of the detection methods. It was concluded that preparative gel chromatography may prove useful for prior fractionation of urine whereby the resultant pooled fractions may be further separated by ion exchange chromatography or other techniques.

5.2.c Identification of Chromatographically Separated Urinary Constituents

C. A. Burtis

The use of a modified Anderson automatic nucleotide analyzer for the analysis of human urine has been developed to a point where 125 peaks are resolved in 44 hr. Identification of the components corresponding to the 125 peaks has been difficult, since the peaks represent microgram quantities of components that are present in a relatively large quantity of acetate buffer. It is planned to collect fractions from urine runs and to separate the urinary constituents from the buffer solution by electrophoresis or by gel-permeation or thin-layer chromatography. The isolated compound will then be purified further and submitted for nuclear magnetic resonance and mass spectrometric analysis.

In addition, a monitoring system for a radioactive flowing system was procured, and several ¹⁴C-labeled urinary compounds were purchased. By spiking a urine run with a ¹⁴C compound and comparing the resultant radiochromatogram with the positions of the ultraviolet chromatographic peaks from the nucleotide analyzer, possibly compounds present in small amounts may also be identified.

5.3 MOLECULAR ANATOMY PROGRAM

5.3.a Electrophoresis of Proteins

A. L. Travaglini

Electrophoresis is the movement of ions in solution under the influence of an electric field. It is frequently used to separate proteins. The test solution is supported in a more or less inert material such as paper, cellulose acetate, starch gel, agar gel, or polyacrylamide gel. Polyacrylamide gel has the advantages of being inert, transparent, mechanically strong, and thermostable; also, it can be prepared to have any of a large range of pore sizes. Thus, proteins similar in charge but differing in size can be separated by a molecular-sieving effect. Disc electrophoresis as described by Ornstein¹⁸ has been used to determine the number of components in protein samples.

A current of 3 to 4 ma is used for each tube; a run requires 1 hr. After the run the gels are stained in Buffalo Blue Black dye to fix the proteins on the gel and to dye the protein bands. Excess dye is removed from the gel by electrophoresis. Figure 5.4 is a diagram of the apparatus currently being used for gel-disc electrophoresis and destaining; Fig. 5.5 shows stained protein bands in a gel slab and in a disc; each band is a different protein from hamster serum.

Future work will involve identifying some of the proteins.

5.3.b Preparative Gel Electrophoresis of Proteins

A. L. Travaglini

Gel slabs (6 × 9 × ³/₄ in.) are used to separate large quantities (200 to 300 mg) of protein. When a run is completed, an edge slice of gel is taken. The remainder of the slab is frozen to -60°C to minimize diffusion of the protein bands. The edge slice is stained, and the positions of the protein bands are determined. The main slab is then cut in sections, each containing one band. Each section is mashed in a small volume of buffer solution, and the protein diffuses into the buffer, from which it can be recovered.

¹⁸L. Ornstein, "Disc Electrophoresis - I. Background and Theory," *Ann. N.Y. Acad. Sci.* 121, 321 (1964).

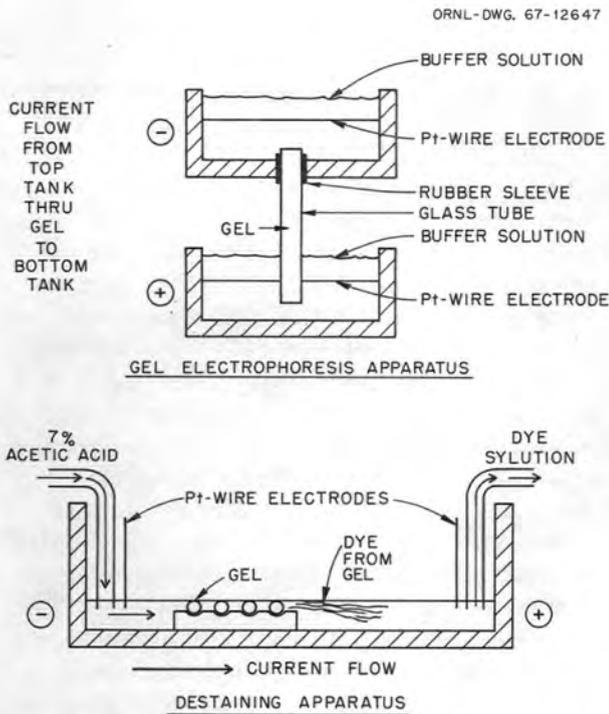


Fig. 5.4. Cross-Sectional Diagrams of Gel-Electrophoresis and Destaining Apparatuses.

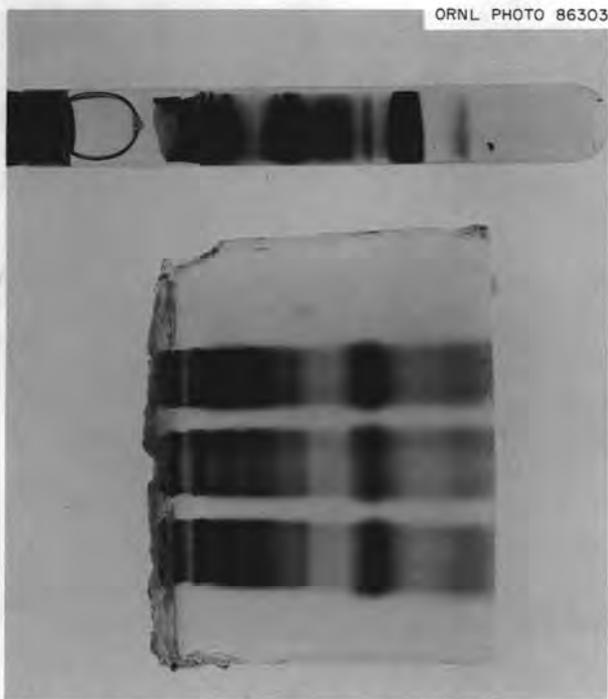


Fig. 5.5. Stained Protein Bands in a Gel Disc and in a Slab.

Equipment is being designed that will permit each band to be removed after it has migrated a fixed distance; the equipment will be built in the near future.

5.3.c Centrifhoresis

N. G. Anderson¹⁶ L. C. Patrick¹⁹
 A. L. Travaglini R. L. Jolley¹
 E. C. Candler, Jr.¹⁶

Centrifhoresis is a new electrophoretic method for separating ions by causing them to migrate in a centrifugally maintained sucrose density gradient. Loading of the sample and removal of the products are fast and easy, and the system can accept gram quantities of protein.

An A-XII zonal-centrifuge rotor was modified for use as a centrifhoresis rotor (Fig. 5.6) to have both an upper and a lower chamber. These two chambers are connected by a series of holes around the outer edge of the divider plates. Current flow is from carbon brushes to a copper ring on the outside surface of the upper plate; through to a platinum ring on the inside surface of the upper plate; through the sample, density gradient, and underlay; and out a platinum ring, copper ring, and carbon-brush arrangement on the lower plate.

For a run the rotor is filled with a 67% sucrose solution buffered to pH 8.5. A preformed 10 to 55 wt % sucrose density gradient is then introduced into the upper chamber and displaces an equal volume of underlay. The rotor is rotating at 1000 rpm all the while, and a very sharp interface between the underlay and the 55 wt % sucrose end of the density gradient can be seen. Next, the sample (20 to 40 ml), which may contain as much as 1 g of protein, and then a buffer solution are introduced. Then a volume of air is placed next to the core in both chambers to isolate the core and to prevent shorting of the current through the core. A current of 450 ma at 200 v is used; the run lasts about 3 hr. Three or four distinct bands can be seen to form and separate as the run proceeds.

Serum from tumor-bearing hamsters and soluble protein from hamster-tumor homogenate have been analyzed. Antigen from the soluble protein was found to concentrate ~90% in the albumin band.

¹⁹Technical Division, ORGDP.

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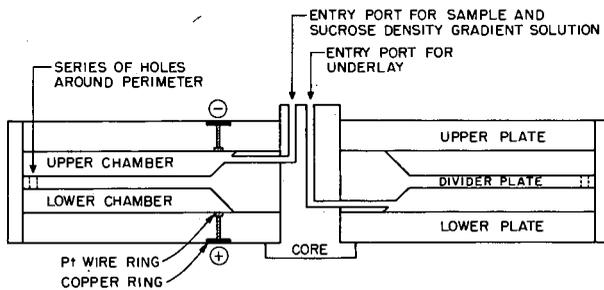


Fig. 5.6. Cross Section of an A-XII Zonal-Centrifuge Rotor Modified for Centrifhoresis.

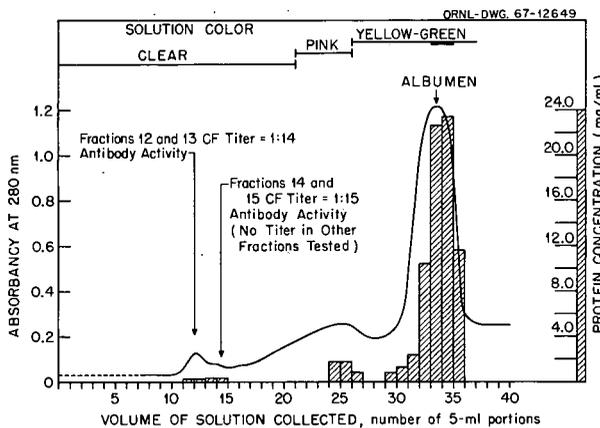


Fig. 5.7. Concentration of Antibody Activity by Centrifhoresis.

Figure 5.7 shows how antibody in serum is concentrated by centrifhoresis. The goal of the work is to isolate milligram quantities of antibody and antigen.

5.3.d Amino Acid Analyses

J. E. Attrill

In an attempt to determine the pattern of free and bound amino acids in mammalian tissues, analyses were made on various tissues from hamsters. Samples of brain, heart, lung, and tumorous material were analyzed for free and bound amino acids. The tumorous material came from tumors originally caused by the injection of an adeno 31 type virus beneath the skin of a hamster. This is a virus associated with humans infected with the common cold. Although thought to be harmless to man, when injected under the skin of a hamster

this virus causes a large tumor that continues to grow and eventually causes the death of the animal. Another group at the laboratory has reported the deficiency of the amino acid arginine in these tumorous tissues. However, an attempt to verify their results was unsuccessful, mainly because arginine existed in such small quantities in *all* the above tissues that no clear-cut difference was established.

Several samples of human urine were analyzed for amino acids. Most of these samples were from patients of the ORAU Medical Division. It was of interest to determine whether any major composition differences exist between the urine of normal persons and that of cancer or mental patients. Since many variables contribute to the chemical content of urine (e.g., water intake, diet, exercise), no immediate conclusions were drawn from these tests. However, the results were given to the National Institutes of Health and to the ORAU Medical Division for study and interpretation.

5.4 AMINO-ACID SEQUENCE ANALYSIS

J. E. Attrill R. A. Popp¹⁶

An important aspect in the analysis of amino acids has been the determination of the amino-acid sequence of the peptides released by enzymatic hydrolysis of the β chain of hemoglobin from the C57Bl type of laboratory control mouse. Hemoglobin is a protein composed of heme prosthetic groups united with polypeptide chains. These peptide chains appear to be homologous for all mammalian hemoglobins²⁰ and usually occur in two forms called α and β chains. The structure of these chains is genetically controlled and differs among mammals. Certain anemias have been definitely traced to the substitution of a single amino acid at various positions in one of these chains. It has thus become important not only to determine the sequence of the amino acids, but also to determine which substitutions may occur without interfering with the function of hemoglobin — its capacity to transport oxygen throughout the body.

²⁰M. F. Perutz, M. G. Rossmann, A. F. Cullis, H. Muirhead, G. Will, and A. C. T. North, "Structure of Haemoglobin — a Three-Dimensional Fourier Synthesis at 5.5-Å Resolution, Obtained by X-Ray Analysis," *Nature* 185, 416 (1960).

Work is now being done to complete the study on the β -chain sequence; the amino acid sequence in the α chain of the hemoglobin of this strain of mouse has already been determined.²¹ The red cells are separated from the whole blood by centrifugation and are lysed in water to obtain the free hemoglobin. The α and β chains of the hemoglobin are separated on an IRC-50 cation exchange column. The β -chain protein is collected and split into 15 smaller peptide fractions by enzymatic digestion with trypsin. These fractions are separated by means of both anion and cation exchange resins. Each individual fraction is then hydrolyzed, and the amino acid content is determined to obtain the integral ratio of amino acids. The larger peptides are also treated with pepsin, papain, or chymotrypsin to obtain smaller fragments. By means of the Edman degradation method,²²⁻²⁴ the *N*-terminal amino acid is split off and is identified. Knowledge of where these enzymatic splits occur and previous information from the α chain and other hemoglobin studies have led to a proposed sequence model shown in Table 5.10. Changes in this model are made as new information is obtained. Other methods of analysis such as starch-gel electrophoresis and paper chromatography are used in the identification, separation, and checks of the purity of these compounds.

²¹R. A. Popp, "The Separation and Amino Acid Composition of the Tryptic Peptides of the α Chain of Hemoglobin from C57Bl Mice," *J. Biol. Chem.* **240**, 2863 (1965).

²²P. Edman, "Preparation of Phenyl Thiohydantoin from Some Natural Amino Acids," *Acta Chem. Scand.* **4**, 277 (1950).

²³P. Edman, "Method for Determination of the Amino Acid Sequence in Peptides," *Acta Chem. Scand.* **4**, 283 (1950).

²⁴C. H. W. Hirs, S. Moore, and W. H. Stein, "The Sequence of Amino Acid Residues in Performic Acid-Oxidized Ribonuclease," *J. Biol. Chem.* **235**, 633 (1960).

Table 5.10. Possible Amino-Acid Sequence of the β Chain of C57Bl Mouse Hemoglobin

Peptide	Sequence
β T-1	Val, His, Leu, Thr, Asp, Glu, Ala, Lys
β T-2	Ser, Ala, Val, Gly, Ala, Leu, Tyr, Gly, Lys
β T-3	Val, Asn, Val, Asp, Glu, Ala, Gly, Gly, Glu, Ala, Leu, Gly, Arg
β T-4	Leu, Leu, Val, Val, Tyr, Pro, Tyr, Thr, Glu, Arg
β T-5	Tyr, Phe, Ser, Ser, Phe, Gly, Asp, Leu, Ser, Ser, Ala, Asp, Ala, Ile, Met, Gly, Asn, Ala, Lys
β T-6	Val, Lys
β T-7	Ala, His, Gly, Lys
β T-8	Lys
β T-9	Val, Ile, Asn, Ala, Phe, Ser, Asp, Glu, Leu, Thr, His, Leu, Asp, Asn, Leu, Lys
β T-10	Gly, Ser, Phe, Ala, Thr, Leu, Ser, Glu, Leu, His, Cys, Asp, Lys
β T-11	Leu, His, Val, Asp, Pro, Glu, Asn, Phe, Arg
β T-12	Leu, Leu, Gly, Asn, Val, Leu, Val, Ile, Gly, Leu, Met, His, His, Ile, Gly, Lys
β T-13	Asp, Phe, Thr, Pro, Ala, Ala, Gly, Ala, Ala, Phe, Glu, Lys
β T-14	Val, Val, Ala, Gly, Val, Ala, Ala, Ala, Leu, Ala, His, Lys
β T-15	Tyr, His

6. X-Ray and Optical Spectrochemistry

Cyrus Feldman

6.1 X-RAY SPECTROSCOPY

H. W. Dunn

A subroutine was written to plot relative intensities and chemical percentages calculated from pure standards; relative intensities or factors obtained by the XRA-E2 code; and chemical percentages calculated from pure standards, absorption coefficients, and fluorescence excitation potentials by the electron probe code EP-2 (a highly modified form of a code written by J. D. Brown¹). This plotting subroutine (DATAPLOT) calculates a least-squares fit and draws the corresponding curve, weighting individual points differently if desired. Code EP-2, a successor to EP-1, now obtains and/or calculates any necessary parameters from fundamental data stored on tape. It makes absorption and fluorescence corrections by several methods and uses the most consistent one in calculating chemical concentrations. It prints out results in final form for reporting, thus saving time and eliminating transcription errors. Standards of wide composition range are being prepared to test the accuracy of EP-2.

6.2 PHOTOGRAPHIC PHOTOMETRY

Cyrus Feldman

6.2.a Revision of ASTM "Tentative Recommended Practices for Photographic Photometry in Spectrochemical Analysis"

The ASTM "Tentative Recommended Practices for Photographic Photometry in Spectrochemical Analysis"² was revised. Sections were added on the diagnosis of difficulties from the appearance of

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

²C. Feldman, "Tentative Recommended Practices for Photographic Photometry in Spectrochemical Analysis (E-116-56T)," pp. 12-25 in *Methods for Emission Spectrochemical Analysis*, 4th ed., American Society for Testing Materials, Philadelphia, Pa., 1964.

the Seidel-function preliminary curve and on the external and internal illumination of the spectrograph. The sections on the step-sector, two-line, and line-group methods were omitted; only the two-step method was retained.

6.2.b Beam Splitter for Use in Emulsion Calibration

The methods most commonly used to calibrate spectrographic emulsions require a light beam of uniform intensity in the vertical direction where it encounters the stepping device (filter, step-sector, etc.) so that the intensity ratio of beams emerging from this device will be known. However, the desired degree of uniformity ($\pm 1\%$) is difficult to achieve over the necessary ranges of height (5-10 mm) and of wavelength (e.g., 2000-4500 Å). Therefore, a way was sought to produce a pair of light beams of known intensity ratio (preferably $\sim 3:2$ at all wavelengths) without first having to produce an illuminating beam of good vertical uniformity.

A beam splitter of the partially transparent mirror type appeared to offer a way to do this. Since suitable devices are not available commercially, an adjustable beam splitter with easily replaceable optical parts was built for developmental work (Fig. 6.1). When the incident beam strikes the partially transparent (or beam-splitting) mirror, part of

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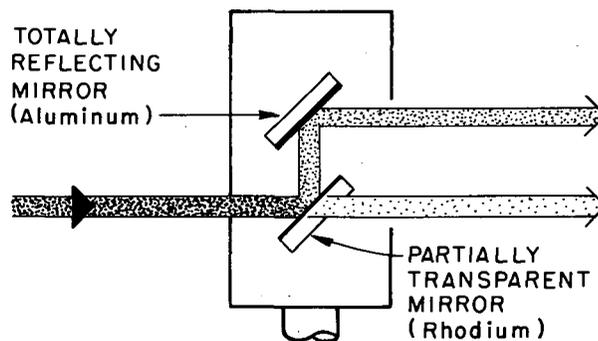


Fig. 6.1. Schematic Diagram of Beam Splitter for Use in Calibrating Spectrographic Emulsions.

the flux (e.g., 30%) is reflected onto the totally reflecting mirror and into the spectrograph. As the remainder of the flux passes through the beam-splitting mirror, about two-thirds is absorbed by the coating on the mirror, and the rest (e.g., 20% of the original flux) emerges almost undeviated. In this example, the intensity ratio of the two emergent beams would thus be 30:20 or 3:2.

The value of this ratio at a given wavelength depends on the density and reflectivity of the coating on the beam-splitting mirror, which in turn depend on the coating material and preparative technique. If it is assumed that the upper mirror gives essentially total reflection at all wavelengths, as is the case for aluminum, the wavelength dependence of the reflectance/transmittance (or beam-intensity) ratio (ideally, a horizontal line) depends primarily on the coating material. Several coating materials (Ag, Pt, Al, Rh) were tested at the 20% transmittance level by measuring separately the spectral transmittance and reflectance curves and then calculating the reflectance/transmittance ratio at each wavelength. Of the elements tested, rhodium gave the flattest reflectance/transmittance vs wavelength curve. Figure 6.2 shows its spectral trans-

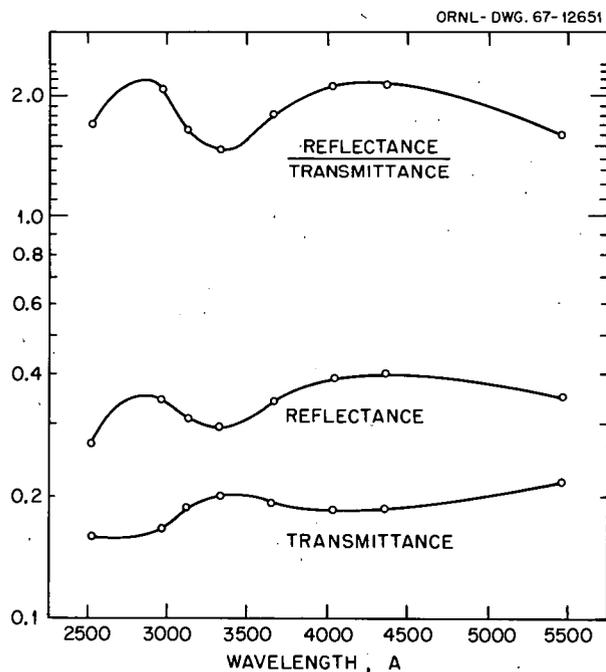


Fig. 6.2. Spectral Characteristics of Rhodium Beam-Splitting Mirror.

mittance, reflectance, and reflectance/transmittance ratio from 2400 to 4400 Å.

This device will now be used to calibrate spectrographic films under working conditions. Other beam-splitter constructions (e.g., prismatic beam splitters and an inclined reflecting light chopper to replace the beam-splitting mirror) are also being considered.

6.3 SPECTROGRAPHIC ANALYSIS OF TISSUE ASH

Cyrus Feldman F. S. Jones

6.3.a Chemical Preconcentration of Trace Elements in Bone Ash

The concentrations of most trace elements in bone ash are too low for their direct spectrographic detection. Use of the Mitchell-Scott procedure³ was attempted to collect quantities sufficient for detection, but high reagent blanks and coprecipitation of calcium phosphate made this impractical. Also, the procedure was ineffective for some trace elements.

Circumvention of the coprecipitation problem by extracting the trace elements from solutions of bone ash was successful for only a few elements. Again, the presence of phosphate hampered otherwise successful extractions.

It was therefore decided that the development of a general collection procedure would be greatly facilitated by first eliminating phosphate from the sample. Electrodialysis through anion-permeable resin membranes appeared to best suit the purpose. This approach would permit the handling of large samples without a commensurate increase in contamination. Although other anions besides phosphate would be removed, few of these are of interest in the present case.

A three-compartment cell was prepared; the sample (a slightly acid solution of natural or synthetic bone ash) was placed in the center compart-

³R. L. Mitchell, *The Spectrographic Analysis of Soils, Plants and Related Materials, with Addendum*, Commonwealth Bureau of Soil Science Technical Communication No. 44 of the Commonwealth Bureau of Soil Science, Harpenden, England; Commonwealth Agricultural Bureaux, Farnham Royal, Buckinghamshire, England (1964).

Table 6.1. Removal of Phosphate from Calcium Phosphate Solution by Electrodialysis Through Anion Exchange Membrane

Voltage (v)	Current (ma)	Time to Reduce $[\text{PO}_4^{3-}]$ by Half (min)	Remarks
10	140-70	820	All compartments stirred
10	135-88	500	Center compartment not stirred
23	365-160	320	
23	410-60	270	20 vol % HCOOH in anode compartment
23	650	80	20 vol % HCOOH in anode compartment; center compartment heated to 65-70°C

ment. To avoid migration of cations through a cation-permeable membrane and their subsequent deposition on the cathode, both walls of the center chamber were made of anion-permeable (i.e., cation-impermeable) resin membrane. The acid used had to be strong enough to dissolve the bone ash yet weak enough to leave most of the phosphate as an anion (e.g., H_2PO_4^-) rather than as neutral H_3PO_4 . The anions of the solvent acid also had to have a relatively low affinity for the anionic resin; the acid itself had to be easily purified and easily eliminated from the solution after processing. Formic acid fulfills these requirements and was chosen. Electrodialysis was carried out at ~ 20 v and 600-700 ma.

Experiments done with ^{32}P tracer showed that the concentration of phosphorus in the center cell decreased almost exponentially with time. Table 6.1 gives typical results. Unless otherwise noted, the sample was prepared by dissolving 1 g of $\text{Ca}_3(\text{PO}_4)_2$ in 5 ml of 90% HCOOH and adjusting the pH to 3. The anode compartment was filled with 10% HCOOH, and the cathode compartment with 1 M NaOH. Under these conditions <4% of the original phosphate remained after 6 hr. This efficiency permits use of the Mitchell-Scott procedure and makes possible improvement of it for collecting manganese and chromium. This procedure will now be applied to bone ash and tested for recovering trace elements.

6.3.b Purification of Water and Other Reagents

Since water and other reagents contribute a large fraction of the trace-element blank encountered in

the procedure for the chemical collection of trace elements, experiments were undertaken to purify water by electrodialysis through anion- and cation-permeable membranes. The first results obtained with ^{64}Cu tracer indicate that this element can be effectively removed from water in this way. Experiments are continuing on other contaminants and reagents.

6.3.c Use of Commercial Freeze-Dried Food as Tissue Standard

If a large amount of a homogeneous tissue standard could be conveniently prepared, stored, subdivided, and shipped, interlaboratory comparative analyses could be made that would include the ashing procedure as well as the determinations of components. Commercially available freeze-dried foods, especially meat, appear to be possible standards. Therefore, work was begun to check the suitability of Kraft freeze-dried chicken for this use. Preliminary results indicate that the chicken can be pulverized and blended satisfactorily by means of a hammer mill.⁴ However, sterilization of the blended product seems to be necessary to ensure adequate shelf life and to facilitate shipping.

⁴F. S. Jones, "Tissue Analysis," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1965*, ORNL-3889, p. 38.

Table 6.2. Limits of Detection for the Spectrographic Determination of Rare-Earth Elements, Ba, Sr, and U in LiF-BeF₂ Mixtures

Element	Limit of Detection		
	Micrograms per Gram of Sample		Absolute (μg)
	Without Preconcentration	With Preconcentration ^a	
Ce	200	2	0.4
Nd, Sm	50	0.5	0.1
Dy, Pr, Tb, Tm	20	0.2	0.04
Eu	5	0.05	0.01
Er, Gd, Lu, Y, Yb	2	0.02	0.004
Ba	4		0.008
Sr	1		0.002
U	230		0.5

^a1-g sample.

6.4 PRECONCENTRATION OF TRACE ELEMENTS IN THE SPECTROCHEMICAL ANALYSIS OF NUCLEAR FUELS

Anna M. Yoakum Cyrus Feldman

6.4.a Determination of Traces of Rare-Earth Elements, Barium, Strontium, and Uranium in LiF-BeF₂-Type Nuclear Fuels

With use of specially synthesized standards and the argon-atmosphere modification of the Kroonen-Vader Li₂CO₃-graphite base technique,⁵ rare-earth elements, Ba, Sr, and U were determined to the limits given in Table 6.2. The sensitivities obtained by direct analysis of sample material (column 2) can be improved by chemical preconcentration of impurities (column 3). Since most of these trace elements are rarely encountered in the blank, the sensitivities can probably be increased by increasing the sample size.

The chemical preconcentration procedure used for collecting rare-earth elements from large samples includes fusion with ammonium bisulfate⁶ to elimi-

nate fluoride and thus to convert the melt to a soluble mixture of sulfates. This melt was dissolved and treated with lanthanum carrier and then with NH₄F and HF. The combined fluorides immediately and quantitatively precipitated the trace elements; HF alone reacted much more slowly, although also quantitatively. The effectiveness of this procedure for collecting traces of barium, strontium, and uranium is being checked.

6.4.b Determination of Trace Impurities in ²³³UO₂ Mixtures by Tributyl Phosphate (TBP) Extraction and Solution-Residue Arc Method

In response to a request for determinations of trace impurities in ²³³UO₂-ThO₂ mixtures at sensitivity levels below the limits attainable by the carrier-distillation technique, an attempt was made to perform this analysis by dissolving the mixture in HNO₃, extracting the ²³³U and Th (in a glove box) into 40% TBP in *n*-hexane, evaporating the aqueous phase to dryness onto graphite powder, and arcing the residue.

The main questions to be answered before performing such an analysis are:

1. Are the ²³³U and Th completely extracted?

Since the aqueous layer is to be evaporated to a residue, even small amounts of uranium or thorium,

⁵J. Kroonen and D. Vader, *Line Interference in Emission Spectrographic Analysis*, Elsevier, New York, 1963.

⁶C. Feldman, "Ammonium Bisulfate Fusion. Application to Trace Analysis by Spectrochemical and Other Techniques," *Anal. Chem.* 32, 1727 (1960).

if still present, would seriously interfere with the spectrographic determination of trace elements. After five extractions of a 4-g sample of $^{233}\text{UO}_2$ - ThO_2 from 10 M HNO_3 with 40% TBP, no alpha radioactivity due to thorium or uranium could be detected, nor could these elements be detected spectrographically (sensitivity limits 0.02 and 0.6 μg respectively).

2. *What is the degree of contamination by reagents, glassware, and environmental dust?* Complete process blanks were run using quartz-distilled HNO_3 , deionized water, quartz laboratory ware when possible, and acid-soaked borosilicate ware when unavoidable. All operations up to the extraction of the ^{233}U and Th were conducted in a glove box, as described above. Table 6.3 shows the total blanks.

3. *How should standards be prepared?* Impurities in samples may occur either in solid solution in the sample lattices, as discrete (and possibly insoluble) particles, or in both ways. When the sample is processed as above, good recovery would be expected for dissolved impurities, but particles that resist dissolution might be lost at liquid-liquid interfaces or on the walls of containers.

In the present procedure the importance of the degree of dissemination of the impurities was tested by analyzing a ThO_2 standard prepared by grinding known amounts of solid impurities with pure ThO_2 . Recoveries were satisfactory for all trace elements except aluminum (40% recovery) and silicon (10% recovery). A second portion of this standard was then treated with soluble forms of aluminum and silicon and analyzed by the above procedures. Recovery of the soluble additions was complete. These experiments indicated that finely divided impurities are usually recovered in this procedure but that highly refractory particles, such

Table 6.3. Magnitude of Blanks for the Determination of Trace Impurities in $^{233}\text{UO}_2$ Mixtures by TBP Extraction and the Solution-Residue Arc Method

Element	Blank	
	Absolute (μg)	Concentrational ($\mu\text{g}/\text{g}$) ^a
Na	>21	>5
Zn	21	5
K	<6	<1.5
Ca	2	0.5
Fe	0.6	0.15
Al	0.6	0.15
Si	0.6	0.15
Mg	0.3	0.075
Cu	0.2	0.05
Ba	0.1	0.025
Cd	0.07	0.018
Cr	0.06	0.015
Ni	0.06	0.015
Pb	<0.03	<0.0075
Sn	<0.03	<0.0075
Ti	0.02	0.005
Mn	0.02	0.005
Mo	0.02	0.005
Zr	0.006	0.0015
Sr	0.006	0.0015
Ag	0.002	0.0005

^aCalculated for a 4-g sample.

as Al_2O_3 and SiO_2 , may be lost. Thus, the most reliable way to prepare standards is to simulate the preparative history of the samples.

4. *Are any trace elements lost by extraction?* This question is now under investigation.

7. Mass Spectrometry

7.1 SPARK-SOURCE MASS SPECTROMETRY

J. R. Sites J. A. Carter

The use of the MS-7 spark-source mass spectrograph increased 30% over the previous year. About a third of the samples being received are Ni-Cr-Fe

base alloys to be analyzed for trace elements with emphasis on their boron content. Twelve metallic samples made from separated stable isotopes (^{26}Mg , ^{57}Fe , ^{90}Zr , ^{150}Sm , ^{156}Gd , ^{164}Dy , ^{167}Er , ^{190}Os , ^{196}Pt) were checked for possible contaminants introduced during production. Other samples included very pure Ni, Cr, and W from different

vendors; ten rare-earth elements in the form of metal, oxide, or salt; and Be, Bi, Pd, Th, Tl, UP, US, and U_2N_3 .

Several unusual alloys (Nb-Ta, Nb-U, W-Re, Ta-Hf-W, V-Ti) were analyzed for minor constitu-

Table 7.1. Elements in W-25%Re Wire

Identity	Concentration (ppm)
Mass 188 (Os?)	1100
Mass 189 (Os?)	0.5
Ir ^a	6
Mo	200
Th	2

^aApproximate relative isotopic abundances of the Ir isotopes: ¹⁹¹Ir, 30%; ¹⁹²Ir, 10%; ¹⁹³Ir, 60%.

Table 7.2. Distribution of Radioactivity in Source

Source Part	Radioactivity Present (%)
First slit	90.0
Electrode clamps	7.5
Shield	1.7
Insulators	0.4
Kidney plate	0.2
Window	0.1
Walls of source	0.1
2d slit	nil
3d slit	nil
Plate	nil

Table 7.3. Results of Spark-Source Mass Spectrographic Determination of Niobium in Niobium-Spiked Uranium and in Purified ²³¹Pa^a

Identity	Sample Number	Niobium (wt %)	
		Added	Found
U	1	2.0	1.9
U	2	0.60	0.52
U	3	0.20	0.27
²³¹ Pa	1		<0.05

^aIndium used as internal standard.

ents. Of particular interest is the amount of osmium that grows into irradiated W-25%Re thermocouple wire. For the spark-source analysis two 1/8-in.-long pieces of 0.020-in.-diam thermocouple wire were mounted on gold wire; Table 7.1 shows the results. This direct analysis saved many man-hours of difficult analytical separations. Table 7.2 shows the distribution of the radioactivity of the residual material from the sparking of the W-25%Re wires.

The analysis of nonradioactive solutions by spark-source techniques was demonstrated previously.¹ This technique has now been applied to the analysis of small samples of radioactive materials. To evaluate the technique for determining niobium in ²³¹Pa ($t_{1/2} = 3.2 \times 10^4$ years), a series of niobium-spiked uranium samples were analyzed. Exactly 2 μ g of uranium, which corresponds to the maximum amount of ²³¹Pa that the Health Physics Division allows to be handled, was evaporated on gold electrodes for the analysis. In the trans-uranium program it was desired to be able to detect as little as 0.5 wt % Nb in ²³¹Pa. Table 7.3 gives the results for the niobium-spiked uranium samples and a purified sample of ²³¹Pa. This spark-source technique for solutions has also been used to study the mechanism whereby constituents of MSRE salts penetrate irradiated graphite.

Significant modifications were made to improve the electronics of the MS-7 mass spectrograph. An easily accessible arrangement for the reference battery of the magnet supply was added. Simple circuits that permit the stabilities of the magnet and high-voltage supplies to be checked to one part in 50,000 were added. Noise-free solid-state rectifiers were also added.

7.2 LOW-RESOLUTION MASS SPECTROMETRY OF ORGANIC COMPOUNDS

W. T. Rainey W. H. Christie
H. S. McKown

During the past year a single-magnetic-stage mass spectrometer was constructed and put into service for the analysis of organic compounds. The instrument (Fig. 7.1) uses a 12-in.-radius 90° magnet with an electron bombardment source and

¹J. R. Sites and J. A. Carter, "Spark-Source Mass Spectrometry," *Anal. Chem. Div. Ann. Progr. Rept.* Oct. 31, 1966, ORNL-4039, p. 47.



Fig. 7.1. Low-Resolution Mass Spectrometer for Analysis of Organic Compounds.

electron multiplier detector. The output is presented in chart form by a six-pen oscillograph, one pen being used as ion-current monitor and mass marker.

Sample-input systems are capable of handling gases, volatile liquids, and solids. Reasonably volatile liquids in milligram quantities can be expanded into a reservoir contained in a thermostatically controlled oven and bled into the source through a variable leak. Solids and liquids in microgram quantity can be introduced into the source by a direct-inlet probe. Samples that are contained in melting-point capillaries can be cooled to liquid-nitrogen temperature or heated to 350°C while they are in the evacuated source can.

A wide variety of samples have been analyzed with satisfactory results. The resolution of the instrument accommodates samples of mass up to 600 provided they are sufficiently volatile. Several series of alkyl-substituted aromatic hydrocarbons, organic phosphate esters, polynuclear hydrocarbons, amines, alcohols, and ketones have been studied to date.

7.3 SURFACE IONIZATION STUDIES

G. R. Hertel

7.3.a Ionization Potential Measurements by Surface Ionization

The surface-ionization comparison technique^{2,3} was used to measure the first ionization potentials

Table 7.4. First Ionization Potentials of the Lanthanides

Element	Ionization Potential (ev)
La	5.55 ± 0.05
Ce	5.54 ± 0.06
Pr	5.40 ± 0.05
Nd	5.49 ± 0.05
Pm	(5.55 estd)
Sm	5.61 ± 0.05
Eu	5.64 ± 0.05
Gd	6.16 ± 0.05
Tb	5.89 ± 0.04
Dy	5.82 ± 0.03
Ho	5.89 ± 0.03
Er	5.95 ± 0.03
Tm	6.03 ± 0.04
Yb	6.04 ± 0.04
Lu	5.32 ± 0.05

of the lanthanides. By use of mixtures of various lanthanide fluorides, the ion intensities of the singly charged lanthanide ions could be measured under identical experimental conditions and could be compared directly. From the relative changes in intensities with temperature, the differences in the ionization potentials among all the lanthanides except promethium were determined. Comparison with a reference element (silver) made possible the calculation of the ionization potentials of the lanthanides (Table 7.4). The value for promethium is an estimate based on the calculated values for the other elements.

Further details of this work and a comparison of all known previously reported values are included in a forthcoming publication.⁴

²I. 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).

³G. R. Hertel, "Surface Ionization II. The First Ionization Potential of Uranium," *J. Chem. Phys.* 47, 335 (1967).

⁴G. R. Hertel, "Surface Ionization III. The First Ionization Potentials of the Lanthanides," accepted for publication in the *Journal of Chemical Physics*.

7.4 MASS SPECTROMETRIC SEARCH FOR NEON IN MINERALS AND IN VOLCANIC GLASSES

L. C. Hall⁵

Several minerals and volcanic glasses were heated at 1800°C in vacuo, and the evolved gases were examined mass spectroscopically for He, Ne, and Ar, with the main emphasis on Ne. Spodumene was examined to determine whether the $^{18}\text{O}(\alpha, n)^{22}\text{Ne}$ reaction, which was suggested by Aldrich and Nier⁶ as a neutron source for the reaction $^6\text{Li}(n, \alpha)^3\text{He}$, would produce detectable amounts of neon in this lithium mineral. Several fluorine-containing minerals, namely, lepidolite, amblygonite, and fluorite (with embedded uraninite), were studied to see if the reaction $^{19}\text{F}(\alpha, n)^{22}\text{Ne} \xrightarrow{(\beta^+)}$ ^{22}Ne would be detectable. Pumice, pearlite, and obsidian were also studied. No conclusive evidence was obtained for the existence of neon in these materials. The extraction, purification, and mass spectrometric systems used in this study allow detection of at least $5 \times 10^{-5} \text{ cm}^3$ (STP) of neon. Since $1 \times 10^{-6} \text{ cm}^3$ (STP) per gram of sample is to date the largest reported amount of neon in crystal samples, extensive changes need to be made. This work indicates that the gas-liberation and purification system needs to be modified extensively to increase the sensitivity by a factor of 10 to 100. A sensitive static mass spectrometer that offers a gain in sensitivity of at least 1000 over dynamic instruments should be used. It is

⁵ORAU Research Participant; Assistant Professor of Chemistry, Vanderbilt University, Nashville, Tenn.

⁶L. T. Aldrich and A. O. Nier, "The Occurrence of He³ in Natural Sources of Helium," *Phys. Rev.* **74**, 1590 (1948).

recommended that helium-containing fluorine minerals (e.g., fluorite with uraninite) be studied closely to evaluate $^{22}\text{Ne}/^{20}\text{Ne}$ and $^{21}\text{Ne}/^{20}\text{Ne}$ ratios. Also, quartz and muscovite samples that have the highest ^{18}O contents should be analyzed for neon isotopes. Such analyses are currently important in understanding the distinction between primordial and radiogenic components and their role in the origin of our atmosphere and its relationship to the origin of the solar system and the genesis of elements.

7.5 DOUBLE-FOCUSING MASS SPECTROMETER

W. T. Rainey W. H. Christie
H. S. McKown

A high-resolution double-focusing mass spectrometer was assembled and is being tested. This type of mass spectrometer consists of an electrostatic deflection sector that is followed by a magnetic deflection sector; low-resolution instruments have only a magnetic stage. This additional electrostatic stage reduces aberrations caused by the spread of energy and of direction of the ions generated in the source. At present a resolution of 4000 has been attained. This means that two peaks of equal height which differ in mass by one part in 4000 can be resolved to the extent that the height of the valley between them is only 0.1% of the peak height.

The source accepts solid and gaseous samples. The detection system uses ion-pulse counting. With the existing instrumentation, only inorganic samples can be accepted, because a spectrum of only four or five mass units can be observed at one time. Instrumentation for the long mass scans required for organic materials is being developed.

8. Optical and Electron Microscopy

T. E. Willmarth

T. G. Harmon

H. W. Wright

8.1 RESEARCH ASSISTANCE IN ELECTRON MICROSCOPY AND ELECTRON DIFFRACTION

Electron microscopy and electron diffraction have continued in use as investigative tools in a wide range of fundamental and applied research of interest to the senior research staff of ORNL and other Union Carbide establishments in Oak Ridge.

8.1.a Surface Chemistry Studies. Adsorption of Water on Porous and Nonporous Samples of Thorium Oxide¹

Electron micrographs were made of samples of thorium oxide prepared by thermal decomposition and by

acid digestion of thorium oxalate. The particle and crystal dimensions so determined correlated well with those determined by other means. In crystal models proposed to explain the difference in water adsorption between the two preparations, the most predominantly exposed crystal face is (100) for the thorium oxide prepared from the oxalate and (111) for the material prepared by acid digestion. In support of the model, electron diffraction studies of relatively large single crystals similar to those formed by acid digestion indicated that for ~80% of the crystals the plane of the exposed faces was (111), that of the remainder of the crystals being (100). Figure 8.1 shows the nature of the preparation used for the electron diffraction studies. The opaque objects are surface crystals of thorium oxide thinned in hot sulfuric acid after a vaporized film of carbon has been deposited on them. The underlying gray structure is a replica of the agglomerate surface. Each crystal mass was isolated for transmission electron diffraction by means of a 10- μ aperture, which gave an effective selected area of ~0.9 μ .

PEM-200-7230

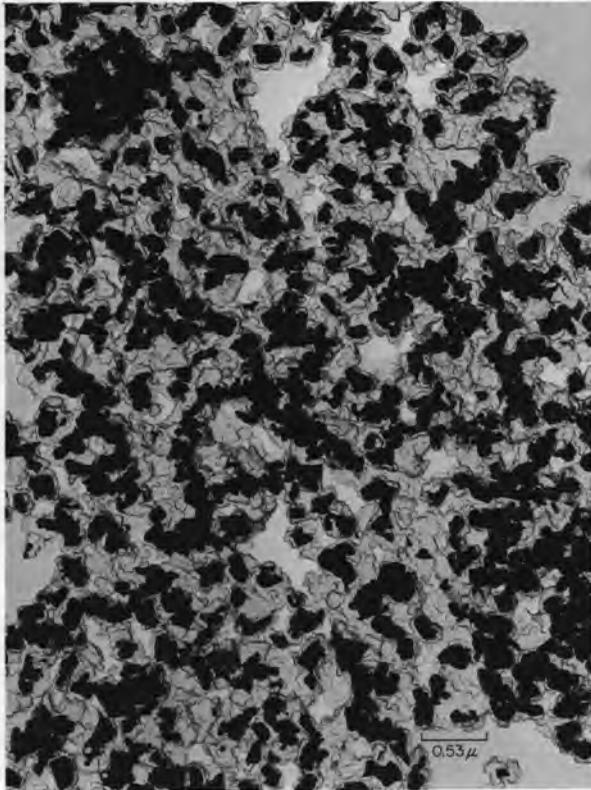


Fig. 8.1. Electron Micrograph That Shows ThO_2 Crystals as Chemically Thinned in Hot H_2SO_4 .

8.1.b Dispersion Hardening Studies. Controlled Oxide Precipitation in Flake Aluminum and Aluminum Alloys

Electron micrographs were made of atomized aluminum and aluminum alloys that had been ball-milled in a high-energy ball mill in the presence of one of several oxides dispersed in petroleum ether with stearic acid as surfactant. The micrographs indicated that the oxide particles were pressed into and retained in or on the metal surface. Masses of such flakes can be vacuum-annealed, cold-pressed, and hot-extruded to form bars or sheets of aluminum or aluminum alloys that contain homogeneous dispersions of the included oxide. Figure 8.2 shows MgO and $\alpha\text{-Al}_2(\text{OOH})_2$ (boehmite) retained in or on the surface of the metal flakes.

¹H. F. Holmes, E. L. Fuller, Jr., and C. H. Secoy, "Gravimetric Adsorption Studies of Thorium Oxide. III. Adsorption of Water on Porous and Non-Porous Samples," submitted to the *Journal of Physical Chemistry*.

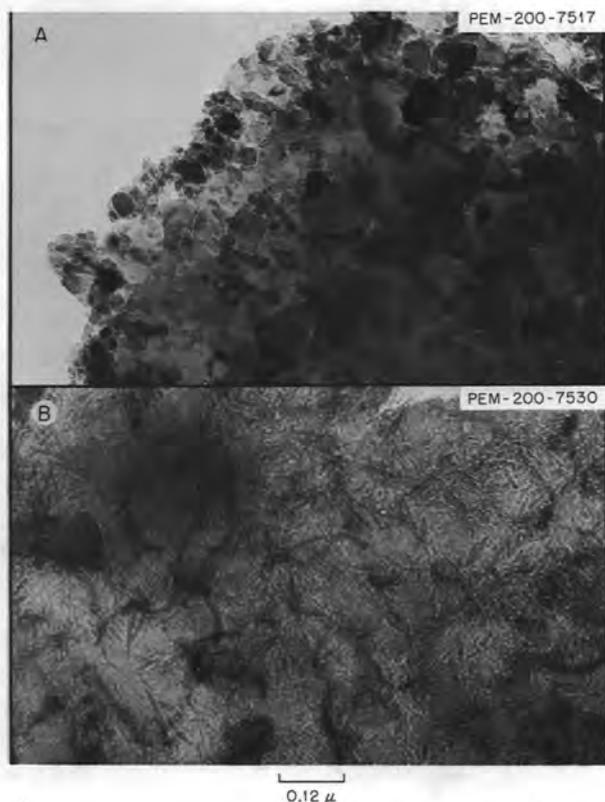


Fig. 8.2. Appearance of Aluminum Flake After High-Energy Ball-Milling with Oxide Particles Dispersed in Petroleum Ether That Contained Stearic Acid as Surfactant. *A*, MgO particles; *B*, α - $\text{Al}_2(\text{OOH})_2$ (boehmite) particles.

8.1.c Aging of Lanthanide Hydroxide Sols

Observations with the electron microscope of the physical characteristics of lanthanide hydroxide sols aged for various periods of time at 25 and 80°C have continued. Erbium hydroxide, aged for prolonged periods at 25°C, has exhibited several changes during the aging (Fig. 8.3); these changes were suspected to have occurred in the lanthanide sols previously studied but were never observed visually. The presence of ragged edged sheets in micrograph *B* and well-formed sheets in micrograph *C* gives credence to previous observations that the final crystalline products are rolled-up tubes or scrolls of sheetlike material.

Work is in progress on the hydroxide sols of samarium, lutetium, gadolinium, and terbium. The time required for the formation of the europium crystalline tube structures has been established as 11 to 12 hr.

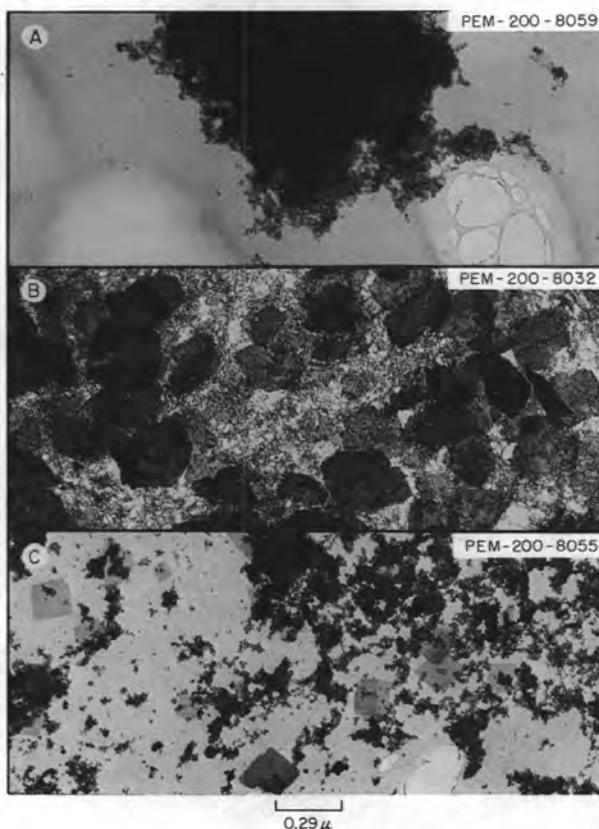


Fig. 8.3. Electron Micrographs of Erbium Hydroxide Sols. *A*, aged 5 min at 25°C; *B*, aged 5 days at 55°C; *C*, aged 19 days 9 hr at 25°C.

8.1.d Nuclear Safety. Retention of Radioactive Iodides by Impregnated Charcoals

As part of a program to determine the ability of impregnated charcoal to capture and retain radioactive methyl iodide-131, activated charcoal was impregnated with a solution of silver salt that was reduced to metallic silver within the pores of the charcoal. Electron microscopy and electron diffraction were used to determine the physical appearance of the metallic silver, its location within the charcoal matrix, and whether the silver salt had been completely reduced. Electron microscopy of sections of the impregnated charcoal prepared by ultratoming with an LKB microtome showed that the metallic silver was present in the form of small spheres lining the pores of the charcoal (Fig. 8.4). Electron diffraction indicated the presence of only metallic silver and carbon.

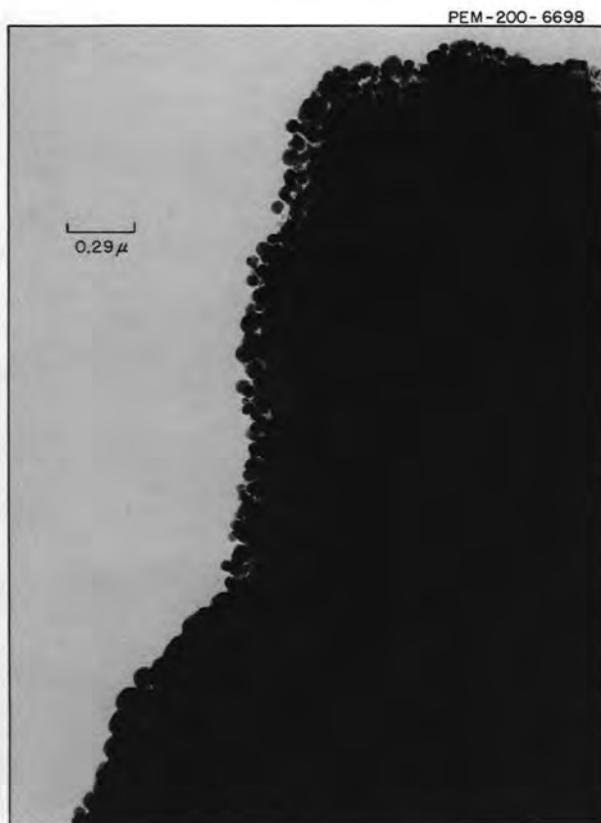


Fig. 8.4. Pore Wall of Activated Charcoal That Shows Deposit of Metallic Silver.

8.1.e Other Research Assistance

Other research assistance in the form of optical and electron microscopy and electron diffraction was given relative to the following work: thoria fuel-cycle studies; anodically deposited coatings for the "moon box"; solid-state surface studies of metals; ferrous-ferric molybdate corrosion inhibitors; solubility of Fe_3O_4 at elevated temperatures; removal of iodide from LiF-BeF_2 melts; development of fuel elements, pressable BN, and radioactive sources for radiographic work; and evaluation of SAP materials, a low-pressure impactor for particle collection, and a contained system for fission product simulation.

8.2 ELECTRON MICROSCOPY OF RADIOACTIVE MATERIALS

The ability to successfully examine, photographically record, identify, and interpret the nature of highly radioactive materials has continued to be demonstrated at the facility for electron microscopy

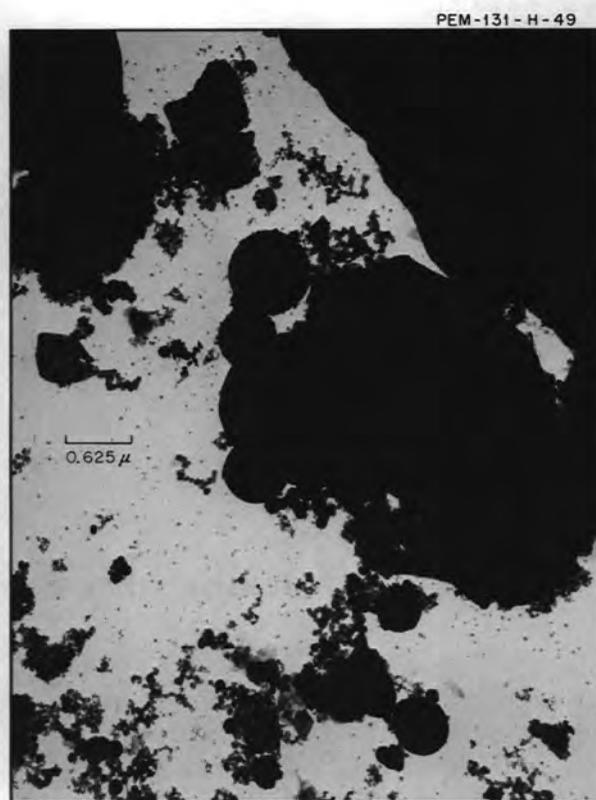


Fig. 8.5. Particles of Highly Radioactive $^{238}\text{PuO}_2$ from Mound Laboratory.

of highly radioactive materials, located in Building 3019. For example, samples of $^{238}\text{PuO}_2$ from the Mound Laboratory were prepared, observed, and recorded photographically with no appreciable contamination of the electron microscope and no difficulties (Fig. 8.5). The electron-diffraction technique was demonstrated to be sufficiently sensitive to identify radioactive corrosion and reaction products on the surfaces of short-cooled MSRE fuel elements and of graphite exposed in the reactor. With numerous samples, the magnification and resolution of the Philips EM 100 electron microscope now being used for this work were not great enough to provide the required information. The microscope now on order (Sect. 8.3) should meet this need.

8.3 INSTRUMENTATION

A beam-tilting device for dark-field microscopy was added to the Philips EM 200 electron microscope. With this accessory one may go from bright

field to dark field by means of a single switch. The beam can be deflected in either of two orthogonal directions to about 6° , so that any diffraction spot can be brought out to about the eighth order onto the optic axis of the objective lens. The device is especially useful in the study of dislocations and stacking faults in thinned metals and in the resolving of subcrystallites in platelet, tube-, and needle-shaped crystals.

To ensure uniform results in the photographic development of electron-micrograph plates, a nitrogen-bubble-agitation system was installed. This device

makes possible temperature control to $\pm 0.5^\circ\text{F}$ and contains a built-in plate dryer and viewer.

A Philips EM 300 electron microscope was ordered; it is to be installed in the Transuranium Processing Plant. It will be used exclusively for the study of radioactive materials, especially the highly radioactive alpha emitters such as americium and curium. To ensure the proper observation and recording of sequential events that may take place while such materials are under observation, the microscope is being equipped with an image-intensifying TV monitor and a video readout.

9. Nuclear and Radiochemical Analyses

W. S. Lyon
H. H. Ross

9.1 NUCLEAR PROPERTIES OF RADIONUCLIDES

9.1.a New Nuclear Data

J. F. Emery

^{95}Zr . — The number of gamma rays per disintegration of ^{95}Zr (ref. 1) was redetermined on a freshly separated ^{95}Zr source by use of two Ge(Li) detectors and an NaI(Tl) detector. After ~ 90 hr a second separation was made to obtain the ^{95m}Nb that had grown in. This ^{95m}Nb was measured to determine the branching of ^{95}Zr to ^{95m}Nb . Table 9.1 summarizes the results.

The value obtained for the ratio of the intensities of the 757- to the 726-keV gamma ray, 1.25, agrees with the value 1.28 reported by Tsaletka and Vrzal.² Also, the value 1.35×10^{-2} for the branching of ^{95}Zr to ^{95m}Nb agrees with the value 1.4×10^{-2} reported by Hudgens and Lyon.³

¹K. Way, "Nuclear Data Sheets," NRC 60-5-119, A = 95, Natl. Acad. Sci.—Natl. Res. Council, Washington, D.C.

²R. Tsaletka and Y. Vrzal, "Measurement of ^{95}Zr and ^{95m}Nb Mixture by Ge(Li) Detectors," *Nucl. Sci. Abstr.* 20, 36170 (1966).

³J. E. Hudgens, Jr., and W. S. Lyon, "Radiation from Columbian 95," *Phys. Rev.* 75, 206 (1949).

^{112}Sn . — The thermal-neutron cross section for the reaction $^{112}\text{Sn}(n,\gamma)^{113}\text{Sn}$ was determined. Irradiations were made in the ORR pneumatic tube ($\phi_{th} = 5 \times 10^{13}$ neutrons $\text{cm}^{-2} \text{sec}^{-1}$; $\phi_r = 1.5 \times 10^{12}$ neutrons $\text{cm}^{-2} \text{sec}^{-1}$) and in the D_2O tank of the BSR ($\phi_{th} = 1.2 \times 10^{12}$ neutrons $\text{cm}^{-2} \text{sec}^{-1}$; $\phi_r \approx 4 \times 10^8$ neutrons $\text{cm}^{-2} \text{sec}^{-1}$). The thermal-neutron cross section found is 0.65 barn; the resonance integral is 24 barns. The reaction $^{197}\text{Au}(n,\gamma)^{198}\text{Au}$ was used for neutron-flux determinations; the values 98 and 1558 barns were used for the thermal-neutron and resonance cross sections of gold for this reaction.

^{249}Bk . — A gamma-ray spectrum of an $\sim 1\text{-}\mu\text{g}$ $^{249}\text{BkO}_2$ source was obtained with a Ge(Li) spectrometer. Gamma rays from the growth of ^{249}Cf were observed. The gamma-ray energies are 108, 122, 254, 333, and 387 keV; those listed in the literature are 260, 340, and 390 keV.

The $^{249}\text{BkO}_2$ was irradiated for 20 min in a thermal-neutron flux of 5×10^{13} neutrons $\text{cm}^{-2} \text{sec}^{-1}$. The effective activation cross section was 210 barns. The gamma-ray emission of ^{250}Bk consisted of a pair of approximately equal-intensity gamma rays of 991 and 1034 keV energy.

The lower limit of detection of activation analysis for ^{249}Bk is about 10^{-12} g.

Table 9.1. Gamma-Ray Intensities in ^{95}Zr

E_{γ} (keV)	Number of Gamma Rays per Disintegration		
	40-cc Ge(Li)	6-cc Ge(Li)	NaI(Tl)
726	0.441	0.434	
757	0.548	0.548	
726 + 757	0.989	0.982	0.987
(Fraction to ^{95m}Nb)			1.35×10^{-2}

9.1.b Alpha Decay of Holmium Nuclei: New Isotope, ^{154}Ho

T. H. Handley R. L. Hahn⁴
K. S. Toth⁵

Neutron-deficient holmium isotopes were produced by $^{156}\text{Dy}(p, xn)$ reactions. Chemical separations verified that the products were holmium nuclei; excitation-function determinations served to assign mass numbers to them. A new isotope, ^{154}Ho , was found; it emits alpha particles of 3.91 ± 0.02 Mev and has a half-life of 11.8 ± 1.0 min. A search was made for an alpha-decay transition from ^{155}Ho , but none was observed. The radionuclides ^{152}Ho and ^{153}Ho , already known to exist, were found to have the following respective alpha energies and half-lives: 4.37 ± 0.02 Mev, 2.7 ± 0.2 min; 3.97 ± 0.02 Mev, 9.3 ± 0.5 min. For ^{153}Ho , the alpha-decay to electron-capture ratio was measured to be $(1.2 \pm 0.7) \times 10^{-3}$; from a comparison with semiempirical estimates of alpha-decay rates, it was concluded that the alpha transition of ^{153}Ho is not hindered. The results of this work were submitted for publication.⁶

9.1.c Nuclear Spectroscopy of Neutron-Deficient Radionuclides

T. H. Handley Ben Harmatz⁵

The systematic study of neutron-deficient radionuclides has continued. The radionuclides

⁴Chemistry Division.

⁵Electronuclear Division.

⁶R. L. Hahn, K. S. Toth, and T. H. Handley, "Alpha Decay of Holmium Nuclei; New Isotope, ^{154}Ho ," accepted for publication in *The Physical Review*.

are produced by proton or alpha bombardment in the ORIC. Following chemical separations, sources are prepared for study by use of permanent-magnet spectrographs and gamma-scintillation spectrometers. During recent months ^{173}Ta and ^{185}Os have been studied. Present work extends earlier work⁷ done with weaker sources of ^{173}Ta and ^{185}Os ; it is now possible to put together more complete decay schemes of these nuclides. A number of possible excited states are proposed. The results of this work will be published eventually in the open literature.

9.1.d Standardization of ^{197}Hg

J. S. Eldridge

The results of the analyses of the interlaboratory comparison samples of ^{197}Hg , distributed by ORNL in 1966,⁸ were reported at an IAEA symposium.⁹ They aroused sufficient concern among many users and suppliers of this medically important radionuclide to justify a second interlaboratory comparison. Samples were prepared and distributed by B. J. Jackson.¹⁰ Results were collected and were reported on an anonymous basis at ORNL. Participants were the laboratories

⁷B. Harmatz, T. H. Handley, and J. W. Mihelich, "Nuclear Spectroscopy of Neutron-Deficient Lu, Ta, and Re Isotopes," *Phys. Rev.* **119**, 1345 (1960).

⁸J. S. Eldridge, "Standardization of ^{197}Hg ," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1966*, ORNL-4039, p. 62.

⁹J. S. Eldridge, "Standardization of Mercury-197," pp. 313-322 in *Standardization of Radionuclides* (Proceedings of the Symposium on Standardization of Radionuclides held by the Atomic Energy Agency at Vienna, October, 1966), IAEA, Vienna, 1965.

¹⁰Commercial Products Branch, Atomic Energy of Canada, Ltd., Ottawa, Ontario.

who took part in the original intercomparison plus two additional laboratories.

Whereas the original intercomparison showed an interlaboratory standard deviation of 16% and a range of values differing by a factor of 2.0, the second intercomparison gave better results – standard deviation, 6.8%; range difference lowered to a factor of 1.3.

The marked improvement of results for interlaboratory comparison of ^{197}Hg attests to the value of such intercomparisons for radionuclide standardizations in those cases where absolute standardizations are not possible or primary standards are not readily available.

Interlaboratory comparisons are planned for ^{99}Mo - ^{99m}Tc and ^{99m}Tc , which are radionuclides that present difficult standardization problems for their average hospital user.

9.2 NEW INSTRUMENTS AND TECHNIQUES

9.2.a A Film-Recording Radiation-Event Monitor for Pulsed X and Gamma Radiation

F. F. Dyer L. C. Bate
L. H. Thacker¹¹

A sentinel-type radiation detector was developed to provide time-stamped records of exposure to rapid pulses of x and gamma radiation. The detector (Fig. 9.1) consists of a CsI(Tl) scintillator, photographic film, mercury microcoulometer, and time-reference scale – all housed in a light-tight enclosure. Visible light generated in the CsI by the radiation pulse is transmitted to the photographic film through the time-reference scale and the transparent gap of electrolyte in the mercury microcoulometer. The image of the time-reference scale and the gap formed on the film together show the time at which the radiation pulse occurred.

The detector responds satisfactorily to radiation doses in the range ~ 0.2 to ~ 50 mr received during intervals from several nanoseconds to several minutes. Full-scale timing can be adjusted to intervals from a few hours to several months. Exposure records can be read with un-

¹¹Instrumentation and Controls Division.

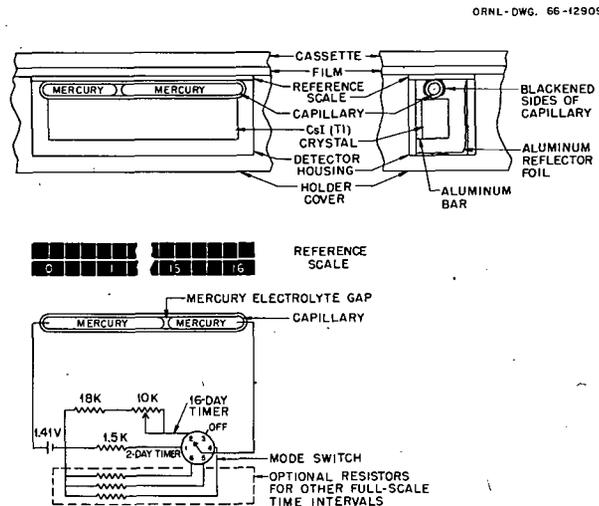


Fig. 9.1. Schematic Diagram of the Detection and Timing Mechanism of the Film-Recording Radiation-Event Monitor for Pulsed X and Gamma Radiation.

certainties of $\pm 0.1\%$ of the full-scale timing interval. The detector is expected to be useful around high-voltage accelerators, x-ray machines, and in other areas where it is desirable to have a record of exposure to rapid radiation pulses. Details of the construction and operation of the device are being published.¹²

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

H. H. Ross

The application of the isotopic-light-source photometer was extended into the ultraviolet region with the development of a new light-source system.¹³ The displacement factor (R) vs concentration curves of several organic substances were determined; all exhibited the expected linear response. Also, an exact analytical expression for the mathematical model of the instrument was developed with the aid of the Mathematics Divi-

¹²F. F. Dyer, L. C. Bate, and L. H. Thacker, "A Film-Recording Radiation Event Monitor for Pulsed X and Gamma Radiation," accepted for publication in *Nuclear Instruments and Methods*.

¹³H. H. Ross, "Precision Photometric Analysis by Use of a Beta-Excited Isotopic Light Source," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1966*, ORNL-4039, p. 70.

sion. The details of this work are given in an invited contribution.¹⁴

9.2. c Analytical Capabilities of a Semiconductor X-Ray Emission Spectrometer

T. H. Handley

Recent improvements in semiconductor crystals and related circuitry have made possible an x-ray emission spectrograph in which characteristic x rays excited by a radioactive source are detected by a semiconductor. Preliminary calibration, resolution determination, limits of detection, and radioactive-source fabrication have been studied with a view to quantitative analytical applications. An annular ¹²⁵I source (9 mc) was used to excite characteristic x rays of the samples; ¹²⁵I is suitable for exciting K α x rays of elements Z = 23 through 49 and L α x rays of the heavy elements. The lithium-drifted silicon semiconductor detector has resolutions of 0.6 and 0.8 kev for 20- and 7-kev x rays respectively. If K α x-ray peaks of equal intensity are to be resolved, the energy separation of the peaks must be equivalent to the resolution of the detector. Therefore, in the region of cobalt and iron the detector cannot resolve mixtures of adjacent elements; however, in the region of molybdenum adjacent elements can be resolved and determined when present in mixtures. A plot of channel number vs energy of x rays (5 to 27 kev) gives a straight line. Standardization with sources of pure molybdenum showed that molybdenum can be determined over the range 5 μ g to 2.0 mg. A plot of count rate vs total amount of molybdenum in the sample is linear from 5 μ g to 2.0 mg. In the region of lead it is possible to determine mixtures of elements three Z numbers apart. However, preliminary chemical separations are always a possibility. Tests also show that this instrument can be used for qualitative identification (e.g., distinction among different grades of steels and alloys). It is planned to also use the instrument in nuclear-decay studies.

¹⁴H. H. Ross, "Analytical Applications of the Secondary Effects of Radiation: Precision Photometry Using a Radioisotopic Light Source," *Record of Chemical Progress*, Kresge-Hooker Science Library, in press (1967).

9.2. d Development of Radiation-Stimulated Light Sources: Excitation with ¹⁴⁷Pm

H. H. Ross S. B. Lupica

The second phase of the program to develop radiation-stimulated light sources, excitation of selected phosphors with ¹⁴⁷Pm, was recently completed.¹⁵ The purpose was to study variables associated with the construction of isotopic light sources. The variables studied included chemical form of the isotope, methods of combining isotope and phosphor, isotope loading, phosphor thickness, and the spectral and intensity characteristics of the stimulated emission. Also, the problems associated with making self-supporting isotope-phosphor pellets were examined.

The results indicate that a number of general rules can be developed for predicting the effects of many of the variables. A careful consideration of these effects permits the construction of highly efficient light-source modules with a minimum of design effort. A complete résumé of the experimental program, including the stimulated emission spectra, is being prepared for publication. Also, the third phase of the program, photovoltaic conversion, has been started.

9.2. e Analytical Alpha Spectrometry

T. H. Handley Ernest Schonfeld¹⁶
J. H. Cooper¹⁷

Efforts have continued toward improving all aspects and reducing the cost of analysis by alpha spectrometry. Studies of reproducibility of spectral response have shown that some electronic equipment in the Analytical Services Laboratories must be improved or replaced to ensure spectral stability and that it is possible to limit spectral shifts to ≤ 2 kev for a period of four days by use of the best electronic equipment. Spectral stability is essential to the quantitative analysis of alpha-spectral data. Spectral instability was shown not to be associated with the sources, because quantitative electrodeposition produced

¹⁵H. H. Ross, "Development of Radiation-Stimulated Light Sources," *Anal. Chem. Div. Ann. Progr. Rept.* Oct. 31, 1966, ORNL-4039, p. 71.

¹⁶Chemical Technology Division.

¹⁷High-Level Alpha Radiation Laboratory.

sources with differences $\leq 1\%$. Several mathematical approaches have been tried for the analysis of complex alpha spectra, and more tests will be necessary before the optimum method can be selected. Some operational improvements include introduction of a vacuum gage into the detector system, zero pole cancellation, change from double-mode to single-mode differentiation, improvement of spectral stability by increasing bias voltage, and installation of a liquid-nitrogen trap to help increase the vacuum.

9.3 NEW FACILITIES FOR ACTIVATION ANALYSIS AND LOW-LEVEL MEASUREMENTS

9.3.a Activation Analysis with a ^{252}Cf Neutron Source

Enzo Ricci T. H. Handley

The possibility of using the neutrons from the spontaneous fission of ^{252}Cf in activation analysis has been considered, and plans have been made for a research and development program in that direction.

A 1-mg ^{252}Cf source is being fabricated at the Transuranium Processing Plant and will be stored in an air-tight hot cell currently being built at the Transuranium Research Laboratory. The ^{252}Cf source is expected to have a total neutron output of about 3×10^9 neutrons/sec. The activation analysis work will be carried out in this cell, behind a shielded window, by using master-slave manipulators. The window and the manipulators have already been installed, as well as most of the concrete shielding of the cell (Fig. 9.2).

The samples to be activated will be conveyed into the cell by a pneumatic rabbit system. Present plans call for a simple switching device, located outside the cell, that will permit delivery of the samples to either of two positions for irradiation with fission-spectrum or thermalized neutrons. It is expected that the integrated fluxes of fast (fission) and of thermal neutrons will be $\sim 10^8$ and $\sim 6 \times 10^7$ neutrons $\text{cm}^{-2} \text{sec}^{-1}$, respectively, at the corresponding irradiation sites.

The conveyor, purchased from Kaman Nuclear, is currently being installed. This unit can be programmed adequately for any chosen sequence or irradiation, delay, and counting time, for



Fig. 9.2. General View of the ^{252}Cf Cell.

standard and sample, and in either of the two irradiation sites. Thus, it can provide an entirely automatic operation. One of its interesting features is the dual-axis rotator for fast-neutron bombardment. The fast-neutron flux depends very strongly on geometry; the rotator ensures that sample and standard are exposed to the same irradiation conditions by spinning them simultaneously in two directions during bombardment. Provision will be made for accurate irradiation timing. The radioactivity measurements will be conducted at a counting station located between two 5- by 5-in. NaI(Tl) detectors. Manual operation of the counting procedure is possible, and the distance between crystals can be varied over a reasonable range.

The potential usefulness of the ^{252}Cf source in remote locations, even including outer space, is being considered. Thus, the sensitivities for fast- and thermal-neutron irradiations will be determined for elements of interest in biology, medicine, geology, and related fields.

9.3.b Experiments Related to the Acquisition of the High-Intensity Analytical Cyclotron

Enzo Ricci

In accordance with its current program on charged-particle activation analysis, the Analytical Chemistry Division is considering the purchase of a small high-intensity accelerator, the High-

Intensity Analytical Cyclotron (HIAC). Two manufacturers offer cyclotrons that meet the specifications set for the HIAC: The Cyclotron Corporation (T.C.C.), Berkeley, California; and Philips, Eindhoven, The Netherlands. Each accelerator is an isochronous, azimuthally varying field (AVF) cyclotron and is priced in the range \$240,000 to \$280,000.

Trials of the machines can be valuable in deciding which accelerator approaches the HIAC specifications more closely. A number of tests were run at Berkeley on the T.C.C. 32-in.-diam cyclotron. These tests were aimed to appraise: (1) ease of operation and of particle switching, (2) maximum beam intensity and beam stability during short and long high-current irradiations with each of the particles, and (3) efficiency of concrete shielding during irradiation of neutron-generating targets. The cyclotron used was designed to deliver 50 μ a of any of the following particles: 21-Mev ^3He ions, 15-Mev protons, and 8-Mev deuterons; it was to be installed later at the Sloan-Kettering Institute.

Neutrons were produced by the reactions $^9\text{Be}(^3\text{He},n)^{11}\text{C}$, $^9\text{Be}(d,n)^{10}\text{B}$, and $^7\text{Li}(p,n)^7\text{Be}$; fast and thermal neutrons and gamma rays were measured at five different locations outside the shielded cyclotron room. These measurements are useful in calculating the shielding necessary for the HIAC; the data of Table 9.2 illustrate the results. It can be seen that, besides its known dependence on shielding and distance, the dose depends strongly on the angle of the observer with the di-

rection of the beam. Also, it is clear that, with the 50- to 100- μ a beam currents specified for the HIAC, the shielding used at the T.C.C. would be insufficient. In fact, it is estimated that 5 ft of ordinary concrete would be required.

A stable 80- μ a deuteron beam and a less-stable 50- μ a proton beam could be obtained rather easily at the T.C.C. cyclotron. Particle switching took ~ 1 hr, and the same time, or less, was necessary to raise and lower the yoke in order to work inside the dee chamber. Operation of the cyclotron is reasonably simple, and its controls are generally straightforward. Unfortunately, ^3He beams more intense than 8 to 10 μ a could not be obtained because of the economical but marginal design of the ion source. Since the cyclotron current integrator was observed to drift, the ^3He and the deuteron irradiations were repeated at the ORIC to check the above intensities by activation. If it is assumed that the ORIC current integrator is correct, the results indicate that the T.C.C. integrator read a current 2.3 to 3.7 times higher than the real one for ^3He particles; for high-intensity deuterons the correct current was found to be 17 to 40% lower than the one read at the T.C.C.

Some other problems arose, apparently always stemming from the difficult compromise between cost and reliability of operation; for example, the power supply for the deflector broke down and had to be replaced by a larger unit, and the deflector resistance was damaged and had to be complemented by another resistance.

Table 9.2. Neutron and Gamma-Ray Doses Measured at the T.C.C. Cyclotron

Normalized to beam intensities of 10 μ a

Nuclear Reaction	Location ^a	Neutron Dose (millirems/hr)		Gamma-Ray Dose (mr/hr)
		Fast	Thermal	
$^9\text{Be}(^3\text{He},n)^{11}\text{C}$	A	157	7	36
	B	1.3	0.2	0.1
$^9\text{Be}(d,n)^{10}\text{B}$	A	230	19	70
	B	3	0.5	4
$^7\text{Li}(p,n)^7\text{Be}$	A	160	15	100
	B	4	0.4	0.8

^aFor A, detectors were behind 40 in. of ordinary concrete, 58 in. from target in forward direction of beam. For B, they were behind 36-in.-thick concrete entrance sliding door, 144 in. from target at 90° angle with beam direction.

This cyclotron seems to be basically sound, generally reliable, and easy to operate. However, redesign of certain components appears to be necessary to assure minimum lost time in performance. In fact, the T.C.C. possibly has already solved most, if not all, these difficulties, because the cyclotron has recently passed the acceptance test required by the Sloan-Kettering Institute.

We hope to run similar experiments with the Philips cyclotron to compare the performance of the two accelerators.

9.3.c Proposed HFIR Activation Analysis Laboratory

F. F. Dyer L. C. Bate

A pneumatic transfer tube and a counting laboratory proposed for the HFIR were evaluated. The facility, designated the High-Flux Activation Analysis Laboratory (HFAAL), would be used for highly sensitive activation analysis and for other neutron-capture studies of interest at ORNL. The pneumatic tube would occupy vertical experimental facility 9 (VXF-9) in the permanent beryllium reflector. The counting laboratory would be located adjacent to the west wall on the first floor of the HFIR building. The Engineering Division has provided engineering drawings and cost estimates (\$102,000 for materials and labor).

For activation analysis this irradiation facility would have significant advantages over the facilities in other ORNL reactors. Table 9.3 compares the thermal-, resonance-, and fast-neutron fluxes for the HFAAL with those of the facilities in the ORR. A major advantage is the large thermal-neutron flux planned for the HFAAL. This flux would permit the activation-analysis sensitivities

for almost all elements that form short-lived radioisotopes to be increased by a factor of ~ 14 over those obtainable in the ORR pneumatic tube. Because irradiations for many hours (e.g., overnight) in graphite and metallic rabbits will be possible, sensitivities for elements that form long-lived radioisotopes will also be increased. For many types of samples the increased sensitivities will reduce the time required for activation analyses and will permit increased use of germanium-diode detectors. For certain types of samples, the proposed high ratio of thermal- to fast-neutron flux in the HFAAL will significantly reduce interferences caused by fast-neutron reactions.

9.3.d Photon and Fast-Neutron Activation Analysis at the Oak Ridge Electron Linear Accelerator (ORELA)

Enzo Ricci

In collaboration with J. A. Harvey,¹⁸ plans are being made to install a facility at the Oak Ridge Electron Linear Accelerator (ORELA) to carry out research and development on photon and fast-neutron activation analysis. The ORELA is currently being built on Laboratory grounds, and the activation facility will be under the control of the Analytical Chemistry Division.

The target room of the ORELA is a heavily shielded cylinder that has provision for three targets to be used alternatively. When one of the targets is in the position to receive the particle bombardment, the other two can be lowered into shielded containers located below floor level. Only one of these targets has been designed; it is the one required for most physics experiments

¹⁸Physics Division.

Table 9.3. Comparison of Neutron Fluxes of the ORR Facilities with Those Proposed for the HFAAL

Facility	Neutron Flux (neutrons cm ⁻² sec ⁻¹)			ϕ_{th}/ϕ_r	ϕ_{th}/ϕ_f
	Thermal (ϕ_{th})	Resonance (ϕ_r)	Fast (ϕ_f)		
ORR					
Pneumatic tube	4.2×10^{13}	1.5×10^{12}	1.6×10^{13}	28	2.6
Hydraulic tube	2×10^{14}	1×10^{13}	1×10^{14}	20	2
HFAAL	6×10^{14}	1×10^{13}	2×10^{13}	60	30

and will have tantalum as electron converter for neutron production. Our present plans call for construction of a second target for photon production, which will use tantalum or tungsten as electron converter; this target will be used mainly for activation analysis.

Samples and standards will be conveyed into and out of the target room by a pneumatic rabbit system. Irradiation locations will be provided both for photon and for fast-neutron activation. The former will be located past the target in the forward direction of the electron beam, where the photon flux is more intense; the latter will be at a 90° angle with the direction of the beam to obtain a higher proportion of neutrons.

9.3.e The 86-in. Cyclotron as a Source of Fast Neutrons

J. E. Strain W. S. Lyon

An experiment was conducted to determine the neutron flux and neutron energy obtainable from the 86-in. proton cyclotron located at Y-12. A thick (9.5-mm) beryllium target was positioned in the external beam of 22-Mev protons. At the maximum beam current of 5 μ a, samples of carbon, silicon, and germanium were irradiated for 30 sec in a position adjacent to the beryllium target. The >3.5 -Mev-neutron flux was only 2.9×10^9 neutrons $\text{cm}^{-2} \text{sec}^{-1}$. No neutron flux of energy greater than 20 Mev was detectable. Because the 150-kv Cockcroft-Walton generator will produce a 14-Mev-neutron flux of this same magnitude, the use of the 86-in. cyclotron for activation analysis appears to be impractical.

9.3.f Low-Level-Radiation Counting Facility

P. R. Bell¹⁹ J. S. Eldridge
V. A. McKay¹¹ G. D. O'Kelley⁴
R. E. Wintenberg¹¹

A low-level-radiation counting facility to serve as a prototype system for the Lunar Receiving Laboratory (LRL) at NASA, Houston, is being assembled at ORNL. The prototype is needed to

test materials and concepts under actual conditions before the final version is assembled. The goal is to achieve for the final facility a counting sensitivity greater than that of any previous device for the assay of natural and induced radioactivity in lunar and meteoritic materials by the detection of gamma rays.

Figure 9.3 is a diagram of the prototype system and gives details of some of its main features. The central detectors, 9- by 5-in. NaI(Tl) crystals, are housed in 5-mil-thick aluminum foil cans. An anticoincidence mantle (34 in. in diameter, 50 in. high) made of NE-102 plastic scintillator surrounds the main detectors. This detector assembly (NaI detectors + anticoincidence mantle) is housed in an 8-in.-thick lead shield of special construction. The lead shield is covered with a thin anticoincidence mantle as an active muon shield.

The lead shield was cast in place by use of a "lead concrete" made of Chemtree 82, lead shot, 1% LiF, and water. The LiF and water serve to thermalize neutrons, which are produced in the lead shield by muon interaction, and thus to shorten their lifetime. The density of the prototype shield is about 9.5 g/cm^3 .

Figure 9.4 shows a preliminary background in the shield without either of the anticoincidence mantles connected. The pulse outputs of the two NaI(Tl) detectors were summed. Under those conditions, the background was 1170 counts/min for the energy range 0.1 to 2.0 Mev or 0.112 counts/min per cubic centimeter of NaI(Tl). Connecting the inner mantle in anticoincidence (with less than optimum performance) lowered the background to 387 counts/min or 0.037 count/min per cubic centimeter of NaI(Tl) for the same energy range. Figure 9.5 shows the spectrum of the background made for the latter conditions. The prominent peak in the spectrum at channel 90 is due mainly to ^{40}K from the glass in the 28 phototubes that are attached to the NaI(Tl) detectors and anticoincidence mantle within the shield. The peak at channel 140 is due to capture gamma rays that accompany the neutron-capture events in the hydrogen of the inner mantle. Final operation with the outer mantle is expected to eliminate or to greatly decrease the contribution from neutron-capture events.

Overall design goals for the final system in Houston call for an effective background level between 0.001 and 0.02 count/min per cubic

¹⁹Chief of Lunar and Earth Sciences Division and Manager of the Lunar Receiving Laboratory, Manned Spacecraft Center, National Aeronautics and Space Administration, Houston, Tex.

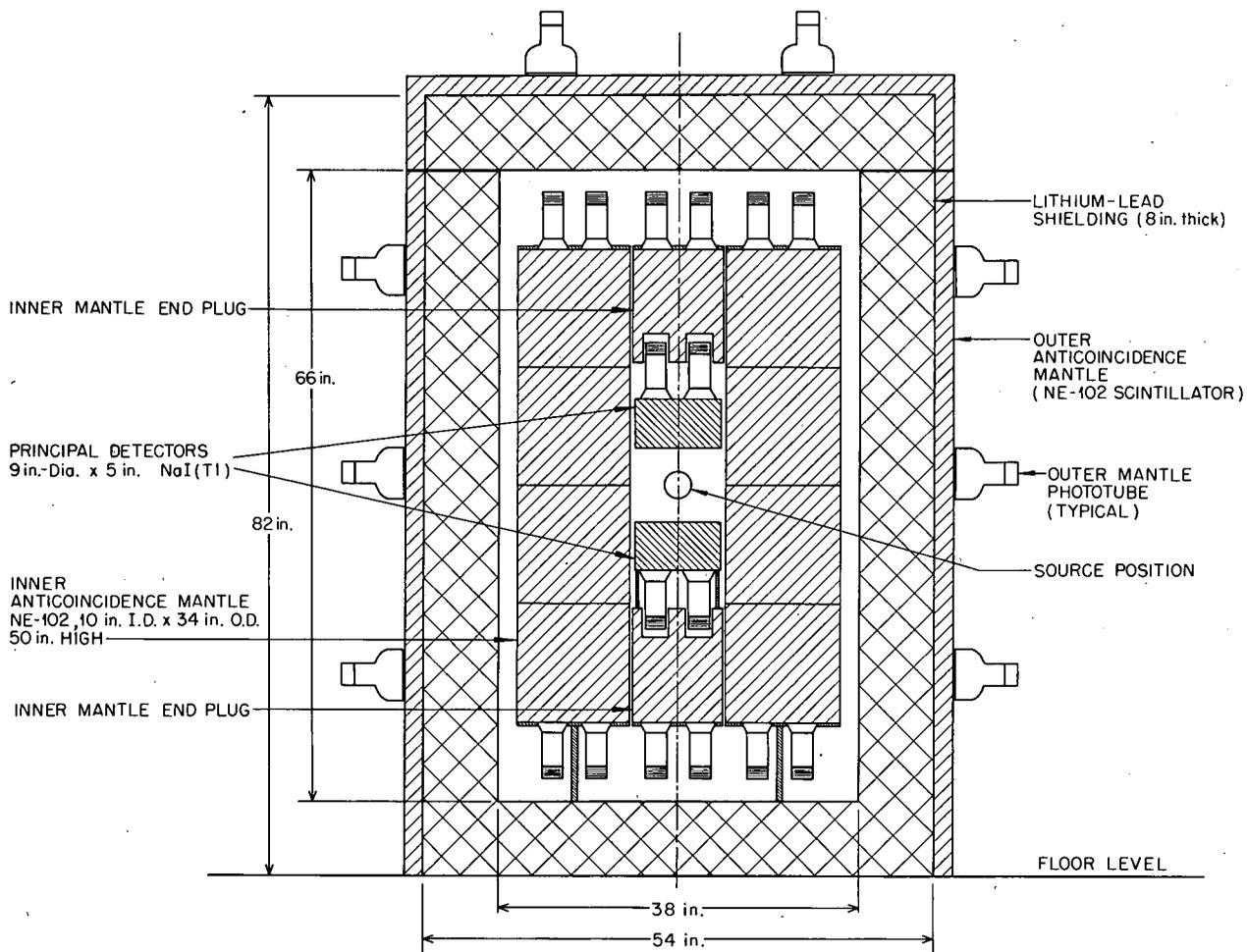


Fig. 9.3. Schematic Diagram of Prototype Low-Level-Radiation Counting Facility. Outer anticoincidence mantle and outer-mantle phototubes are out of proportion.

centimeter of detector in the energy range 0.1 to 2.0 Mev.²⁰ Since measurements under less than ideal conditions at ORNL have produced a background as low as 0.037 count/min per cubic centimeter of NaI(Tl), the design goals for the LRL should be attained with the entire system operational and the extra shielding afforded by the 40 ft of overburden (the LRL counting room is 50 ft below ground).

The prototype counting system will be a permanent installation at ORNL and will be operated with a 4096-channel dual-parameter analyzer system. It will be useful in the quantitative analysis of numerous low-level radionuclide mixtures. It

can be used to study the natural and induced radioactivity in meteorites and the natural and artificial radionuclides in many types of bulk samples such as ocean sediments and various environmental materials.

9.4 ACTIVATION ANALYSIS

9.4.a Interactions of ³He Particles with Boron, Nitrogen, Sodium, and Beryllium

R. L. Hahn⁴ Enzo Ricci

Thick-target yields for reactions of ³He particles with B, N, Be, and Na had been determined by irradiating boron, beryllium nitride, and sodium iodide

²⁰J. C. McLane, Jr., "Collecting and Processing Samples of the Moon," *Astronaut. Aeron.* 5(8), 34 (1967).

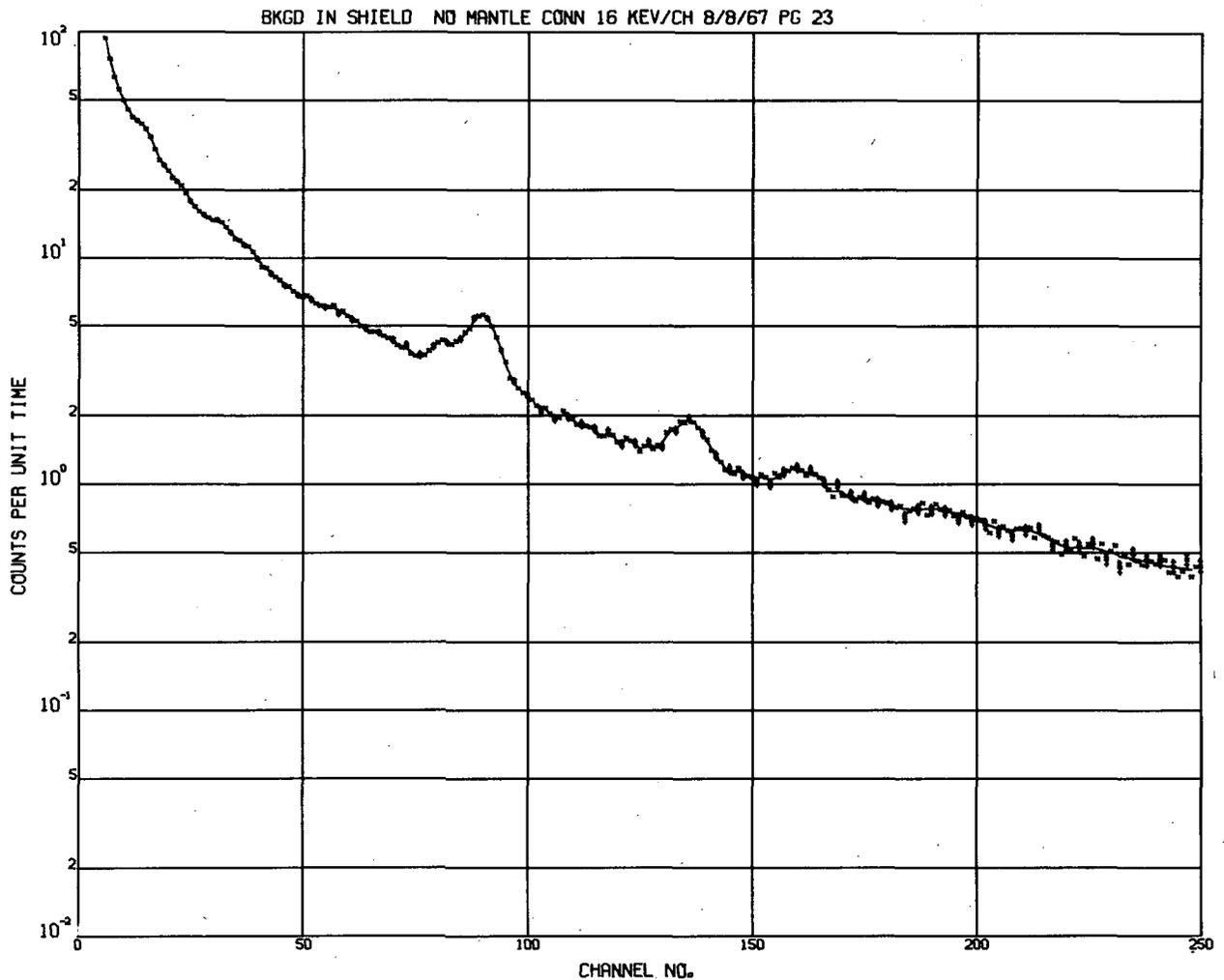


Fig. 9.4. Background Spectrum with Two 9- by 5-in. NaI(Tl) Detectors Summed Without Anticoincidence Inside ORNL Shield. Ordinate gives counts per minute.

with ^3He ions of energies from 6 to 18 Mev.²¹ Excitation functions for the reactions $^{11}\text{B}(^3\text{He},n)^{13}\text{N}$, $^{10}\text{B}(^3\text{He},d)^{11}\text{C}$, $^{14}\text{N}(^3\text{He},\alpha)^{13}\text{N}$, $^{14}\text{N}(^3\text{He},d)^{15}\text{O}$, $^9\text{Be}(^3\text{He},n)^{11}\text{C}$, and $^{23}\text{Na}(^3\text{He},2p)^{24}\text{Na}$ were obtained by numerical differentiation of the smoothed yield vs energy curves. The distorted-wave theory of nuclear reactions²² was used to compute excitation functions for comparison with the data for the $^{14}\text{N}(^3\text{He},\alpha)^{13}\text{N}$ reaction and for

the $(^3\text{He},d)$ reactions on ^{14}N and ^{10}B . Optical-model potentials, not for the particular nuclides observed, but determined for neighboring light nuclei, were used in the calculations. This comparison of theoretical with experimental data indicates that the $^{14}\text{N}(^3\text{He},\alpha)$ and $^{10}\text{B}(^3\text{He},d)$ reactions proceed mainly by direct pickup and by stripping processes respectively. For the $^{14}\text{N}(^3\text{He},d)$ reaction a stripping calculation, in which were considered only those ^{15}O states with known spins and parities, yielded a lower limit of $\sim 30\%$ of the observed excitation function.

A paper on this work was published.²³

²¹R. L. Hahn and E. Ricci, "Thick Target Yields from Reactions of ^3He Particles with Boron, Nitrogen, and Sodium," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1966*, ORNL-4039, p. 64.

²²R. 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).

²³R. L. Hahn and E. Ricci, "Interaction of ^3He -Particles with Boron, Nitrogen, Sodium, and Beryllium," *Nucl. Phys. A101(2)*, 353 (1967).

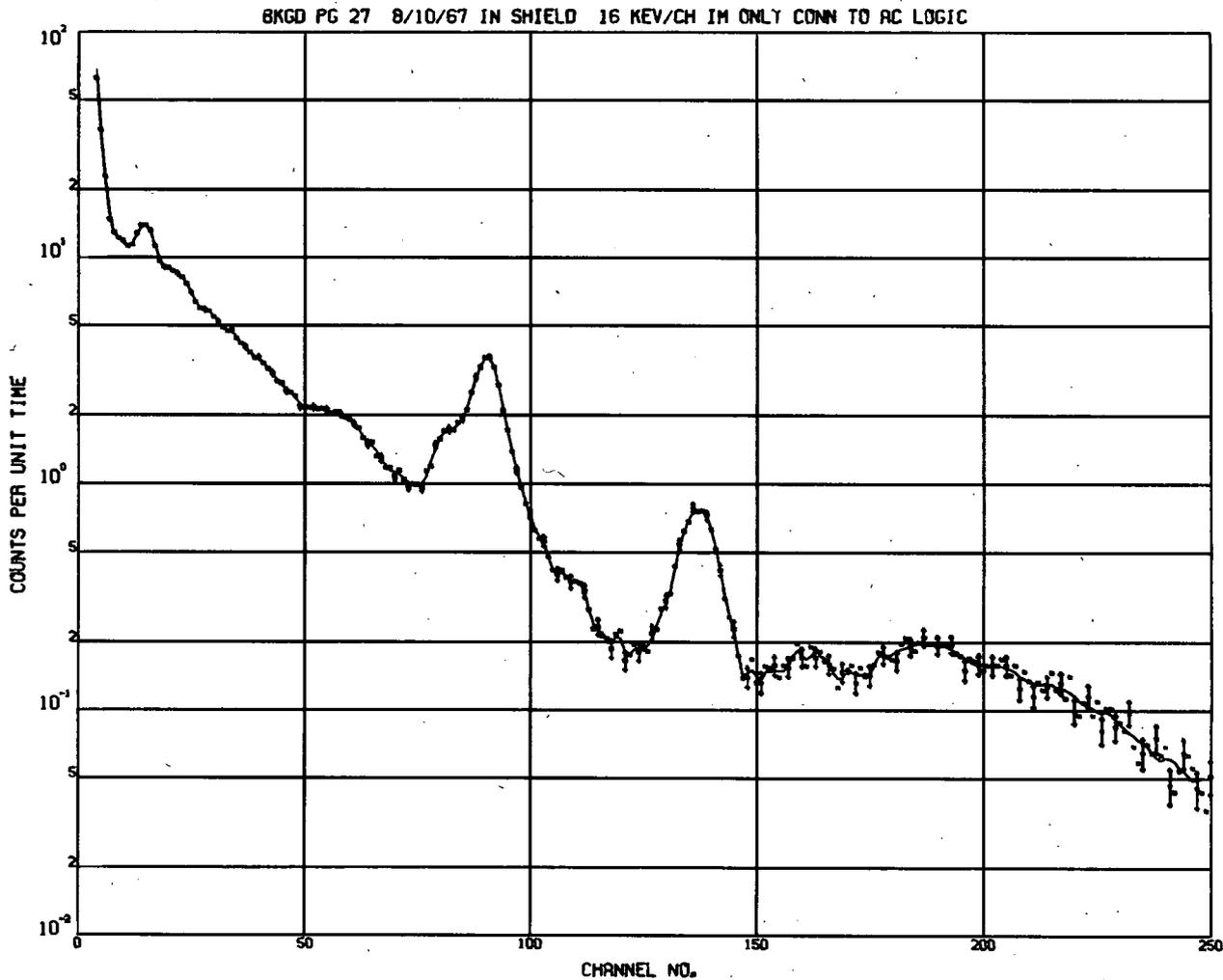


Fig. 9.5. Background Spectrum with Two 9- by 5-in. NaI(Tl) Detectors Summed with Inner Mantle Anticoincidence Inside ORNL Shield. Ordinate gives counts per minute.

9.4. b Simple Method to Calculate Sensitivities, Interferences, and Optimum Bombardment Energies in ^3He -Activation Analysis

Enzo Ricci R. L. Hahn⁴

To facilitate calculations of sensitivities and interferences in terms of bombarding energies in ^3He -activation analysis, an approximate method was developed for the rapid computation of the average cross section, $\bar{\sigma}$.^{24,25} This energy-

²⁴E. Ricci and R. L. Hahn, "Theory and Experiment in Rapid, Sensitive Helium-3 Activation Analysis," *Anal. Chem.* **37**, 742 (1965).

²⁵E. Ricci and R. L. Hahn, "Sensitivities for Activation Analysis of 15 Light Elements with 18 Mev Helium-3 Particles," *Anal. Chem.* **39**, 794 (1967).

dependent variable is difficult to obtain by rigorous mathematics, and yet it is vital in these calculations, because the analytical sensitivity is directly proportional to $\bar{\sigma}$ (ref. 24) and because the interference depends on the ratio of cross sections, $\bar{\sigma}_i/\bar{\sigma}_s$, for the interfering and the sought reactions. In fact, the familiar charged-particle activation equation²⁴ leads to the following expression for the relative interference F :

$$F = \frac{D_i}{D_s} = \frac{n_i(1 - e^{-\lambda_i t})}{n_s(1 - e^{-\lambda_s t})} \frac{\bar{\sigma}_i}{\bar{\sigma}_s}, \quad (9.1)$$

where D_i and D_s are the disintegration rates of the interfering and the sought radionuclides, re-

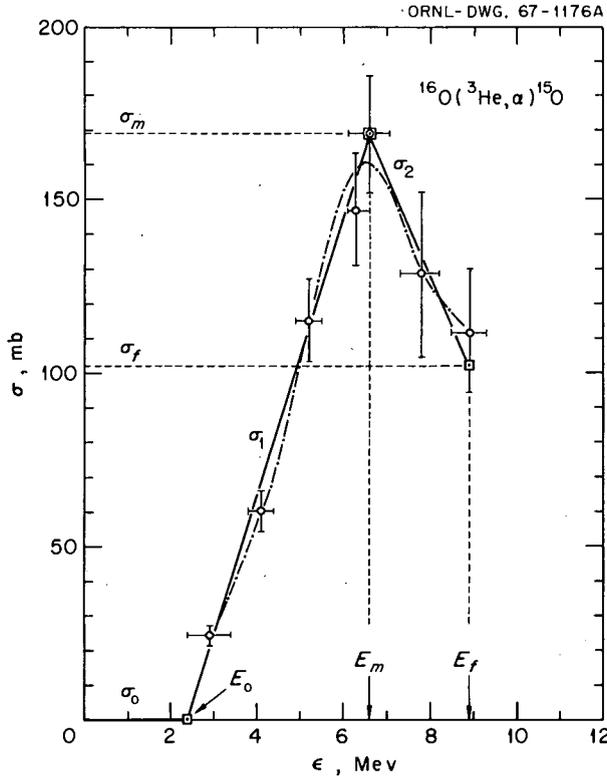


Fig. 9.6. Straight-Line (—) Fitting of the Excitation Function (---) for the Reaction $^{16}\text{O}(^3\text{He}, \alpha)^{15}\text{O}$. \circ designates experimental points taken from ref. 26.

spectively, t is the length of irradiation, the n 's are numbers of target atoms per unit weight of sample, and the λ 's are decay constants. Clearly, the optimum bombarding energy is one that corresponds to a minimum value for F .

Interference may become serious in ^3He activation, because most of its products are positron emitters, which can be determined best by measuring their common 0.511-Mev gamma rays or by beta counting. Moreover, elements of neighboring Z may produce the same radioactive product. In this particular case, because $\lambda_i = \lambda_s$, Eq. (9.1) reduces to

$$F = \frac{n_i \bar{\sigma}_i}{n_s \bar{\sigma}_s} \quad (9.2)$$

Our method is based on the straight-line fitting of excitation functions,^{26,23} which facilitates

²⁶R. L. Hahn and E. Ricci, "Interactions of ^3He Particles with ^9Be , ^{12}C , ^{16}O , and ^{19}F ," *Phys. Rev.* 146, 650 (1966).

integration of the definition of $\bar{\sigma}$:

$$\bar{\sigma} = \frac{\int_0^E \sigma \epsilon d\epsilon}{\int_0^E \epsilon d\epsilon} = \frac{2}{E^2} \int_{E_0}^E \sigma \epsilon d\epsilon, \quad (9.3)$$

where E is the bombarding energy, σ is the value of the excitation function at energy ϵ , and E_0 is either the corrected Coulomb barrier²⁷ or the threshold energy of the reaction, whichever is greater. In Fig. 9.6 the fitting procedure is illustrated for the reaction $^{16}\text{O}(^3\text{He}, \alpha)^{15}\text{O}$, where σ_0 , σ_1 , and σ_2 are the approximated values of the cross section within different energy intervals of the excitation function, σ_m is the cross section at the maximum of the fitting line and corresponds to energy E_m , and σ_f is the cross-section value that corresponds to E_f , which is the maximum particle energy used in the fitting procedure.

The excitation curves can be expressed by the equations

$$\sigma_0 = 0 \quad \text{for the interval } 0 \leq \epsilon \leq E_0, \quad (9.4)$$

$$\sigma_1 = m_1 (\epsilon - E_0) \quad \text{for the interval } E_0 \leq \epsilon \leq E_m, \quad (9.5)$$

$$\sigma_2 = m_2 (\epsilon - E_m) + \sigma_m \quad \text{for the interval } E_m \leq \epsilon \leq E_f, \quad (9.6)$$

where

$$m_1 = \frac{\sigma_m}{E_m - E_0}$$

and

$$m_2 = \frac{\sigma_f - \sigma_m}{E_f - E_m}.$$

Substituting Eqs. (9.4), (9.5), and (9.6) in Eq. (9.3) and integrating leads to the simple equations

$$\bar{\sigma} = \frac{2}{3} m_1 E - a_1 + \frac{b_1}{E^2}$$

$$\text{for the interval } E_0 \leq E \leq E_m, \quad (9.7)$$

²⁷I. Dostrovsky, Z. Fraenkel, and G. Friedlander, "Monte Carlo Calculations of Nuclear Evaporation Processes. III. Applications to Low-Energy Reactions," *Phys. Rev.* 116, 683 (1959).

$$\bar{\sigma} = \frac{2}{3} m_2 E - a_2 + \frac{b_2}{E^2}$$

$$\text{for the interval } E_m \leq E \leq E_f, \quad (9.8)$$

which can be used directly to calculate $\bar{\sigma}$ in terms of E . The constants m_1 , m_2 , a_1 , a_2 , b_1 , and b_2 have been computed for the 12 most significant nuclear reactions of ^3He ions with the elements that display the highest sensitivities in ^3He activation: Be, B, C, N, O, and F. These values can be easily applied to calculations of ^3He -activation sensitivities, interference ratios, and optimum bombarding energies. By comparison of the results of this method with data obtained either by experiment or by graphical integration of excitation functions, the average error was found to be $\leq 13.5\%$. A paper on this subject was accepted for publication.²⁸

9.4.c Possible Use of Recoil Nuclei in Activation Analysis

R. L. Hahn⁴

The nuclei-recoil technique,²⁹ used extensively in nuclear research, involves the collection and assay of radioactive product nuclei that recoil out of a target during irradiation. As a consequence of the physics of the recoil process, for a given irradiation energy, light recoil nuclei in most instances have larger kinetic energies than do heavy recoil nuclei (see Table 9.4). It may be possible to use these differences in recoil properties to separate the different radioactive products of an irradiation.

To evaluate this possibility, foils of copper metal, previously heated at 900°C to cover their surfaces with CuO, were irradiated with ^3He particles at the ORIC. After each irradiation the Cu-CuO target and an accompanying aluminum recoil catcher were assayed with a gamma-ray spectrometer. Figure 9.7 shows typical results; the differences between target and catcher are

²⁸E. Ricci and R. L. Hahn, "Rapid Calculation of Sensitivities, Interferences, and Optimum Bombarding Energies in ^3He -Activation Analysis," accepted for publication in *Analytical Chemistry*.

²⁹B. G. Harvey, "Recoil Techniques in Nuclear Reaction and Fission Studies," *Ann. Rev. Nucl. Sci.* 10, 235 (1960).

Table 9.4. Calculated Maximum Recoil Energies (E) for Reactions Induced by 20-Mev ^3He Particles^a

Reaction	Q^b (Mev)	E^a (Mev)
$^{16}\text{O}(^3\text{He},p)^{18}\text{F}$	+2.0	7.4
$^{65}\text{Cu}(^3\text{He},2n)^{66}\text{Ga}$	-4.8	2.5 ^c
$^{65}\text{Cu}(^3\text{He},\alpha)^{64}\text{Cu}$	+10.7	5.0
$^{65}\text{Cu}(^3\text{He},n)^{67}\text{Ga}$	+6.5	2.4
$^{63}\text{Cu}(^3\text{He},n)^{65}\text{Ga}$	+3.9	2.4
$^{63}\text{Cu}(^3\text{He},2n)^{64}\text{Ga}$	-7.9	2.3 ^c
$^{63}\text{Cu}(^3\text{He},\alpha)^{62}\text{Cu}$	+9.7	5.0
$^{12}\text{C}(^3\text{He},\alpha)^{11}\text{C}$	+1.9	15.2
$^{12}\text{C}(^3\text{He},d)^{13}\text{N}$	+10.0	17.1
$^{16}\text{O}(^3\text{He},\alpha)^{15}\text{O}$	+4.9	13.8
$^{28}\text{Si}(^3\text{He},p)^{30}\text{P}$	+6.3	5.1
$^{31}\text{P}(^3\text{He},\alpha)^{30}\text{P}$	+8.3	9.1
$^{127}\text{I}(^3\text{He},n)^{129}\text{Cs}$	+5.5	1.2
$^{127}\text{I}(^3\text{He},\alpha)^{126}\text{I}$	+11.4	2.7
$^{238}\text{U}(^3\text{He},n)^{240}\text{Pu}$	+4.0	0.7 ^d
$^{238}\text{U}(^3\text{He},\alpha)^{237}\text{U}$	+14.4	1.6 ^d

^aThe recoiling nucleus has maximum energy when it emerges from the target at an angle of 0° with respect to the ^3He beam.

^b Q is the energy released during the nuclear reaction.

^cValues based on the assumption that both neutrons were emitted at the same angle, 180° .

^dThe classical Coulomb barrier has the value 23 Mev for the reaction of ^3He with ^{238}U . Because of quantum-mechanical effects, the reaction will proceed at 20 Mev.

readily apparent. The Cu-CuO target has a decay curve that is complex and a gamma-ray spectrum that contains the prominent 1.04- and 2.75-Mev peaks of ^{66}Ga from the reaction $^{65}\text{Cu}(^3\text{He},2n)^{66}\text{Ga}$. The catcher spectrum, on the other hand, consists almost entirely of the 0.51-Mev gamma ray, with small contributions from higher-energy peaks. The decay data show that $\sim 98\%$ of the catcher activity is due to ^{18}F from the $^{16}\text{O}(^3\text{He},p)^{18}\text{F}$ reaction, whereas in the target the $^{18}\text{F}/^{66}\text{Ga}$ activity ratio is about 2/1. Thus, differences between the recoil properties of the ^{18}F and ^{66}Ga nuclei served to effectively separate these radio-nuclides.

This result indicates that the nuclei-recoil technique has potential use in activation analysis, especially for the determination of surface impurities, such as oxygen or carbon, on medium-weight elements. Of course, details of the recoil

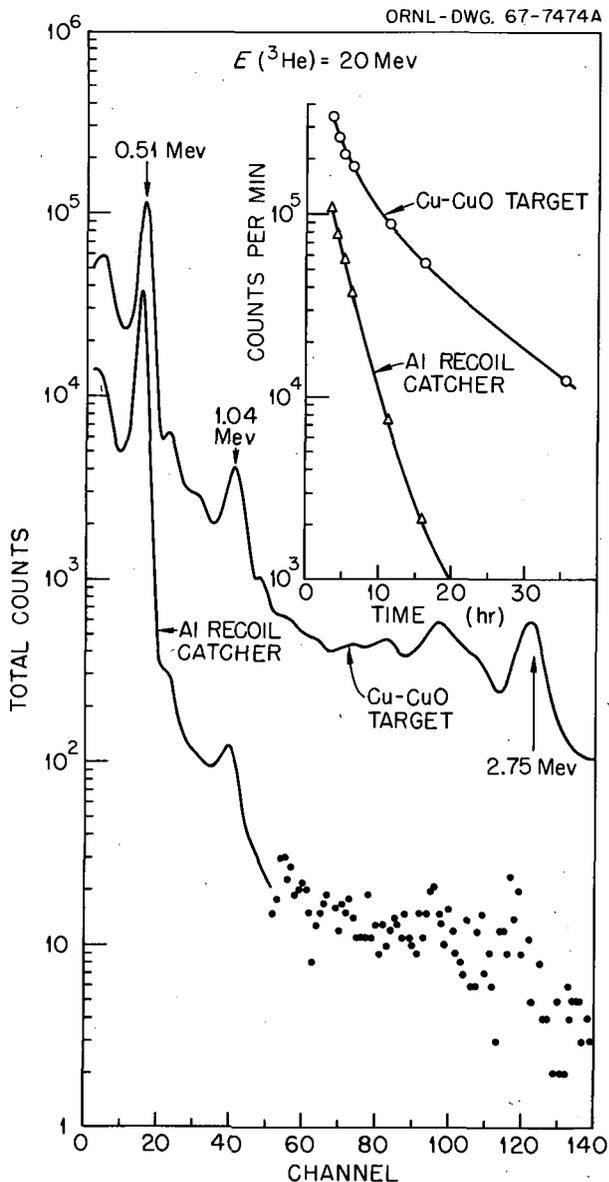


Fig. 9.7. Gamma-Ray Spectra for a Cu-CuO Target and an Aluminum Recoil Catcher. Inset shows corresponding decay curves for the 0.51-Mev gamma rays.

phenomena must be studied before the method can be applied to an actual analysis.

A note that describes this work will be published.³⁰

9.4.d Applied Activation Analysis

J. F. Emery

Samples of paint from two paintings, one thought to be a Rembrandt and the other from the Dutch

School, were analyzed for silver. That from the possible Rembrandt contained 32 ppm Ag and 70 ppm Co; that from the other contained 16 ppm Ag and 20 ppm Co. Paints produced before ~1850 and after ~1850 are known to contain 18 to 27 and 0.5 to 20 ppm Ag respectively.³¹ Other elements found in these paints were Al, Au, Ba, Cl, Co, Mn, Na, and Sb; all seemed to be present to the same extent in each specimen.

The pneumatic tube of the ORR is used to determine ^{235}U by counting the delayed neutrons produced after fission. The delayed-neutron background of this system was equivalent to 1×10^{-9} g of ^{235}U . After rupture of a rabbit that contained ^{233}U , used in an experiment for the Chemistry Division to determine independent fission yields, and subsequent cleanup of the system, the delayed-neutron background was equivalent to 70×10^{-9} g of ^{235}U . Any further cleanup was considered to be impossible, and a new in-pile section of the pneumatic tube was installed. The delayed-neutron background of this system is equivalent to 0.5×10^{-9} g of ^{235}U .

Several samples of bismuth metal powder to be used for shielding in a gamma-ray telescope were examined for the presence of low-level radioactivity. They contained 0.1 picocurie of ^{207}Bi per gram of powder. Samples of bismuth powder received later and marked "obtained from virgin ore" were free of ^{207}Bi . No explanation was given by the manufacturer for the presence of ^{207}Bi in the original samples.

9.4.e Contribution to the *International Encyclopedia of Pharmacology and Therapeutics*

Enzo Ricci

A chapter titled "Radioactivation" was written to be included in the *International Encyclopedia of Pharmacology and Therapeutics*.³²

³⁰R. L. Hahn, "The Recoil Technique and Its Possible Use in Activation Analysis," accepted for publication in *Analytical Chemistry*.

³¹J. P. W. Houtman and J. Turkstra, "Neutron Activation Analysis and Its Possible Application for Age Determination of Paintings," p. 85 in *Radiochemical Method of Analysis*, vol. I (Proceedings of the Symposium on Radiochemical Methods of Analysis held by the International Atomic Energy Agency at Salzburg, Oct. 19-23, 1964), IAEA, Vienna, 1965.

³²E. Ricci, "Radioactivation," to be included as chap. IIa of the *International Encyclopedia of Pharmacology and Therapeutics*, to be published by Pergamon Press Ltd.

9.5 14-Mev NEUTRON GENERATOR

9.5.a 14-Mev Neutron Activation Analysis

J. E. Strain

The 14-Mev neutron generator for research and for neutron activation analysis (Texas Nuclear Corporation model 9900) has been used in a variety of programs to determine oxygen in aluminum, in zirconium, and in ^{99}Tc and chlorine in organic compounds that are resistant to conventional pyrolysis methods.

In collaboration with N. C. Dyer³³ the formation and decay characteristics of ^{58}Mn are being studied. This isotope is formed by neutron bombardment of an enriched ^{58}Fe target.

9.5.b 14-Mev Neutron Activation Analysis for Oxygen in Alkali and Refractory Metals

J. E. Strain

The facility³⁴ designed for the determination of oxygen in alkali and refractory metals by 14-Mev neutron activation is now complete. Operational tests showed the necessity to increase the neutron shielding and to redesign the rabbit and the rabbit-stopping mechanism. The analysis of standard samples that contained known amounts of oxygen indicated that the sensitivity of the method is 2350 counts per milligram of oxygen (10-sec irradiation, 1.7-ma beam current, and 30-sec counting interval). The sensitivity will increase as higher beam currents are attained. The counter background is 200 counts per 30 sec and is independent of the neutron flux.

9.5.c Fluorine Determination by 14-Mev Neutron Activation Analysis

L. C. Bate F. F. Dyer

At the request of the U.S. Bureau of Customs a study was made to ascertain whether 14-Mev neutron activation analysis is suitable for measur-

³³Research Associate, Vanderbilt University, Nashville, Tenn.

³⁴J. E. Strain, "14-Mev Neutron Activation Analysis for Oxygen in Alkali Metals," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1966, ORNL-4039*, p. 75.

ing fluorine in fluorspar ores. The Bureau desires a method that is faster than the currently used wet-chemistry methods and that has an error no greater than about $\pm 0.1\%$. The method was shown to be precise and rapid, but the results may be slightly biased by activation of impurities in the ore.

The activation method is based on the measurement of ^{18}F produced by the reaction $^{19}\text{F}(n,2n)^{18}\text{F}$. Two standards and three unknown samples are irradiated simultaneously in the biaxial sample rotator (Sect. 9.5.d). Crystalline CaF_2 obtained from Harshaw Chemical Company was used for standards. The induced ^{18}F is measured in a well-type scintillation detector after a 1-hr decay period. Measurements of fluorine in reagent-grade CaF_2 have consistently indicated a bias of about +1.0%. Results obtained for three samples of fluorspar were within $\pm 0.5\%$ of the values obtained by wet-chemistry methods.

9.5.d A Biaxial Sample Rotator for 14-Mev Neutron Irradiation

F. F. Dyer L. C. Bate
J. E. Strain

Because the neutron flux of 14-Mev neutron generators changes markedly with distance from the target and with duration of the irradiation, the measurement and reproduction of the neutron dose to which samples are exposed are difficult. To obtain uniform activation of samples, a new type of sample holder was developed that rotates several samples around two axes.

The rotator (Fig. 9.8) consists of two concentric plastic cylinders; the inner one holds the samples in cylindrical form (molded or machined material, powders in vials, etc.). The outer cylinder is rotated end over end by a motor-driven hollow shaft, the inner one by an air jet. The air jet is generated by applying a vacuum through the hollow drive shaft of the outer cylinder. Air enters the outer cylinder through a hole bored tangentially to the vaned surface of the inner cylinder, strikes this surface, and causes the inner cylinder to rotate.

The achievement of highly uniform neutron activation of samples with the rotator has been demonstrated by measurements of ^{18}F induced in samples of Teflon and of CaF_2 . The device has

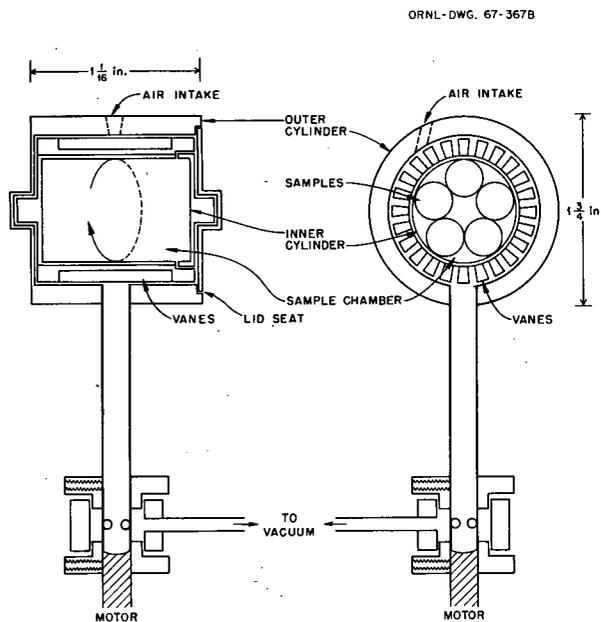


Fig. 9.8. Schematic Diagram of Biaxial Sample Rotator for 14-Mev Neutron Irradiations.

been used to precisely determine fluorine in synthetic and natural fluorspar (Sect. 9.5.c). The method of use and results obtained with the rotator are being published.³⁵

9.5.e Monitoring of Leached Fuel Elements with a 14-Mev Neutron Generator

J. E. Strain W. J. Ross

Development of the leached-fuel-element monitor was completed; a report summarizes its design and performance.³⁶ The most significant advance during the year was the use of a ^{10}B -lined neutron detector in the monitor. This detector, developed by the Reuter-Stokes Corporation, is essentially independent of gamma-radiation dose rates as high as 10^5 r/hr. Its inclusion in the leached-hull monitor allows the use of the monitor on any type of fuel cladding independently of fuel burnup or cooling time.

As a result of the recovery and reprocessing of Ni-Th alloy used in aircraft turbine blades, ^{232}Th is becoming an increasingly frequent contaminant

³⁵F. F. Dyer, L. C. Bate, and J. E. Strain, "A 3-Dimensionally Rotating Sample Holder for 14-Mev Neutron Irradiations," accepted for publication in *Analytical Chemistry*.

in stainless steel. To evaluate the effect of ^{232}Th in the leach-hull monitor, the delayed-neutron yields of ^{232}Th and of ^{235}U were compared. One gram of ^{235}U was found to produce the same delayed-neutron count as 1620 g of ^{232}Th . Therefore, ^{232}Th will not introduce an analysis error at any foreseeable contamination level.

9.6 COOPERATIVE ISOTOPES PROGRAM

9.6.a Technical Assistance

S. A. Reynolds

The joint program³⁷ of the Isotopes and Analytical Chemistry Divisions to characterize and analyze isotopes has continued. Technical assistance includes answering inquiries, making calculations relative to production and purity of radioisotope products, and reviewing and participating in research on properties and analysis of isotopes. Some of these activities are reported in the following subsections and in other sections of this report, as well as in a paper.³⁸ The problem of measurement of fission-spectrum neutron flux continues to be studied by ASTM Committee E-10. The work is being followed through membership on the appropriate task group.

9.6.b Quality Control

S. A. Reynolds

The topic of quality control encompasses analytical methods for radioactive and stable constituents of isotope products. Radioactivity standards were obtained from various suppliers; agreement among their measurements and local ones continues to be good. A summary of the availability of radioactivity standards was accepted for publication.³⁹ Ninety-three nuclides were listed as

³⁶J. E. Strain, W. J. Ross, G. A. West, and J. W. Landry, *Design and Evaluation of a Delayed-Neutron Leached-Hull Monitor*, ORNL-4135, in press (1967).

³⁷"Cooperative Isotopes Program," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1966*, ORNL-4039, pp. 60-62.

³⁸S. A. Reynolds, "Radioisotope Characteristics, Measurements, and Standards," *Isotopes Radiation Technol.* 4(1), 46 (1966).

³⁹S. A. Reynolds, "Availability of Standards for Common Radionuclides," accepted for publication in *Isotopes and Radiation Technology*.

having significant current interest. Of these, 21 standards – solution or solid – are available from the NBS and 18 from a leading commercial supplier; six of the latter duplicate the former. The International Atomic Energy Agency (IAEA), Vienna, supplies standards for eight of the nuclides, and the Radiochemical Centre, Amersham, England, supplies them for 67. Twelve that are not available in the United States were listed as being urgently needed because of substantial use of the nuclides, difficulty of precise measurement, or both; they include ^{47}Ca , ^{75}Se , $^{99\text{m}}\text{Tc}$, ^{113}Sn , ^{133}Xe , and ^{241}Pu . Because of the perennial problem of poor quality of some commercial standards, the criteria⁴⁰ of the National Research Council subcommittee on standards were briefly reviewed in the paper.

In connection with production of ^{132}Te - ^{132}I , methods of assay and determination of impurities were reviewed. Products in radioactive equilibrium can be measured by a calibrated gamma ionization chamber. Determination of ^{131}I requires radiochemical separation and overnight decay to remove ^{132}I . The possible contaminants ^{95}Zr - ^{95}Nb , ^{99}Mo , and ^{103}Ru can be determined by conventional methods. Other products for which methods have been reviewed or modified, or special analyses done, have included ^{36}Cl , ^{45}Ca , ^{47}Ca , ^{84}Rb , ^{91}Y , ^{95}Zr - ^{95}Nb , and ^{181}Hf .

9.6.c Characterization of Isotope Materials

S. A. Reynolds J. F. Emery L. G. Farrar¹⁷
 J. E. Morton⁴¹ J. S. Wike⁴²

The heat output of the ^{90}Sr - ^{90}Y equilibrium mixture was redetermined. The calorimetric measurement will be described elsewhere by J. A. Setaro⁴³ and C. L. Ottinger.⁴³ Analytical work consisted in confirmation of purity by extraction and measurement of ^{90}Y and by gamma spectrometry (only bremsstrahlung was found). Attainment of equilibrium was confirmed by re-counting selected beta mounts and observing that no growth or decay occurred. The actual measurement was

⁴⁰S. B. Garfinkel, A. P. Baerg, and P. E. Zigman, *Certificates of Radioactivity Standards*, Natl. Acad. Sci.-Natl. Res. Council, Washington, D.C., 1966.

⁴¹Radioisotopes-Radiochemistry Laboratory.

⁴²General Hot-Analyses Laboratory.

⁴³Isotopes Division.

made by gross beta counting with end-window proportional counters that had been calibrated previously. The calibration was checked by use of an IAEA ^{90}Sr standard; the result was 1% lower than the IAEA value, which was certified to $\pm 1\%$; the relative standard deviation was $\sim 2\%$ overall, and this is very satisfactory. The value is 6.86 w/kilocurie, in good agreement with values obtained by other investigators who used calorimetric and beta-spectral determinations.

A sample of dust suspected to contain erbium tritide was analyzed for erbium by emission spectroscopy and for tritium by thermal decomposition, oxidation over hot CuO ,⁴⁴ and trapping in chilled water, which was subsequently counted by liquid scintillation. The tritium content was estimated as about one atom per atom of erbium. Thus the material contained a substantial amount of tritide as well as an inert erbium salt, presumably the hydrated oxide.

By combustion and by wet oxidation $^{137}\text{CsCl}$ products were shown to contain < 0.1 wt % carbon. Work is continuing in an effort to increase sensitivity of the method for carbon and to determine the chemical form of any trace carbon that may be found.

Activation analysis was used to determine inactive phosphorus in an old ^{32}P product; the value was 0.20 ppm. The original ^{32}P content was 1.35 ppm (386 mc/ml); this concentration corresponds to an isotopic abundance of 2.5×10^5 curies/g, which is 88% of that of pure ^{32}P .

9.6.d High-Energy Gamma Radiations from ^{233}U , ^{239}Pu , and ^{241}Am

S. A. Reynolds J. F. Emery

Samples of high-purity ^{233}U , ^{239}Pu , and ^{241}Am were examined by gamma spectrometry in a search for high-energy gamma radiations that might interfere with the intended uses of these radionuclides. The gammas detected are listed in Table 9.5, together with the familiar low-energy ones associated with the principal components.

The 0.44- and 1.56-Mev radiations in the ^{233}U sample are from its daughters ^{213}Bi and ^{209}Tl . If available decay-scheme data⁴⁵ are assumed to

⁴⁴T. H. Handley, *Release of Tritium from Tritium-Labeled Luminous Paints*, ORNL-TM-1478 (January 1966).

⁴⁵*Nuclear Data B*1(5), 1,3 (1966).

Table 9.5. Gamma Radiation Intensities in Samples

Sample	Gamma Radiation	
	E_γ (Mev)	Percent of Principal Radioactivity
^{233}U	0.44	4
	1.56	0.3
^{239}Pu	0.38	6.7
	0.76	1
	0.63-0.66	0.08
^{241}Am	0.34	1.6
	0.66	0.6

be correct, the bismuth and thallium levels correspond to a time of ingrowth since last separation of ~ 1.5 years, which is in agreement with the history of the material.

It is well known that in ^{239}Pu there is a group of gammas of average energy ~ 0.38 Mev. The observed intensity was in good agreement with the recently reported values $6.6 \times 10^{-3}\%$ ⁴⁶ and $6.4 \times 10^{-3}\%$.⁴⁷ The peak at ~ 0.76 Mev was first thought to be due to ^{238}Pu ,⁴⁸ but its intensity was too high. It was surmised that the peak came from the contaminant fission products ^{95}Zr and ^{95}Nb , and this was confirmed by use of a Ge(Li) spectrometer. Also, several peaks were detected between 0.63 and 0.66 Mev that are believed to be identical with radiation of "650 \pm 20 kev," which is reported to have an intensity of $(8 \pm 3) \times 10^{-5}\%$ in ^{239}Pu .⁴⁹ Gammas of similar energies reported for ^{240}Pu and ^{241}Am would not have so high an intensity in this plutonium sample, based on its reported mass analysis.

In ^{241}Am , the 0.34-Mev radiation is due to a combination of radiation from the ^{233}Pa daughter

⁴⁶R. S. Forsyth and N. Ronquist, *A Study of the "384" Kev Complex Gamma Emission from Plutonium-239*, AE-202 (November 1965).

⁴⁷A. J. Fudge, A. Phillips, and E. Foster, *Non-Destructive Determination of Plutonium-239 in Ceramic Fuel Element Pellets by Gamma Spectrometry*, AERE-R-3838 (January 1962).

⁴⁸W. W. Strohm, "765-keV Gamma Ray Assay of ^{238}Pu in Waste Drums," *Trans. Am. Nucl. Soc.* 10, 41 (1967).

⁴⁹C. M. Lederer, *The Structure of Heavy Nuclei: A Study of Very Weak Alpha Branching*, UCRL-11028 (Sept. 27, 1963).

and the actual ^{241}Am . The intensity of the 0.66-Mev gamma is consistent with published values.^{50,51}

9.6.e Half-Life Measurements

S. A. Reynolds E. I. Wyatt⁴¹

The measurement⁵² has continued of the half-lives of radionuclides of importance in local programs. The value for ^{170}Tm was 128.6 ± 0.3 days, which is the average of determinations by decay measurements made with an ionization chamber, a scintillation spectrometer, and scintillation counters. This value is in agreement with 128 ± 1 days⁵³ and with the adopted value in the new "Table of Isotopes,"⁵⁴ 130 days. The value for ^{109}Cd was 450 ± 5 days, in good accord with 453 ± 2 days.⁵⁵ The half-life of ^{92}Nb was determined to be 10.14 ± 0.03 days by ionization-chamber and gamma-spectrometer measurements. The half-life of ^{151}Sm was measured (against an ^{63}Ni standard) in a solution that contained a weighed amount of fission product samarium in which abundances had been determined by mass spectrometry; the value calculated from the measured specific activity was 93 ± 8 years.

The half-life of ^{95}Nb was redetermined because the value 34.85 days was reported.⁵⁶ Frustratingly, our new value, 35.15 ± 0.03 days, agrees with neither our old one, 35.0 ± 0.1 days,⁵⁷ nor with

⁵⁰C. M. Lederer, J. K. Poggenburg, F. Asaro, J. O. Rasmussen, and I. Perlman, "Energy Levels of ^{237}Np (I). The Alpha Decay of ^{241}Am ," *Nucl. Phys.* 84, 481 (1966).

⁵¹W. Michaelis, "Precision Measurement of the ^{241}Am Gamma Spectrum," *Z. Physik* 194, 395 (1966).

⁵²S. A. Reynolds and E. I. Wyatt, "Half-Life Measurements," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1966*, ORNL-4039, p. 59.

⁵³W. R. Cornman, DP-1052 (February 1967) (classified).

⁵⁴C. M. Lederer, J. M. Hollander, and I. Perlman, *Table of Isotopes*, 6th ed., Wiley, New York, 1967.

⁵⁵H. Leutz, K. Schneckenberger, and H. Wenninger, "Electron Capture Ratios in Cd^{109} and Internal Conversion Coefficients in Ag^{109m} ," *Nucl. Phys.* 63, 263 (1965).

⁵⁶S. C. Anspach, L. M. Cavallo, S. B. Garfinkel, J. M. R. Hutchinson, and C. N. Smith, "Half-Lives of Materials Used in the Preparation of Standard Reference Materials of Nineteen Radioactive Nuclides Issued by the National Bureau of Standards," *Natl. Bur. Std., Misc. Pub.* 260, 9 (1965).

⁵⁷E. I. Wyatt, S. A. Reynolds, T. H. Handley, W. S. Lyon, and H. A. Parker, "Half-Lives of Radionuclides - II," *Nucl. Sci. Eng.* 11, 74 (1961).

34.85 days. The data were recomputed several times, and no trend with time or other indication of systematic error was found. (Data for the first few days were discarded because of the ingrowth of ^{95}Nb from 90-hr ^{95m}Nb .) If it is assumed that the value of 34.85 days is correct, computation indicates that over 1% of ^{95}Zr or another long-lived nuclide would have been present. However, examination with Ge(Li) and NaI(Tl) gamma spectrometers excluded that possibility. Specifically, the upper limit for Zr was 0.1%. Continuation of the use of 35.0 days as the half-life is recommended.

The decay of four sources of ^{137}Cs , originally prepared from homogeneous-reactor off-gas, has been followed for eight years. The present value, 30.23 ± 0.16 years, supersedes last year's.⁵² Various determinations of the half-life and related decay-scheme data can now be reconciled. As an example, a preparation called RC-137-ASTM contained 1.72×10^{14} atoms of ^{137}Cs per milliliter and emitted 1.07×10^5 gamma photons $\text{sec}^{-1} \text{ml}^{-1}$.⁵⁸ By use of the "best" value for gamma branching (85%⁵⁹), the apparent half-life of ^{137}Cs is 30.0 years; use of this value is recommended.

We and other early investigators⁵⁴ were unaware of the presence of significant amounts of ^{175}Hf in ^{181}Hf preparations, and our original value⁶⁰ was therefore incorrect and must be lowered to 42.4 ± 0.2 days. Determinations of the ^{99}Mo half-life as low as 66 hr⁵⁴ led to computer treatment of decay data from five early preparations and taking new data on a contemporary one. The new value is 66.69 ± 0.06 hr; our old value of 66.96 hr must be discarded.

The determinations reported here and other recent ones require that a few significant changes be made in our list of recommended half-life values.⁶¹ The corrected values are: ^{226}Ra , 1600 years;⁵⁹ ^{93m}Nb , 13.6 years;⁶² ^{146}Pm , 5.5 years;⁶³ ^{170}Tm , 129 days; ^{181}Hf , 42.5 days; ^{241}Pu , 14.2 ± 0.2 years; and ^{241}Am , 433 years.⁶⁴ The ^{241}Pu value was deduced from the new value for ^{241}Am and from two determinations on ^{241}Pu itself, the results of which have not yet been released.

9.6.f Study of HFIR Coolant Water

S. A. Reynolds J. F. Emery

At the request of the Operations Division, analyses and calculations were made relative to the

primary coolant water of the High Flux Isotope Reactor (HFIR), especially with respect to the detection of leaks in target rods that contain transplutonium elements. The ideal was to find an in-line monitoring device to immediately signal a leak. Other objectives were to devise methods of decontaminating the water and the coolant system and to establish "background" conditions to permit detection of possible future anomalies. For in-line or "real time" monitoring, detection of neutrons from spontaneous-fission nuclides appears to be the best approach. Neutrons were detected at the top of one of the coolant demineralizers, and the source was removable by regeneration. Installation of a neutron counter on each of the two demineralizers was recommended.

Batch analyses are normally required for confirmation of leakage. The concentration levels of components determined by such analyses must override existing levels, whether the existing levels result from prior target ruptures or fuel background (fission products). The appearance of alpha radioactivity is a good indicator of the leakage of transuranic elements, but, once in the system, the alpha radioactivity behaves erratically. Two approaches were taken in the search for a key fission product to distinguish target leaks from ^{235}U contamination. In one, a nuclide was sought whose ^{235}U fission yield is low but whose ^{245}Cm yield is appreciable because of the upshift of the lower-mass peak. Such a one is ^{111}Ag (0.019% in ^{235}U , 3.6% in ^{245}Cm). Silver was separated chemically from a sample of coolant, and no significant gamma counts were found; the method therefore appears to be impracticable. The other method involves sep-

⁵⁸D. S. Kim, Nuclear-Chicago Corp., personal communication to S. A. Reynolds, March 2, 1965.

⁵⁹Personal communications to S. A. Reynolds from members of the ORNL Nuclear Data Group, directed by Dr. Katharine Way.

⁶⁰H. W. Wright, E. I. Wyatt, S. A. Reynolds, W. S. Lyon, and T. H. Handley, "Half-Lives of Radionuclides. I," *Nucl. Sci. Eng.* 2, 427 (1957).

⁶¹G. Goldstein and S. A. Reynolds, "Specific Activities and Half-Lives of Common Radionuclides," *Nuclear Data A1*, 435 (1966).

⁶²K. F. Flynn, L. E. Glendenin, and E. P. Steinberg, "Half-Life Determinations by Direct Decay," *Nucl. Sci. Eng.* 22, 416 (1965).

⁶³L. R. Bunney and E. M. Scadden, *Decay Characteristics of Pm¹⁴⁶*, USNRDL-TR-1109 (Nov. 7, 1966).

⁶⁴S. R. Gunn and F. L. Oetting, quoted by D. L. Davis and F. L. Oetting, *Analysis of Americium by Calorimetry*, RFP-895 (Apr. 13, 1967).

aration of cesium and measurement of the radioactivity ratio $^{134}\text{Cs} : ^{136}\text{Cs} : ^{137}\text{Cs}$. The relative concentration of ^{136}Cs in a target is much higher than in ^{235}U fuel because of higher independent yield and buildup by successive capture in cesium isotopes beginning with fission product ^{133}Cs . Analytical data confirmed the expected ratios and showed that the method is practicable.

An ion exchange method⁶⁵ for removal of cesium from low-level waste was adapted for concentrating cesium from 1-liter samples and for the simultaneous separation of ^{24}Na , the principal radionuclide in the coolant. Carrier is added, and the sample is made 0.01 M in NaOH and is passed through a 5-cm-long column of Duolite CS-100 resin at a rate of ~ 10 ml/min. Over 90% of the cesium but only part of the ^{24}Na is retained by the resin; most of the latter is washed off by 0.1 M NaOH, overall decontamination being $>10^3$. Cesium is eluted in 0.5 M HCl, in which solution it is ready for final isolation by the usual chlorostannate method and for subsequent determination of ^{134}Cs , ^{136}Cs , and ^{137}Cs by gamma spectrometry. The ion exchange decontamination from ^{24}Na is sufficient to permit performing the latter part of the separation on the bench.

The appearance of unusually high amounts of radioactivity in the primary coolant on one occasion indicated release of fission products from fuel. Several iodine isotopes, notably ^{134}I , were identified in the cation exchanger effluent. It was desired to interpret the fission product concentrations in terms of exposed uranium. Only a crude approximation could be made because of lack of knowledge of the location of the uranium, and hence of the magnitude and character of the flux. All concentrations indicated that only a few milligrams of uranium was exposed. Ratios of concentrations of the volatile iodine, gas daughters ^{91}Sr and ^{140}Ba , and nonvolatile ^{99}Mo and ^{239}Np indicated that the uranium was in a thin film, that is, surface contamination.

Spectrographic and flame spectrophotometric analyses yielded the information that only ppb levels of such elements as Na, Al, Be, Ca, and Fe were present in solution. Centrifugation of a water sample gave no indication of removal of ^{51}Cr and ^{60}Co , the principal long-lived induced radioactivities, but gross-alpha content was re-

duced about 40% and barium by about half. The centrifugate was made 0.01 M in NaOH and was passed through a Duolite CS-100 resin column. All the chromium and $\sim 20\%$ of the cobalt were unadsorbed. Chromium is surely anionic, and cobalt may be carried in part by a colloid (perhaps iron oxide or alumina) that exists in the water or is formed on adding base.

Efforts to estimate the average thermal-neutron flux to which the water is exposed resulted in a very crude estimate of 10^{12} neutrons $\text{cm}^{-2} \text{sec}^{-1}$. This estimate was derived from the residence times of coolant water in the core and in the rest of the system, plus a slow migration from the region outside the beryllium reflector. An attempt to estimate the flux from ^3H concentration, 1.5×10^5 dis $\text{min}^{-1} \text{ml}^{-1}$, led to an unrealistically high value, which indicates that $^2\text{H}(n,\gamma)$ is not the principal source. Production from fission in contaminating ^{235}U is insignificant. Based on crude estimates of fission-spectrum- and thermal-neutron fluxes at beryllium surfaces in contact with water, the following reaction sequence approximately accounts for the observed ^3H : $^9\text{Be}(n,\alpha)^6\text{Li}$, $^6\text{Li}(n,\alpha)^3\text{H}$. It was assumed that all tritons produced within $\sim \frac{1}{4}$ range (~ 1 mg/cm²) of the beryllium surface escape and remain in the water.

The circulating curium was removed successfully⁶⁶ from the system by using the "solubilizing" effect of lanthanum at ~ 1 ppm.⁶⁷ Thus no other study of decontamination was required. Further details may be obtained from reports of the Operations Division.

9.6.g Radioisotope Tagging of Copper Wire

L. C. Bate F. F. Dyer

At the request of a power company, a study is being made to determine whether copper wire can be safely tagged with a radioisotope to aid in the location and identification of stolen wire. Tests indicate that ^{110m}Ag , ^{106}Ru , and ^{192}Ir might be suitable tags. About 50 to 100 μc of a radioisotope that has a high yield of energetic gamma rays is required for detection at a distance of ~ 1 m.

⁶⁶A. G. Samartseva, "Adsorption of Curium on a Polished Platinum Surface," *Sov. Radiochem.* **8**, 253 (1966).

⁶⁷J. A. Cox, Operations Division, personal communication to S. A. Reynolds, Aug. 17, 1967.

⁶⁵R. R. Holcomb, *Low-Radioactivity-Level Waste Treatment*, ORNL-3322 (June 25, 1963).

With such a tag, copper junk piles could be scanned fairly rapidly with a beta or gamma detector.

The radioisotope tag is applied between the strands of the multistrand copper wire with a hypodermic syringe; the tag seeks the surfaces of the inner wires where it dries. The strand is then lacquered. After repeated exposure of the strand to sun and to washing with water to simulate the effects of rain, the radioactivity decreases $\sim 8\%$. Tagging of single copper wire results in decreases as great as 40%.

9.7 RADIOCHEMICAL STUDIES

9.7.a Separation of Californium from Curium and of Berkelium from Cerium by Extraction Chromatography

F. L. Moore Aart Jurriaanse⁶⁸

An extremely simple method was developed for the separation of Cf(III) from Cm(III) and of Bk(III) from Ce(III).⁶⁹ The new method is based on extraction chromatography by use of small Teflon-packed columns that contain di(2-ethylhexyl)orthophosphoric acid as the stationary phase. Excellent separations are achieved with dilute nitric acid eluents. Among the significant advantages of the system are room-temperature operation, use of nonchloride media, relatively high flow rates, and applicability for glove-box or hot-cell work. The method has many useful analytical and process applications.

9.7.b Separation Method for Americium Based on Liquid-Liquid Extraction Behavior of Americium(V)

J. R. Stokely F. L. Moore

A new method for the separation and radiochemical determination of ^{241}Am and ^{243}Am was developed.⁷⁰ Americium(III) is oxidized to Am(VI)

⁶⁸Alien Guest from South African Atomic Energy Board, Pelindaba, Private Mail Bag 256, Pretoria, South Africa.

⁶⁹F. L. Moore and A. Jurriaanse, "Separation of Californium from Curium and Berkelium from Cerium by Extraction Chromatography," *Anal. Chem.* **39**, 733 (1967).

⁷⁰J. R. Stokely and F. L. Moore, "New Separation Method for Americium Based on Liquid-Liquid Extraction Behavior of Americium(V)," *Anal. Chem.* **39**, 994 (1967).

with ammonium persulfate; Ag^+ is used as a catalyst. Addition of an acetate buffer solution to the cooled solution of Am(VI) reduces Am(VI) to Am(V) and raises the pH of the solution to between 4.8 and 5.2. Actinide(III), (IV), and (VI) and lanthanide(III) and (IV) ions are extracted into a 0.5 M solution of 2-thenoyltrifluoroacetone in xylene. Americium(V) remains in the aqueous phase and thus is efficiently separated from many metal ions. Absorption spectrophotometry was used to ascertain the valence states of americium.

9.7.c New Method for the Rapid Separation of Berkelium(IV) from Cerium(IV) by Anion Exchange

F. L. Moore

A paper on this subject was submitted to *Analytical Chemistry*.⁷¹

9.7.d Use of Organic Additives to Induce Selective Liquid-Liquid Extraction of Niobium with 2-Thenoyltrifluoroacetone

Aart Jurriaanse⁶⁸ F. L. Moore

A new highly selective method for the liquid-liquid extraction of Nb(V) was developed.⁷² The method is based on the ability of *n*-butanol in the aqueous phase to enhance the formation of an extractable chelate of niobium with 2-thenoyltrifluoroacetone. Niobium can be recovered quantitatively from aqueous solutions of concentrated hydrochloric acid or hydrochloric acid-sulfuric acid mixtures. Excellent separation of Nb(V) from most metal ions is effected with 0.5 M 2-thenoyltrifluoroacetone-xylene solution.

9.7.e Radiochemical Purification of ^{245}Pu - ^{246}Pu

J. R. Stokely C. E. Bemis⁴

Chemical separation of ^{245}Pu - ^{246}Pu from fission products produced in the neutron irradiation

⁷¹F. L. Moore, "New Method for the Rapid Separation of Berkelium(IV) from Cerium(IV) by Anion Exchange," *Anal. Chem.* **39**, in press (1967).

⁷²A. Jurriaanse and F. L. Moore, "Use of Organic Additives to Induce Selective Liquid-Liquid Extraction of Niobium with Thenoyltrifluoroacetone," *Anal. Chem.* **39**, 494 (1967).

of ^{244}Pu was performed to permit study of the nuclear properties of the heavier plutonium isotopes. Solvent extraction with 2-thenoyltrifluoroacetone and precipitation with lanthanum fluoride effectively eliminated all fission product radionuclides from the plutonium. The purified plutonium, having americium daughters in equilibrium, was absorbed on an anion exchange column from 8 M HNO_3 , and the americium daughters were eluted continuously from the column with 8 M HNO_3 . The column was positioned next to a Ge(Li) detector connected to a multichannel analyzer. Gamma-ray spectra of mixtures of ^{245}Pu and ^{246}Pu and of pure ^{246}Pu were recorded without appreciable contribution from the gamma radioactivity of the americium daughters. Gamma-ray spectra of ^{245}Am and ^{246}Am were also obtained.

9.7.f Evidence of Photofission of Iron

C. B. Fulmer⁵ I. R. Williams⁷³ T. H. Handley
G. F. Dell⁷⁴ L. N. Blumberg⁷⁵

It has been pointed out that any nucleus can be made to fission provided it is supplied with sufficient excitation energy. Studies of proton-induced reactions in the GeV energy region have given evidence that fission occurs in nuclei at least as light as silver. We have now obtained evidence of photofission in iron foils that were bombarded with high-energy electrons.

In a study of residual radioactivity produced in targets exposed to electrons of energy in the GeV region, radionuclides were identified over a wide range of mass number. It was demonstrated that the residual nuclei are produced predominantly by photonuclear reactions that are induced by the electron bremsstrahlung photons.

The experiments are done by exposing targets to monitored beams of electrons at the Cambridge Electron Accelerator, Cambridge, Massachusetts, and subsequently measuring the gamma spectra from the exposed targets with a Ge(Li) gamma-ray spectrometer. Radionuclides are identified by

⁷³Chairman, Department of Mathematics and Physics; Associate Professor of Physics, Knoxville College, Tenn.

⁷⁴Cambridge Electron Accelerator, Cambridge, Mass.

⁷⁵Present address: Accelerator Department, Brookhaven National Laboratory.

gamma energy and half-life. The results of this work were published.⁷⁶

9.7.g Aqueous Liquid-Scintillation Counting System for Weak-Beta Emitters

S. B. Lupica

Effort was made to develop an aqueous scintillation-counting system for weak-beta emitters (^3H , ^{14}C , ^{33}P , ^{35}S , ^{63}Ni , ^{147}Pm , etc.). The use of a Cab-O-Sil gel in which various inorganic rare-earth-element phosphors were dispersed and suspended gave low results. In addition a long pre-counting period was required to allow for the disappearance of the effect of room light on the phosphors. Emulsions of anthracene, of 2,5-diphenyloxazole (PPO), and of other powdered organic scintillators in triethanolamine stearate gave results that were no better than those obtained with Cab-O-Sil gel. With colorless Triton X-100 as a solvent common for water and the scintillator, promising results were obtained. To date, the two organic scintillators 2,5-bis-[2-(5-*tert*-butylbenzoxazolyl)]-thiophene (BBOT) and PPO, each in *p*-dioxane, have been investigated. A Triton X-100 solution that contained 3 to 5 vol % of a solution of PPO in *p*-dioxane (12 g/liter) has given the highest efficiencies; examples of the results follow:

Water in Aqueous Solution of Triton X-100 (vol %)	Counting Efficiency for ^3H (%)
5.6	4.9
10.6	4.1
15.0	3.3

A Triton X-100-PPO-*n*-dioxane mixture offers possibilities for counting certain aqueous samples that at present cannot be counted in an all-organic system.

9.7.h Analysis of Water Systems

W. J. Ross

The increasing involvement of ORNL in projects related to desalination and environmental control is resulting in the initiation of a wide variety of

⁷⁶C. B. Fulmer, I. R. Williams, T. H. Handley, G. F. Dell, and L. N. Blumberg, "Evidence for Photofission of Iron," *Phys. Rev. Letters* 19, 522 (1967).

programs related to water chemistry. Consequently, the Analytical Chemistry Division is being approached more frequently for advice, development of methods, and service analyses pertaining to various aspects of these water programs. This participation has been related primarily to (1) the construction and testing of components of desalination plants, (2) ecological investigations of local watersheds, and (3) basic investigations of the application of radiotracers and activation analysis in tracing the flow of streams and rivers.

Desalination Program. — Participation in this program has involved (1) developing chemical methods for the determination of 1 to 7000 ppb of dissolved O_2 and 1 to 200 ppm of dissolved CO_2 in seawater and brackish artesian water; (2) using these methods to evaluate the performance of a deaerator in two series of engineering tests at the Freeport Desalination Facility operated by the Office of Saline Water; (3) contributing technical advice for the solicitation of instruments for determining O_2 and CO_2 in saline water; (4) evaluating an instrumented oxygen analyzer at the Freeport and Wrightsville Beach Desalination Facilities and installing and testing one of these analyzers in a facility at ORGDP; (5) investigating the effect of interferences on the Winkler method in an effort to establish a "standard" chemical method for O_2 to be incorporated in operating procedures of future desalination plants; (6) studying the effects of temperature, pH, and turbulence on the liberation of CO_2 from saline solution; (7) applying the information relating to carbonate equilibria to the design of a test facility at Freeport; and (8) investigating the carbonate-hydroxide equilibria of saline solutions that contain calcium and magnesium in an effort to interpret the results of analyses of precipitates formed in a test facility at Wrightsville Beach. Much of this work is described in greater detail in ORNL-TM-1953.⁷⁷

Ecological Program. — A cooperative effort has been initiated to investigate thoroughly the origin and movement of certain ions that are important in the pollution of local watersheds. Plans have been made with the Health Physics Division to analyze local rivers and creeks for nitrate and phosphate on an extensive and continuous scale. For these determinations, methods are being se-

lected that can be adapted for use with a Technicon AutoAnalyzer, which has been purchased for this purpose.

Attempts are still being made to develop an ultrasensitive method for the determination of PO_4^{3-} ; however, little progress has been made.

Application of Activation Analysis to Water Tracing. — The investigation of the use of trace amounts of elements and activation analysis to follow stream movement is continuing.⁷⁸ Completed experiments with cadmium, cobalt, and copper reveal that these elements are not sufficiently stable to remain in solution in streams that are slightly alkaline or that contain large amounts of adsorbents such as clay and hydrous oxides of iron or aluminum. However, these same elements, when complexed with EDTA, exhibit greatly enhanced stability in all these environments.

Experiments designed to establish the sensitivity with which selenium can be detected in water by activation analysis revealed that as little as 0.001 μg of selenium can be measured by using ^{77m}Se ($t_{1/2} = 18$ sec).

9.7.i Separation of Sodium with Antimony Pentoxide

W. J. Ross

The sorptive properties of Sb_2O_5 have been studied in an effort to devise a method for removing the interfering gamma radioactivity of ^{24}Na from samples that are being analyzed for trace elements by activation analysis. Preliminary results show that sodium is retained quantitatively on columns prepared with Sb_2O_5 , whereas cesium is eluted. This work will be extended to study the adsorption characteristics of several other ions of interest, especially in biological samples.

9.8 ANALYTICAL CHEMISTRY OF RADIONUCLIDES

9.8.a Analytical Chemical Research Facilities in the Transuranium Research Laboratory

J. R. Stokely

A laboratory in the Transuranium Research Laboratory was equipped for performing specialized

⁷⁷B. E. Mitchell, R. K. Sood, and W. J. Ross, *Tests of the Deaerator at the Freeport Seawater Desalting Plant and Evaluation of Dissolved Gas Analyses*, ORNL-TM-1953 (October 1967).

⁷⁸W. J. Ross, "Analytical Chemistry of Water Environments," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1966*, ORNL-4039, p. 72.

chemical analyses and analytical chemical research on the transuranium elements. The laboratory contains six glove boxes, each supplied with normal laboratory services and capable of containing curie levels of alpha radioactivity.

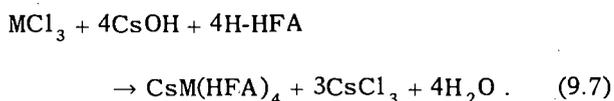
One glove box was modified to house the furnace section of a Perkin-Elmer model 240 Automatic Elemental Analyzer. This analyzer is used for the microchemical determination of carbon, hydrogen, and nitrogen in organometallic compounds of the transuranium elements. Associated with the analyzer is a remotely operated microbalance, which is also enclosed in a glove box. A future report will describe the operation of the analyzer.

Electrochemical studies on the transuranium elements are carried out in a specially designed glove box. As many as ten electrical leads may be used in the electrochemical studies.

9.8.b Preparation of Lanthanide and Actinide β -Diketones

J. R. Stokely J. H. Burns⁴ L. J. Nugent⁴

β -Diketone chelates of some lanthanide and actinide elements were prepared for x-ray crystallographic and fluorescence spectroscopic studies. The general formula of these compounds is $CsM(HFA)_4$, where HFA is the hexafluoroacetylacetonate anion and M is a trivalent actinide or lanthanide metal ion. The reaction that leads to the formation of a compound of this type is



Chelates of Eu(III), Tb(III), Sm(III), and Am(III) have been prepared.

9.8.c Controlled-Potential Coulometric Determination of Americium⁷⁹

J. R. Stokely W. D. Shults⁸⁰

Application of controlled-potential coulometry to the accurate and precise determination of ameri-

cium was reported recently.⁸¹ The procedure involves a rapid chemical oxidation of americium to Am(VI), destruction of excess oxidant by heating, and electrolytic reduction of Am(VI) to Am(V) at a platinum electrode controlled at +1.05 v vs S.C.E. Details of this work will be published.

9.8.d Feasibility of Determining the Formal Potential of the Bk(IV)-Bk(III) Couple

J. R. Stokely

Preliminary experiments were performed to determine whether the potential of the Bk(IV)-Bk(III) couple can be measured electrochemically with $\sim 50 \mu\text{g}$ of berkelium. Cerium was used as a substitute for berkelium. The experimental procedure involves complete oxidation of Ce(III) to Ce(IV) at +1.60 v vs S.C.E. at a platinum electrode by use of the ORNL model 2564 coulometric titrator and a micro coulometric cell. Cerium(III) was produced in known concentration ratios to Ce(IV) by reduction at +1.0 v vs S.C.E. with integration of the current. The potential of the platinum electrode is determined at each Ce(IV):Ce(III) ratio. By this procedure the formal potential of the Ce(IV)-Ce(III) couple in 0.5 M H_2SO_4 was measured to be +1.4406 v vs N.H.E.; the literature value is +1.4435 v vs N.H.E.⁸² One problem is the slow reduction of Ce(IV) by water at the low concentration used (15 $\mu\text{g}/\text{ml}$). In 0.5 M H_2SO_4 the reduction rate is 0.02 $\mu\text{g}/\text{min}$ for a Ce(IV):Ce(III) ratio of 1. In 1 M HNO_3 the reduction rate is much faster, 0.6 $\mu\text{g}/\text{min}$. It appears that although the potential of the Bk(IV)-Bk(III) couple can be measured accurately in sulfuric acid, measurements in nitric acid will be susceptible to large errors because of the reduction of Bk(IV) to Bk(III) by water.

⁷⁹This work was done in the Transuranium Research Laboratory.

⁸⁰Methodology Group.

⁸¹J. R. Stokely and W. D. Shults, "Controlled-Potential Coulometric Determination of Americium," presented at the Eleventh Conference on Analytical Chemistry in Nuclear Technology, Oct. 10-12, 1967, Gatlinburg, Tenn.

⁸²A. H. Kunz, "The Reduction Potential of the Cerico-Cerous Electrode," *J. Am. Chem. Soc.* **53**, 98 (1931).

10. Inorganic Preparations

D. E. LaValle

R. B. Quincy

The fused-salts program continued with little change. It consisted chiefly in the preparation of anhydrous rare-earth-metal halides for the High Temperature and Structural Chemistry Group of the Chemistry Division and of a few anhydrous salts such as MgCl_2 , CdCl_2 , CaCl_2 , and CsCl for the Ion Spectroscopy Group of the Metals and Ceramics Division.

Most of the programs for the Solid State Division were also continued without significant change. For the Neutron Spectrometry Group more compounds of the spinel type (MgAl_2O_4) structure were prepared; various combinations of Fe, Mn, Co, Ni, Ti, and Li were used in place of Mg and Al. Additional glasses of GeO_2 , GeS_2 , and $^{73}\text{GeO}_2$ were made for the Spin Resonance Group. Details of these preparations were described last year.¹ For the Neutron Diffraction Studies Group, a new project was undertaken in addition to the usual program of heat treatment of alloys that contain rare-earth metals. Equipment was obtained and procedures were developed to prepare gadolinium metal from Gd_2O_3 by reduction of the intermediate GdF_3 with calcium metal. These steps were in preparation for the eventual treatment of 50 g of ^{160}Gd when it shall have been isolated and obtained as $^{160}\text{Gd}_2\text{O}_3$.

For the Low Temperature, Nuclear, and Solid State Physics Group of the Physics Division, the preparation, made last year, of the solid solution of 3 mole % FeBr_3 in CrBr_3 was repeated using ^{57}Fe ; also, the alloy AuLi (0.5 at. % Au) was made. For another section of this group a number of isotopic compounds and alloys were prepared:

$^{73}\text{GeI}_2$, $^{73}\text{GeCr}$ (5 at. % ^{73}Ge), $^{61}\text{NiF}_2$, $^{64}\text{NiAl}$ (50 at. % ^{64}Ni), and ^{64}NiV (85 at. % ^{64}Ni).

Compounds prepared by well-known methods included Na_2O and ZrN for the Materials Compatibility Laboratory of the Metals and Ceramics Division and Mg_3N_2 for the Insulating Crystals Group of the Solid State Division. The Special Electromagnetic Separations of Heavy Elements Group of the Isotopes Division was provided with 100 g of UCl_3 made from depleted U.

Among preparations of particular interest, VF_2 was made for the Neutron Diffraction Group of the Solid State Division by passing a gas mixture of 10 vol % HF and 90 vol % H_2 over VF_3 at 1000°C . Both the temperature and composition of the gas mixture are critical in obtaining a pure product. For the Neutron Scattering Cross Sections Studies Group of the Neutron Physics Division, Be_3N_2 was prepared by passing N_2 over 50-mesh beryllium metal contained in a molybdenum boat at 1250°C . A preparation of 65 g of very pure (98%) SmF_2 was achieved for the High Temperature and Structural Chemistry Group of the Chemistry Division by the solid-state reaction of finely divided SmF_3 and SmH_2 .

The production of high-purity KCl was continued on a routine basis for the Research and Development of Pure Materials Group of the Solid State Division. The preparation of high-purity MgO has been resumed after a lapse of five years.² The new objective is to produce much greater amounts of material at less cost.

¹M. T. Kelley, D. E. LaValle, and R. B. Quincy, "Inorganic Preparations," *Anal. Chem. Div. Ann. Progr. Rept.* Oct. 31, 1966, ORNL-4039, p. 78.

²R. B. Quincy, Jr., *Preparation of High-Purity Magnesium Oxide*, ORNL-TM-302 (Aug. 15, 1962).

II. Organic Preparations

P. F. Thomason

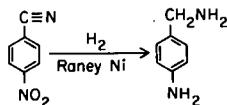
H. L. Holsopple

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, and Metals and Ceramics Divisions.

11.1 COMPOUNDS SYNTHESIZED

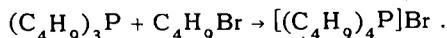
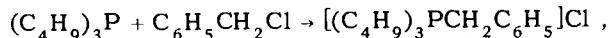
11.1.a *p*-Aminobenzylamine

p-Aminobenzylamine (~100 g) was prepared for the Physics Group of the K-25 Technical Division. *p*-Nitrobenzonitrile was hydrogenated in ethyl acetate at 750 psi and 90 to 100°C for 3 hr over Raney nickel. The reaction is



11.1.b Quaternary Phosphonium Halides

Two quaternary phosphonium compounds, tri-*n*-butylbenzylphosphonium chloride and tetra-*n*-butylphosphonium bromide, were prepared for the Metals and Ceramics Division according to the reactions



Results of elemental analysis were in good agreement with calculated values (Table 11.1) based on the formulas given above. The details for the preparation of these compounds are similar to those given elsewhere for analogous substances.¹

11.1.c Heptadecylamine

Heptadecylamine was prepared for the Chemical Technology Division according to the three-step

Table 11.1. Compositions of Two Quaternary Phosphonium Halides

Component	Weight Percent			
	In A ^a		In B ^b	
	Theoretical	Found	Theoretical	Found
Carbon	69.4	69.3	56.6	57.3
Hydrogen	10.4	10.9	10.7	11.0
Phosphorus	9.4	9.6	9.1	9.1
Chlorine	10.8	10.8		
Bromine			23.6	22.9

^aA = tri-*n*-butylbenzylphosphonium chloride, C₁₉H₃₄PCl.

^bB = tetra-*n*-butylphosphonium bromide, C₁₆H₃₆PBr.

process of Barnes² in which heptadecanol is converted to heptadecanone, from which *N*-(heptadecylidene)-2-ethylhexylamine is prepared. The latter is then catalytically hydrogenated to heptadecylamine. The yield based on imine was 95% (400 g, 1.55 moles).

11.1.d α, α' -Dipiperidyl

Dipiperidyl (60 g) was prepared for the Chemical Separation of Isotopes Group of the Chemistry Division by catalytic hydrogenation of α, α' -dipyridyl in ethyl alcohol.

The product was purified by extraction into diethyl ether; its purity was established by infrared-absorption and elemental analyses. The details of this preparation are given elsewhere.³

¹H. L. Holsopple, *Syntheses of Three Quaternary Phosphonium Halides*, ORNL-TM-1572 (July 12, 1966).

²R. K. Barnes, Union Carbide, Chemicals Division, South Charleston, W. Va., personal communication to F. G. Seeley, Chemical Technology Division, March 1965.

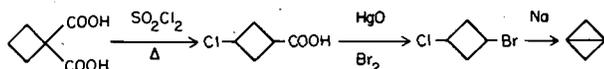
³H. L. Holsopple, *Synthesis of α, α' -Dipiperidyl*, ORNL-TM-1470 (Mar. 30, 1966).

11.1.e Cesium *p*-Ethylbenzenesulfonate

Cesium *p*-ethylbenzenesulfonate (25 g) was prepared for the Chemistry Division. Barium *p*-ethylbenzenesulfonate was passed through a Dowex 50W-X4 resin column in H⁺ form. Cesium carbonate was neutralized with the acid solution; the cesium *p*-ethylbenzenesulfonate thus formed was recovered by filtration on a Büchner funnel.

11.1.f Bicyclo[1.1.0]butane

Bicyclo[1.1.0]butane (~7 ml) was prepared for research members of the Chemistry Division. This compound is unique, because it has the greatest amount of thermochemical destabilization ("strain energy") per carbon atom of any known saturated organic compound. It was prepared in three stages: 1,1-cyclobutanedicarboxylic acid to 3-chlorocyclobutane-1-carboxylic acid to 1-bromo-3-chlorocyclobutane to bicyclo[1.1.0]butane according to the reaction sequence



1,1-Cyclobutanedicarboxylic acid (88 g) was chlorinated with sulfuryl chloride (51 ml) in the presence of benzoyl peroxide (1.5 g) in benzene. The benzene was distilled off, and the residue was decarboxylated at 180°C for 30 min. The mixture was distilled, and the product was collected at 130 to 148°C/17 mm.

The resulting 3-chlorocyclobutane-1-carboxylic acid was then brominated with bromine (21 ml) in CCl₄ (300 ml) that contained a slurry of red mercuric oxide (61 g) in CCl₄ (550 ml). Double distillation gave a dihalide, which was collected at 38 to 40°C/5 mm.

The dihalide was treated with sodium metal in dioxane to give bicyclo[1.1.0]butane. The volatile (bp, 8°C) product was collected in gas-collection bulbs at -78°C. Infrared analysis of the product showed strong C-H bands in the positions indicated in the literature for bicyclo[1.1.0]butane.

12. Mass Spectrometric Analyses

A. E. Cameron

12.1 MASS SPECTROMETRY SERVICE

J. R. Sites E. J. Spitzer

The number of analyses reported by the Mass Spectrometry Service Laboratory increased 25% over last year. This total increase includes a 15% increase for the Isotopes Division and about a tenfold increase for the Reactor Chemistry Division.

The relative isotopic abundances of samples of Cr, Fe, and Ni from irradiated HFIR components were determined to help decide whether such values might be used to indicate the magnitude of the reactor's very high neutron flux.

The major sample load for the gas mass spectrometers was the certification for Stores of the purity of the compressed gases He (15 trailers, 200 cylinders), N₂ (50 cylinders), and Ar (500 cylinders).

An electron bombardment source that uses a five-sample wheel was developed and tested for the use with the 12-in. 90° mass spectrometer. This source is an important complement to the usual surface-ionization source.¹

12.2 TRANSURANIUM MASS SPECTROMETRY

R. E. Eby

The Transuranium Mass Spectrometry Laboratory reported about 4500 results, an 88% increase over the previous year. The cost per analysis decreased from \$20.86 in FY-1966 to \$12.86 in FY-1967.

About a third of the work was for the Isotopes Division, mostly analysis of separated isotopes of uranium and plutonium; about a quarter was for the Chemical Technology Division.

The types of samples received have become increasingly diverse; many more of them are radioactive samples of the lighter elements. Samples of irradiated thulium have been analyzed to verify values for cross sections and to indicate the possibility of producing ¹⁷¹Tm, which is being considered as an isotopic power source for a mechanical heart. Radioactive samples of gadolinium were analyzed to determine the possibility of producing ¹⁵³Gd, which is a candidate for use in a density-measuring device on the Mars probe. Radioactive samples of strontium and samarium have also been analyzed.

The isotopic analysis of all elements from thorium through californium is done routinely. Samples of synthetically mixed standards of ²³⁹Pu, ²⁴²Pu, and ²⁴⁴Pu were analyzed for the Chemistry Division in a program to determine a better value for the half-lives of ²⁴²Pu and ²⁴⁴Pu. Uranium and plutonium isotopic analyses were made on a series of samples for Westinghouse Atomic Power Development Division as a part of the Yankee core evaluation program. Another series of samples were analyzed for plutonium isotopic content for the Los Alamos Scientific Laboratory.

The first sample of einsteinium was received and analyzed with no difficulties.

¹W. H. Christie and A. E. Cameron, "A Reliable Sample Changer for Mass Spectrometer," *Rev. Sci. Instr.* 37, 336 (1966).

13. Emission Spectrochemical Analyses

A. E. Cameron

J. A. Carter

S. A. MacIntyre

The Spectrochemistry Laboratory reported about 42,000 results on some 2100 samples, which represents an increase of 13.4%. The Isotopes Division submitted about 18% more samples than last year. The stable isotopes of the following 41 elements were analyzed for trace impurities: Ag, B, Ba, Ca, Cd, Ce, Cl, Cr, Cu, Dy, Gd, Ge, Hf, Hg, In, Ir, K, La, Lu, Mg, Mo, Nd, Ni, Os, Pb, Pt, Rb, Ru, S, Sb, Si, Sm, Sn, Sr, Te, Ti, Tl, W, Yb, Zn, and Zr. Over 500 samples of air and water were analyzed

for the Industrial Hygiene Department of the Health Division. The analysis of metals and alloys increased by some 400% over the previous year.

A number of unusual alloys whose base metals are Co, Ti, V, Cb, Bi, Pb, Ta, and W are being analyzed routinely by use of a modified rotating-disk technique and the Paschen Direct Reader. Developmental high-temperature alloys for the Wright-Patterson Air Force Base are also being analyzed.

14. Process Analyses

L. T. Corbin

14.1 HIGH-LEVEL ALPHA RADIATION LABORATORY

J. H. Cooper

The High-Level Alpha Radiation Laboratory reported some 20,000 results, more than twice as many as were reported the previous year; ~95% were for the Chemical Technology Division. The large increase was due to the full-scale operation of the Transuranium Processing Plant (TRU).

Samples that simulated TRU process samples were analyzed for gross alpha activity, plutonium alpha activity, and acid to determine whether biases exist in the methods. Although the simulated samples did not contain all the constituents of process samples, their use demonstrated the consistency of the methods and showed that no biases exist.

A method for the determination of californium was tested on highly radioactive samples and found to be precise. Californium was separated from curium and fission products by extraction chromatography. A 0.3 M HNO₃ solution of the sample was passed through a column of di(2-ethyl-hexyl)orthophosphoric acid on Teflon powder. Californium and einsteinium were adsorbed, whereas curium, americium, and fission products were

eluted with 0.3 M HNO₃. A 4 M HNO₃ wash removed the californium and einsteinium; the amount of each was determined by alpha counting and pulse-height analysis. Californium was determined rapidly by counting the solution directly in a neutron counter.

Fluoride in the ppm range was determined in samples from the Fluoride Volatility Process after separation from aluminum oxide by pyrohydrolysis. The fluoride-containing sample was heated at 1000°C in a flux of sodium tungstate-tungstic acid. A stream of moist oxygen swept the fluoride into a solution of sodium hydroxide. The fluoride was measured colorimetrically.

Plutonium in low concentration (1 to 2 µg/ml) was determined for the Isotopes Division by isotopic dilution and mass spectrometry.

14.2 GENERAL ANALYSES LABORATORY

W. R. Laing

Development work was completed on the vacuum-fusion-gas-chromatographic analyzer. It was operated for the past year with less than five days downtime. A total of 1700 samples and standards were analyzed; the results obtained were in good

agreement with predicted values. The cost of vacuum-fusion analyses was cut in half when this instrument was put into service.

The work load in analytical biochemistry increased sharply in the latter part of the year. Each month since February at least 4000 results were reported; one month the number was 6100. The total for the year was 46,464.

Several small but significant improvements in the analyses for transfer ribonucleic acids (tRNA) were made. The standard deviation for the determination of phenylalanine-accepting tRNA was reduced from 10 to 4% by these changes.

Molten-salt development projects required several new methods. Small amounts of bismuth in fuel salts were measured polarographically in 5 vol % H_2SO_4 solution. In this supporting medium the half-wave potential of Bi(III) ($E_{1/2} = -0.03$ v vs S.C.E.) is well separated from that of U(VI) ($E_{1/2} = -0.20$ v vs S.C.E.). Lithium metal in fluoride salts was measured by a hydrogen-evolution technique. Fluoride in $NaBF_4$ was titrated directly with Th(IV) after fusion with Na_2CO_3 .

To aid in the study of the growth of algae in lakes, samples of water were analyzed for free ammonia, total ammonia, inorganic phosphate, and total phosphate. The concentrations ranged from 10 to 200 ppb.

The method of Schreyer and Baes¹ was used to determine total uranium in phosphoric acid solutions of sol-gel uranium. This method proved very useful. After a 3.5 M H_3PO_4 –4.5 M H_2SO_4 solution of the urania was passed through a Jones reductor, no U(III) was found to be present in the solution. Before the use of this method, all total uranium analyses in H_3PO_4 solution were done coulometrically.

The methods for determining carbon and formate in aqueous solutions were improved by using a nitrogen stream to sweep the CO_2 into a caustic absorber. With the Conway diffusion flasks formerly used, only a limited amount of heat can be applied and the diffusion rate of the CO_2 is slow.

The physical and chemical analyses of coated particles were continued. Of the analyses, the most useful were measurement of mercury density at 15, 80, and 15,000 psi and of grams of carbon per gram of core. The density values were ob-

tained with an American Instrument Company mercury porosimeter. The carbon/core ratio was obtained by burning a weighed sample and then weighing the residue.

A cutoff wheel was obtained to cut samples of tungsten, molybdenum, niobium, and titanium alloys.

Several other methods were modified by D. C. Canada for use by the General Analyses Laboratory (Sects. 4.4.e, 4.4.h, 4.5.a, and 4.6.e).

The addition of the analytical biochemistry work increased the number of reported results from 50,134 for last year to 99,125 for this year.

14.3 GENERAL HOT-ANALYSES LABORATORY

C. E. Lamb

In the General Hot-Analyses Laboratory about 19,000 analyses were made on some 7100 samples, an increase of 6% over the number done last year. This increase was attained in spite of a 25% reduction in the staff. About two-thirds of the samples were received from the Operations, Chemical Technology, Reactor, and Reactor Chemistry Divisions; the balance was from seven other sources.

A glove-box facility consisting of four boxes was installed. Equipment for measuring certain physical properties of solid samples, mainly from the Chemical Technology Division's Plutonia Sol-Gel Program, was placed in the boxes. The measurements include density by means of a mercury porosimeter, gas released, and surface area. Also, carbon was determined by a Leco combustion-gas-chromatographic method. A fifth box is now being equipped with a combustion furnace and a Nitrox-6 Analyzer for the determination of nitrogen and oxygen. A sealed glove box in Laboratory 103 was used most of the past year by H. H. Ross² and S. B. Lupica² for special work with ^{147}Pm .

One of the major sources of work was from the MSRE of the Reactor Division. Power runs 8 through 12 were completed, and about 200 fuel samples were submitted. With few problems, the samples were analyzed for major constituents, corrosion products, and oxygen.

A method to determine the U^{3+}/U^{4+} ratio in MSRE fuel samples was developed by the Reactor Projects Group (Sect. 3.1.b). The apparatus used

¹J. M. Schreyer and C. F. Baes, Jr., "Volumetric Determination of Uranium(VI) in Phosphate Solutions," *Anal. Chem.* 25, 644 (1953).

²Nuclear and Radiochemistry Group.

for this determination was installed in cell 3. Because of the potential radiation hazard of removing the traps from the hot cell, the apparatus will be modified to allow the entire operation to be made within the hot cell.

Some work was done for facilities outside the Oak Ridge National Laboratory. Total gas released was determined in $\text{PuO}_2\text{-UO}_2$ samples received from Nuclear Fuel Services, Erwin, Tennessee. More tantalum-clad samples from the Argonne National Laboratory were analyzed for Cu, Mg, Pu, and U. Some EBR-II fuel (95% enriched uranium and 5% fission products) samples for the determination of Al and Fe were also received from Argonne. Aluminum was determined by the spectrophotometric aluminum method after the interfering ions were removed by liquid-liquid extraction with tricapriline (Alamine). Carbon was determined on some scrapings from the surface of several fuel elements from the Puerto Rican Bonus Reactor. Carbon dioxide was evolved by oxidation of the carbon, trapped on a 5A molecular-sieve column, and determined by gas chromatography.

Carbon was also determined on some metal carbides and cesium chloride product samples from the Isotopes Division. The carbides of metals such as Nb, Ta, Ti, and W were dissolved in a mixture of HNO_3 and HF. The resulting solution was filtered through fine quartz, and the residue was analyzed for carbon.

14.4 RADIOISOTOPES-RADIOCHEMISTRY LABORATORY

E. I. Wyatt

The types of work performed in the Radioisotopes-Radiochemistry Laboratory were essentially unchanged this year. The routine determination of oxygen with the 14-Mev neutron generator was begun. The new experimental products ^{33}P , $^{69,69m}\text{Zn}$, ^{132}Te , and ^{171}Tm are now being assayed. New equipment on order includes a 4096-channel pulse-height analyzer and a new flame photometer.

A large part of the work consisted in fission product analyses in support of nuclear safety experiments done by the Reactor and the Reactor Chemistry Divisions. Another large fraction resulted from fission product analysis associated with both the fuel and graphite-moderator specimens from the Molten-Salt Loop Experiment and

the MSRE. The total number of analyses increased from 58,000 in 1966 to 70,000 in 1967.

A paper was presented orally on the application of computer programs by this laboratory.³ A chapter titled "Equipment and Techniques for Handling Liquid Samples" was completed for inclusion in Vol. 10 of SERIES IX, **Progress in Nuclear Energy**.⁴

14.5 QUALITY CONTROL

G. R. Wilson

In January a weighted average 2S% was calculated for each control program by using the 2S values obtained during the four quarters of 1966. Based on these calculations, new limits were established for some of the control programs. The limit for the colorimetric thorium control program in the General Analyses Laboratory was increased from 2 to 3%, and the colorimetric uranium limit was decreased from 4 to 3%. The control limit for high carbon content by the Leco method was

³M. E. Pruitt, "Application of Computer Programs for Analysis of Radioisotopes," presented at 153d Meeting of the American Chemical Society, Miami Beach, Fla., Apr. 9-14, 1967.

⁴R. R. Rickard, "Equipment and Techniques for Handling Liquid Samples," to be included in Vol. 10, *Remote Analysis of Radioactive Materials*, of SERIES IX, ANALYTICAL CHEMISTRY, ed. by H. A. Elion and D. C. Stewart, in **Progress in Nuclear Energy**, Pergamon Press, New York.

Table 14.1. Distribution by Laboratories of Control Tests for July 1966-June 1967

Laboratory	Number of		Quality	
	Control Results		Level (%) ^a	
	Total	Outside Fixed Limits	1966	1967
General Hot-Analyses	2129	130	85.9	93.89
General Analyses	2118	90	97.2	95.75
Total	4247	220	92.6	94.82

^aControl results within prescribed 2S limits.

increased from 5 to 7%. The control limits for the amperometric chromium, colorimetric iron, and colorimetric nickel determinations in the MSRE control program of the General Hot-Analyses Laboratory were decreased from 15 to 10%.

Although the number of control results decreased by 14% from the like period a year ago, the overall quality level has improved as shown by Table 14.1. Table 14.2 gives the number of control results and their specific characteristics.

Table 14.2. Distribution by Methods of Control Results for July 1966-June 1967

Type of Method	Constituent	Number of Control Programs		Number of Control Results	
Amperometric	Chromium	1		238	
	Zirconium	1	2	210	448
Colorimetric (spectrophotometric)	Aluminum	1		107	
	Chromium	2		109	
	Iron	3		364	
	Molybdenum	1		30	
	Nickel	3		285	
	Thorium	2		337	
	Uranium	2	14	391	1623
Coulometric	Uranium	3	3	1239	1239
Fluorometric	Uranium	2	2	305	305
Gravimetric	Carbon	2	2	285	285
Photoneutron	Beryllium	1	1	278	278
Volumetric	Aluminum	1		67	
	Sulfate	1	2	2	69
Total			26		4247

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 1966. The updated indexes are titled "Indexes to the *Oak Ridge National Laboratory Master Analytical Manual* (1953-1966), TID-7015 (Indexes), Revision 4." 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 \$3.00. The indexes were issued as a companion document to Supplement 9 of TID-7015.

The computer work required for the preparation of the updated indexes was supervised by Ann S. Klein.¹

15.2 MAINTENANCE OF THE ORNL MASTER ANALYTICAL MANUAL

The ninth supplement to the reprinted form of the *ORNL Master Analytical Manual* was published. It contains the new methods and the revisions issued in 1966. This supplement, designated TID-7015 (Suppl. 9), 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 \$3.00.

Altogether, three new methods were added to the *Manual*; two of these were for the purpose of record only. Revisions were made to one method. 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.

¹Technical Information 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

H. A. Laitinen*
University of Illinois

Gleb Mamantov
University of Tennessee

G. H. Morrison*
Cornell University

M. L. Moss*
Institute for Muscle Disease, Inc.

A. O. C. Nier
University of Minnesota

L. B. Rogers*
Purdue University

*Advisory Committee Member.

16.2 ORAU RESEARCH PARTICIPANTS

Two ORAU Research Participants were with us in the summer of 1967.

J. C. Guyon, Professor of Chemistry, University of Missouri, worked with *W. D. Shults*, Methodology Group, to develop fluorometric methods for phosphate with aluminum-morin and tin-flavonol systems (Sect. 4.4.d) and to study the spectrophotometric determination of plutonium with arsenazo (Sect. 4.4.c).

L. C. Hall, Professor of Chemistry, Vanderbilt University, worked with *A. E. Cameron* in a mass spectrometric search for neon in minerals and in volcanic glasses (Sect. 7.4).

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 1966 on a two-year assignment to work in the Analytical Biochemistry Group with *Gerald Goldstein*. On October 1, 1967, he became a permanent member of the Division.

16.4 GRADUATE THESIS RESEARCH PROGRAMS

16.4.a ORAU Graduate Fellowship Program

H. W. Jenkins, a graduate student in the Department of Chemistry with *Professor Gleb Mamantov* at the University of Tennessee, is continuing his work toward the Ph.D. degree. In the Reactor Projects Group with *J. C. White* and *D. L. Manning*, he is studying reference electrodes in fluoride melts to develop a practical reference electrode. Also, he is investigating the behavior of several metallic couples with the objective of establishing an electromotive series for fluoride melts.

16.5 ORNL LOANEEES

L. P. Turner III, Department of Chemistry, University of Tennessee, Knoxville, continued the pyrolytic gas chromatography of macromolecules with *A. S. Meyer*, Reactor Projects Group.

F. L. Whiting, a graduate student in the Department of Chemistry with *Professor Gleb Mamantov* at the University of Tennessee, is continuing his doctoral thesis research. In the Reactor Projects Group with *J. C. White* and *J. P. Young*, he is studying the simultaneous electrochemical generation and absorption spectrophotometric identification and characterization of solute ions of unusual oxidation states in molten fluoride melts.

16.6 SUMMER STUDENT PROGRAMS

In the summer of 1967, nine students participated in summer student programs.

16.6.a ORAU Student Trainee Program

D. R. Anderson, a senior at Elizabethtown College, Elizabethtown, Pennsylvania, assisted in the gas chromatographic work of the Reactor Projects Group. With *W. F. Peed* he studied the gas chromatography of corrosive gases, especially interhalogen compounds; with *A. S. Meyer* he assisted in evaluating a helium-discharge detector; and with *A. D. Horton* he used gas chromatography as a preparative technique.

Frances E. Kraus, a senior at Nazareth College, Nazareth, Kentucky, worked with *Helen P. Raaen*, Methodology Group, to investigate the thin-layer chromatography of transfer ribonucleic acids and of proteins (Sect. 4.1.b).

Karen E. Pashman, a senior at McMurry College, Abilene, Texas, worked with *W. D. Shults*, Methodology Group, in the study of the controlled-potential coulometric determination of U(VI) in the presence of Cu(II) (Sect. 4.2.b).

Susan S. Potterton, a senior at Randolph-Macon Woman's College, Lynchburg, Virginia, also worked with *W. D. Shults* to evaluate the performance of ion-selective electrodes (Sect. 4.2.c).

16.6.b ORNL Graduate Program

G. C. Giesler, a second-year graduate student at Michigan State University, East Lansing, did special problems in radiochemical separations and measurements, for example, the separation of ^{32}Si and ^{32}P and the measurement of the decay scheme of ^{19}O . He worked with *J. S. Eldridge* of the Nuclear and Radiochemistry Group.

Janice C. Laney, a graduate of Ouachita University, Arkadelphia, Arkansas, now a first-year graduate student at the University of Tennessee, Memphis, worked with *Gerald Goldstein*, Analytical Biochemistry Group. She assisted in research to develop methods for the determination of partially purified individual transfer ribonucleic acids.

J. B. Pressly, formerly a student at the University of Tennessee, Knoxville, did gamma-ray spectrometry and data analysis with *E. I. Wyatt*, Radioisotope-Radiochemistry Laboratory.

E. S. Wolfe, who has completed one year of graduate study at the University of North Carolina, Chapel Hill, worked with *D. J. Fisher*, Analytical Instrumentation Group, to investigate the short- and long-term precision obtainable by single-cell first-derivative dc polarography and to explore ways of improving the precision (Sect. 1.7).

16.6.c ORNL Undergraduate Program

T. G. Fox, a junior at the University of Tennessee, Knoxville, assisted *J. R. Sites*, Mass Spectrometry Service Group, and *J. A. Carter*, Spectrochemistry Group, in mass spectrometric analyses with the spark-source mass spectrometer and also in the analysis of gases by mass spectrometry with electron bombardment sources.

16.7 ALIEN GUESTS IN RESIDENCY

Three scientists from three countries were guests part of the year in the Analytical Chemistry Division.

Guest	Sponsor	Division Group	See Section
T. M. Florence Australian Atomic Energy Commission Research Establishment, Sutherland, New South Wales	Australian Atomic Energy Commission	Methods Development Group	2.1
Aart Jurriaanse Atomic Energy Board, Pretoria, Republic of South Africa	South African Atomic Energy Board	Nuclear and Radio- chemistry Group	9.7.a, 9.7.d
Jun-Lan Wang (<i>Mrs. Kian-Chu Li</i>) Institute of Nuclear Science, National Tsing Hua University, Hsinchu, Taiwan, Republic of China	International Atomic Energy Agency	Nuclear and Radio- chemistry Group	

Part E. Extralaboratory Professional Activities

The extralaboratory professional activities of members of the Division are numerous and varied. They reflect participation in a diversity of work, especially at the national and international levels.

L. C. Bate

Member: Lithium Metal Task Force
Division N, Nonferrous Metals
Committee E-3 on Chemical Analysis of Metals
American Society for Testing and Materials (ASTM)

A. E. Cameron

Member: International Commission on Atomic Weights
International Union of Pure and Applied Chemistry (IUPAC)
Advisory Board (1967–1969)
Analytical Chemistry

L. T. Corbin

Member: Subcommittee N5.1, Fuel Manufacture and Fabrication
American Standards Association (ASA)
ASTM Committee E-10 on Radioisotopes and Radiation Effects

J. S. Eldridge

Secretary: Subcommittee on the Use of Radioactivity Standards
Committee on Nuclear Sciences
National Academy of Sciences–National Research Council (NAS-NRC)

D. J. Fisher

Member: Editorial Board
Chemical Instrumentation

Gerald Goldberg

Member: Division N, Nonferrous Metals
ASTM Committee E-3 on Chemical Analysis of Metals
Subcommittee I, Research
ASTM Committee G-1 on Corrosion of Metals
Task Group B, Liquid Sodium Systems–Alkali Metals Corrosion
Subcommittee VIII, Corrosion of Nuclear Materials
(Also of Committee G-1)
Task Group C, Liquid Potassium Systems–Alkali Metals Corrosion
(Also of Subcommittee VIII)

A. D. Horton

Member: Subcommittee III, Research
ASTM Committee E-19 on Chromatography

M. T. Kelley

Member: Chemistry Directors' Meeting
Division of Research
United States Atomic Energy Commission
Washington, D.C.

Analytical Specialists Group
Technology Subcommittee
Union Carbide Corporation

Program Review Committee
Chemistry Section
Laboratory of Nuclear Science
Massachusetts Institute of Technology

Committee on Analytical Chemistry (1966–1969)
Division of Chemistry and Chemical Technology
National Academy of Sciences–National Research Council (NAS-NRC)

Advisory Committee
Microchemical Journal

Board of Editorial Advisors
Analytica Chimica Acta

C. E. Lamb

Member: Executive Committee (1967–1970)
Remote Systems Technology Division
American Nuclear Society (ANS)

Chairman: Membership Committee (1966–1967)
(Also of ANS Remote Systems Technology Division)

W. S. Lyon

Liaison Officer, ASTM Committee E-10 on Radioisotopes and Radiation Effects

Member: Subcommittee I, Burnup
ASTM Committee E-10

Subcommittee III, Tracer Applications
(Also of Committee E-10)

Subcommittee V, Neutron Dosimetry
(Also of Committee E-10)

Editorial Board
Chemical Instrumentation

Scientific Committee 25 on Radiation Protection in the Use of Small Neutron Generators
National Council on Radiation Protection and Measurement

Committee N5-4, Use and Handling of Radioisotopes
United States of America Standards Institute

Chairman: Referee Committee F, Industrial Wastes
Subcommittee on Radiological Methods for Biological and Environmental Samples
American Public Health Association (APHA)

Emissary on activation analysis to:

Chemistry and Biology Divisions
 Thai Atomic Energy for Peace Laboratories
 Bangkok, Thailand

(Negotiated by the Agency for International Development between the Thailand Atomic Energy Commission for Peace and ORNL)

S. A. Reynolds

Member: Subcommittee on Radiological Methods for Biological and Environmental Samples
 American Public Health Association (APHA)

ASTM Committee D-19 on Industrial Water

Subcommittee IX, Methods of Radiochemical Analysis

ASTM Committee D-19

ASTM Committee E-10 on Radioisotopes and Radiation Effects

Subcommittee III, Tracer Applications

ASTM Committee E-10

Task Group on Neutron Cross Sections

(Also of Committee E-10)

H. H. Ross

Emissary on Radiochemistry to:

Chemistry Division
 Thai Atomic Energy for Peace Laboratories
 Bangkok, Thailand

(Negotiated by the Agency for International Development between the Thailand Atomic Energy Commission for Peace and ORNL)

Participant: 1967 I-R 100 Competition sponsored by *Industrial Research*
 (The Laboratory's winning entry was the ORNL-Ross radioisotopic light source photometer.)

E. J. Spitzer

Chairman: Analytical Group
 East Tennessee Section
 American Chemical Society

J. R. Walton

Member: Subcommittee VI, Practices and Procedures
 ASTM Committee E-14 on Mass Spectrometry

J. C. White

Secretary-Treasurer:

Division of Analytical Chemistry (1967-1969)
 American Chemical Society (ACS)

Representative from the ACS to:

Project N11, Basic Materials and Materials Testing for Nuclear Applications
 Nuclear Standards Board
 United States of America Standards Institute
 (Sponsored by ASTM)

Member: Editorial Board
Analytical Letters
Standing Committee for Sodium Impurities
New York Operations Office
United States Atomic Energy Commission
Committee on Analytical Chemistry
National Academy of Sciences—National Research Council (NAS-NRC)
Panel 310.00, Analytical Chemistry, of NAS-NRC Panels Advisory to:
Institute for Materials Research
United States National Bureau of Standards

Chairman: Subcommittee on Reference Materials
NAS-NRC Committee on Analytical Chemistry

Anna M. Yoakum

Secretary-Treasurer:
Southeastern Section
Society for Applied Spectroscopy

Delegate: National Governing Board
Society for Applied Spectroscopy

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

Thesis, Contributions to Books

AUTHOR(S)	TITLE	PUBLISHER
1 Carter, J. A.	"Analysis of Solution and Radioactive Samples by Spark Source Mass Spectrometry," pp. 12-20 in <i>MS-7 Users Meeting April 10-11, 1967</i>	Picker Nuclear, White Plains, N.Y., 1967
2 Costanzo, D. A.	<i>A Resonant Cavity High Frequency Oscillometer</i> (Ph.D. thesis)	Michigan State University, June 1967
3 Eldridge, J. S.	"Standardization of Mercury-197," pp. 313-322 in <i>Standardization of Radionuclides</i> (Proceedings of a Symposium Organized by the International Atomic Energy Agency, Vienna, October 14-16, 1966)	International Atomic Energy Agency, Vienna, March 1967
4	"Secondary Standardizations with Digital Computer Techniques to Normalize Multiple Gamma-Ray Spectra for Direct Efficiency Determination," pp. 639-646 in <i>Standardization of Radionuclides</i> (Proceedings of a Symposium Organized by the International Atomic Energy Agency, Vienna, October 14-16, 1966)	International Atomic Energy Agency, Vienna, March 1967
5 Horton, A. D., A. S. Meyer, Jr., M. T. Kelley	"Applications of Gas Chromatography in Nuclear Technology," pp. 337-349 in <i>Chromatography and Methods of Immediate Separation</i> [Proceedings of the Meeting Journées Hellènes d'Étude des Méthodes de Séparation Immédiate et de Chromatographie (III ^e J.I.S.I.C.) held at the National Technical University, Athens, Greece, September 19-24, 1965. Union of the Greek Chemists, Athens] ed. by G. Parissakis	Patric Press, Athens, 1966

- 6 Kelley, M. T. "The Accurate and Precise Determination of Uranium with Special Reference to Coulometry," pp. 54-71 in Technical Reports Series No. 62, *Analytical Chemistry of Nuclear Materials* (Report of Panel on Analytical Chemistry of Nuclear Materials Held in Vienna March 23-26, 1964) International Atomic Energy Agency, Vienna, 1966
- 7 "Current Capabilities in Analysis of Trace Substances: Electroanalytical Methods," pp. 45-50 in *Proceedings University of Missouri's 1st Annual Conference on Trace Substances in Environmental Health* University of Missouri, Columbia, 1967
- 8 Laing, W. R., E. C. Lynn "Alkyl Benzene Sulfonate (ABS) Control for the Foam Separation Process," pp. 158-159 in *Technicon Symposia 1965, Automation in Analytical Chemistry*, New York, September 1965, ed. by L. T. Skeggs, Jr. Mediad, New York, 1966
- 9 Ricci, E., R. L. Hahn, J. E. Strain, F. F. Dyer "Helium-3 Activation Analysis," pp. 200-205 in *Proceedings of the 1965 International Conference on Modern Trends in Activation Analysis*, College Station, Texas, April 19-22, 1965 Activation Research Laboratory, Texas A & M, College Station, Tex.
- 10 Ross, H. H. "Theoretical and Experimental Aspects of Quenching Variables from Biomedical Samples in Liquid Scintillator Systems," pp. 409-426 in *Radioisotope Sample Measurement Techniques in Medicine and Biology* (Proceedings of the Symposium on Radioisotope Sample Measurement Techniques in Medicine and Biology held by the International Atomic Energy Agency in Vienna, May 24-28, 1965) International Atomic Energy Agency, Vienna, 1966

Articles

AUTHOR(S)	TITLE	PUBLISHER
11 Bate, L. C., W. B. Healy, ¹ T. G. Ludwig ²	"Microelement Content of Hair from New Zealand Boys as Determined by Neutron Activation Analysis"	<i>New Zealand J. Sci.</i> 9 , 559 (1966)
12 Bell, J. T.,* R. E. Biggers	"The Absorption Spectrum of the Uranyl Ion in Perchlorate Media. Part II. The Effects of Hydrolysis on the Resolved Spectral Bands"	<i>J. Mol. Spectry.</i> 22 , 262 (1967)
13 Canada, D. C.	"Spectrophotometric Thiocyanate Determination of Niobium in the Presence of Fluoride"	<i>Anal. Chem.</i> 39 , 381 (1967)
14 Canada, D. C., W. R. Laing	"Use of a Density Gradient Column to Measure the Density of Microspheres"	<i>Anal. Chem.</i> 39 , 691 (1967)

¹Alien Guest, 1963-64 from Soil Bureau, Department of Scientific and Industrial Research, Private Bag, Lower Hutt, New Zealand.

²Dental Research Unit, Medical Council, P.O. Box 3155, Wellington, New Zealand.

- 15 Fulmer, C. B.,* I. R. Williams,³ T. H. Handley, G. F. Dell,⁴ L. N. Blumberg⁴ "Evidence for Photofission of Iron" *Phys. Rev. Letters* **19**, 522 (1967)
- 16 Goldstein, G. "Ligand-Exchange Chromatography of Nucleotides, Nucleosides, and Nucleic Acid Bases" *Anal. Biochem.* **20**, 477 (1967)
- 17 Hahn, R. L., E. Ricci "Interactions of ³He Particles with Boron, Nitrogen, Sodium, and Beryllium" *Nucl. Phys.* **A101**, 353 (1967)
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- 19 Hertel, G. R. "Surface Ionization. I. Desorption of U⁺ Ions from W and Re Surfaces" *J. Chem. Phys.* **47**, 133 (1967)
- 20 "Surface Ionization. II. The First Ionization Potential of Uranium" *J. Chem. Phys.* **47**, 335 (1967)
- 21 Jurriaanse, A.,⁵ F. L. Moore "Use of Organic Additives to Induce the Selective Liquid-Liquid Extraction of Niobium with Thenoyltrifluoroacetone" *Anal. Chem.* **39**, 494 (1967)
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- 24 Kubota, H. "Gamma Dosimetry at 150-250°C with Deaerated Ferrous Sulfate Solution" *J. Inorg. Nucl. Chem.* **28**, 3053 (1966)
- 25 Lyon, W. S. Book Review of: *Radioisotope Instruments in Industry and Geophysics*, vols. I and II, International Publications, New York, 1966 *Mater. Res. Std.* **7**(2), 92 (1967)
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- 27 Manahan, S. E.⁶ "Electron Exchange Between Copper(I) and Copper(II) in Acetonitrile" *Can. J. Chem.* **45**, 2451 (1967)
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- 29 Moore, F. L., A. Jurriaanse⁵ "Separation of Californium from Curium and Berkelium from Cerium by Extraction Chromatography" *Anal. Chem.* **39**, 733 (1967)

³Chairman, Department of Mathematics and Physics, Associate Professor of Physics, Knoxville College, Tennessee.

⁴Cambridge Electron Accelerator, Cambridge, Mass.

⁵Alien Guest from the South African Atomic Energy Board, Pretoria, Republic of South Africa.

⁶ORAU Research Participant, summer 1966; Assistant Professor of Chemistry, Department of Chemistry, University of Missouri, Columbia.

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R. E. Druschel,* *J. Inorg. Nucl. Chem.* 29,
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- 32 Pappas, W. S.,⁷ "A Comparative Condensation Pressure
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C. W. Weber⁷ *Anal. Chem.* 38, 1570 (1966)
- 33 Reynolds, S. A. "Radioisotope Characteristics, Measurements,
and Standards" *Isotopes Radiation Technol.*
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- 34 Book Review of: *Radiometric Titrations*, T.
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- 36 "Simple Method to Calculate Sensitivities and
Interferences vs Bombardment Energy in ³He
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- 38 Rubin, I. B., "The Determination of Transfer Ribonucleic
A. D. Kelmers,* Acid by Aminoacylation. I. Leucine and
G. Goldstein Phenylalanine Transfer Ribonucleic Acid
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- 39 Scott, C. D.,* "Automatic, High Resolution Analysis of Urine
J. E. Attrill, for Its Ultraviolet-Absorbing Constituents"
N. G. Anderson* *Proc. Soc. Exptl. Biol. Med.*
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- 40 Shults, W. D., "Controlled-Potential Differential DC Polarog-
W. B. Schaap⁸ raphy. II. The ΔE-Differential Polarographic
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in Comparative Polarography, Theory and Ex-
periment" *Anal. Chem.* 39, 1384 (1967)
- 42 Shults, W. D., "Controlled-Potential Differential DC Polarog-
D. J. Fisher, raphy. Comparative Polarography"
W. B. Schaap⁸ *Anal. Chem.* 39, 1379 (1967)
- 43 Shults, W. D., "Controlled-Potential Differential DC Polarog-
D. J. Fisher, raphy. I. Instrumentation, Apparatus, and
H. C. Jones, Techniques"
M. T. Kelley,
W. B. Schaap⁸ *Z. Anal. Chem.* 224, 1 (1967)

⁷ORGDP.⁸Professor of Chemistry, Indiana University, Bloomington.

- 44 Stokely, J. R., Jr., F. L. Moore "New Separation Method for Americium Based on the Liquid-Liquid Extraction Behavior of Americium(V)" *Anal. Chem.* **39**, 994 (1967)
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- 46 White, J. C. Book Review of: *Trace Analysis: Physical Methods*, G. H. Morrison, ed., Interscience, New York, 1965 *Anal. Chem.* **38**(13), 77A (1966)
- 47 Book Review of: *Progress in Nuclear Energy, SERIES IX, ANALYTICAL CHEMISTRY, vol. 7*, H. A. Eliön, D. C. Stewart, eds., Pergamon, New York, 1966 *Anal. Chem.* **39**(3), 84A (1967)
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- 49 Young, J. P. "Spectra of Uranium(IV) and Uranium(III) in Molten Fluoride Solvents" *Inorg. Chem.* **6**, 1486 (1967)
- 50 Young, J. P., G. Mamantov,⁹ F. L. Whiting¹⁰ "Simultaneous Voltammetric Generation of U(III) and Spectrophotometric Observation of the U(III)-U(IV) System in Molten Lithium Fluoride-Beryllium Fluoride-Zirconium Fluoride" *J. Phys. Chem.* **81**, 782 (1967)
- 51 Zittel, H. E., T. M. Florence¹¹ "Voltammetric and Spectrophotometric Study of the Zirconium-Alizarin Red S Complex" *Anal. Chem.* **39**, 320 (1967)
- 52 "Voltammetric Method for the Determination of Zirconium" *Anal. Chem.* **39**, 355 (1967)
- 53 Zittel, H. E., F. J. Miller "Anodic Reactions of the Halides in Dimethyl Sulfoxide at the Pyrolytic-Graphite Electrode" *Anal. Chim. Acta* **37**, 141 (1967)
- 54 "Chronopotentiometry of the Iodine System at the Pyrolytic-Graphite and Glassy-Carbon Electrodes" *J. Electroanal. Chem.* **13**, 193 (1967)
- 55 Anonymous (work by J. R. Stokely and W. D. Shults) "Controlled-Potential Coulometric Titration of Americium" *Chem. Eng. News*, Oct. 23, 1967, p. 67

Reports

AUTHOR(S)	TITLE	REPORT NO. AND DATE
56 Fisher, D. J.	<i>Report on Foreign Travel of Dale J. Fisher July 11, 1964 to August 30, 1964</i>	April 10, 1967 (unpublished)
57 Goldstein, G., S. A. Reynolds	<i>Half-Lives and Specific Activities of Common Radionuclides</i>	ORNL-TM-1318

⁹Consultant; Associate Professor of Chemistry, University of Tennessee, Knoxville.

¹⁰ORNL Loanee; Graduate Student, Department of Chemistry, University of Tennessee, Knoxville.

¹¹Alien Guest from Australian Atomic Energy Commission, Sutherland, New South Wales, Australia.

- 58 Kelley, M. T. *Statistical Quality Control Report, Analytical Chemistry Division, October Through December 1966* Jan. 10, 1967 (unpublished)
- 59 *Statistical Quality Control Report, Analytical Chemistry Division, January Through March, 1967* Apr. 14, 1967 (unpublished)
- 60 *Statistical Quality Control Report, Analytical Chemistry Division, April Through June, 1967* July 11, 1967 (unpublished)
- 61 Lewis, R. E.,*
S. A. Reynolds *Preparation of Phosphorus-33 by Irradiation of Enriched Sulfur-33 in Highly Thermalized Flux* ORNL-4051 (January 1967)
- 62 Lyon, W. S. *Report of Foreign Travel of W. S. Lyon April 26, 1967 to May 17, 1967* May 26, 1967 (unpublished)
- 63 *Report of Foreign Travel of W. S. Lyon October 14, 1966 to November 15, 1966* Nov. 30, 1966 (unpublished)
- 64 Mitchell, B. E.,⁷
R. K. Sood,*
W. J. Ross *Tests of the Deaerator at the Freeport Seawater Desalting Plant and Evaluation of Dissolved Gas Analysis* ORNL-TM-1953 (Oct. 3, 1967)
- 65 Raaen, Helen P., ed. *Analytical Chemistry Division Annual Progress Report for Period Ending October 31, 1966* ORNL-4039 (January 1967)
- 66 *Oak Ridge National Laboratory Master Analytical Manual, Supplement 9* TID-7015, suppl. 9 (March 1967)
- 67 Raaen, Helen P.,
Ann K. Haas,* eds. *Cumulative Indexes to the Analytical Chemistry Division Annual Progress Reports 1964-1966* ORNL-3904, rev. 1 (January 1967)
- 68 Raaen, Helen P.,
Ann S. Klein,* eds. *Indexes to the Oak Ridge National Laboratory Master Analytical Manual (1953-1966)* TID-7015, Indexes, rev. 4 (March 1967)
- 69 Stelzner, R. W. *Report of Foreign Assignment of Robert W. Stelzner November 27, 1964 to December 5, 1965* Aug. 25, 1967 (unpublished)
- 70 Stokely, J. R., Jr. *Solvent Extraction and Gas Chromatography of Metal Fluoroacetylacetonates* TID-23663 (May 1966)
- 71 White, J. C. *Analytical Chemistry Research and Development Group Monthly Summary - November 1966* Nov. 30, 1966 (unpublished)
- 72 *Analytical Chemistry Research and Development Group Monthly Summary - December, 1966* Jan. 4, 1966 (unpublished)
- 73 *Analytical Chemistry Research and Development Group Monthly Summary - January, 1967* Jan. 27, 1967 (unpublished)
- 74 *Analytical Chemistry Research and Development Group Monthly Summary - February, 1967* Mar. 2, 1967 (unpublished)
- 75 *Analytical Chemistry Research and Development Group Monthly Summary - May, 1967* May 26, 1967 (unpublished)
- 76 *Analytical Chemistry Research and Development Group Monthly Summary - June, 1967* June 28, 1967 (unpublished)

77		<i>Analytical Chemistry Research and Development Group Monthly Summary – August, 1967</i>	Aug. 28, 1967 (unpublished)
78		<i>Analytical Chemistry Research and Development Group Monthly Summary – September, 1967</i>	Sept. 26, 1967 (unpublished)
79		<i>Analytical Chemistry Research and Development Group Monthly Summary – October, 1967</i>	Oct. 27, 1967 (unpublished)
80	White, J. C., C. Feldman	<i>Analytical Chemistry Research and Development Group Monthly Summary – March, 1967</i>	Mar. 28, 1967 (unpublished)
81		<i>Analytical Chemistry Research and Development Group Monthly Summary – July, 1967</i>	July 29, 1967 (unpublished)
82	White, J. C., D. J. Fisher	<i>Analytical Chemistry Research and Development Group Monthly Summary – April, 1967</i>	April 27, 1967 (unpublished)
83	Anonymous	"Pipetters" in <i>Engineering Materials List</i> , TID-4100 (Supplement 37)	CAPE-1220 (March 1967)
84		"Filter Photometer" in <i>Engineering Materials List</i> , TID-4100 (Supplement 33)	CAPE-1313 (May 1966)

Methods Issued to the ORNL Master Analytical Manual

REVISED METHODS

AUTHOR(S)	TITLE	NUMBER	DATE
85 Wyatt, E. I.	"Iodine-131, Product Analysis Guide"	9 0733391	R. 3-27-67

RECORD-COPY METHODS

AUTHOR(S)	TITLE	NUMBER	DATE
86 Raaen, Helen P.	"Separation of Amanita Toxins by Thin-Layer Chromatography on Silica Gel G Chromatoplates"	1 00702	8-23-67
87 Raaen, Helen P.	"Separation of Complex Mixtures of Nucleic Acid Bases, Nucleosides, and Nucleotides by Two-Dimensional Thin-Layer Chromatography on Polyethyleneimine-Cellulose"	1 00703	10-1-67

ORAL PRESENTATIONS

Eleventh Conference on Analytical Chemistry in Nuclear Technology

More than 340 scientists, including 23 representatives from 9 foreign countries, attended the Eleventh Conference on Analytical Chemistry in Nuclear Technology, which was held in Gatlinburg, Tennessee, on October 10–12, 1967. Again, the Analytical Chemistry Division of ORNL sponsored the Conference.

The theme of the Conference was "Instrumental Methods in Analytical Chemistry." In addition to the 63 papers that were presented, panel discussions on the topics "Non-Reactor Methods of Activation Analysis" and "The Apathy in Isotope Applications: What Can Be Done?" were informative contributions, which added interest in the Conference.

The Conference Committee members were: M. T. Kelley, Division Director; L. J. Brady, Chairman; H. L. Holsopple, Treasurer; R. R. Rickard, H. H. Ross, W. D. Shults, E. J. Spitzer, and R. W. Stelzner.

Although the proceedings of the Conference will not be published, tape recordings were made of all the presentations. Copies of these recordings are available on a loan basis.

The Twelfth Conference in this series is tentatively scheduled to be held at the Mountain View Hotel and Motor Lodge, Gatlinburg, Tennessee, on October 8-10, 1968.

At Meetings of Professional Societies, Conferences, and the Like

AUTHOR(S)	TITLE	PRESENTED AT
88 Belew, W. L.	"A Solid-State, Controlled-Potential DC Polarograph and a Precision Drop-Time Controller"	11th Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Oct. 10-12, 1967
89	"Controlled-Potential DC Polarograph-Voltammeter (Q-2792)"	Analytical Chemistry Instrument Demonstration Conference. An Industrial Cooperation Conference, Oak Ridge National Laboratory, Oak Ridge, Tenn., Oct. 16-17, 1967
90 Botts, J. L., ¹² H. G. Davis, W. R. Laing	"A Vacuum Fusion-Gas Chromatographic Apparatus for Low Levels of Interstitial Gases"	11th Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Oct. 10-12, 1967
91 Canada, D. C., ¹² W. R. Laing	"Use of a Density Gradient Column to Measure the Density of Microspheres"	Southeastern Regional Meeting, American Chemical Society, Louisville, Ky., Oct. 27-29, 1967
92 Carter, J. A.	"Analysis of Solution and Radioactive Samples by Spark Source Mass Spectrometry"	MS-7 Users Meeting, White Plains, N.Y., Apr. 10-11, 1967
93 Dyer, F. F.	"Principles of Activation Analysis" (Invited Lecture)	South-Central Independent College Association of Chemists, Tennessee Wesleyan College, Athens, Tenn., Oct. 6, 1967
94 Eldridge, J. S.	"Gamma-Ray Spectroscopy" (Invited Lecture)	South-Central Independent College Association of Chemists, Tennessee Wesleyan College, Athens, Tenn., Oct. 6, 1967
95 Emery, J. F.	"Nuclear Analysis Using a Large Volume Ge(Li) Detector"	153d Meeting, American Chemical Society, Miami Beach, Fla., Apr. 10-14, 1967
96 Goldstein, G., ¹² W. L. Maddox, I. B. Rubin	"A Semi-Automated Filter Paper Disk Technique for the Determination of Transfer Ribonucleic Acids"	Technicon Symposium on Automation in Analytical Chemistry, New York, Oct. 2-4, 1967

¹²Speaker.

- 97 Guyon, J. C.,^{12,13}
W. D. Shults "The Fluorometric Determination of the Phosphate Ion" 11th Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Oct. 10-12, 1967
- 98 Hahn, R. L.,¹²
E. Ricci "Interactions of ³He Particles with Low-Z Elements - Excitation Functions from Experiment and from Direct-Interaction Theory" 153d Meeting, American Chemical Society, Miami Beach, Fla., Apr. 10-14, 1967
- 99 Hardy, C. J.,¹⁴
S. R. Buxton,*
T. E. Willmarth¹² "Chemical and Electron Optical Studies of the Sols, Gels, and Oxide Microspheres Prepared from the Rare-Earth Hydroxides" 6th Rare-Earth Conference, Gatlinburg, Tenn., May 3-5, 1967
- 100 Jones, H. C. "Controlled-Potential Coulometric Titrators (Q-2005-X50 and Q-2564)" Analytical Chemistry Instrument Demonstration Conference. An Industrial Cooperation Conference, Oak Ridge National Laboratory, Oak Ridge, Tenn., Oct. 16-17, 1967
- 101 Kelley, M. T. "Comparison of Analytical Chemistry and Activation Analysis" ORAU Neutron Activation Analysis Course, Special Training Division, Oak Ridge, Tenn., May 29-June 16, 1967
- 102 "Comparison of Activation Analysis to Other Analytical Methods" ORAU Summer Institute in Isotope Technology, ORAU Special Training Division, Oak Ridge, Tenn., June 26-Aug. 14, 1967
- 103 "Current Capabilities in Analysis of Trace Substances: Electroanalytical Methods" 1st Annual Conference on Trace Substances in Environmental Health, University of Missouri, Columbia, July 10-11, 1967
- 104 "Electroanalytical Research at Oak Ridge National Laboratory" Institut Ruder Bosković, Zagreb, Croatia, Yugoslavia, Sept. 11, 1967
- 105 Lund, J. R.,¹²
H. Kubota "Properties of Rock Salt of Interest to Radioactive Waste Disposal" 76th Meeting, Tennessee Academy of Science, Johnson City, Nov. 25-26, 1966
- 106 Lyon, W. S. "Systems Development - Analytical" 8th Annual Contractors' Meeting, Division of Isotopes Development, U.S. Atomic Energy Commission, Washington, D.C., Dec. 5-7, 1966
- 107 "Physical Techniques of Activation Analysis" International Atomic Energy Agency's Symposium on Nuclear Activation Techniques in Life Sciences, Amsterdam, May 7-13, 1967

¹³ORAU Research Participant; Associate Professor of Chemistry, University of Missouri, Columbia.

¹⁴Guest Scientist, 1965-66, from Chemistry Division, Atomic Energy Research Establishment, Harwell, England.

- 108 "Practice of Activation" ORAU Neutron Activation Analysis Course, ORAU Special Training Division, Oak Ridge, Tenn., May 29--June 16, 1967
- 109 "Activation Interferences" ORAU Summer Institute in Isotope Technology, ORAU Special Training Division, Oak Ridge, Tenn., Aug. 14, 1967
- 110 Maddox, W. L. "Precision Pipetter, Remotely Controlled, Corrosion Resistant" Analytical Chemistry Instrument Demonstration Conference. An Industrial Cooperation Conference, Oak Ridge National Laboratory, Oak Ridge, Tenn., Oct. 16--17, 1967
- 111 "Velocity-Servo Potentiometric Titrator" Analytical Chemistry Instrument Demonstration Conference. An Industrial Cooperation Conference, Oak Ridge National Laboratory, Oak Ridge, Tenn., Oct. 16--17, 1967
- 112 Maddox, W. L., M. T. Kelley¹² "An Output Sampler for the Technicon Auto-Analyzer" 21st International Congress of Pure and Applied Chemistry, Prague, Sept. 4--10, 1967
- 113 Mamantov, G.,^{9,12} D. L. Manning, H. W. Jenkins¹⁵ "Exchange Current Measurements on Nickel-Nickel(II) Couple in Molten Fluorides" 153d Meeting, American Chemical Society, Miami Beach, Fla., Apr. 10--14, 1967
- 114 Mamantov, G.,^{9,12} D. L. Manning, H. W. Jenkins,¹⁵ J. M. Dale "Electroanalytical Chemistry in Molten Fluorides" Southwest Regional Meeting, American Chemical Society, Albuquerque, N.M., Nov. 30--Dec. 2, 1966
- 115 Manning, D. L.,¹² G. Mamantov,⁹ H. W. Jenkins¹⁵ "Voltammetric and EMF Measurements on Nickel-Nickel(II) Couple in Molten Fluorides" 153d Meeting, American Chemical Society, Miami Beach, Fla., Apr. 10--14, 1967
- 116 Meyer, A. S., Jr.,¹² C. M. Boyd, J. E. Attrill "Automation of Gas Chromatographic Analyses at the Oak Ridge National Laboratory" 21st International Congress of Pure and Applied Chemistry, Prague, Sept. 4--10, 1967
- 117 Moore, F. L. "Recent Developments in the Analytical Chemistry of the Transplutonium Elements" 153d Meeting, American Chemical Society, Miami Beach, Fla., Apr. 10--14, 1967
- 118--120 Mueller, T. R. "Instrumentation for Electrochemical Kinetics Studies" (Series of Three Invited Lectures) Course No. 645 Department of Chemistry, University of Tennessee, Knoxville, May 20, 25, and 27, 1967

¹⁵ORAU Graduate Fellow; Graduate Student, Department of Chemistry, University of Tennessee, Knoxville.

- 121 Mueller, T. R. "Controlled-Potential and Controlled-Current Cyclic Voltammeter" Analytical Chemistry Instrument Demonstration Conference. An Industrial Cooperation Conference, Oak Ridge National Laboratory, Oak Ridge, Tenn., Oct. 16-17, 1967
- 122 Pruitt, M. E. "Application of Computer Programs for Analysis of Radioisotopes" 153d Meeting, American Chemical Society, Miami Beach, Fla., Apr. 10-14, 1967
- 123 Raaen, Helen P. "Teflon Polarographic Dropping-Mercury Electrode" Analytical Chemistry Instrument Demonstration Conference. An Industrial Cooperation Conference, Oak Ridge National Laboratory, Oak Ridge, Tenn., Oct. 16-17, 1967
- 124 Ricci, E. "³He Activation' in Panel Discussion on 'Non-Reactor Methods of Activation Analysis'" 11th Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Oct. 10-12, 1967
- 125 Ricci, E.,¹²
R. L. Hahn "Simple Method to Calculate Sensitivities and Interferences vs Bombardment Energy in ³He Activation Analyses" 13th Annual Meeting of American Nuclear Society, San Diego, Calif., June 11-15, 1967
- 126 "Present Status of the Helium-3 Activation Analysis Program at ORNL" EURATOM Conference on Practical Aspects of Activation Analysis with Charged Particles, II, Liege, Belgium, Sept. 21-22, 1967
- 127 Ross, H. H. "Development of Radiation Stimulated Light Sources" 8th Annual Contractors' Meeting, Division of Isotopes Development, U.S. Atomic Energy Commission, Washington, D.C., Dec. 5-7, 1966
- 128 "Precision Photometric Analysis in the Ultra-violet Using a Radioisotopic Light Source Photometer" 153d Meeting, American Chemical Society, Miami Beach, Fla., Apr. 10-14, 1967
- 129 "The Analytical Applications of the Secondary Effects of Radiation: Precision Photometry Using a Radioisotopic Light Source" Frontiers in Chemistry Lecture, Wayne State University, Detroit, Mich., June 5, 1967
- 130 "Radiochemical Separations" Thai Atoms for Peace Laboratory, Bangkok, Thailand, Aug. 24, 1967
- 131 "Application of Radioisotopes to Problems of Chemical and Physical Research" National Tsing Hua University, Hsinchu, Taiwan, Republic of China, Aug. 29, 1967
- 132 "ORNL-Ross Radioisotopic Light Source Photometer" Analytical Chemistry Instrument Demonstration Conference. An Industrial Cooperation Conference, Oak Ridge National Laboratory, Oak Ridge, Tenn., Oct. 16-17, 1967

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| 133 Sites, J. R., ¹²
J. A. Carter | "Spark Source Mass Spectrometry of Radioactive Samples" | 11th Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Oct. 10-12, 1967 |
| 134 Stelzner, R. W. | "Fluorophotometer (Q-1165) and Fusion Device (Q-1187)" | Analytical Chemistry Instrument Demonstration Conference. An Industrial Cooperation Conference, Oak Ridge National Laboratory, Oak Ridge, Tenn., Oct. 16-17, 1967 |
| 135 Stokely, J. R., ¹²
W. D. Shults | "Controlled-Potential Coulometric Determination of Americium" | 11th Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Oct. 10-12, 1967 |
| 136 White, J. C. | "General Requirements of Modern Day Analytical Chemistry" | 76th Meeting, Tennessee Academy of Science, Johnson City, Nov. 25-26, 1966 |
| 137 | "The Analytical Chemist Looks at Standard Reference Materials" | Analytical Group, Chemical Society of Washington, D.C., Feb. 9, 1967 |
| 138 Yoakum, Anna M. | "Recent Advances in Analytical Emission Spectroscopy" | 18th Annual Mid-America Symposium on Spectroscopy, Chicago, May 15-18, 1967 |
| 139 | "Recent Advances in Emission Spectroscopy" | Fall Meeting, Southeastern Section of the Society for Applied Spectroscopy, Birmingham, Ala., Sept. 30, 1967 |
| 140 Young, J. P. | "Windowless Spectrophotometric Cells for Use with Corrosive Liquids" | Analytical Chemistry Instrument Demonstration Conference. An Industrial Cooperation Conference, Oak Ridge National Laboratory, Oak Ridge, Tenn., Oct. 16-17, 1967 |

Under the Traveling Lecture Program

LECTURER	TITLE	PRESENTED AT
141 Cameron, A. E.	"Geological Age Determination by Isotopic Measurements"	Vanderbilt University, Nashville, Tenn., Apr. 28, 1967
142 Hahn, R. L.	"Interaction of ³ He Particles with Low-Z Elements: A Nuclear Chemist Looks at Direct Reactions"	Purdue University, Lafayette, Ind., Nov. 19, 1966
143 Ricci, E.	"Recent Developments in Activation Analysis"	University of Virginia, Charlottesville, Feb. 27, 1967
144 White, J. C.	"The New Image of Analytical Chemistry in Science"	Alabama College, Montevallo, Dec. 12, 1966
145		Virginia Military Institute, Lexington, Feb. 28, 1967
146		Southern Illinois University, Carbondale, Oct. 23, 1967

Patents

AUTHOR	TITLE	PATENT NO.	DATE ISSUED
147 Moore, F. L.	"Method for Removing Lanthanides and Tri-valent Actinides from Aqueous Nitrate Solutions"	3,294,494	Dec. 27, 1966

Translations

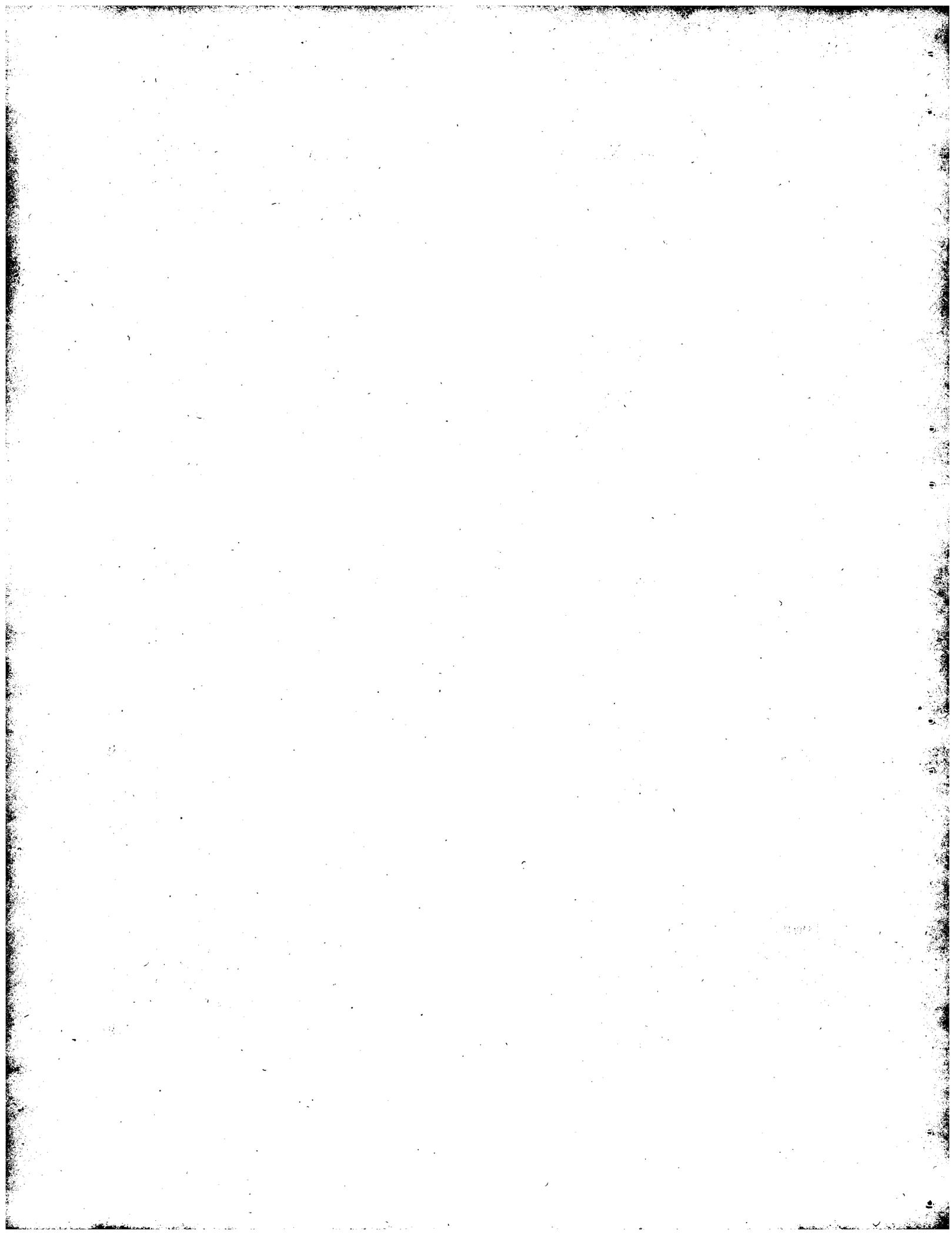
TRANSLATOR	ORIGINAL REFERENCE			TRANSLATION		
	Author (Transliterated)	Source	Language	Language	Translated Title	ORNL-tr Number
1 Raaen, Helen P.	K. Randerath and H. Struck	<i>J. Chromatog.</i> 6, 365-367 (1961)	German	English	"Thin-Layer Chromatographic Separation of Nucleic Acid Derivatives on Cellulose Layers"	1713
2	K. Randerath	<i>Biochim. Biophys. Acta</i> 61, 852-854 (1962)	German	English	"Polyethyleneimine-Cellulose—A New Anion Exchanger for the Chromatography of Nucleotides"	1703
3 Susano, C. D. ¹	I. Mezzasoma and B. Farina	<i>Boll. Soc. Ital. Biol. Sper.</i> 42(20), 1449- 1452 (1966)	Italian	English	"Separation of Bases, Nucleosides and Nucleotides on a Column of Dextran (Sephadex G-10)"	1667

¹Retired.

Articles Reviewed or Refereed for Periodicals

Reviewer or Referee	Number of Articles Reviewed or Refereed for Indicated Periodical													
	Advan. Chem. Ser.	Anal. Chem.	Anal. Chim. Acta	Appl. Spectry.	Australian J. Chem.	Croat. Chem. Acta	Inorg. Chem.	Isotopes Radiation Technol.	Nucl. Appl.	Nucl. Sci. Eng.	Nucleonics	Rev. Sci. Instr.	Science	Total
Attrill, J. E.		1												1
Dunn, H. W.		1												1
Eldridge, J. S.											1			1
Emery, J. F.		1												1
Feldman, Cyrus		3		3								1		7
Florence, T. M. ¹		1			1									2
Goldstein, Gerald		3												3
Horton, A. D.		1												1
Jones, H. C.		1												1
Kelley, M. T.		1												1
Kubota, Hisashi		1												1
Lyon, W. S.		7											1	8
Maddox, W. S.		2												2
Manning, D. L.		3												3
Miller, F. J.		2												2
Moore, F. L.	1	3				1								5
Mueller, T. R.		6												6
Reynolds, S. A.		2					3		1	1				7
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Shults, W. D.		2	1											3
Stelzner, R. W.		5												5
White, J. C.		3												3
Young, J. P.		1					1							2
Zittel, H. E.								1						1
Total	1	59	1	3	1	1	1	3	2	1	2	1	1	77

¹Alien Guest from Australian Atomic Energy Commission, Sutherland, New South Wales, Australia.



Indexes

Helen P. Raaen

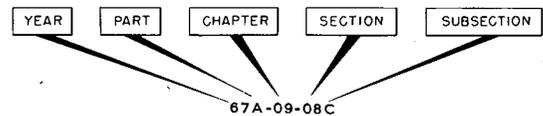
Ann S. Klein¹

Indexes are a part of the Analytical Chemistry Division annual report for the fourth 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 1964-1967 are bound separately and are designated ORNL-3904 (Rev. 2).

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.

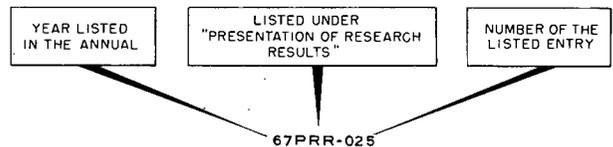
The Author Index is an alphabetical listing of authors, together with number entries that specify the material to which each author contributed. This index includes the authors of material in the body of the report, the authors of the entries listed in the Presentation of Research Results, and the translators 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 67A-09-08C, where 67 indicates the year of publication of the annual report, A 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 67PRR-025, where 67 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

¹Technical 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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 /PHOSPHORUS-33 / SULFUR-35 / NICKEL-63 / PROMETHIUM-147 / 2,5 DI PHENYL OXAZOLE (PPO) / 2,5 BIS (2 (5 / 67A-09-C7G
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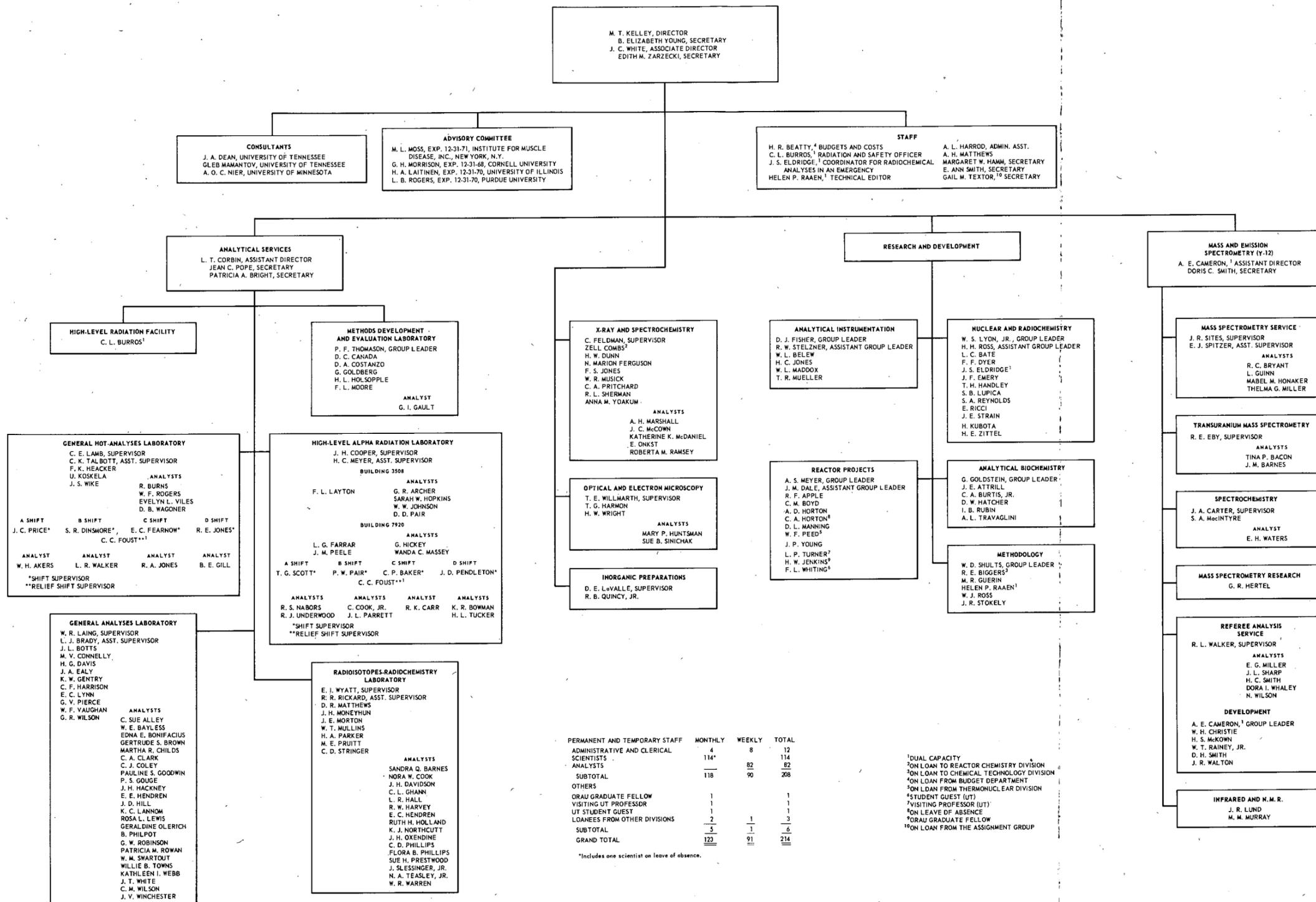
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ANALYTICAL CHEMISTRY DIVISION

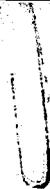
OCTOBER 1, 1967



PERMANENT AND TEMPORARY STAFF	MONTHLY	WEEKLY	TOTAL
ADMINISTRATIVE AND CLERICAL	4	8	12
SCIENTISTS	114*		114
ANALYSTS		82	82
SUBTOTAL	118	90	208
OTHERS			
ORAU GRADUATE FELLOW	1		1
VISITING UT PROFESSOR	1		1
UT STUDENT GUEST	1		1
LOANEEES FROM OTHER DIVISIONS	2	1	3
SUBTOTAL	5	1	6
GRAND TOTAL	123	91	214

*Includes one scientist on leave of absence.

- ¹DUAL CAPACITY
- ²ON LOAN TO REACTOR CHEMISTRY DIVISION
- ³ON LOAN TO CHEMICAL TECHNOLOGY DIVISION
- ⁴ON LOAN FROM BUDGET DEPARTMENT
- ⁵ON LOAN FROM THERMONUCLEAR DIVISION
- ⁶STUDENT GUEST (UT)
- ⁷VISITING PROFESSOR (UT)
- ⁸ON LEAVE DF ABSENCE
- ⁹ORAU GRADUATE FELLOW
- ¹⁰ON LOAN FROM THE ASSIGNMENT GROUP



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ATC 100-100-100
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