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ANALYTICAL CHEMISTRY DIVISION
ANNUAL PROGRESS REPORT
FOR PERIOD ENDING SEPTEMBER 30, 1968

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UC-4 – Chemistry

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ANALYTICAL CHEMISTRY DIVISION
ANNUAL PROGRESS REPORT
For Period Ending September 30, 1968

M. T. Kelley, Director
J. C. White, Associate Director

Compiled and Edited by
Helen P. Raaen

DECEMBER 1968

OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee
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Preface

The general mission of the Analytical Chemistry Division is to provide complete analytical support for the many research programs at ORNL. This support comprises research in analytical chemistry, development of new and improved methods for analysis, and a chemical analysis service that employs a wide variety of analytical techniques. The great diversity in the research programs at ORNL is likewise reflected in a similar diversity in the activities of the Division.

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 made constantly to provide more precise and more rapid analyses by employing the most advanced instrumentation available and by using automated or semiautomated procedures. Efforts are being made to use electronic data acquisition and processing equipment wherever possible. Considerable progress has been made in this regard this year in that three small dedicated computers have been acquired and commitments have been made for the acquisition of two more such devices. Initially, these will be used primarily for acquisition of data in digital form, but at least one of these will be used to explore the feasibility of computer-controlled analytical instrumentation. It is gratifying to note that the preeminence of the work of the Division in the field of analytical instrumentation has been recognized by the announcement that Dale J. Fisher, Group Leader of the Analytical Instrumentation Group, will be the recipient of the 1969 ACS Award in Chemical Instrumentation sponsored by the Sargent-Welch Scientific Company.

The Division is continuing major research and development programs with the objectives of providing chemical analyses for the MSRE and for bioengineering programs. Efforts in analytical biochemistry continue to occupy a major place in the program of the Division with constant exploration being made of areas that would constitute a logical expansion of present work in this field. This emphasis is in recognition of the increasing importance of biology and biochemistry in the research efforts of ORNL and of the entire scientific community.

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ORGANIZATION CHART

Summary

PART A. ANALYTICAL RESEARCH

1. Analytical Instrumentation

The design work of the Analytical Instrumentation Group was continued with stress on operational-amplifier modules and increasing emphasis on digital electronic techniques. Considerable time was devoted to modifying and upgrading several of the field instruments, to writing new and revising existing methods for the *ORNL Master Analytical Manual*, assembling CAPE packages, and preparing technical articles for outside publication. A substantial number of cooperative efforts were undertaken that involved several projects and groups – both within and outside the Division.

The prototype of a modular solid-state coulometric titrator in the NIM system will be fabricated for ultimate replacement of the older Q-2005-X50 instruments that have seen as much as ten years of continuous laboratory service. Optimum and compatible electrochemical cell design is aimed at shorter titration times and the achievement of accuracy and precision on smaller amounts ($\leqslant$1 mg) of electroactive material. Also, five of the high-sensitivity coulometric titrators (Q-2564) have been field modified.

The study of single-cell high-precision dc polarography was continued with development of a prototype mercury-flow-rate controller. An instrument computer (Fabri-Tek model 1064) has been obtained to serve as a diagnostic tool in this project. The FT-1064 instrument will be used also as a laboratory test instrument and in the study of analytical techniques including digital polarography. Our controlled-potential differential dc polarograph has been applied to a survey of certain biochemicals of interest in the Body Fluids Analyses Program. Packages of fabrication information on the ORNL model Q-2792 controlled-potential dc polarograph and the Q-2942 polarographic drop-time controller were sent to 18 requesters in the U.S.A. and ten abroad. Technical papers that describe the two instruments are being prepared. Both instruments have been duplicated at the Lucas Heights Research Establishment of the Australian Atomic Energy Commission. The polarography of zirconium in acetonitrile and dimethyl sulfoxide is being investigated in a cooperative study at the Lucas Heights laboratory.

Work in flame photometry was continued with the assembly of a model IX flame spectrophotometer to replace an older model Q-1457 instrument in the hot laboratories of the Radioisotopes-Radiochemistry Laboratory. A paper that describes signal/noise enhancement in flame photometry by statistical techniques is being prepared.

For the analysis of tRNA's, a second-generation automatic sampler was developed. This instrument uses electrical synchronization and proper phasing instead of the earlier photometric sensing. A new titrant-delivery unit and elevator (Q-1728A potentiometric titrator) and a new ORNL model Q-1348C servo-pipetter were fabricated in the ORNL machine shops for the High-Radiation-Level Analytical Laboratory.

Operational amplifiers were used in an instrument for end-point detection in bipotentiometric titrations and for an analog integrator with ranges from 0.1 to 100 μC.

In the study of high-speed voltammetry, new derivative circuits are under investigation, the Q-2950 dual set-point comparator has been modified to be more reliable, and a paper that describes the controlled-potential controlled-current cyclic voltammeter is being readied for publication.

A new digital computer (model PDP 8/I) and a system of logic modules were obtained for study of possible computer-assisted applications in the Division. The first project will involve a molten-salt voltammetric problem.

2. Analytical Methodology

The Analytical Methodology Group is charged with the responsibility to develop competencies in areas that will prove beneficial to the Division, to carry out nonroutine projects that do not logically fall to another Group, and to conduct new programs when they

develop. Consequently, the activities of this group are very diverse and often supplement the efforts of other groups. For example, a new research-quality gas chromatograph was acquired for use by both the Analytical Biochemistry and the Analytical Methodology Groups. We have initiated programs of study in biomedical applications of gas chromatography (GC), in derivative GC, and in aqueous-injection GC. Other GC developments include a method for determining perfluoromethylcyclohexane in methane, resolution of the methyl esters of the phosphoric acids that derive from degradation of tributyl phosphate, and the purification of some 16 compounds by preparative GC. A laboratory has been remodeled specifically for gas chromatographic work.

Means for concentrating, desalting, and separating materials, especially tiny amounts of organic materials, are needed to an increasing extent. We have continued our work in thin-layer chromatography. Several fruitful applications of TLC have been made, and we are currently evaluating its utility when conjoined with other analytical techniques, such as mass and infrared spectroscopy. We anticipate studies in quantitative TLC and have acquired an instrument for such studies. The lipophilic resin Amberlite XAD-2 has been explored as a means for collecting and/or desalting solutions of organic materials. We anticipate a continuing program of study, evaluation, and application of techniques and materials for isolation of organic compounds.

We are participating, along with several other members of the Division, in a new program to develop an analytical scheme for characterizing tobacco smoke and smoke condensates. Our efforts in this program have been largely exploratory to date, with emphasis on fractionation and separation of the multitudinous compounds into classes. Gas-liquid and thin-layer chromatography will be used extensively in this program.

Our efforts in water analysis fall broadly into two areas: analysis of deaerated and saline waters and analysis of waters of ecological significance. Efforts to improve the methods used to determine oxygen in very low concentrations in water have continued, because such methods are needed to calibrate and evaluate any instrumental method that may be considered. We have evaluated several instrumental methods and have continued to provide on-site analytical support for tests of several desalination plants. The new aqueous-injection gas chromatography project mentioned above offers considerable promise for analysis of waters of ecological importance.

We conducted a brief air-quality survey for the neighboring Melton Hill Regional Development Corporation and found that the air in the Eagle Bend Industrial Park is of typical nonurban quality.

Our activities in the Transuranium Research Laboratory (TRL) involve both analytical research and general chemical support. We have conducted several electroanalytical studies of the lighter transuraniums. With these elements, precise analytical methods are needed so that their fundamental characteristics can be better determined. We hope ultimately to use thin-layer electrochemical techniques to study these elements. Considerable assistance of both an analytical and a preparative nature has been given to many members of the TRL staff.

3. Analytical Chemistry for Reactor Projects

Oxide concentrations in the MSRE fuel, as determined by the hydrofluorination method, remained near the 50-ppm level. The essentially identical result obtained in the analysis of a sample maintained at an elevated temperature to minimize radiolysis virtually proved that the radioactivity of the samples does not produce a negative bias in the results. Oxide was also determined on $LiF\text{-}BeF_2$ preparations and on samples of $LiF\text{-}UF_4$ from the production of ^{233}U fuel concentrates. These latter samples required the development of new sampling techniques and the adaptation of the method to smaller samples and to new forms of sample containers. An evaluation of the operating history of the hot-cell apparatus for oxide determination indicated that electrolytic moisture-monitor cells are desensitized rapidly during the analysis of highly radioactive samples; however, similar desensitization of a test cell could not be produced by exposure to radioactivity of comparable and higher levels. All current and previous results of the determinations of the U^{3+}/U^{4+} ratio in the MSRE fuel were reevaluated by means of a modified computer program that calculates ratios at the operating temperature of the reactor rather than at the lower temperatures at which the analyses were performed. With the exception of three results, apparently associated with samples of the highest radioactivity, the recomputed ratios are in reasonable agreement with the ratios calculated on the basis of the operating history of the reactor (i.e., beryllium additions, corrosion-product increases, and allowance for burnup). A method for the remote measurement of traces of HF in gas streams has been developed to cover the range of concentrations (8 to 400 ppm) found in hydrogen-transpiration effluents. The method is based on the preconcentration of the HF on an NaF trap and thermal-conductivity measurement of the pulse of HF generated on desorption. The

hydrogen-evolution method, which is a measure of both U(III) and dispersed electropositive metals, is being modified for adaptation to radioactive samples of reprocessed MSRE fuel solvent. To avoid loss of reducing power of samples during pulverizations, sample fragments are dissolved in $HCl\text{-}HBO_3$ at 250°C in a sealed tube. An in-line analyzer that was installed to measure traces of hydrocarbons in the MSRE off-gas was found to be subject to substantial radiation interference. This interference was determined to be caused by radiolysis of moisture on the reagents used to absorb the combustion products of hydrocarbons and will be minimized by the substitution of a small regenerable molecular-sieve trap.

The electrochemical oxidation of U(IV) in molten $LiF\text{-}BeF_2\text{-}ZrF_4$ has been studied. Experimental evidence indicates that the U(V), which is formed, subsequently disproportionates into U(IV) and U(VI). Also, the electrochemical reduction of Cr(II) in this same solvent was studied by conventional and first-derivative voltammetry. Although U(IV) interferes to some extent, it is believed that the Cr(II) → Cr(0) reduction wave can be used to monitor chromium in fluoride melts. The emf's of several metal–metal ion couples vs an Ni/Ni(II) reference electrode were measured in molten fluorides at 500°C. Because boron nitride does not appear to be entirely satisfactory as a sheath for the reference electrode, it is planned to investigate the use of metal frits for separating the reference electrode from the bulk of the melt.

The designing of the in-cell equipment of a facility for spectral studies of highly radioactive materials is essentially complete, and some of these components are now being fabricated. This equipment will permit the transfer of samples of radioactive fluoride salts to spectrophotometric cells without exposure of the samples to the atmosphere and will simultaneously maintain them at a temperature sufficient to eliminate significant radiolysis. A diamond-windowed cell has been developed for use with molten fluoride salts.

The study of the spectra of Fe(II), Ni(II), Cr(II), and Cr(III) in various $LiF\text{-}BeF_2$ melts has been concluded. Studies of the spectra of U(V), U(VI), UO_2^{2+}, and Mo(III) have been initiated or continued. Solvent contaminants, primarily products of hydrolysis, have been found to greatly affect the electrochemical generation of solute species for spectral characterization. Vacuum pretreatment of LiF-NaF-KF at 450°C is effective in removing the H_2O precursor. The electrochemical generation of tantalum and copper in their various oxidation states for spectral study is presently being investigated.

4. Analytical Biochemistry

Analytical assistance to the Biology and Chemical Technology Divisions was continued in support of the Macromolecular Separations and Molecular Anatomy Programs. In the analysis of transfer ribonucleic acids (tRNA's), the optimum conditions for the assay of tyrosine- and methionine-accepting tRNA's were established, and a statistically designed program for optimization studies is now being used. Our automated analyzer has been modified to operate more economically, and a technique for recovering ^{14}C-labeled amino acids was developed. The raw assay data are now being processed by computer; the processed data are presented as both a tabulation and a plot. Studies of the kinetics of aminoacylation are continuing to provide a better understanding of the assay. Methods for determining impurities in tRNA have been improved. Future plans for the Macromolecular Separations Program include separations of ribosomal RNA and various enzymes; consequently, we are developing methods to measure high-molecular-weight RNA by gel electrophoresis and are investigating the assay of the enzyme L-asparaginase. As a "spinoff" of this program, we find that the aminoacylation reaction can be used in an ultrasensitive isotope-dilution method for the determination of L-amino acids.

Further research into the principles and applications of ligand-exchange chromatography is being carried out.

For the Body Fluids Analysis Group of the Molecular Anatomy Program, we are attempting to isolate and characterize compounds separated from urine by anion-exchange chromatography. Two compounds were identified definitely and several others tentatively. Conjugates of various types were located from chromatograms by chromatography before and after enzymatic or acid hydrolysis. Prefractionation of urine by gel chromatography into six fractions followed by anion-exchange chromatography of the individual fractions improves the purity of the compounds isolated. We will also attempt the gas chromatographic analysis of fractions from anion-exchange chromatography after extraction or forming derivatives in the desalted and lyophilized material.

Gel electrophoresis and centriphoresis were used to concentrate and isolate tumor antigen activity and to separate other large proteins. Amino acid sequence analysis of mouse hemoglobins was continued. Consideration of the frequency of certain substitutions in the β chain led to the conclusion that gene duplication is the primary cause of chain heterogeneity. Amino acid analyses were also done for several members of the Biology Division.

5. X-Ray and Optical Spectrochemistry

A code has been written for converting x-ray diffraction data to lattice parameters. A technique has been developed for determining sulfur-to-phosphorus ratios in small organic samples with the electron probe. The optical and mechanical design of a beam splitter for calibrating spectrographic film has been optimized, and the best conditions for its use were determined for different types of spectrographs. Preconcentration procedures were devised for traces of rare-earth elements in bismuth, cadmium in plutonium, and bismuth in $LiF\text{-}BeF_2$. In the electrodialysis–Mitchell-Scott procedure for determining trace elements in bone, the removal of phosphate was accelerated by use of a two-compartment electrodialysis cell. Recoveries and total process blanks were determined for numerous trace impurities. A procedure was developed for determining silicon in curium oxide.

6. Mass Spectrometry

A trace characterization analysis of ^{242}Pu ($t_{1/2}$ = 3.79×10^5 y) was conducted by use of the solution spark-source technique. Twenty-one general contaminants were found. The distribution of rare-earth elements in different leaf and wood ash samples was determined.

The low-resolution mass spectrometer has been used to record the spectra of a wide variety of organic compounds for identification or elucidation of structure and to accumulate reference spectra for known compounds. For samples of adequate size and purity, the mass spectrometry has been coordinated with nuclear magnetic resonance and infrared spectrometry. All three of these techniques have been applied with some success to service problems for ORNL and Y-12. The analytical effort has been slanted toward support of the Body Fluids Analyses Program. The difficulty so far has been that the samples available are small and frequently of dubious purity. Informal sessions on techniques have been held at approximately monthly intervals to bring together Division personnel involved in the bioanalytical work and to keep them informed on techniques and results.

Surface ionization studies have shown that, in the presence of oxygen, the emission of UO_2^+ from tungsten filaments exhibits two maxima as the temperature is increased. The temperatures at which these maxima occur are very weakly dependent on the partial pressure of oxygen. Below ~1500°K, the evaporation energy of UO_2^+ is deduced to be ~6.9 ± 0.4 eV. Between ~1500° and ~2250°K, the effects of competing reactions hinder satisfactory interpretation of the data. A measurement of the ionization potential of thorium has yielded a tentative value of 7.5 ± 0.3 eV.

7. Optical and Electron Microscopy

Optical and electron microscopy and electron diffraction have been applied to many problems arising in fundamental and applied research. These include studies of lanthanide hydroxide sols and absolute filters at elevated temperatures and the characterization of "good" and "bad" Varcum, which is a furfuryl alcohol derivative. Observations of radioactive materials have continued in connection with a NASA project and with the MSRE. An electron-beam vaporizer is being used to produce ultrapure thin films. The "hot" microscope in Building 3019 is being replaced with a newer model, and the Philips EM 300 electron microscope is being installed in the Transuranium Processing Plant.

8. Nuclear and Radiochemistry

An air-floating sphere for homogeneous photon and neutron irradiations in nonuniform neutron-flux environments was designed for use in the ORELA. Cross sections for activation-analysis experiments with a ^{252}Cf source were measured by use of an 0.87-mg source. Investigation of ^3He activation analysis continued with the study of channeling effects. Among the applied activation-analysis programs are two cooperative efforts: trace elements in drugs (with the Bureau of Drug Abuse Control) and copper and manganese in brain tissue (with ORAU).

Preliminary studies of the use of Cerenkov radiation for radionuclide measurement and the study of alpha damage in inorganic phosphors were begun. The investigation of photovoltaic conversion of radioisotope decay energy was completed. Actinide electrodeposition from dimethyl sulfoxide for preparation of alpha-counting sources and the variables that affect alpha spectrometry were studied. Counting of weak-beta emitters in an aqueous solution was improved through use of Triton X-100, and the low-level-radiation counting facility at Houston for returned lunar samples was completed and equipment was installed in it.

Interlaboratory comparison of measurements of the nuclides ^{57}Co, ^{99}Mo-^{99m}Tc, and ^{99m}Tc was made. Among new nuclear data reported are energies of gamma rays in ^{38}Cl and ^{116}In, cross sections for ^{64}Ni and ^{208}Pb, gamma branchings and conversion coefficients in ^{103}Pd-^{103m}Rh, and gamma-ray intensities in the decay of ^{133}Ba. By use of a thin-window Ge(Li)

detector, the 53-keV gamma ray of ^{73}As was measured in the presence of other arsenic radioisotopes; this detector has been calibrated for efficiency. The investigation of nuclear properties of neutron-deficient rare-earth elements continued with studies in the $173 \leqslant A \leqslant 185$ region.

Considerable effort was spent in applying nuclear methods to reactor program problems. Delayed-neutron counting made possible a highly precise ^{235}U determination. The 3-MeV Van de Graaff generator was used to determine lithium and fluorine in MSRE graphite, and a highly collimated Ge(Li) spectrometer was used to determine deposited fission products in the MSRE heat exchanger. In addition, in-reactor fission-product penetration of specimen graphite was determined by gamma-ray spectrometry. A 10-cc Ge(Li) detector was set up to assist in measuring fission-product release from reactor-fuel microspheres. The 14-MeV neutron generator has been used to determine oxygen in a variety of materials and fluorine in fluorspar and to evaluate the usefulness of new targets. In addition, a new target into which tritium continuously diffuses has been designed. Both the Texas Nuclear Corporation and the Kaman machines have been used by a number of extradivision experimenters.

Quality control for products of the Isotopes Division continued as an important assistance, as did checking of commercially available radioactivity standards used therein. Data have been evaluated and, in some instances, measurements made for inclusion in the *Isotopes Users' Guide*; for example, half-life values for 36 radionuclides were published. Techniques were developed for production of ^{123}I for medical purposes. Study of radionuclides in HFIR coolant water was continued.

Investigation of the radiolysis of radiation-safeguard spray solutions continued as a contribution to spray and absorption technology. The radiolysis of nitrate solutions and of perchloric acid solutions and the stability of some inert metals in irradiated mineral acids also received attention. A chapter concerned with effect of radiation on common analytical reagents was prepared for a book, and the effect of gamma radiation on spectrophotometric methods for iron was studied. The effect of moisture in the salt beds of interest in Project Salt Vault was evaluated.

9. Inorganic Preparations

The preparation of fused salts continued but on a reduced scale and mostly for the Metals and Ceramics Division. The main activities for the Solid State Division were the preparations of ferrites (Fe_3O_4-type compounds) and some high-purity fluorides, the purification of MgO, and the heat treatment of rare-earth-metal alloys. The production of 50 g of ^{160}Gd was accomplished. For the Physics Division, numerous AuLi alloys were prepared and also some isotopic intermetallic compounds that contain ^{61}Ni, ^{64}Ni, and ^{73}Ge. The decontamination of ~7 kg of platinum was undertaken for the Stores Department.

PART B. SERVICE ANALYSES

The Methods Development and Evaluation Group was united with the Service groups this past year, and the union of these groups has worked out very satisfactorily.

10. Methods Development and Evaluation

Several new radiochemical methods have been developed; they include the anion separation of Bk(IV) from Ce(IV), isolation of ^{249}Bk by solvent extraction and its determination by beta counting, and separation of americium from other transuranium elements by extraction chromatography.

A variety of special work included the thermogravimetric analysis of thorium iodate, gravimetric determination of tetrafluoroborate, determination of fluoride in molybdenum fluoride, and determination of hafnium and zirconium in their mixed oxides.

Two of the newer electroanalytical methods were evaluated for special applications: rapid-scan derivative polarography for determining uranium in synthetic MSRE fuel and thin-film mercury-electrode voltammetry for bismuth.

A literature survey was made for the Smoking and Health Program. A smoking machine was fabricated and used to collect cigarette-smoke condensates. The components of tobacco extracts were separated by gel-permeation chromatography.

The work in absorption spectrophotometry of alpha-emitting materials has continued.

Uranium was separated from radioactive MSRE fuel by fluorination in a shielded hot cell. The resulting UF_6 was absorbed in a sodium fluoride trap, dissolved in sulfuric acid, and determined by controlled-potential coulometric titration at a relative standard deviation of 0.1%.

11. Spectrometry

In mass spectrometry, the Transuranium Mass Spectrometry Laboratory continued to make isotopic analyses for a diverse group of customers. The half-lives of

^{242}Pu and ^{244}Pu were measured, and the new values were published. Work was also done to measure the half-lives of some other transuranium elements. The isotopic analysis of all elements from thorium through einsteinium was done routinely. The total number of analyses reported by the Mass Spectrometry Service Laboratory increased 20% over last year. The increase included gas samples and stable and radioactive solid samples.

The Spectrochemistry Laboratory (Y-12) reported 47,000 results of emission spectrochemical analyses on 2500 samples, which represents increases of 12 and 19%, respectively. More than 300 bismuth samples from the MSRE salt cleanup program were analyzed for thorium, lithium, and beryllium.

In the Infrared Laboratory, various samples of organic, organometallic, and inorganic nature were examined. The information desired is primarily qualitative in nature and usually involves identification, structure, changes in structure, and presence of impurities. Occasional quantitative work also was performed. Development or modification of instruments or techniques is done when it is pertinent to the service work.

In nuclear magnetic resonance (NMR) spectrometry, the NMR Spectrometry Laboratory rendered support to several ORNL, Y-12, and ORAU research divisions through the recording and interpretation of the NMR spectra of organic compounds that resulted from studies being carried out in these divisions. Often this service was a combined effort of the Mass, Infrared, and NMR Laboratories.

12. Process Analyses

The laboratories doing process analyses performed 207,100 analyses, which is about the same as the past year, but we had a 12% decrease in personnel. The largest number of samples came from the Transuranium Process and biochemical programs. Brief statements about the work and new developments in each laboratory follow.

The High-Level Alpha Radiation Laboratory put into service a method for the determination of berkelium in transuranium process solutions. Oxygen-to-metal ratios were determined on PuO_2-UO_2 pellets. A new procedure for dissolving high-fired PuO_2-UO_2 was adopted.

The work load of the General Analyses Laboratory increased ~35%. A method for bulk-density measurement of large porous graphite specimens was developed. A pycnometer was constructed for measuring the density of 0.1-cc samples. The arsenazo III method was used to determine thorium in bismuth metal.

The General Hot-Analyses Laboratory continued the same type of work as was done last year. Equipment for fluorinating uranium from MSRE fuel-salt samples was set up for use in a work cell for determining uranium. A Kollmorgen periscope was installed through the cell-block wall for visually examining radioactive materials.

The Radioisotopes-Radiochemistry Laboratory continued to use computer resolution of gamma-ray spectrometric data for the divisions requesting radioactive data. To facilitate the acquisition and processing of radiometric data, a 4096-channel pulse-height analyzer and an IBM card punch were purchased. Computer programs have been written for qualitative and quantitative Ge(Li) spectrometry.

The Radiation Control and Safety personnel continued their work, primarily in Buildings 3019 and 2026. In July, construction of the West Annex to Building 2026 (HRLAL) was begun.

The number of control samples included in the Statistical Quality Control Program decreased 22.6%; the apparent overall quality of the work decreased slightly as a result of the initiation of several new control programs for which the 2*S* limits have not yet been established.

PART C. ORNL MASTER ANALYTICAL MANUAL

13. *ORNL Master Analytical Manual*

Five new methods were added to the *Manual*; one of these was for the purpose of record only. Revisions were made to six methods. The status of nine methods for the Transuranium Process was changed from tentative to formal.

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, electroanalytical chemistry of molten salts, radiation effects on analytical methods, and mass spectrometry are carried out for the Division of Research 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 and National Cancer Institute 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 SOLID-STATE CONTROLLED-POTENTIAL COULOMETRIC TITRATOR

T. R. Mueller H. C. Jones

The circuit designs and tentative panel layouts of three of the four modules for the solid-state controlled-potential coulometric titrator have been completed.[1] The modules will be fabricated by the Instrument Department of the Instrumentation and Controls Division. The potentiostat module contains the reference-electrode-follower amplifier, control amplifier, and current-limiting amplifier. The current module contains the current amplifier, current booster, and part of the automatic cutoff circuitry. The meter module contains the current meter, meter-driver amplifier, automatic-cutoff-detector amplifier, and part of the automatic cutoff circuitry. A fourth module will house an analog integrator.

Two modes of current integration will be available. One will use a voltage-to-frequency converter followed by an electronic counter; the other will use an operational amplifier to perform analog integration. For operation, only one mode of integration is needed. However, for I vs Q recordings, the analog integrator module is required.

After the first instrument is fabricated, the cell-design studies will be continued in an effort to decrease

[1] H. C. Jones and T. R. Mueller, "Solid-State Controlled-Potential Coulometric Titrator," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967,* ORNL-4196, p. 3.

significantly the total titration time and to increase the precision and accuracy of the titration of samples that contain <1 mg of the substance to be determined.

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

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

The five model Q-2564 coulometric titrators[2] in use in the Division were modified by the Instrument Department of the Instrumentation and Controls Division and returned to operation. The modifications have been described.[3,4]

1.3 INSTRUMENTATION FOR THE HIGH-RADIATION-LEVEL ANALYTICAL LABORATORY (HRLAL)

W. L. Maddox

1.3.a New Titrant-Delivery Unit and Elevator

A new titrant-delivery unit (TDU) and elevator assembly of the type described previously[5] were fabricated and checked out for use with the automatic potentiometric titrator (Q-1728A) in the HRLAL. Plexiglas shields, to give increased protection against inadvertent spills and mechanical mishandling, were added to the new assembly. The drawings that describe the TDU and elevator were revised to show the new parts.

1.3.b New ORNL Model Q-1348C Pipetter

A new ORNL model Q-1348C pipetter was fabricated in the ORNL shops. Stainless-steel plungers were made for this unit, since it will be used with a secondary displacement liquid and therefore does not require the higher corrosion resistance of the usual Graphitar plungers. Check-out of the pipetter was completed. For it, a pipet that has a displaceable volume of 3.0 ml (1.0 ml is standard) was built and is being evaluated.

1.4 APPARATUS FOR PRECISE CONTROL OF MERCURY FLOW RATE OF DROPPING-MERCURY ELECTRODES (D.M.E.'s)

D. J. Fisher W. L. Maddox

We are continuing to study the precision attainable in single-cell first-derivative dc polarography.[6] For this work, a positive-displacement pump was developed to deliver mercury at a constant rate through the D.M.E. capillary. A prototype apparatus has been assembled that uses a high-precision Gilmont piston buret connected to the capillary through an all-glass system. The capillary is mounted in the model Q-2942 polarographic drop-time controller described earlier.[7] The evaluation of this assembly is in the preliminary stages.

1.5 ELECTRICAL AND ELECTROCHEMICAL TESTS OF PERFORMANCE OF POLAROGRAPHS

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

The abstract from a paper accepted for publication follows:[8]

"Both electrical and electrochemical tests are required in order to define, describe, and specify the performance of a polarograph, when a polarograph or a cell or an electrode is newly constructed or placed in service, when work is to be started that is of special importance, or when poor results are obtained with a combination that has been in use. In the last case, the first question is whether the malfunction originates in the polarograph itself or in the cell-electrode assembly or is due to electrochemical problems specific to the sample just introduced. The polarograph is tested by means of electrical tests. If it is performing according to specifications, the overall system

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

[3] T. R. Mueller and H. C. Jones, "Modifications to the High-Sensitivity Controlled-Potential Coulometric Titrator (ORNL Model Q-2564)," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967,* ORNL-4196, p. 3.

[4] T. R. Mueller, H. C. Jones, D. J. Fisher, and R. W. Stelzner, "Modifications of the ORNL Model Q-2564 High Sensitivity Coulometric Titrator," ORNL-TM-2175 (Mar. 18, 1968).

[5] W. L. Maddox, "Titrant-Delivery Unit and Elevator for the ORNL Model Q-1728 Potentiometric Titrators," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1966,* ORNL-4039, p. 6.

[6] W. L. Belew, D. J. Fisher, M. T. Kelley, R. W. Stelzner, and E. S. Wolfe, "Investigation of Precision Attainable in Single-Cell First-Derivative DC Polarography," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967,* ORNL-4196, p. 7.

[7] W. L. Belew, H. C. Jones, D. J. Fisher, and M. T. Kelley, "Apparatus for Precise Control of Drop Time of a Dropping-Mercury Electrode (D.M.E.) (ORNL Model Q-2942)," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967,* ORNL-4196, p. 6.

[8] D. J. Fisher, W. L. Belew, and M. T. Kelley, "Electrical and Electrochemical Tests of Performance of Polarographs. I. Needs and Methods. II. Diagnostic Criteria for Establishing the Degree to Which Recorded Polarograms Are Adversely Affected by Circuit Performance," accepted for publication in *Chemical Instrumentation.*

(and thus the cell and electrodes) is checked out by means of obtaining polarograms with reactions of well known characteristics. Satisfactory electrical and electrochemical test methods which have evolved at Oak Ridge National Laboratory (ORNL) are set forth in this paper. For any specific polarograph, each of these tests is generally applicable, provided, of course, that the polarograph includes the feature or mode of operation for which the test is designed. Also, diagnostic criteria are given for establishing the degree to which the forms of recorded regular, first derivative, and second derivative dc polarograms (for simple, polarographically reversible test reactions) are adversely affected by circuit performance."

1.6 A CONTROLLED-POTENTIAL AND DERIVATIVE DC POLAROGRAPH – ADVANTAGES OF RAPID AND DERIVATIVE DC POLAROGRAPHY

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

The abstract from a paper accepted for publication follows:[9]

"The circuits, theory, and performance characteristics are described of a controlled-potential dc polarograph (ORNL model Q-1988-FES) which is designed to take advantage of the inherent high sensitivity and resolution of derivative dc polarography. Several circuit modifications for specific applications are described. Cell and electrode design considerations are given. The advantages of potentiostatic electrolysis in polarography, the advantages of the use of current-averaging and analog derivative circuits in improving the measurement and the interpretation of recorded dc polarograms, the attainment of ultimate high resolution through the recording of first derivative dc polarograms of good wave form, the achievement of high sensitivity by means of rapid derivatice dc polarography, the practical advantages of first derivative over regular dc polarography, the relative advantages of first and second derivative dc polarography, and the practical advantages of rapid dc polarography are discussed. Conclusions are illustrated and substantiated by data.

"With a 2 to 5 sec D.M.E., controlled-potential undamped, maximum-current, or average-current regular dc polarograms or first derivative dc polarograms can be recorded. With a low average mercury flow rate 0.5 sec D.M.E., controlled-potential rapid average-current regular dc polarograms or first or second derivative dc polarograms can be recorded and instrumental conditions can be selected that result in optimizing either resolution or sensitivity (signal-to-noise ratio). At scan rates up to 0.3 v/min, the recorded forms of regular and derivative waves agree well with the usual, accepted equations. Under these conditions, regular average-current and first derivative dc polarograms are particularly useful, since they are highly reproducible and the inherently superior resolution for first derivative dc polarography is achieved. Because superior signal-to-noise ratio is achieved at faster scan rates, up to 3 v/min, the first and second derivative dc polarograms obtained at these scan rates are more useful when the sensitivity of measurement is of principal interest. Due to the greater ease of measurement at high sensitivity, rapid second derivative dc polarography is of particular advantage. Such polarograms can be recorded in as little as 10 sec. By using an XY recorder, the X axis is independent of scan rate and replicate polarograms are superimposed.

"Data is presented showing the ultimate resolution is achieved with first derivative dc polarograms and that the peak-to-peak height of second derivative dc polarograms is linearly related to concentration over the range tested, 5×10^{-7} to 1×10^{-3} *M*."

[9] D. J. Fisher, W. L. Belew, and M. T. Kelley, "A Controlled-Potential and Derivative DC Polarograph for Regular and First and Second Derivative DC Polarography: Circuits, Theory and Performance Characteristics. Advantages of Rapid and Derivative DC Polarography," accepted for publication in *Chemical Instrumentation.*

1.7 POLAROGRAPHY OF ZIRCONIUM IN ACETONITRILE AND DIMETHYL SULFOXIDE

W. L. Belew T. M. Florence[10]

The possibility was investigated of obtaining polarographic waves due to the reduction of zirconium species to lower valence states in nonaqueous solvents. Reduction waves are reported for zirconium in dimethyl sulfoxide (DMSO)[11] and in acetonitrile;[12] therefore, preliminary work was done with these compounds as solvents.

Since traces of water are expected to greatly affect waves for a substance in nonaqueous media as easily hydrolyzed as $ZrCl_4$, effort was made to ensure at all times the lowest possible water content of the supporting media. All solutions were prepared in a dry box. Their water contents were determined by Karl Fischer titration before and after each series of polarograms. DMSO with a water content as low as 5×10^{-4} *M* was obtained by shaking the DMSO with freshly activated Linde type 5A molecular sieve. Acetonitrile with a water content of 2×10^{-3} *M* was obtained by fractional distillation from P_2O_5 and CaH_2.

In DMSO, 0.1 *M* in tetrapropylammonium perchlorate and with a water concentration of 6×10^{-3} *M* or greater, two waves for zirconium tetrachloride are observed. Their $E_{1/2}$ values are −1.04 and −1.60 V vs an aqueous S.C.E. The wave at −1.04 V is identical

[10] Australian Atomic Energy Commission Research Establishment, Sutherland, New South Wales, Australia.

[11] G. Schöber and V. Gutmann, "Die Polarographie in Wasserfreiem Dimethylsulfoxyd," pp. 940–47 in *Advances in Polarography* (Proceedings of the Second International Congress, Cambridge, 1959), ed. by I. S. Longmuir, vol. 3, Pergamon, London, 1960.

[12] J. W. Olver and J. W. Ross, Jr., "Polarography of Zirconium and Hafnium in Acetonitrile," *J. Inorg. Nucl. Chem.* 25, 1515 (1963).

with that obtained when only free HCl is added to the supporting medium, and therefore this wave is believed to be due to free HCl formed by the reaction of $ZrCl_4$ with water. The wave at −1.60 V is ill-formed and very irreversible; it is believed to be a "hydrolytic wave," because both the height and the form of the wave depend on the water concentration. In DMSO that has a water content of $<1 \times 10^{-3}$ *M*, $ZrCl_4$ appears to be only slightly soluble.

Although three reduction waves had been reported for $ZrCl_4$ in acetonitrile (tetraethylammonium perchlorate as supporting electrolyte),[12] we observed only one irreversible wave for $ZrCl_4$ in the system acetonitrile–0.1 *M* tetrapropylammonium perchlorate–0.1 *M* water. When the water content was reduced to 2×10^{-3} *M*, $ZrCl_4$ appeared to be only slightly soluble.

Even though the $ZrCl_4$ is reagent grade (99.9% pure), some impurity may be present. Further studies will be done with reagent-grade $ZrCl_4$ that has been sublimed.

1.8 POLAROGRAPHIC SURVEY OF BIOCHEMICALS

C. R. Spell[13] D. J. Fisher

In the ORNL Body Fluids Analyses Program,[14] biochemicals that are urinary constituents are separated in high-resolution chromatographic columns. A survey has been made of the dc polarographic characteristics of selected examples of these biochemicals. Also, the feasibilities have been assessed of using dc polarography to qualitatively and quantitatively monitor column-effluent streams and for laboratory identification and concentration analyses of biochemicals in column fractions.

Survey polarograms were obtained with the ORNL controlled-potential differential dc polarograph.[15] The instrument was used in its regular and first-derivative modes for single-cell operation and its differential subtractive modes[16] for two-cell operation. Buffer systems (0.1 *M* sodium acetate and 0.1 *M* sodium acetate–0.1 *M* acetic acid) were chosen to match conditions in the elution step of chromatographic processes. Gelatin (0.01%) and Triton X-100 (0.001%) were used as maximum suppressors when needed.

The differential subtractive two-cell modes of operation were tried with and without sparging and with and without maximum suppressors. For adequate reproducibility, sparging and a maximum suppressor were found to be necessary, in addition to careful control of D.M.E. and cell operation.

Survey polarograms were run on 23 compounds selected from five major groups: pyrimidines, purines, pyridine derivatives, benzene derivatives, and amino acids. Reduction waves were obtained for ten compounds in 0.1 *M* acetic acid–0.1 *M* sodium acetate and for eleven compounds in 0.1 *M* sodium acetate. Only three compounds gave clearly defined reduction waves in acid media: pyridoxal, cystine, and uric acid. Reduction waves for all other compounds were associated with the hydrogen-discharge wave. Waves in 0.1 *M* sodium acetate were better defined but not completely resolved from the hydrogen-discharge wave. Half-wave potentials vs S.C.E. were obtained for all waves that were well enough defined for measurement.

Nicotinamide and pyridoxal were selected for quantitative study in the acid buffer system with the differential subtractive modes. Nicotinamide gives a reduction wave on the slope of the hydrogen-discharge wave; pyridoxal gives one somewhat before that slope. For the concentrations 0.02, 0.04, and 0.08 mg/ml, the linearity of response indicates that quantitative estimation is possible.

Survey polarograms were run on 5-ml samples collected from a chromatographic column. To check the feasibility of the polarographic technique as a monitoring tool without the addition of either supporting electrolyte or maximum suppressor, the 5-ml samples were diluted to 50 ml with distilled water and placed in the cells. The samples were sparged with argon, and polarograms were recorded by the regular single-cell method; first-derivative curves were obtained on some of the samples. It was discovered that, without additional supporting electrolyte, apparent reduction waves appeared on the derivative curves. They were not reproducible and were drawn out. This behavior was attributed to migration currents and was checked by taking 25 ml of the sample from the polarographic cell and adding sufficient acetic acid and sodium acetate to make the solution 0.1 *M* with respect to each when diluted to 50 ml with distilled water. The solutions so prepared contained the equivalent of 2.5 ml of the

[13] ORAU Research Participant; Professor of Chemistry, Emory and Henry College, Emory, Va.

[14] C. D. Scott, *Progress Report for the Period March 1, 1967–March 1, 1968, Body Fluid Analysis Program*, ORNL-TM-2184 (April 1968).

[15] W. D. Shults, D. J. Fisher, H. C. Jones, M. T. Kelley, and W. B. Schaap, "Controlled-Potential Differential DC Polarography. I. Instrumentation, Apparatus, and Techniques," *Z. Anal. Chem.* **224**, 1 (1967).

[16] W. D. Shults, D. J. Fisher, and W. B. Schaap, "Controlled-Potential Differential DC Polarography. V. Subtractive Polarography," *Chem. Instr.* 1, 7 (1968).

original sample. Polarograms were again recorded; in no case were reduction waves noted. To get valid polarograms, additional supporting electrolyte was necessary for all fractions tested. From the survey of fractions collected from the chromatographic column, it seems that the polarographic technique can not be used as a direct monitor of the column effluent. This is especially true for the elution conditions (i.e., buffer system) now in use. The concentration of buffer salts up to fraction 40 are not adequate as a supporting electrolyte. Also, sparging and the addition of a maximum suppressor seems to be necessary. The two-cell modes, although workable under laboratory conditions, do not seem feasible for direct monitoring with the restrictive conditions imposed by the current elution system. Indications are, however, that more pertinent monitoring information could be obtained under column elution conditions that would provide a more basic effluent and one that has a higher concentration of buffer salts.

Useful information, especially for compounds in the pyridine group, can be secured by the polarographic technique in laboratory analyses. Both qualitative identification and quantitative estimation of some of the compounds eluted from the column should be possible. It is evident, however, that fractions from the column may have to be concentrated or possibly extracted from the elution medium and then examined under more favorable polarographic conditions. More clearly defined reduction waves appear in solutions more basic than 0.1 *M* sodium acetate.

If further polarographic work is contemplated on these compounds, it is recommended that a complete polarographic survey of each compound be made in well-buffered solutions over the pH range 1 to 12. Only in this way can all the pertinent identifying characteristics of these compounds be determined, that is, the influence of pH on half-wave potentials, the separation of true and catalytic hydrogen-reduction waves, the explanation of the reduction mechanisms, and the determination of the number of electrons involved in the reductions. Additional polarographic work on these compounds is strongly recommended.

1.9 ORNL MODEL Q-2792 CONTROLLED-POTENTIAL DC POLAROGRAPH AND ORNL MODEL Q-2942 POLAROGRAPHIC DROP-TIME CONTROLLER

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

Companion papers that describe the ORNL model Q-2792 controlled-potential dc polarograph[17] and Q-2942 polarographic drop-time controller[7] are being prepared for publication. Packages of diagrams and specifications for fabrication of the polarograph and drop-time controller were sent to 18 requesters in the U.S.A. and to 10 requesters abroad. At the Lucas Heights Research Establishment of the Australian Atomic Energy Commission, both the Q-2792 and the Q-2942 units were duplicated and are working well.

1.10 DESIGN OF TIME-AVERAGING AND TIME-DERIVATIVE CIRCUITS FOR DC POLAROGRAPHS

R. W. Stelzner

A paper is being prepared for publication that describes the mathematical design techniques which have produced the analog circuitry in several of the ORNL dc polarographs.

1.11 INSTRUMENT FOR END-POINT DETECTION IN BIPOTENTIOMETRIC TITRATIONS

W. L. Belew T. M. Florence[10]

Bipotentiometric titrations are done by observing the potential developed across two indicator electrodes in a stirred solution during the passage of a small highly

[17] W. L. Belew, D. J. Fisher, H. C. Jones, M. T. Kelley, T. R. Mueller, and R. W. Stelzner, "Controlled Potential DC Polarograph-Voltammeter (ORNL Model Q-2792)," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967*, ORNL-4196, p. 3.

stabilized current. The apparatus usually consists of a pair of platinum electrodes, a large ballast resistor, and a source potential – all in series.[18,19] The developed potential is measured with a high-impedance voltmeter such as a pH meter. When high sensitivity and accuracy are desired, a stable current is required. In the simple apparatus just described, the current is made increasingly independent of cell characteristics by increasing the source potential and the size of the ballast resistor. However, at very high resistances, response times increase and leakage currents may become a factor. An instrument was fabricated that includes an operational amplifier which produces a highly stable cell current that is essentially independent of the electrode or cell characteristics.

The instrument incorporates a Philbrick P65QU operational amplifier to force a constant current through the cell. This amplifier has unusually low power requirements; therefore, mercury batteries can be used efficiently as a power supply, thus making the apparatus inexpensive and portable. The cell is placed in the circuit by connecting one electrode to the amplifier output and the other electrode to the inverting input of the amplifier. A small bias current (~0.5 μA) is applied at the inverting input by a 30-MΩ resistor connected to the +15-V power supply. Thus, a constant current of ~0.5 μA is forced through the cell. The potential developed across the electrodes is measured at the output of the amplifier with a microammeter and suitable scaling resistors to provide a full-scale output of 0.5 and 1.0 V.

This simple circuit has several advantages over the use of large resistors in series with a potential source. The cell current is independent of the conditions of the cell and electrodes. The potential developed across the cell can be measured with a low-impedance voltmeter, thus eliminating the need for a high-impedance instrument, such as a pH meter, to measure the cell potential. The cell current is easily adjusted to provide the optimum current density at the electrodes without affecting current stability (within the error limits of the amplifier).

The instrument has been used for Karl Fischer determinations of water in organic solvents and for iodine-thiosulfate titrations. The sensitivity and accuracy compare well with those attained by other methods. In the Karl Fischer titration, a 200-mV change in potential at the end point is observed for additions of as little as 0.003 ml of Karl Fischer reagent (≈15 μg of H_2O). Thiosulfate solutions as dilute as 1×10^{-5} N were titrated with 0.005 N iodate solutions with an end point error <0.01 ml. Further work will be done with the instrument to evaluate its sensitivity and stability.

1.12 CONTROLLED-POTENTIAL CONTROLLED-CURRENT CYCLIC VOLTAMMETER

T. R. Mueller H. C. Jones

A paper that describes the cyclic voltammeter discussed previously[20–22] was submitted for publication. Engineering drawings and a parts list are available.[23]

1.13 DERIVATIVE CIRCUITS FOR USE WITH THE CONTROLLED-POTENTIAL CONTROLLED-CURRENT CYCLIC VOLTAMMETER

T. R. Mueller D. L. Manning[24]

Preliminary studies are in progress to determine the requirements for the design of a differentiator (or purchase of a commercially available unit) to be used with the cyclic voltammeter (Sect. 1.12). The use of derivative techniques is expected to increase the accuracy[25] and sensitivity[26] of the determination of low-level constituents such as corrosion products in MSRE fuels.

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[18] J. T. Stock, *Amperometric Titrations,* Interscience, New York (1965) pp. 59–60; vol. XX of *Chemical Analysis*, ed. by P. J. Elving and I. M. Kolthoff.

[19] E. Bishop, "Differential Electrolytic Potentiometry. Part II. Precision and Accuracy of Applications to Redox Titrimetry," *Analyst* 83, 212 (1958).

[20] T. 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.

[21] T. 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.

[22] T. R. Mueller and H. C. Jones, "Controlled-Potential Controlled-Current Cyclic Voltammeter," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967,* ORNL-4196, p. 1.

[23] Anonymous, "Controlled-Potential and Controlled-Current Cyclic Voltammeter," revised CAPE-1653, *Engineering Materials List*, TID-4100.

[24] Reactor Projects Group.

[25] T. R. Mueller, "On the Separation of Half-Wave Potentials Required for Resolution of Simple, Reversible, Overlapping Waves in Stationary Electrode Voltammetry," *Chem. Instr.* 1, 113 (1968).

[26] S. P. Perone and T. R. Mueller, "Application of Derivative Techniques to Stationary Electrode Polarography," *Anal. Chem.* 37, 2 (1965).

1.14 ON THE SEPARATION OF HALF-WAVE POTENTIALS REQUIRED FOR RESOLUTION OF SIMPLE, REVERSIBLE, OVERLAPPING WAVES IN STATIONARY-ELECTRODE VOLTAMMETRY

T. R. Mueller

The requirements for the simultaneous determination of two or more species by linear-potential-sweep voltammetry at stationary (plane or sphere) or quasi-stationary (expanding sphere) electrodes are discussed in a note published in *Chemical Instrumentation.*[25]

1.15 MODIFICATION OF DUAL SET-POINT VOLTAGE COMPARATOR (ORNL MODEL Q-2950)

H. C. Jones T. R. Mueller

The operational-amplifier feedback network in the ORNL model Q-2950 comparator[27] was modified to eliminate occasional double-triggering that occurred when it was used with input signals that change very slowly. The comparator now operates reliably and has been tested with inputs in the range 100 mV/min to 20,000 V/sec.

1.16 INTEGRATOR MODULE

H. C. Jones T. R. Mueller

An inexpensive operational-amplifier current integrator was designed for H. H. Ross.[28] The integrator has three ranges. The ratio of the time constants of the three ranges can be trimmed to within ±0.01%. Total charges of 0.1 to 100 microcoulombs can be measured with a maximum error for worst-case design of ±1% in 4 min. The minimum input resistance is 3 MΩ. Full-scale output voltage is ±10 V. The integrator is very compact; it is packaged in a 5 × 6 × 8¾ in. box.

1.17 MODEL IX FLAME SPECTROPHOTOMETER

H. C. Jones R. W. Stelzner

The flame spectrophotometer designated model IX, formerly model VI-C,[29] was assembled and has been installed in Building 3026. A dc-amplifier system that consists of a Philbrick P25AU operational amplifier and PR-30C power supply provides full-scale output at the recorder for a photomultiplier-tube current of 10^{-9} amp. The following commercial components are incorporated into the model IX system: EMI type 9558QA photomultiplier tube, Fluke model 412B high-voltage power supply, Jarrell-Ash Ebert model 82-000 scanning spectrometer, and a Heath EUW20A recorder. A Beckman oxy-hydrogen burner and burner housing are mounted on an optical bench from the Jarrell-Ash Company. One quartz lens is used to focus the image of the flame on the entrance slit of the monochromator.

This instrument, now being used by the Radioisotopes-Radiochemistry Laboratory, was constructed to be a reliable replacement for an ORNL model Q-1457 flame spectrophotometer that had been in use for several years.

1.18 USE OF STATISTICAL TECHNIQUES IN FLAME PHOTOMETRY

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

The sensitivity limit of any measurement is set by the statistical characteristics of the noises that inevitably accompany the analytical signal. Statistical techniques, including correlation in the frequency and in the time domains, can optimize and enchance the signal-to-noise ratio and thus increase sensitivity. A paper has been prepared which describes a demonstration that ensemble averaging increases the sensitivity of flame photometry.[30]

1.19 INSTRUMENTATION FOR ANALYTICAL BIOCHEMISTRY

W. L. Maddox

The prototype tRNA analyzer described previously[31,32] has been replaced by an improved model. The new apparatus functions in the same way in the overall analytical scheme, that is, it removes fractions from the analytical product stream by depositing them

[27] T. R. Mueller, "Dual Set-Point Voltage Comparator (ORNL Model Q-2950)," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967,* ORNL-4196, p. 2.

[28] Nuclear and Radiochemistry Group.

[29] R. W. Stelzner, M. T. Kelley, and H. C. Jones, "Model VI Flame Spectrophotometers," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967,* ORNL-4196, p. 10.

[30] D. J. Fisher, R. W. Stelzner, and H. C. Jones, "The Use of Ensemble Averaging to Increase the Sensitivity of Measurements with Flame Photometers," submitted to *Chemical Instrumentation.*

[31] W. L. Maddox, "Instrumentation for Analytical Biochemistry," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967,* ORNL-4196, p. 9.

[32] W. L. Maddox and M. T. Kelley, "An Automatic Sampler for Obtaining Discrete Samples from the Output of a Continuous Flow Analyzer," *Chem. Instr.* 1, 105 (1968).

on filter-paper disks. The precision and speed of analysis with the new unit are similar to those of the prototype. The new unit differs in that the sampling operation is electrically synchronized with the Technicon input sampler rather than with a photometric controller at the proportioning-pump output. Provision is made to automatically start the output sampler 14 to 20 samples after the input sampler has been started; also, once started, the output sampler will continue automatically until 40 samples – the number contained in the input-sampler tray – have been taken. The output sampler then stops automatically. Adjustments are provided to phase the sample taken with the desired portion of the output stream.

1.20 COMPUTER-ASSISTED ANALYTICAL CHEMISTRY

R. W. Stelzner

A 4K-word-memory, integrated-circuit, digital computer – the Digital Equipment Corporation's model PDP 8/I – has been ordered for a study of possible applications of data acquisition and on-line real-time computing in the Division. It has been demonstrated[33] that where analytical methods are computer-assisted they have greater accuracy and precision as well as greater throughput. After an initial period of familiarization, the PDP 8/I computer will be evaluated for a molten-salt voltammetric problem. To facilitate the development of suitable interfacing, a system of logic modules has been purchased also.

1.21 ANALYTICAL APPLICATIONS OF A DIGITAL INSTRUMENT COMPUTER SYSTEM

D. J. Fisher D. R. Martin[34]
W. L. Maddox R. W. Stelzner

It has been shown that flame-photometric sensitivity can be improved by statistical means. Sensitivity is increased with a flame spectrophotometer that produces an optimized signal-to-noise ratio (S/N) and that is followed by ensemble averaging,[30,35] which gives substantial further enhancement of S/N. This work was done with a specific wired-program computer – a signal-averaging computer. A program has been initiated to explore analytical applications of a general-purpose digital instrument computer system.

1.21.a Capabilities

Their Instrument Computer system has been purchased from Fabri-Tek Instruments, Inc., Madison, Wisconsin 53711. The system is a wired-program digital computer that combines the flexibility of a general-purpose computer and the ease of operation of a laboratory instrument. The program can be varied by selection of switch positions and plug-in modules.

The system consists of:

Component	Description
FT-1064 main-frame unit	4096-word magnetic-core memory (18 bits/word, 1-μsec access time)
Plug-in module, digitizer	SD-1 digitizer, (12 or 8)-bit, resolution, linearity of ±0.05% of full scale SD-2 digitizer, (10 or 6)-bit resolution, linearity of ±0.1% of full scale (Each digitizer can function as an experiment interface)
Plug-in module, sweep-control	SW-1 control, dwell-time ranges 200 μsec to 0.8 sec SW-2 control, dwell-time range 50 μsec to 200 msec (Each unit programs the digitizer, arithmetic, and memory operations)
Oscilloscope	Tektronix type 503 (Monitors on expanded calibrated scales the analog-to-digital converter output, memory contents, and trials of data-processing options)
FT-201 decimal printer	Prints on paper 120 characters per second (as many as 4096 data prints in <4 min)
X-Y recorder (Other read-out devices, including tape can be obtained.)	Linear or logarithmic readout

For digital acquisition of each set of data, quarter, half or full memory can be used. Data processing can be

[33] J. W. Frazer, "Digital Control Computers in Analytical Chemistry," *Anal. Chem.* 40(8), 26A (1968).

[34] ORAU Student Trainee from Sacramento State College, Sacramento, Calif.

[35] D. J. Fisher, R. W. Stelzner, and H. C. Jones, "Enhancement of Flame-Photometric Sensitivity by Increasing Signal-to-Noise Ratio (S/N) with a Signal-Averaging Computer," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967*, ORNL-4196, pp. 11–12.

performed with the main-frame unit prior to readout. Processing options include: additive or subtractive acquisition of successive sets of data, data normalization, scaling, complementation, single or multiple integration, and data transfer. The original or processed data stored in a quarter or half of the memory can be added to or subtracted from information stored in any other quarter or half of the memory. Important separate processing capabilities are the correction of data for background and the quantitative comparison of data with other data. The FT-201 prints out the original and/or processed data and also the number of measurement sweeps made on the data.

The system is applicable to the measurement of any variable that can be locked to a sync pulse and observed as a function of a second variable. Just as software can be written to specifically program a general-purpose digital computer, so can plug-in modules be purchased or designed and wired for specific hardware programming of this digital computer system. There are two chief modes of digital data acquisition: one sweep per measurement (for fast, precise measurement followed by processing and/or readout to slower analog or digital recording devices) and ensemble averaging (with or without further data processing). Each measurement sweep can start either with the sync pulse that is locked to the experimental variable or after a selected delay time. As many as 8192 sweeps can be programmed by the Autostop switch. Also, the system can be used to control or automate experiments and, at the same time, to acquire and process data from the experiments. Its output can be interfaced to a general-purpose digital computer.

1.21.b Planned Use with Special Interfaces

It is expedient for the experimenter to acquire the ability to design, build, and modify interfaces between experimental equipment and a digital-computer system. Digital Equipment Corporation's Logic Kits and associated digital and analog equipment have been purchased so that interfaces for specific purposes can be built on a standard breadboard panel. It is planned to use the Fabri-Tek Instrument Computer system with interfaces built for special applications.

1.21.c Use as a Laboratory Test Instrument

The Fabri-Tek Instrument Computer system will be used to collect data essential to the rational design and testing of components, circuits, and systems for other instruments. In this application, it serves as an excellent laboratory test instrument. The output of a portion of an instrument, such as a voltage follower, precision peak follower, sample-and-hold circuit, or amplifier, can be quantitatively compared with its input. Also, the output of a derivative-computing circuit can be integrated and compared with the input. The system can be used to obtain the logarithmic or the n'th integral transform of a measurement. The response (single or averaged observations) of instrument circuits to standard test signals can be obtained and then read out to slower recording devices. During the design of an instrument, the system can be used to establish which measurement conditions are optimum. The performance of D.M.E.'s has been examined and tested with this system.

1.21.d Use for Digital Polarography

Work was begun to determine what use digital data acquisition has in polarography. The enhancement of sensitivity that can be obtained by ensemble averaging is being investigated. Later, possibly in conjunction with the average-mercury-flow-rate control device (Sect. 1.4) the precision obtainable with an optimum time scale will be determined. Also, it is planned to compare current-time D.M.E. wave forms. Special attention will be given to the study of the early portion of drop growth in relation to the various accepted equations. Results obtained by the mathematical resolution of overlapping digital polarographic waves will be compared with first-derivative dc polarograms obtained earlier.[36] Other polarographic work planned with this system includes the reevaluation of third-derivative dc polarography and the design of D.M.E.'s having improved S/N ratios.

1.22 INFORMATION PACKAGES SUBMITTED TO THE DIVISION OF TECHNICAL INFORMATION EXTENSION

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

Each CAPE package consists of a description of an instrument or apparatus, a list of references, copies of the drawings, a parts list, and specifications for fabrication. The material is duplicated and offered for sale by the Clearinghouse for Federal Scientific and Technical

[36] D. 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.

Information (CFSTI), Springfield, Virginia 22151. The *Engineering Materials List* (TID-4100 and its supplements) is a catalog of the items available from CFSTI.

We have submitted to DTIE the following revised or new packages:

CAPE No.	Title
368	"Automatic Potentiometric Titrator" (revised)
1196	"High-Sensitivity Coulometric Titrator /Q-2564/" (revised)
1220	"Servo-Controlled Pipetters" (revised)
1651	"Polarographic Drop Time Controller /Q-2942/"
1652	"Controlled-Potential D.C. Polarograph-Voltammeter /Q-2792/"
1653	"Controlled-Potential and Controlled-Current Cyclic Voltammeter"

1.23 COOPERATIVE PROJECTS

D. J. Fisher
and
Group

Through consultation and design efforts, the Analytical Instrumentation Group has cooperated with groups in the Analytical Chemistry Division and in other ORNL divisions and with people outside the Laboratory. Within our own division, we have consulted with the Reactor Projects Group on molten-salt instrumentation, the Methodology Group on a constant-current coulometric problem, the Methods Development and Evaluation Laboratory on the measurement of uranium by high-precision single-cell dc polarography, and the General Analyses Laboratory on a special valve problem in vacuum-fusion technology. With the various divisional groups who are using the instruments and apparatus developed in our group, we have consulted concerning their operation and maintenance.

Within the ORNL organization, we have assisted the Chemistry Division in the design of a voltage-follower amplifier system for an electrochemical measurement; we have cooperated with the Instrumentation and Controls Division in the check-out of a cyclic voltammeter (ORNL model Q-2942); and we have consulted with Reactor Chemistry Division personnel concerning the application of the cyclic voltammeter and have assisted in the design of a high-input impedance amplifier system. For the Process Instruments Maintenance Group (4500S Instrument Shop) of the Instrumentation and Controls Division, a series of six lectures on operational amplifiers in analytical instrumentation were delivered – their purpose was to help the instrument technicians who must service the equipment.

Outside the laboratory, we have had fabricated for the USAEC New Brunswick Laboratory some critical parts for an ORNL model Q-1348B servo-pipetter; for the University of Tennessee Hospital in Knoxville, we checked out an ORNL model Q-1988A polarograph prior to its loan. We have sent on loan to the Department of Chemistry at Purdue University the prototype model of the conductivity titrator, and we have arranged the loan of a servo-pipetter display to the United Technical Corporation, West Concord, Mass. With personnel of Lawrence Radiation Laboratory, Livermore, Calif., we have cooperated in the interpretation of voltammetric data from multicomponent systems through use of our computer program CHICAL and CURRENTS. To Professor A. F. Krivis of the University of Akron, diagrams and information about the ORNL model Q-2792 polarograph were furnished for use in a book he is writing.

2. Analytical Methodology

W. D. Shults

2.1 GAS CHROMATOGRAPHY

2.1.a Instrumentation

M. R. Guerin

The gas chromatographic capabilities of the Division have been extended with the acquisition of a biomedical gas chromatographic system. It consists of two column-bath units and an electronics unit and has the usual thermal-conductivity and dual hydrogen-flame-ionization detectors for routine use. The system features gas- and solid-sampling capability, an all-glass analytical flow train, capillary-column capability, multilinear temperature programming, high-temperature electron-capture detection, flame-photometric detection (phosphorus- and sulfur-specific), and simultaneous operation of all detectors. The system allows the separation, partial characterization, and quantitation of microgram and submicrogram amounts of most nonmacromolecular organic compounds.

2.1.b A Remodeled Laboratory for Gas Chromatography

A. D. Horton[1] C. M. Boyd[1] A. S. Meyer[1]

One of the "wet-chemistry" laboratories in Building 4500S is being remodeled specifically for gas chromatography to effect more efficient use of the floor and storage areas and to eliminate potential safety hazards now associated with instrument maintenance.

A major modification is the separation of the two center benches to form a 2-ft-wide unobstructed aisle between their backs. This aisle gives safe access for maintenance of the bench-top chromatographs. All services for these instruments are conducted through a 6-in.-wide chase that runs full length at the rear of each bench. There are no backsplashes on the benches, and very short connections can be used to provide the gas and electrical services to each instrument.

All unneeded services have been eliminated, for example, water with accompanying drains and about half the air, natural-gas, and house-vacuum outlets. The services required to operate the chromatographs were added, namely: high-purity helium, argon, and nitrogen; two unassigned high-purity gas systems; and clean air. A high-vacuum system and outlets for natural gas, oxygen, high-pressure process air, and house vacuum complete the services on the center benches. Similar services are installed at wall stations to provide for the operation of console instruments.

All gas cylinders and vacuum pumps are located in the attic above the laboratory, but the associated regulators and electrical switches are located within the laboratory. The purity of the service gases to the chromatographic system is ensured by constructing connecting lines of stainless steel and by Heliarc welding all connections that are not easily accessible.

One 8-ft laboratory hood was removed from each side of the laboratory. On one side, the hood was replaced with storage cabinets; on the other, the Varian-Aerograph preparative gas chromatograph and vented cabinets for the storage of organic solvents will be installed.

The modifications were designed with the assistance of G. R. McNutt,[2] and the changes are being made under the supervision of F. L. Hannon.[2]

2.1.c Device for Sampling Gases Contained in Sealed Metal Tubes

A. D. Horton[1] C. M. Boyd[1] M. R. Guerin

The gases contained in Geiger-Mueller (G-M) tubes were collected for mass spectrometric analysis by use of a puncturing device (Fig. 2.1). The G-M tube is inserted in a $^3/_4$-in.-OD copper tube that is connected at one end to a vacuum pump and at the other end to a sample bulb. The entire system is evacuated, and the device is isolated from the vacuum system by closing a valve (not shown). The G-M tube is punctured by a sharp blow on the welding rod with a hammer. A portion of the sample enters the sample bulb, which is sealed off with a torch. A bulb with a vacuum stopcock may also be used. This device has been used to puncture copper tubing having 0.035-in.-thick walls. It can be used for any soft-metal tubing or for thin-walled tubing of any metal if the outside diameter of the tube does not exceed $^9/_{16}$ in.

[1] Reactor Projects Group.

[2] General Engineering Division.

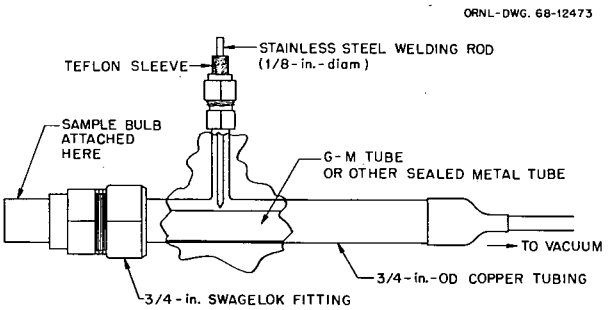

Fig. 2.1. Device for Sampling Gases in Sealed Metal Tubes.

2.1.d Biomedical Applications

M. R. Guerin

The biomedical gas chromatographic system has been used to study free fatty acids, phenylthiohydantoin derivatives of amino acids, and trimethylsilylphenylthiohydantoin derivatives of amino acids. Valeric, isovaleric, myristic, palmitic, and stearic acids were used to establish conditions for the separation and detection of submicrogram quantities of C_4 to C_{30} saturated fatty acids. The conjoint use of silylation and flame-photometric detection appears to be highly promising for the analysis of phenylthiohydantoin derivatives of amino acids.

2.1.e Determination of the Methyl Esters of Butyl Phosphoric Acids

A. D. Horton[1]

The Chemical Technology Division is studying extensively the degradation of tributyl phosphate (TBP) by nitric acid. The decomposition products are phosphoric, butyl phosphoric, and dibutyl phosphoric acids. That Division prepared methyl esters of these acids by diazotization; trimethyl phosphate (TMP), dimethylbutyl phosphate (MMBP), and methyldibutyl phosphate (MBBP) are obtained.

These esters are separated from each other and from TBP on a 10-ft by ¼-in. stainless steel column that is packed with 60- to 80-mesh siliconized glass beads coated with 0.15 wt % Carbowax 20-M. The column is operated at a helium flow rate of 60 cc/min and is programmed to 235°C at 9°/min after an isothermal period of 8 min at 110°C. Figure 2.2 shows the chromatogram.

The outstanding feature of the low-loaded glass-bead column is that the easily decomposed compounds are eluted at temperatures as much as 125°C below their boiling points. The column is operated routinely with ester concentrations as low as 0.01 mole % when the flame-ionization detector is used. If the sample size is increased, concentrations as low as 0.001 mole % can be measured.

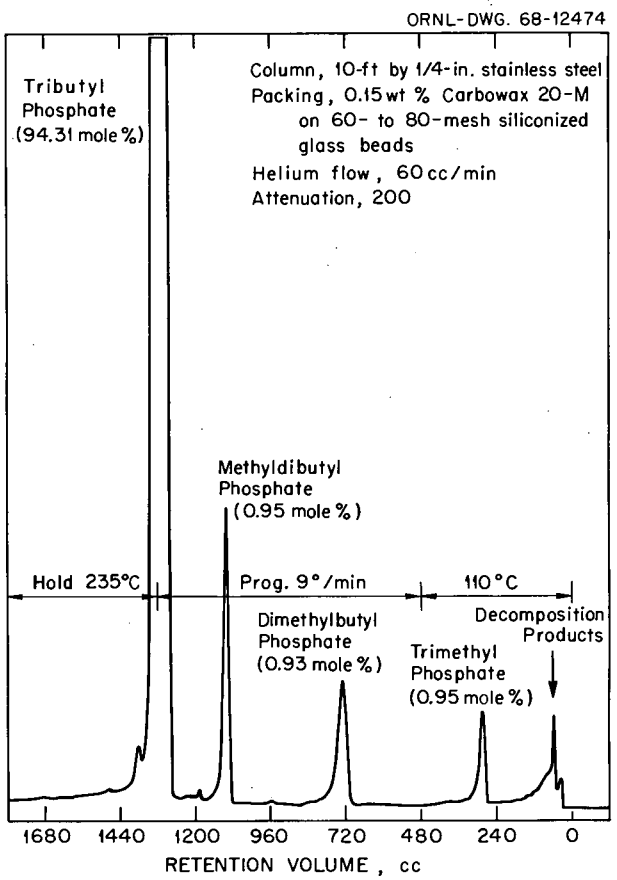

Fig. 2.2. Gas Chromatogram of a Mixture of Trimethyl Phosphate, Dimethylbutyl Phosphate, Methyldibutyl Phosphate, and Tributyl Phosphate.

2.1.f Determination of Perfluoromethylcyclohexane in Methane

A. D. Horton[1]

The Radiation Physics Section of the Health Physics Division is studying electron attachment to fluorinated hydrocarbons and the life of the attachment to these compounds. A sample of methane that contained perfluoromethylcyclohexane (C_7F_{14}) in very low concentration was submitted for the determination of C_7F_{14}. This compound is an analog of methylcyclo-

hexane and has about the same boiling point. However, columns that are suitable for separating methylcyclohexane from methane did not adequately resolve methane and C_7F_{14}; the C_7F_{14} peak appeared on the tail of the relatively large methane peak.

Perfluoromethylcyclohexane was separated satisfactorily from methane on an 8-ft by ¼-in. stainless steel column packed with 30- to 60-mesh Burrell medium-activity silica gel coated with 3 wt % squalene. The helium flow was 60 cc/min; the column was operated isothermally at 62°C for 4 min and then was temperature programmed at 6°/min to 125°C. The flame-ionization detector was used. The retention volume of methane was 66 cc and that of C_7F_{14} was 630 cc. The concentration of C_7F_{14} in the only methane sample submitted was 0.17 μl (liquid) per liter of methane.

2.1.g Preparative Gas Chromatography

A. D. Horton[1] J. D. Lodmell[3]

The organic compounds listed in Table 2.1 were purified by preparative gas chromatography.[4]

Table 2.1. Organic Compounds Purified by Preparative Gas Chromatography

Compound	Purity Achieved (mole %)
Acetonitrile	>99.99
Aniline	>99.99
1-Bromobenzene	99.96
1-Bromobutane	99.98
1,6-Dimethylnaphthalene	99.96
Dimethylbutyl phosphate (crude)	96.76
2-Ethylnaphthalene	>99.99
Methyl formate	>99.99
Propionitrile	>99.99
n-Propylbenzene	>99.99
2,2,6,6-Tetramethylheptane-3,5-dione	>99.99
1,1,1-Trichloroethane	99.98
1,1,2-Trichloroethane	>99.99
N-Valeric acid	99.95
p-Xylene	>99.99

Only one significant change has been made in the Varian-Aerograph preparative gas chromatograph. The neoprene diaphragm in the pressure-time sampling valve, which is normally closed, was replaced with a Viton diaphragm. Benzene and its derivatives cause neoprene to swell. An inordinately high pressure is required to inject a sample onto the column through a swollen neoprene diaphragm. The Viton diaphragm allows a uniform low pressure to be used and thus provides more reproducible injection volumes. The life of the Viton diaphragm has not been determined; the first one has given satisfactory service since its installation in February 1968.

2.1.h Derivative Gas Chromatography

R. W. Morrow[5] J. A. Dean[6]
M. R. Guerin W. D. Shults

We have initiated a program of study in derivative gas chromatography. This program will comprise the doctoral research for R. W. Morrow. The objective of the first phase of this project has been to develop a detector that is specific for organosilicon compounds. A flame-photometric detector for silica has been built and tested. Its performance is adequate, but work is continuing with the intent of improving sensitivity. This phase of the work has been conducted largely at the University of Tennessee, but the subsequent work will be done within the Division laboratories.

2.2 THIN-LAYER CHROMATOGRAPHY

2.2.a Instrumentation

Helen P. Raaen

The Chromoscan was received from Joyce, Loebl & Co., Ltd. on April 1. The instrument is a recording and integrating densitometer, absorptiometer, and colorimeter for reflectance and transmittance operations in the visible and ultraviolet on various types of samples. The thin-layer scanner attachment arrived on July 26, and the power supply for its ultraviolet light source was received on August 16. However, the ultraviolet light source has not yet come. The scanner attachment will be suitable for reflectance and transmittance measurements in the visible and ultraviolet on thin-layer chromatograms of various types.

[3]ORNL Student Trainee under the ORNL Undergraduate Program; present address, University of Tennessee, Knoxville.

[4]A. D. Horton, "Preparative Gas Chromatography," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967,* ORNL-4196, p. 33.

[5]Student Guest from the Department of Chemistry, University of Tennessee, Knoxville.

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

2.2.b Polytetrafluoroethylene (Teflon) as an Adsorbent

Helen P. Raaen

We have done experimental work to develop polytetrafluoroethylene (Teflon) layers for thin-layer chromatography (TLC). A description of the details of the work will be reserved for publication as a journal article; a summary follows:

Teflon slurries of numerous compositions were evaluated for forming the Teflon layer. Layers were made whose properties are entirely satisfactory for TLC. These properties include rate of drying, uniformity, adherence to a support (glass, and Mylar or vinyl film), wettability by test-solution aliquot and developer, rate of development, and resolution of the components of a test mixture. It was demonstrated that the layers are suitable for both partition and reversed-phase partition chromatography, depending on how the liquid components of the chromatographic system are distributed between the stationary phase on the thin layer and the mobile developer. A thin layer of Teflon was found to be a suitable solid support for the selected liquid anion-exchanger di-(2-ethylhexyl)-orthophosphoric acid (HDEHP); two multicomponent mixtures of cations were separated on such a layer by both partition and reversed-phase partition TLC. This finding invites a detailed study of the use in TLC of liquid ion exchangers, of which many exist. In addition, it indicates the potential usefulness of thin layers of Teflon (a chemically inert material) for studying the mechanism of extractions with liquid ion exchangers by means of TLC without the need to consider the reactivity of the solvent system with the solid support, a factor that must be considered when most other TLC adsorbents are used. Also, Teflon was shown to be compatible in thin layers with alumina and with silica gel in any ratio. The possibility therefore exists of including Teflon as a component of mixed-adsorbent and gradient layers for TLC and thus, on a single layer, of simultaneously effecting adsorption and partition TLC in any controlled ratio to each other.

Extensive and exciting experimental work with Teflon as a TLC adsorbent is possible. First consideration is being given to corrosive (*e.g.*, HF-containing) systems for which existing adsorbents (*e.g.*, silica, alumina, the celluloses, and the like) are not suitable and to the difficultly separable transuranium elements.

2.2.c Constituents of Transfer Ribonucleic Acid (tRNA)

Helen P. Raaen Frances E. Kraus[7]

Our work[8] to resolve complex mixtures of nucleic acid bases, nucleosides, and nucleotides by two-dimensional TLC on polyethyleneimine-cellulose is described in a published article.[9] The summary from the article follows:

"A method is given for resolving a complex mixture of nucleic acid bases, nucleosides, and nucleotides on polyethyleneimine-cellulose MN-300 chromatofilms. The nucleotides are prevented from migrating when the individual nucleic acid bases and nucleosides are separated from each other. Also, in the course of the development that resolves the nucleotides, the nucleic acid bases and nucleosides disappear. Thus, the procedure simplifies the isolation and detection of individual components of a complex mixture of nucleic acid derivatives. The method was applied satisfactorily to the analysis of alkali hydrolysates of the transfer ribonucleic acids of E. coli B and of yeast."

2.2.d Amanita Toxins

Helen P. Raaen

Thin-layer chromatography was used to follow the progress of the production of amanita toxins by fermentation growth of the mycelium of the mushroom *Galerina marginata.*[10] During this work, some improvement in an existing TLC separation was achieved. In a note accepted for publication,[11] this improvement is described and is illustrated with color photographs of pertinent chromatograms.

2.2.e Steroids

Linda J. Crist[12] Helen P. Raaen

In the Body Fluids Analyses Program, it is of interest to detect, identify, and quantitatively measure steroids in urine. Work was begun to apply TLC in the solution of these problems. Because of the large number (>600) and the complexity of the steroids that may be present in urine, their separation by TLC can be exceedingly difficult. The experimentation to date has consisted in showing that a selected TLC system is satisfactory for the immediate work, obtaining reference data for pure

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[7] ORAU Student Trainee from Nazareth College, Nazareth, Ky.; summer, 1967.

[8] H. P. Raaen and F. E. Kraus, "Thin-Layer Chromatography of Constituents of Transfer Ribonucleic Acid (tRNA)," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967,* ORNL-4196, pp. 34–35.

[9] H. P. Raaen and F. E. Kraus, "Resolution of Complex Mixtures of Nucleic Acid Bases, Nucleosides, and Nucleotides by Two-Dimensional Thin-Layer Chromatography on Polyethyleneimine-Cellulose," *J. Chromatog.* 35, 531 (1968).

[10] H. P. Raaen, "Thin-Layer Chromatography of Amanita Toxins," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967,* ORNL-4196, p. 35.

[11] H. P. Raaen, "Improved Thin-Layer Chromatographic Separation of Amanita Toxins on Silica Gel G Chromatoplates," accepted for publication in the *Journal of Chromatography.*

[12] ORAU Student Trainee from Arkansas State University, Jonesboro.

steroids and steroid conjugates, determining the limit of detection for four steroids and a steroid conjugate, analyzing samples provided from the column chromatography of urines by the Body Fluids Analyses Group, and evaluating the TLC system as a means to follow the course of acid and enzymatic hydrolysis of steroid conjugates. Also, information was accumulated on possible methods for the quantitative measurements of submicrogram amounts of steroids isolated by TLC.

The TLC system found to give satisfactory resolution of 20 steroids of primary interest includes: adsorbent, Merck Silica Gel G in 250-μ-thick layers on Mylar film (or glass); developer, chloroform-methanol (97:3 v/v); and development, 15 cm in a developer-saturated atmosphere. Gelman ITLC-SA medium is an equally satisfactory adsorbent. The developer dichloromethane-acetone (70:30 v/v) also causes the steroids to migrate on Merck Silica Gel G, but in a slightly different order. Therefore, it may be a suitable developer for use in a two-dimensional system for resolving complex mixtures of steroids.

The chromatograms are analyzed both before and after they are sprayed with 1:1 H_2SO_4 (conc.) and heated to 78°C. The analysis consists in observing the chromatograms for color, absorbance, fluorescence, and phosphorescence both in air and immersed in liquid nitrogen under visible, 366-nm ultraviolet, and 254-nm ultraviolet light.

For the 21 pure reference steroids and the 10 conjugated steroids that constitute the collection of the Body Fluids Analyses Group, reference TLC information has been accumulated and cataloged. It can be retrieved when sought according to either the name of the compound or the conditions under which the presence of a resolved component on the thin-layer chromatogram is evident.

For some selected compounds, their limits of detection on the thin layer by observation of fluorescence at room temperature were determined by sequential dilution. The developed chromatogram was sprayed with 1:1 H_2SO_4 (conc.) and then heated at 78°C on a hot plate to form products that are colored in visible light and that fluoresce under 254-nm ultraviolet light. The compounds and their detection limits were:

Compound	Micrograms	
	Detectable	Not Detectable
17-Hydroxydeoxycorticosterone	0.1	0.01
11-Dehydrocorticosterone	1	0.1
Cortisone	1	0.1
5α-Pregnane-3β,20β-diol	1	0.1
Pregnanediol glucuronide	1	0.1

Fluorescence is among the most sensitive means for detection.

Of particular interest is the observation that androsterone, 11-dehydrocorticosterone, 5β-pregnane-3α,20α-diol diacetate, Δ^4-androsterone-3,11,17-trione, 17β-estradiol, estriol, and estrone, on layers sprayed with 1:1 H_2SO_4 (conc.) and heated at 78°C, phosphoresce under liquid nitrogen after excitation with ultraviolet light. This characteristic of these steroids has not, to our knowledge, been reported before. A note on this observation has been prepared for publication in the *Journal of Chromatography*. Low-temperature phosphorescence of steroids appears to have received only limited study; it should have most interesting possibilities.

The TLC system was applied to the analysis of 16 samples of interest in the column chromatography of urine. The results did not show conclusively the presence of any one of the standard steroids studied. However, several components were isolated that are now undergoing identification tests.

The TLC system was shown to be a means to follow the hydrolysis of steroid conjugates. Samples of three pregnanediols and a pregnanediol glucuronide were chromatographed simultaneously with the HCl-hydrolysis product of the pregnanediol glucuronide. Each of the pregnanediols and the glucuronide were isolated satisfactorily. In the product, pregnanediols were identified, but numerous other products of the hydrolysis were also indicated to be present.

2.2.f Mass Spectrometry of Substances Isolated by Thin-Layer Chromatography

W. T. Rainey[13] Helen P. Raaen
Linda J. Crist[12]

Mass spectrometry can be used to identify and possibly to quantitatively measure some substances isolated by TLC. The method is of increasing interest for organic substances. The mass spectra of several TLC adsorbents (polytetrafluoroethylene, polyethyleneimine-cellulose, Sephadex G-75, polyolefin, Merck Silica Gel G, Corning porous glass 7935, Gelman ITLC-SA medium, alumina) were recorded on the low-resolution mass spectrometer to determine the extent of their background interferences in the mass spectrometry of substances held on them. The inorganic adsorbents behave better than the organic. The silicic adsorbents cause little or no interference; the interference from

[13] Mass and Emission Spectrometry Group.

polytetrafluoroethylene is acceptable, and that from polyolefin is not.

2.2.g Infrared Spectrometry of Substances Isolated by Thin-Layer Chromatography

M. M. Murray[14] Helen P. Raaen
Linda J. Crist[12]

Infrared spectrometry is an established way to identify organic substances. A technique in which Wick-Sticks (wedge-shaped pieces of compressed KBr) are used effects the removal of a TLC-isolated substance from the adsorbent onto KBr for subsequent infrared spectrometry. This technique is being evaluated with corticosterone isolated by TLC on a Merck Silica Gel G layer and on Gelman ITLC-SA medium.

2.3 CHARACTERIZATION OF TOBACCO SMOKE AND SMOKE CONDENSATES

A program was begun to develop analytical methodology to definitively characterize tobacco smoke. The approach proposed is to selectively remove compounds of given classes, such as polynuclear aromatic hydrocarbons, inorganic gases, and alkaloids, from tobacco smoke or smoke condensate and to determine the compounds simultaneously as classes and individual components. Gas chromatography, with its high resolving power, high sensitivity, and general applicability, appears to be the analytical method of choice.

2.3.a Exploratory Studies

M. R. Guerin

Thus far, experimental work has been done to gain a further appreciation of analytical difficulties peculiar to cigarette smoke and smoke-condensate matrices. The condensate from 500 cigarettes in each of the five trap sections of a smoking apparatus (Sect. 10.11.b) was divided into ether-soluble and ether-insoluble portions; the ether-insoluble condensate was dissolved in methanol. The ethereal solutions and methanolic solutions were concentrated. Samples of the ether-soluble fraction and methanol-soluble fraction of the total condensate were obtained by combining aliquots of the ethereal concentrates and methanolic concentrates, respectively.

The total condensate samples were subjected to chemical fractionation. An aliquot of the methanolic total-condensate sample was extracted with cyclohexane. The cyclohexane extract was then extracted with nitromethane to give a solution enriched in polynuclear aromatic hydrocarbons. Part of the cyclohexane-extracted methanolic solution was filtered through a lipophilic resin to give a fraction enriched in polar components. An aliquot of the ethereal total-condensate sample was fractionated into basic, phenolic, acid, and neutral portions by solvent extraction. The neutral portion was subjected to further fractionation on silica gel. Portions of each solution were taken at appropriate stages in the fractionation and concentration procedures to yield samples that corresponded to various levels of condensate handling. Gas chromatographic profiles are to be obtained for each fraction to find whether significant chemical fractionation occurs in the condensate-collection apparatus and to measure the influence of the various fractionation and concentration procedures on the contents of the fractions.

2.3.b Thin-Layer Chromatography

Helen P. Raaen

Information has been collected on the application of TLC to the isolation and identification of the components of tobacco-smoke condensates. Bibliographic information was received from the National Clearinghouse for Smoking and Health, the National Library of Medicine, and 21 organizations listed in the publication "1967 Directory of On-Going Research in Smoking and Health" as doing analyses on tobacco smoke. Also, references were extracted from the "1967 Bibliography on Smoking and Health." The most abundant information came from Dr. R. L. Stedman of the Tobacco Laboratories, Agricultural Research Service, U.S. Department of Agriculture, and from Dr. E. Sawicki of the Particulate Chemistry Section of the National Center for Air Pollution Control. The material was searched carefully and indexed. Unfortunately, it includes very few references on the application of TLC.

This scarcity prompted a search of the entire *Journal of Chromatography*. Information was sought in particular on ways to separate polynuclear aromatic hydrocarbons, alkaloids, and phenols, which are three types of compounds of importance in the analysis of tobacco smoke condensates. Also, attention was given to articles that describe unusual techniques of analysis or that suggest unique overall approaches to the general problem of the analysis of tobacco-smoke condensates. The search was very fruitful, and the references of interest have been indexed.

[14] Infrared and N.M.R. Group.

A memorandum was prepared on the possible role of TLC in the analysis of tobacco-smoke condensates. The memorandum calls attention to newer techniques that warrant consideration during our work in this area.

2.4 ANALYSIS OF WATER SYSTEMS

2.4.a Aqueous-Injection Gas Chromatography

M. R. Guerin W. J. Ross W. D. Shults

Samples for gas chromatography are often in organic solvents. When the compounds to be determined are present in aqueous solution, they are normally recovered in an organic solvent and subsequently determined in that solvent. Applications of the direct gas chromatographic analysis of aqueous solutions (aqueous-injection gas chromatography) are becoming increasingly important with the intensified interest in determining the organic components of biological fluids and natural water systems. Accordingly, we have begun to study means for, and applications of, aqueous-injection gas chromatography.

An aqueous-injection gas chromatographic technique[15] for the quantitative determination of the total carbon content of aqueous solutions has been studied. Microliter volumes of aqueous samples are injected onto a bed of hot (850°C) copper oxide over which a carrier gas (nitrogen or helium) passes continuously. The carbon in the sample is converted by the hot copper oxide to carbon dioxide. The carbon dioxide is mixed with hydrogen, and the mixture is swept by the carrier gas through a heated (300°C) nickel packing to produce methane. The methane is swept onto a gas chromatographic column and is measured in the column effluent with a flame-ionization detector. Whereas for methane the limit of detection (<1 ppm total carbon) and the shape of the detector response are good, it has been difficult to obtain reproducible quantitative results for total carbon. The problem may be associated with the necessity to inject the sample by syringe through a silicone rubber septum.

The possibility of applying aqueous-injection gas chromatography to the determination of dissolved gases has also been examined. Conditions have been established that allow methane, air, carbon dioxide, hydrogen sulfide, and phosphine to reach an analytical column while water is being retarded long enough to be vented from the system.

The results of the study of the carbon analyzer and relative ease with which methane, hydrogen sulfide, and phosphine can be stripped from water suggest that it may be possible to develop an aqueous-injection gas chromatographic system for estimating the total carbon, sulfur, and phosphorus (and perhaps total nitrogen) contents of aqueous solutions.

2.4.b Eutrophic Water Systems

W. J. Ross

In association with members of the Health Physics Division, 15 sites have been monitored for phosphorus and nitrogen to study the origin and movement of algal nutrients in local watersheds.[16] A Technicon Auto-Analyzer was acquired to measure very low concentrations (5 to 100 ppb) of phosphorus as PO_4^{3-} and nitrogen as NO_2^- or NO_3^- by the molybdenum blue and diazo (sulfanilamide-naphthylethylenediamine) colorimetric methods, respectively. Measurements were made on untreated samples, as well as on those that had been treated to convert organic phosphates and inorganic metaphosphates to orthophosphate ions.

A highly polluted water system was analyzed by activation analysis. The objectives were to establish the effect of Mo, Cu, P, Mn, and Zn on the growth of *chlorella* algae and to determine the effectiveness of hydroponic beds of rye and fescue to remove these elements from flowing streams of water. This work was done in cooperation with members of the U.S. Department of Agriculture, who initiated the program.

2.4.c Deaeration Systems

W. J. Ross

A reliable chemical method was needed to evaluate instruments designed to measure very low concentrations (1 to 100 ppb) of dissolved O_2 in saline water and also as a "standard" method to be used in acceptance tests of future desalination plants. The Winkler method was adapted for the determination of dissolved O_2 in water from any source. The work has consisted principally in identifying and eliminating all interferences and has included the modification of colorimetric methods for Fe^{2+}, Fe^{3+}, Cu^+, and Cu^{2+} for use in the determination by field laboratories of ppb amounts of these ions. Methods are also being developed to remove these interferences from discrete samples or flowing

[15] F. R. Cropper, D. M. Heinekey, and A. Westwell, "The Determination of Total Organic Matter (Carbon Content) in Aqueous Media," *Analyst* 92, 436 (1967).

[16] W. J. Ross, "Analysis of Water Systems," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967*, ORNL-4196, p. 87.

streams of deaerated water without altering the concentration of dissolved O_2. Chelating reagents and columns of chelating ion-exchange resins have been applied successfully to fresh-water systems but have not been tested with saline water. Continued investigation of the $H_2CO_3 \rightleftharpoons HCO_3 \rightleftharpoons CO_2$ equilibria and additional modification of a revised ASTM method[17] for determining CO_2 in saline water have been required to ensure reliable analyses of brackish water that contains very high concentrations (200 to 500 ppm) of dissolved CO_2.

These analytical methods were used in a large number of performance tests to evaluate the deaeration system of the Desalination Facility at Roswell, New Mexico, which is operated by the Office of Saline Water, and the experimental deaeration equipment designed and installed at ORNL.

2.4.d Fluorometric Determination of Phosphate

J. C. Guyon[18] W. D. Shults

The work on the fluorometric determination of phosphate was reported previously.[19] An article on it has now been submitted for publication in the *Journal of the American Water Works Association.*[20]

2.5 SEPARATION AND CONCENTRATION STUDIES

2.5.a Column Chromatography with Antimony Pentoxide

W. J. Ross

A study of the efficacy of Sb_2O_5 for isolating sodium ions[21] – especially dangerous or interfering amounts of $^{24}Na^+$ – has been continued as part of a program to determine manganese and copper in samples of human tissue by activation analysis. Complete removal of milligram amounts of sodium from 1 to 12 *N* solutions of HCl, HNO_3, and H_2SO_4 can be achieved by adsorption of sodium on small (0.5-g) columns of Sb_2O_5. The ions Mn^{2+} and Cu^{2+} are not retained when the acidity is 6 to 12 *N*; therefore, the gamma radioactivity from ^{56}Mn and ^{64}Cu can be measured in the eluate without interference from ^{24}Na. Related experiments have shown that K^+ and Cs^+ are completely adsorbed on a column of Sb_2O_5 when the acid concentration of the elutriant is 1 *M*. However, the degree of adsorption diminishes with increasing acidity. Therefore, both K^+ and Cs^+ are completely eluted in >9 *M* HNO_3 and can be separated quantitatively from Na^+.

The ions Mn^{2+} and Cu^{2+} can also be separated from Na^+ by adsorption on a column of Chelex-100 cation-chelating resin. When the pH of the sample solution is in the range 2 to 4, Mn^{2+} and Cu^{2+} are completely adsorbed and Na^+ is rejected. Sodium ions are not adsorbed at pH's <4; Mn^{2+} and Cu^{2+} are retained completely only at pH's >2. Therefore, the buffer 0.1 *M* acetic acid–0.1 *M* sodium acetate is used to ensure quantitative separation.

2.5.b Evaluation of a Lipophilic Adsorption Resin for Desalting and Removing Organic Compounds from Water

M. R. Guerin

Amerlite XAD-2, a highly lipophilic polystyrene adsorption resin manufactured by the Rohm and Haas Company, has been examined as a medium for removing low-molecular-weight water-soluble organic compounds from large volumes of acetate buffer. The ability of the resin to adsorb organic compounds from dilute aqueous solutions suggests that the resin may also be useful for removing organic compounds from natural waters for further analysis.

Batch-extraction procedures were used to assess the potential utility of XAD-2. When 7-ml volumes of wet XAD-2 were in contact for 24 hr with 40 ml each of $\sim 5 \times 10^{-3}$ *M* aqueous solutions of *sec*-butylamine, piperidine, 3-indoleacetic acid, and hippuric acid, the resin adsorbed 65, 54, 72, and 94%, respectively, of the test compound. For 0.05, 0.1, 1, and 2 *M* solutions of acetate buffer (pH 4.4) under the same conditions, the resin extracted 16, 15, 17, and 17% of the acetate, respectively. When the resin and test solution were transferred to a glass-wool-plugged separatory funnel and washed with distilled water, 99% of the acetate

[17] "Total Carbon Dioxide and Calculation of the Carbonate and Bicarbonate Ions in Industrial Water," ASTM Method No. D 513-57, pp. 29–34 of *1964 Book of ASTM Standards,* pt. 23, American Society for Testing and Materials, Philadelphia, Pa., 1964.

[18] ORAU Research Participant; Associate Professor of Chemistry, University of Missouri, Columbia.

[19] J. C. Guyon and W. D. Shults, "Fluorometric Determination of Phosphate," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967,* ORNL-4196, p. 40.

[20] J. C. Guyon and W. D. Shults, "Rapid Survey Methods for the Fluorometric Determination of Phosphate," submitted for publication in the *Journal of the American Water Works Association.*

[21] W. J. Ross, "Separation of Sodium with Antimony Pentoxide," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967,* ORNL-4196, p. 88.

buffer was removed from the sample, whereas of that originally present ~40% each of the *sec*-butylamine, piperidine, and hippuric acid and ~90% of the indoleacetic acid were left on the resin. The facts that acetate is removed from the resin and the test compounds were not completely stripped from the resin strongly suggest that large amounts of inorganic salts could be very easily removed from very small amounts of organic compounds by use of XAD-2.

It is concluded that this liquid-solid extraction procedure affords a simple and effective approach to greatly reducing the contamination of small amounts of organic compounds by common salts. The conclusion is based on considerations of the influence of pH, sample volume, resin volume, and sample concentration on resin uptake and on a comparison of liquid-liquid extraction by use of ethyl acetate, ether, *n*-butanol, and benzene with liquid-solid extraction using XAD-2. Although filtration through XAD-2 is definitely a way to remove organic compounds from natural waters, its relative merit for this purpose compared with filtration through carbon remains to be determined.

Amberlite XAD-2 is being evaluated further as a scavenger of organic compounds from natural waters and as a medium for adsorption chromatography.

2.5.c Device for the Unattended Concentration of Solutions

M. R. Guerin

A means has been devised for the unattended 100- to 1000-fold concentration of the solutes in organic solutions. By an infusion pump that drives a 10-ml syringe, the solution is fed continuously into a small evaporation chamber while the solvent is removed by an aspirating stream of air through the chamber. Loss of solute to the walls of the chamber is minimized by maintaining a very small liquid volume within the chamber (50 to 100 μl). An evaluation of the technique by use of *n*-eicosane as a solute in diethyl ether indicates that solute recovery can be quantitative.

Chamber geometry influences the rate of evaporation of the solvent. A higher rate is achieved by venting the aspirating air parallel to and directly above rather than perpendicular to the sample zone of the chamber. The rate of evaporation is highly dependent on the flow rate of the aspirating air. A study of 22 solvents showed that the time necessary to reduce 10 ml of solvent to 10 to 100 μl ranges from 30 to 90 min for solvents such as diethyl ether and *n*-hexane to 15 to 20 hr for solvents such as dimethyl sulfoxide and *n*-butanol.

Caution must be exercised in applying the technique to solutions that contain large amounts of inorganic salts. Salt deposited in the chamber alters the evaporation rate and thus necessitates frequent adjustments in the flow rate of the air and/or delivery rate of the solution.

Because the chamber remains cool throughout the concentration procedure and because concentration is achieved at atmospheric pressure, the dangers of chemically altering or volatilizing the solute are minimized. An inert gas may be substituted for air in those cases where contact with atmospheric constituents must be avoided.

2.5.d Concentrating Hydrochloric Acid

W. J. Ross

In the course of a series of experiments in which ^{36}Cl was adsorbed on tin oxide electrodes, a technique was developed whereby ^{36}Cl, as $H^{36}Cl$ in aqueous solution, can be concentrated by removal of water without loss of the valuable ^{36}Cl radionuclide. This procedure consists simply in controlled dehydration, in a closed system, of the $H^{36}Cl$ solution through preferential removal of water vapor from the solution by a pool of concentrated H_2SO_4. Solutions that were 0.1 to 0.5 *N* in $H^{36}Cl$ were concentrated to 1 to 5 *N* in four days with a loss from the $H^{36}Cl$ of <1% of the ^{36}Cl originally present.

2.6 AIR QUALITY SURVEY

M. R. Guerin W. D. Shults

At the request of the Melton Hill Regional Industrial Development Association, the levels of potential air pollutants were surveyed at Eagle Bend Industrial Park, Clinton, Tennessee. Airborne sulfur, nitrogen dioxide, halogens, and reducible metals were collected by drawing air through gas-dispersion bottles filled with solutions of sodium hydroxide. Particulate matter was collected by drawing air through 0.45-μ Millipore filters. Air-sampling rates were controlled with needle valves on the vacuum pumps used to sample the air and were measured with calibrated flowmeters. Collection times varied from 24 hr to 5 days over a period of about 30 days. Airborne low-molecular-weight organic compounds were collected by opening a previously evacuated 40-liter bomb sampler at the sampling site.

Several capabilities within the Division – including service analysis, mass spectroscopy, and gas chromatography – were applied to the determinations of the

compounds of interest. Table 2.2 summarizes the results.

Table 2.2. Levels of Air Contaminants at the Eagle Bend Industrial Park, Clinton, Tennessee

Contaminant	Level ($\mu g/m^3$)
Sulfur	30
Nitrogen dioxide	<17
Fluoride	Undetectable
Halide other than F^-	Undetectable
Metals (Cd)	3
Total particulates	10
Total hydrocarbons	(0.10 ppm v/v)

It is concluded that the quality of the air at the Eagle Bend Industrial Park is far superior to that of typical urban areas and, in many respects, is superior to that of most nonurban areas for which comparable information is available. The detection of measurable amounts of what seems to be cadmium is consistent with reports that a nearby industry makes heavy use of cadmium alloys. The operation of the Bull Run Steam Plant does not appear to contribute significantly to ground-level sulfur.

2.7 RESEARCH AND SUPPORT ACTIVITIES IN THE TRANSURANIUM RESEARCH LABORATORY

2.7.a Controlled-Potential Coulometric Determination of Americium

J. R. Stokely W. D. Shults

Further work on the controlled-potential coulometric determination of americium[22] comprised a study of precision, accuracy, and several possible interferences. The work is now complete. An article on it has been submitted for publication;[23] the abstract follows:

"Controlled-potential coulometry has been studied as a means for the precise and accurate determination of americium. Americium is chemically oxidized to the hexavalent state, and then coulometrically reduced from the hexa- to the pentavalent state at a platinum electrode controlled at 1.05 V *vs.* S.C.E. The results of repetitive analysis of standard solutions prepared from high-purity americium dioxide agree within a few tenths percent with standard values for 0.4 to 1.0 mg of americium."

[22] J. R. Stokely and W. D. Shults, "Controlled-Potential Coulometric Determination of Americium," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967,* ORNL-4196, p. 89.

[23] J. R. Stokely and W. D. Shults, "Controlled-Potential Coulometric Determination of Americium," submitted for publication in *Analytica Chimica Acta.*

Work is under way to redetermine the half-lives of ^{241}Am and ^{243}Am by measurement of absolute alpha radioactivities and coulometric determinations of masses.

2.7.b Titrimetric Determination of Americium

J. R. Stokely W. D. Shults

We investigated the possibility of determining americium by redox titrimetry. Direct titration of Am(VI) with Fe^{2+} proved unsatisfactory, because the reaction is sluggish in the end-point region. Attempts to accelerate the reaction by heating and by titrating in strong-acid medium were unsuccessful. Similar behavior was observed with several types of indicator-electrode systems. An indirect procedure that involves adding excess Fe^{2+} to reduce the Am(VI) and then titrating the excess Fe^{2+} with a standard solution of dichromate was also studied. This approach gave analytical results that were erratic and inaccurate by several percent. We believe that the limitation common to both these procedures is that it is impossible to prepare Am(VI) quantitatively and free of excess oxidant. Accordingly, the procedures do not appear to have promise, and this work has been terminated.

2.7.c Micro Titration of Plutonium

J. R. Stokely W. D. Shults

Work is under way to extend the titrimetric method of Drummond and Grant[24] to the determination of semimicro and micro amounts of plutonium. The method consists in chemical oxidation of Pu(III) to Pu(VI), reduction of Pu(VI) to Pu(IV) with an excess of Fe^{2+}, and titration of the excess Fe^{2+} with a standard solution of dichromate. The purpose of this work is to offer an accurate and precise alternate to the presently used coulometric method in which iron and large quantities of sulfate interfere. The method appears to have potential usefulness.

2.7.d Voltammetric Studies of Plutonium, Neptunium, and Americium

J. C. Guyon[18] W. D. Shults

We have initiated a program to study the voltammetric behavior of the lighter transuranium elements.

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

We tested a rotating (600 rpm) pyrolytic-graphite electrode and a rotating platinum-disk electrode for this work and found the platinum electrode to be more satisfactory. The voltammetric behavior of plutonium in mineral-acid media has been examined in some detail, and that of neptunium has been examined in an exploratory manner. Our results indicate that voltammetry may be useful for rapid ionic determinations of these ions in various oxidation states and for studying the effects of various reagents and complexing agents on these ions. These studies have revealed possible ways to eliminate the interferences of iron and Pu(VI) in the controlled-potential coulometric procedure for determining plutonium; these means are being pursued. We have also examined briefly the voltammetric behavior of Am(III) but did not obtain promising results with that ion.

2.7.e Modification and Installation of Perkin-Elmer Model 240 Elemental Analyzer in a Glove Box

J. R. Stokely

Several modifications were made on the Perkin-Elmer model 240 Elemental Analyzer to permit analysis of samples inside a glove box. The major alterations were to place the combustion furnace inside the box and to extend the gas lines outside to the Analyzer. The purpose of the alterations was to prevent contamination of the electronics and gas-analysis sections of the Analyzer. Since water is a product of combustion, it was necessary to heat the exit gas line to 300 to 350°C to prevent condensation and adsorption of water vapor in the line.

Standard acetanilide from the National Bureau of Standards was used to check the calibration of the Analyzer. The calibration factors, which relate recorder readout from thermal-conductivity detectors to weight of carbon, hydrogen, and nitrogen in the sample, were the same as values obtained before modifications were made. The consistency indicates that the instrument is operating properly. In addition, several pure organic compounds were analyzed, and the results obtained were within the Perkin-Elmer specifications. Several highly radioactive samples have been analyzed, and no radioactivity has been transferred out of the glove box through the gas lines. Liquids that have low vapor pressures can be analyzed for carbon and hydrogen with the instrument by altering the automatic combustion cycle.

2.7.f Separation of Californium

J. R. Stokely

To assist members of the Chemistry Division in determining the (n,γ) cross section of ^{252}Cf, californium was separated chemically from fission products and actinides. The separation consisted in a lanthanum fluoride precipitation, separation of the actinides from the lanthanides on a cation-exchange column with 12.5 *M* HCl–20% ethyl alcohol as the eluting solvent, and finally the separation of californium from einsteinium on a small "but" column. The purified californium fraction contained <1% of the original einsteinium and ~80% of the californium. Other details of this work are presented in a recent report.[25]

2.7.g Preparation and Analysis of the β-Diketones of the Lanthanides and Actinides

J. R. Stokely

Assistance was given in the preparation of the americium and the europium chelates of hexafluoroacetylacetone and 2,2,6,6-tetramethyl-3,5-heptanedione. The compounds were analyzed for americium, carbon, hydrogen, nitrogen, and water. One of the compounds is reasonably volatile and probably can be used for vapor-pressure and gas-chromatographic studies. Details of this work are being published.[26]

2.7.h Purification of ^{241}Am and ^{243}Am

J. R. Stokely

Several batches of ^{241}Am and ^{243}Am were purified to meet a need for high-purity americium in the analytical chemistry and other research programs at the Transuranium Research Laboratory. Because of the radiation hazard, the sizes of the batches had to be limited to 300 to 500 mg. The purification method consisted in three precipitations of potassium americium(V) carbonate, elution of Am(III) from an anion-exchange column

[25] C. E. Bemis, J. Halperin, and J. R. Stokely, "The Neutron Capture Thermal Cross Section and Resonance Integral of ^{252}Cf," *Chem. Div. Ann. Progr. Rept. May 20, 1968,* ORNL-4306, p. 22.

[26] M. D. Danford, J. H. Burns, O. L. Keller, Jr., J. R. Stokely, and W. H. Baldwin, "Preparation and Volatilities of Some Rare-Earth and Americium Chelates," submitted for publication in the *Journal of Physical Chemistry.*

with 8 *N* HCl, several precipitations of americium hydroxide with ammonium gas, and a final precipitation of americium oxalate from weak acid solution. The oxalate was converted to the dioxide by ignition in air at 900°C. Emission spectrographic and radiochemical analyses revealed no detectable impurities except that one batch contained ~1 wt % calcium.

2.7.i Spectrophotometric Determination of Plutonium

J. C. Guyon[18] J. R. Stokely W. D. Shults

The work on the spectrophotometric determinations of plutonium was reported previously[27] and now has been published.[28]

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

Helen P. Raaen

2.8.a Evaluation of Vertical-Orifice Rapid Teflon D.M.E.'s for Obtaining Fundamental Polarographic Data

An article[29] on the evaluation of vertical-orifice Teflon D.M.E.'s was accepted by *Chemical Instrumentation* for publication. The summary from the article follows:

"A vertical-orifice dropping-mercury electrode (dme) of polytetrafluoroethylene (Teflon, Du Pont trade-mark) was fabricated and evaluated for rapid polarography in glass-corroding media. The orifice is in a vertical face of the Teflon segment of the dme capillary, which is positioned vertically. Capillaries of orifice diameter from about 70 to 120 μ were made. The geometric and performance characteristics of the dme were studied. Polarograms of Tl^+, Cd^{2+}, and UO_2^{2+} were recorded; the supporting media were aqueous hydrofluoric acid or acid chloride solutions. The polarographic characteristics of the reference reaction $Tl^+ \rightleftharpoons Tl^0$ in 0.1 *M* KCl–1m*M* HCl were determined to permit comparison of this dme with horizontal-orifice glass and Teflon dme's, for which similar data have been published. Results obtained with polyacrylamide present as a maximum suppressor agree with the known polarographic characteristics of the $Tl^+ \rightleftharpoons Tl^0$ reaction. They thus show that the vertical-orifice dme of Teflon is satisfactory for the rapid polarography of glass-corroding media."

2.8.b Rapid Polarography of Uranium in 1 *M* Hydrofluoric Acid with a Vertical-Orifice Telfon Dropping-Mercury Electrode

An article[30] that describes the rapid polarography of uranium in 1 *M* HF with a vertical-orifice Teflon D.M.E. is to be submitted for publication; the abstract follows:

"The polarographic characteristics of uranium, as $UO_2(NO_3)_2$, in 1*M* HF were studied with a rapid, vertical-orifice dropping-mercury electrode (dme) of polytetrafluoroethylene (Teflon, du Pont trade-name). The study shows that this type of dme is satisfactory for rapid polarography in glass-corroding media. Within the span of potential from +0.6 to −1.5 V *vs.* a saturated calomel electrode (sce), two polarographic waves were observed. At $E_{1/2} \cong -0.55$ V *vs.* sce a reproducible wave of excellent form exists, for which the relations of C to $\bar{i}_d$ and of C to $(d\bar{i}/dt)_{max}$ are linear and the plots pass through the origins. The wave is therefore both qualitatively and quantitatively useful. Dissymmetry in the first-derivative peak of the wave suggests that the reduction is irreversible. A second polarographic wave appears at $E_{1/2} \cong$ +0.3 V *vs.* sce. This wave is reproducible and its height is a function of uranium concentration, although the C *vs.* $\bar{i}_d$ relation appears not to be linear. The first-derivative peak of the wave is unsymmetrical. The wave is easily distinguished from the mercury-dissolution wave. It is relatively large – possibly catalytic – and may be the basis for a highly sensitive polarographic method for uranium."

2.8.c Polarography of Sulfur Dioxide (Sulfite) in Aqueous Hydrofluoric Acid

The following is the summary from an article accepted for publication:[31]

"A well-formed polarographic wave exists for sulfur dioxide (sulfite) in aqueous hydrofluoric acid at a half-wave potential ($E_{1/2}$) of about −0.4 V *vs.* a saturated calomel electrode (S.C.E.). The characteristics of the wave are similar to those of the wave for sulfite in aqueous solutions of hydrochloric acid and of nitric acid. The relation of sulfite concentration (C) to diffusion current (i_d) is linear, and the wave is reproducible. Polarography in aqueous hydrofluoric acid media is indicated to be a suitable method for the detection and direct quantitative measurement of sulfur dioxide. Possible significant applications of the method are to determine contaminative sulfur dioxide in hydrogen fluoride gas, in reagent-grade hydrofluoric acid and fluorosulfuric acid, and in the atmosphere."

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[27] J. C. Guyon and W. D. Shults, "Spectrophotometric Determination of Plutonium," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967*, ORNL-4196, p. 40.

[28] J. C. Guyon, J. R. Stokely, 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-2108 (Jan. 2, 1968).

[29] H. P. Raaen, "Vertical-Orifice Dropping-Mercury Electrode of Polytetrafluoroethylene for Rapid Polarography in Glass-Corroding Media," to be published in *Chemical Instrumentation*.

[30] H. P. Raaen, "Rapid Polarography of Uranium in 1*M* Hydrofluoric Acid with a Vertical-Orifice Polytetrafluoroethylene Dropping-Mercury Electrode," to be submitted for publication.

[31] Helen P. Raaen, "Polarography of Sulfur Dioxide (Sulfite) in Aqueous Hydrofluoric Acid," accepted for publication in *Analytica Chimica Acta*.

2.9 EVALUATION OF THE PERFORMANCE OF ION-SELECTIVE ELECTRODES

W. D. Shults

We have evaluated the performance of the Orion sulfide electrode. This is a solid-membrane type of electrode that is advertised to measure sulfide concentrations as small as 10^{-17} *M* with no interferences. The electrode does respond to very low concentrations of sulfide, but the *total sulfur* concentration must not be $<10^{-6}$ *M* for satisfactory response. For several reasons, use of this electrode is much more complicated than is the use of the other ion-selective electrodes we have studied.[32] First, there is no completely satisfactory standard material for sulfide; hence, one is forced to "standardize" the standard solution. This standardization is a nuisance, especially because standard sulfide solutions are not stable. Second, concentrations of sulfide in solution depend very much on pH – H_2S, HS^-, and S^{2-} are in mutual equilibria – and therefore the acidity must be either controlled very carefully or measured. At some pH's, the sulfide concentration may be extremely low and yet total sulfur concentration may be very large. This condition may lead to gross inaccuracies in determining total sulfur, since one measures sulfide and computes total sulfur from the sulfide measurement for the pH that prevails. Third, calibration curves may exhibit marked curvature. Because of these difficulties, we prefer to use the sulfide electrode as an indicator electrode for titrations rather than as a device for direct potentiometry. In the indicator function, the electrode performs beautifully. When sulfide is titrated with silver, potential changes that are ~1 V in magnitude occur near the end point.

[32]W. D. Shults, A. F. Roemer, Jr., and S. S. Potterton, "Evaluation of the Performance of Ion-Selective Electrodes," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967*, ORNL-4196, p. 36.

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 by Hydrofluorination

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

Because the earlier analyses by the hydrofluorination method[1] had established that a low and essentially constant concentration of oxide could be maintained in the MSRE fuel, the determination of oxide in reactor salt samples was assigned a lower priority during this period of operation. Table 3.1 compares the results of the two oxide analyses performed on the fuel with the result for an earlier sample (FP-7-9) whose analysis was deliberately delayed to accentuate the effects of radiation and with the average of the results of all fuel analyses since the reactor achieved power operation.

Whereas the absolute standard deviation (±8 ppm) of the sampling-analysis procedure is satisfactory, the

Table 3.1. Oxide Concentrations of MSRE Fuel Salt

Sample Designation	Date	Special Treatment	Oxide Concentration (ppm)
FP-14-39	12-2-68	None	46
FP-14-53	2-7-68	Heated carrier	58
FP-7-9	7-4-66	Stored 24 hr at cell temperature	50
Average of all samples (15) analyzed since reactor start-up			54 ± 8

[1]R. F. Apple and J. M. Dale, "Determination of Oxide in MSRE Salts," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967*, ORNL-4196, p. 15.

accuracy of the method for radioactive samples is difficult to establish. A possible source of error is the loss of oxygen because of radiolysis during the ~8-hr interval between sampling and analysis; it is known that fluorine is generated radiolytically during this period. Sample FP-14-53 was transported in a carrier that maintains the sample at a temperature above 500°F to eliminate the radiolytic generation of fluorine. Because of a crowded sampling schedule, it was not possible to analyze many samples transported in the heated carrier before the scheduled reactor shutdown. The facts that minimization of the effects of radiation on the sample caused no significant change in the results and that the overall reproducibility is good strongly suggest that the radioactivity of the samples introduces no determinate error in the method.

When the reactor was not in operation, the hydrofluorinator was used to analyze a number of radioactive and nonradioactive samples. Solvent salt ($LiF\text{-}BeF_2$) samples for the In-Pile Test Loop were analyzed to determine the source of oxide contamination. The results are:

Sample	Oxide Concentration (ppm)
1	42
2	375
3	62
4	60
5	62

Sample 1 was taken from the original salt preparation. Sample 2 was exposed in the molten state for 96 hr to the atmosphere of a helium-filled dry box. Samples 3 through 5 were purged, in series, with helium for 48 hr.

The method for oxide was also adapted to smaller samples taken to determine the completeness of removal of oxide from fuel concentrates ($LiF\text{-}^{233}UF_4$) by batch hydrofluorination. Table 3.2 gives the results.

The first five analyses were run to establish optimum sampling techniques; the samples came from a test preparation of concentrate prepared from normal uranium. This development work was performed with G. I. Cathers.[2] Initial samples were taken in a special sampling system designed to withdraw a filtered sample in a small copper ladle. To ensure that $HF\text{-}H_2$ mixture bubbled through the molten sample, it was necessary to either transfer the sample to a standard ladle or couple a copper sleeve to the ladle. Larger and more representative samples were provided by drawing the molten salt into a ½-in.-OD Inconel tube. The melt was cooled, and 1-in. sections were cut from the salt-filled tube and taken for analysis. These sections were placed in standard ladles that were prefitted with a welded delivery tube which extended to the bottom of the ladle. After the usual preliminary hydrofluorination at 300°C, the sample was melted and allowed to flow to the bottom of the ladle for efficient purging. The results confirm the effectiveness of the preliminary hydrofluorination step to remove surface contamination that results from atmospheric exposure.

Table 3.2. Oxide Concentrations of Samples from Test Runs and from ^{233}U Fuel Concentrates

Sample Designation	Sampler	Sample Weight (g)	Oxide Concentration (ppm)
$LiF\text{-}UF_4$	Pellet	3.5[a]	1000
25A	Copper-sleeve	3.5[a]	85
40	Copper-brass funnel	3.5[a]	105
43	Inconel	12.1[b]	107
44	Inconel	12.1[b]	117
RU 33-1-S-2 Fuel	Inconel	12.1[b]	62
RU 33-2-S-1 Fuel	Inconel	12.1[b]	33
RU 33-2-S-2 Fuel	Inconel	12.1[b]	37
RU 33-3-S-1 Fuel	Inconel	12.1[b]	31
RU 33-3-S-2 Fuel	Inconel	12.1[b]	32

[a]Estimated.
[b]Calculated.

The evaluation of the effects of radiation on the components of the hydrofluorinator is of considerable interest, because it is planned to adapt the technique to the in-line analysis of radioactive salt streams. In general, the experience has been satisfactory. In this third year of operation, only three repairs of in-cell components were needed. Two of the failures – leaks in the remote coupler resulting from the corrosion of nickel ball joint by fumes from an adjacent cell and an open circuit in the valve-compartment heater system – do not appear to be associated with radiation damage. The cause of the desensitization of the electrolytic moisture-monitor cell, the third since the apparatus was installed, is subject to question. The first and last (present) of these cells, which were used primarily for the analysis of samples of relatively low radioactivity, survived at least 20 analyses and eight-month service life. Conversely, the other cells, which were used for samples taken during extended power operation, failed after four to ten analyses and two- to four-month usage.

All failures have been of the same general type. Plastic components exhibited some evidence of noncritical radiation damage, and the P_2O_5 electrolyte was re-

[2]Chemical Technology Division.

moved from the electrodes. In an attempt to reproduce these failures under controlled conditions, a moisture-monitor cell was operated in the vertical position at 65°C. After the repeated analysis of a series of relatively large SnO_2 standards had failed to reduce the cell's efficiency, the cell was removed and subjected to $\sim 10^7$ rads of ^{60}Co radiation. The irradiated cell continued to function for several months and finally was desensitized after the passage of a total of about 1.5 g of water. This treatment represents an exposure to both radiation and water that is at least an order of magnitude greater than that which produced in-cell failure. Although the in-cell failures may have been due to the characteristics of the individual moisture monitors, it is desirable to investigate other techniques for the measurement of water for application to in-line oxide analysis of molten-salt reactor fuels.

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

J. M. Dale R. F. Apple
A. S. Meyer J. E. Caton
C. M. Boyd

The theory of the hydrogen-reduction method for U(III) in radioactive MSRE fuel and the initial evaluation of the experimental results were described previously.[3] It was noted at that time that the results of the U^{3+} determinations did not reflect the beryllium additions made to the MSRE fuel salt to reduce fractional percentages of the U^{4+} to U^{3+}. However, the computer program used initially to evaluate the experimental results did not take into account the change in equilibrium concentrations of the corrosion products and uranium that must occur when the fuel is cooled from reactor temperature to the temperature at which the first hydrogen-reduction step was made. As a result, the values previously reported for the U^{3+} concentrations are those for 500°C, the temperature of the first reduction step, and not for 663°C, the temperature of the fuel in the reactor.

The computer program has since been modified, and the experimental results were reevaluated. The new ratios of U^{3+} to total U are plotted in Fig. 3.1 and are compared with the calculated ratios that would be expected to exist by virtue of the beryllium additions and the fuel burnup. After the first sample was run, a

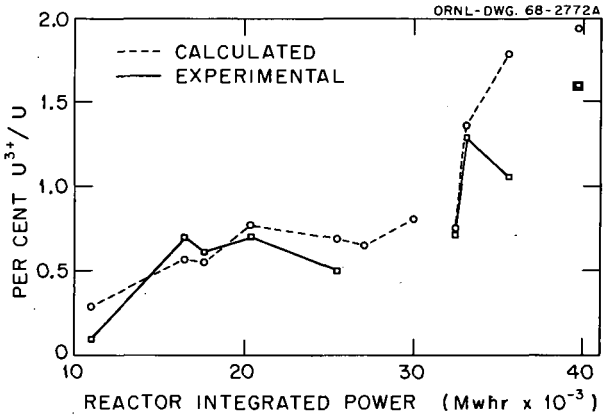

Fig. 3.1. Concentration of U^{3+} in MSRE Fuel Salt.

rod of beryllium metal was lowered into the fuel in the reactor. The beryllium is oxidized by the fuel; this reaction results in an increase in the U^{3+} concentrations. All increases in the calculated U^{3+} concentrations are a result of beryllium additions, whereas the decreases result from fuel burnup. The analysis of the sixth and seventh samples gave HF yields in the hundreds of micromoles, which do not correlate with U^{3+} concentrations. At this point, difficulty was experienced with the sampler station, and the reactor was shut down for about a month and a half for maintenance. The first two U^{3+} results determined after the reactor was brought back to power were close to the calculated values, and the result for the third sample was low. At this point, the reactor was down again for a short time. The next U^{3+} results were increased but were still lower than the estimate. One explanation for the low U^{3+} results is that they are caused by the radiolytic production of fluorine. This hypothesis was discussed previously.[4] To eliminate this source of error, a heated sample carrier was made to transport the fuel samples from the reactor to the hot-cell facilities (Sect. 3.1.a). Due to sampling problems at the reactor, only one U^{3+} sample so transported was taken before the final reactor shutdown. The U^{3+} result for this sample was low compared with the calculated value. On the basis of only one analysis, no conclusions can be drawn about the importance of using the heated transport container.

Work was continued to develop a method for the remote measurement of ppm concentrations of HF in helium and in hydrogen gas streams.[5] The construction

[3] J. M. Dale, R. F. Apple, and A. S. Meyer, "Determination of Uranium(III) in Radioactive MSRE Fuel by a Hydrogen-Reduction Method," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967*, ORNL-4196, p. 16.

[4] *Ibid.*, p. 19.

[5] *Ibid.*, p. 20.

and calibration of the dual trapping system for the continuous absorption and desorption of HF on sodium fluoride were completed. The trapping system consists of a dual selector valve, a special monel thermal-conductivity (TC) cell (Gow Mac model 9454) with nickel filaments and two sodium fluoride traps. The valve and TC cell are contained in a Boekel model 1078 laboratory oven at 50°C and are connected to the sodium fluoride traps, which are mounted on the outside walls. This arrangement permits the simultaneous absorption of HF on one trap at room temperature and the desorption of HF from the alternate trap at 300°C by means of self-resistance heating. Figure 3.2 shows a curve typical of the response of the TC cell to the desorption of HF from a sodium fluoride trap. The initial smaller peak is due to a small amount of water impurity and is almost completely resolved from the HF peak. As shown in Fig. 3.3, integration of the TC-cell response vs the number of micromoles of HF desorbed from the traps is linear over the range of interest (1 to 50 micromoles of HF). This range corresponds to an HF concentration of about 8 to 400 ppm at a carrier-gas flow of 100 cc/min and an absorption period of 30 min.

When this trapping system for the U^{3+} determinations is installed in the hot-cell facilities, it will be necessary to have some means to check periodically for any deterioration in the TC-cell response. We have found that the sampling of a helium gas stream will serve for this purpose. At a cell current of 270 mA and a flush-gas flow of 100 cc/min, the agreement of the cell responses for repetitive samplings of a pure helium gas stream was better than 0.1%. A 10% change in the flush-gas flow causes a change in the cell response of only 1%.

Provisions are now being made to use the trapping system for the analysis of the HF evolved from the hydrogen reduction of standard fuel-salt mixtures. This analysis should provide a check of the validity of the hydrogen-reduction method and reveal any of its limitations.

3.1.c Determination of Total Reducing Power of Radioactive MSRE Salts

R. F. Apple A. S. Meyer

A method is required to determine whether active colloidal metals, particularly excess zirconium, are present in reprocessed MSRE fuel solvent. The estab-

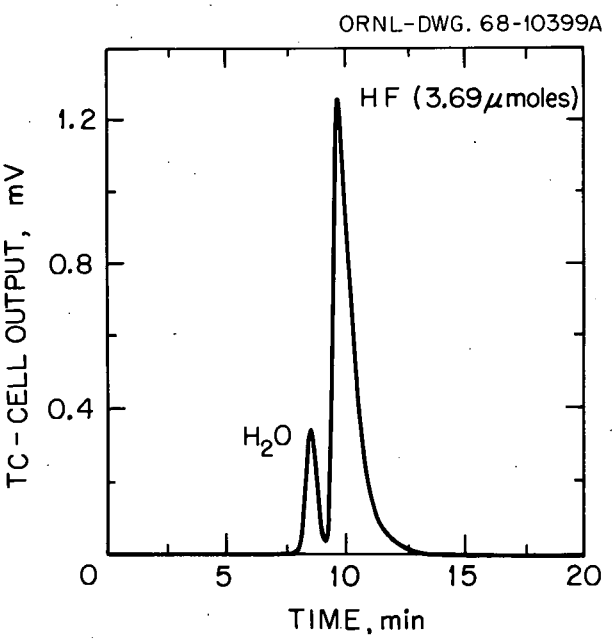

Fig. 3.2. Thermal-Conductivity-Cell Response Curve for the Desorption of HF from a Sodium Fluoride Trap.

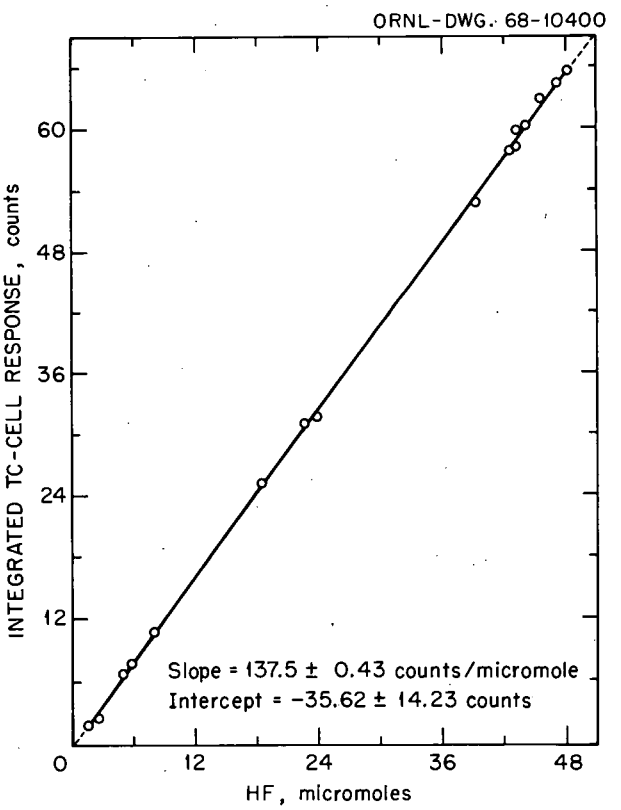

Fig. 3.3. Thermal-Conductivity-Cell Response vs HF Desorbed from Sodium Fluoride.

lished hydrogen-evolution method[6] is not applicable to radioactive samples, because a finely powdered sample is required. No facilities are available to pulverize radioactive samples under a controlled atmosphere. A modification of this technique is being developed in which an unpulverized solid sample is dissolved in a sealed tube in an HCl-HBO_3 mixture. We have demonstrated that 250-mg fragments of fuel, solvent, and fuel concentrate are completely disintegrated by a 24-hr treatment at 250°C. The sealed tube is protected by an overpressure of CO_2 during the dissolution. In the hot-cell determination, the evolved hydrogen will be mixed with a standard addition of helium, and the H_2/He ratio will be determined mass spectrographically. To simulate the hot-cell operation, samples were added to a tube that was fitted with a breakseal and that contained a thin-walled ampoule of deaerated acid. The tube was evacuated and sealed off by using a small (~300-W) platinum-wound furnace. The acid-containing ampoule was then broken by shaking. The dissolution was completed successfully. Since all these operations appear to be feasible by remote manipulation, an apparatus is being fabricated to perform the dissolutions and standard additions of helium within the hot cell.

3.1.d Analysis of MSRE Helium for Hydrocarbons

C. M. Boyd

A system to measure continuously the total hydrocarbon concentration in the radioactive off-gas of the MSRE has been installed in the gas-sampling station. In this system (Fig. 3.4), the reactor off-gas is passed through a combustion tube that contains CuO maintained at 700°C. This combustion converts hydrocarbons in the helium to CO_2 and H_2O. The oxidized gas stream is passed through one side of a thermal-conductivity (TC) cell, through a 3A molecular sieve–Ascarite–$Mg(ClO_4)_2$ trap, which removes the CO_2 and H_2O, and back through the reference side of the TC cell. The difference between the thermal conductivity of the gas in the two sides of the cell is a measure of the hydrocarbon concentration in the off-gas; the detection limit is ~10 ppm. A length of brass tubing is installed upstream of the reference side of the TC cell. After the gas stream is switched by means of valves to bypass the trap, a step response in the TC signal is obtained. This plateau represents the period required to equilibrate the oxidized surface of the brass tubing with moisture (Fig. 3.5). By use of standard gas mixtures, it was demonstrated in a laboratory system that the relative step heights are proportional (within ±10%) to the ratio of CO_2 to H_2O. Therefore, an estimate of the ratio of carbon to hydrogen in the reactor off-gas can be made from such measurements.

The initial tests of the system on MSRE off-gas have shown that the fission-product radioactivity in this stream causes serious interference. This interference results primarily from the radiolysis of traces of absorbed water in the trap rather than from the radiolysis of the reagents themselves and indicates an impurity level of the order of 100 ppm. This level increased only slightly on oxidation, indicating only about 10 ppm of combustibles.

Tests have been made on the materials contained in the trap to determine the effect of radiation. Samples (5-g) were sealed in 20-cc glass vials filled with helium at 50-mm pressure and were subjected to 2×10^7 rads

[6]W. J. Ross and J. C. White, "Uranium Trifluoride in the Presence of Uranium Tetrafluoride, Hydrogen Evolution Method," Method No. 9 00720003 (3-28-56), *ORNL Master Analytical Manual,* TID-7015, sect. 9.

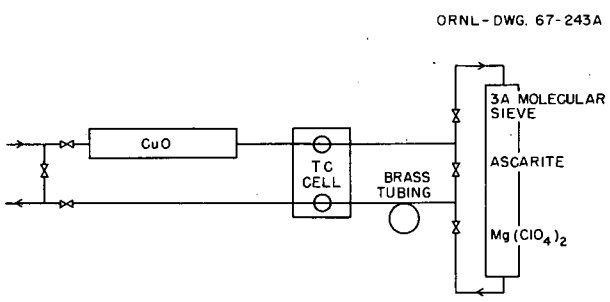

Fig. 3.4. Flow Diagram of Hydrocarbon Analyzer for MSRE Off-Gas.

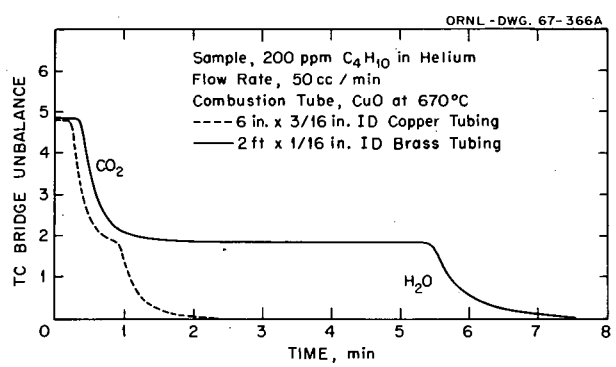

Fig. 3.5. Results of the Determination of Hydrocarbons in Helium by Measuring the Thermal Conductivity of Their Combustion Products.

of ^{60}Co radiation. Mass spectrometry showed that hydrogen is the major radiolysis product from Ascarite, whereas oxygen was produced from both wet and dry samples of $Mg(ClO_4)_2$. Wet molecular sieve gave some H_2, N_2, CO, and O_2. Dry molecular sieve produced very little contaminant gases on irradiation. The large trap that contained the mixture of absorbing materials is being replaced with a small trap that contains only molecular sieve. A heater on this trap will keep it free of water and CO_2 by a bake-out between tests. The amount of contaminant gases produced by radiolysis in this small dry trap should be greatly decreased.

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

3.2.a Disproportionation of Electrochemically Generated Uranium(V) in Molten $LiF-BeF_2-ZrF_4$ at 500°C

D. L. Manning Gleb Mamantov[7]

An article was published[8] that describes our study of the disproportionation of electrochemically generated U(V) in molten $LiF-BeF_2-ZrF_4$ at 500°C. Certain of the results are consistent with theoretical predictions of Fischer and Drǎcka.[9] The summary from the article follows:

"Electrochemical oxidation of U(IV) has been studied in molten $LiF-BeF_2-ZrF_4$ (65.6-29.4-5.0 mole %) at 500° by means of voltammetry (scan rate, ν, 0.01–100 V/sec), chronopotentiometry, and chronoamperometry at unsheathed pyrolytic graphite and Pt–10% Rh electrodes. The following results point to the disproportionation of the electrochemically-produced U(V): (a) no voltammetric currents owing to the re-reduction of the product at $\nu \ll 1$ V/sec, the peak currents (i_p) for the oxidation of U(IV) and re-reduction of the product being approximately equal at $\nu \gg 1$ V/sec; (b) peak currents for the reduction of U(IV) to U(III) and the oxidation of U(IV) are the same at large scan rates; (c) the observed increase in $i_p/\nu^{1/2}$ vs. ν at $\nu < 1$ V/sec; (d) the observed variation of $i_0\tau^{1/2}$ with $i_0^{2/3}$ is in agreement with theoretical predictions of Fischer and Dracka[8]. At Pt–10% Rh electrodes, evidence for the adsorption of U(IV) has been obtained. The reproducibility of voltammograms and chronopotentiograms for the oxidation process is poor, presumably owing to attack of the working electrode by U(VI) produced in the disproportionation."

3.2.b Voltammetry of Chromium(II) in Molten $LiF-BeF_2-ZrF_4$ at 500°C

D. L. Manning J. M. Dale

The electrochemical reduction of Cr(II) in molten $LiF-BeF_2-ZrF_4$ (65.6-29.4-5.0 mole %) was studied by voltammetry with linearly varying potential. Well-defined voltammograms were obtained with pyrolytic-graphite and glassy-carbon indicator electrodes at a potential of about −0.9 V vs a Ni/NiF_2 reference electrode.[10] Linear plots of peak current (i_p) vs concentration were obtained over the concentration range studied, ~0.008 to ~0.03 *F* Cr(II). Linear plots of i_p vs (scan rate)$^{1/2}$ obeyed the Randles-Sevcik equation from about 0.02 to 1 V/sec. The diffusion coefficient calculated from the slope of the line is of the order of 1×10^{-6} cm^2/sec at 500°C. In the presence of U(IV), the chromium wave is enhanced, because the Cr(II) → Cr(0) reduction occurs at the foot of the U(IV) → U(III) reduction wave.

In cooperation with T. R. Mueller,[11] a first-derivative circuit was assembled and added to the controlled-potential controlled-current cyclic voltammeter. Use of the derivative circuit gave much better resolution of the chromium wave in the presence of U(IV); however, the first-derivative peak current for the Cr(II) → Cr(0) reduction was still enhanced by U(IV).

From the results obtained thus far, it is believed that the Cr(II) → Cr(0) reduction wave can be used analytically to monitor Cr(II) in fluoride melts; in the presence of U(IV), it could be useful on an empirical basis.

3.2.c Electromotive Force (EMF) Measurements in Molten Fluorides

H. W. Jenkins[12] D. L. Manning Gleb Mamantov[7]

The program of emf measurements in molten fluorides[10] was continued; Table 3.3 summarizes the

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

[8]D. L. Manning and G. Mamantov, "Disproportionation of Electrochemically-Generated Uranium(V) in Molten $LiF-BeF_2-ZrF_4$ at 500°," *J. Electroanal. Chem.* 18, 137 (1968).

[9]O. Fischer and O. Drǎcka, "Studium der Reaktionskinetik von Elektrodenvorgängen mit hilfe der Elektrolyse bei Konstantem Strom I. Studium der Dismutation der Ionen des Fünfwertigen Urans," *Collection Czech. Chem. Commun.* 24, 3046 (1959).

[10]H. W. Jenkins, D. L. Manning, and G. Mamantov, "Electromotive Force (EMF) Measurements on the Nickel-Nickel(II) Couple in Molten Fluorides," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967,* ORNL-4196, p. 23.

[11]Analytical Instrumentation Group.

[12]ORAU Graduate Fellow from the Department of Chemistry, University of Tennessee, Knoxville.

Table 3.3. Electrode Potentials in Molten Fluorides at 500°C

Couple	Emf (V)[a] Measured in $LiF-BeF_2-ZrF_4$ (65.6-29.4-5.0 mole %)	Emf (V)[a] Calculated[b] in $LiF-BeF_2$ (66-34 mole %)[c]	Standard State
Be/Be(II)	−2.120	−2.168	$LiF-BeF_2$ (66-34 mole %)
Zr/Zr(IV)	−1.742	−1.772	1 Mole fraction
Cr/Cr(II)	−0.754	−0.789	1 Mole fraction
Fe/Fe(II)	−0.408	−0.413	1 Mole fraction
Ni/Ni(II)	0	0	1 Mole fraction

[a]Values vs Ni/Ni(II) reference electrode.
[b]Private communication with H. R. Bronstein, Chemistry Division.
[c]Values converted to 500°C.

electrode potentials measured thus far. From a comparison of the emf's in Table 3.3, it is believed that the agreement between the measured values and those calculated by Baes[13] is reasonably good, even though the solvent systems were not the same.

The Ni/NiF_2 reference electrode consists of a thin-walled ($^1/_{32}$-in.-thick) boron nitride (BN) compartment that contains the fluoride melt, NiF_2 in a fixed concentration, and a nickel electrode. Electrical contact is made when the BN becomes wetted by the melt. Although the majority of the BN sheaths used thus far have been satisfactory, in a few cases the melt extensively penetrated the sheath. The reason for this behavior is not yet understood. The extent of penetration appears to be related to the free oxide content of the fluoride melts as well as to the previous history of the BN (e.g., grade and amount of exposure to atmospheric moisture during machining). Therefore, it was decided to seek other ways to separate the half-cells.

H. R. Bronstein[14] successfully used tantalum sheathes with tantalum frits for half-cell separation in chloride melts for several hours without significant diffusion. It seemed worthwhile to investigate this approach for fluoride melts. C. J. King[15] fabricated for us copper frits (~$^3/_8$-in.-OD by $^1/_4$-in. thickness) with a pore radius of ~1 μ and a void fraction of ~0.30 (comparable with Bronstein's tantalum frits). The frits will be fitted into copper tubes and tested for half-cell separation along with additional BN sheathes. If the penetration problem can be overcome, an attempt will be made to obtain emf measurements on additional redox couples, including U(IV)/U(III), in molten $LiF-BeF_2-ZrF_4$.

3.2.d Facility for Spectrophotometry of Radioactive Samples

J. P. Young

The development of a spectrophotometric facility for hot-cell use has continued.[16] The scope of the project has broadened somewhat. The facility will make use of an extended double-beam arrangement; both sample and reference beams will pass into and out of the hot cell. The conceptual optical design of the extended light beams has been completed by Cary Instruments, Monrovia, Calif., but a contract for its detail design and construction has not. The spectrophotometer itself will be outside the cell. The facility can be used with solid, liquid, or gaseous samples at temperatures up to ~800°C. As pointed out previously,[16] the facility is designed primarily for use with molten-fluoride salts and will be used to demonstrate the spectrophotometric determination of U(III) in molten-salt reactor (MSR) fuels (e.g., $LiF-BeF-ZrF_4-UF_4$), which contain U(IV). The spectrum of U(V) in molten-fluoride salts has recently been characterized (Sect. 3.2.e); it is now

[13]C. F. Baes, Jr., "The Chemistry and Thermodynamics of Molten-Salt-Reactor Fluoride Solutions," pp. 409–433 in vol. 1 of *Thermodynamics,* International Atomic Energy Agency, Vienna, 1966.

[14]Private communication with H. R. Bronstein, Chemistry Division.

[15]Oak Ridge Gaseous Diffusion Plant.

[16]J. P. Young, "Spectrophotometric Studies of Molten-Salt Systems," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967,* ORNL-4196, p. 25.

possible to identify U(V) in MSR salt if it is present in at least 1000-ppm concentration.

Equipment has been designed and techniques are being developed to take a sample of fuel from the MSRE reactor, divide it into as many as five portions, and transfer each of the portions to a spectrophotometric furnace assembly without exposing the portion to the atmosphere or to temperatures below 200°C for periods longer than 0.5 hr. Exclusion of air is necessary to prevent the reaction of U(III) with H_2O and/or O_2. Elevated temperature is required to prevent the radiolytic evolution of fluorine, which occurs if the sample is allowed to remain at temperatures below ~200°C for a long time. With evolution of fluorine, the oxidation state of uranium would most likely change on remelting the sample.

The apparatus required for this sample transfer has been designed with the assistance of F. L. Hannon[17] and E. F. Marguerat.[17] It consists of six components: (1) Sample ladle, which removes a 5-g molten-salt sample from the pump bowl of the reactor. (2) Transport container, which receives the sample ladle and maintains it under inert gas. (3) Heated transfer carrier, which receives the transport container, provides radiation shielding during transfer of the sample from reactor to the hot cell, and heats the sample to ~200°C during the transfer. (4) Sample-loading furnace, which receives the transport container in the hot cell. Within this assembly the 5-g sample is melted, and test portions are transferred into as many as five containers for spectrophotometric study. (5) Spectrophotometric furnace lid, which fits both the sample-loading and spectrophotometric furnaces; the lids receive the spectral sample for transfer between the two furnaces. (6) Spectrophotometric furnace, which receives the sample for melting and spectral study.

All the sample-handling operations must, of course, be carried out with manipulators behind suitable shielding. The apparatus is designed in such a way that the manipulations required at this reactor site fall within their routine procedure requirements. At the hot cell, the apparatus can be operated and general maintenance can be done with master-slave manipulators. All these components except the sample-loading furnace have been designed completely; components 1, 2, 5, and 6 are presently being fabricated for testing prior to hot-cell use.

The optimum orientation of apparatus within the hot cell is being determined so that all operations, sample handling, and maintenance can be carried out properly. The obvious applicability of this installation to other types of samples is also being considered in this design work.

For initial spectra analysis in this facility, the molten fluoride sample will be contained in a modified captive-liquid cell[18] adapted for remote-handling techniques. However, the system is sufficiently versatile that it will accept other types of optical cells which are now being developed to contain molten salts, for example, the diamond-windowed cell (Sect. 3.2.f).

3.2.e Spectral Studies of Uranium(V) and Uranium(VI) in Molten Li_2BeF_4

J. P. Young

The spectra of uranium in its higher oxidation states, that is, >U(IV), in molten Li_2BeF_4 have been studied further in collaboration with G. I. Cathers,[2] who supplied the solute Na_2UF_8 (UF_6 adsorbed on NaF). It was found that Na_2UF_8 undergoes a rapid and vigorous reaction when placed in molten Li_2BeF_4 that is held in a graphite windowless container. Since gas is evolved in the process, it is assumed that Na_2UF_8 oxidizes a portion of the graphite container. The spectrum of the colorless solution that results from this reaction is that of U(V); it consists of two overlapping peaks centered at 950 and 1240 nm and a sharp peak at 1465 nm. The corresponding molar absorptives (ϵ's) are ~6, 3, and 15 liters $mole^{-1}$ cm^{-1}, respectively. They were calculated by determining the decrease in absorbance of U(V) for a given increase in absorbance of U(IV). The U(V) species is not stable in molten Li_2BeF_4 at 550°C; it slowly disappears, apparently by disproportionation.

From the spectral study, the rate constant for the disproportionation of U(V) in molten Li_2BeF_4 was calculated to be 0.005 liter $mole^{-1}$ sec^{-1}, a rate that is several orders of magnitude slower than that measured in an electrochemical study of U(IV) oxidation.[8] The large discrepancy between the two values is real and can be explained if one assumes that the rate obtained in the electrochemical study is the result of catalysis at the surface of the electrode, whereas the spectrally recorded rate is the rate for a noncatalyzed reaction in bulk solution.

The spectra of solutions of Na_2UF_8 in molten Li_2BeF_4 contained in a windowless LaF_3 cell were studied; LaF_3 is slightly soluble in the solvent but

[17] General Engineering Division.

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

should not be oxidized by U(VI). No reaction was observed in this melt, unlike the case in graphite. The spectra of the resultant solutions showed strong absorption in the ultraviolet region with shoulders near 270 and 315 nm. Some contamination of the solution with U(IV) and U(V) was evident from the spectra and apparently was caused by reaction of U(VI) with impurities in the melt. It is assumed that the spectra seen were those of U(VI) in some form of UF_n^{6-n}. The absorbing species is not UO_2^{2+}, which has been observed in other studies.[16]

3.2.f Containers for Molten Fluoride Salts

J. P. Young

The use of silica containers for molten fluoride salts has been reported.[19] As pointed out, the compatibility of silica with each component of a particular solution must be considered. Such things as oxide solubility and silicate formation can influence a system that may appear to be thermodynamically stable with respect to the simple reaction

$$4F^- + SiO_2 \rightleftharpoons SiF_4 \uparrow + 2O^{2-}. \qquad (3.1)$$

For example, it has been observed that solutions of Ni(II) and of Co(II) in molten Li_2BeF_4 cannot be maintained in a silica cell. Presumably, the low solubility of NiO and of CoO results in the loss of these ions by precipitation as oxide. The O^{2-} level is maintained by the equilibrium indicated by Eq. (3.1).

In cooperation with L. M. Toth[20] and G. P. Smith,[21] a diamond-windowed spectrophotometric cell is being developed for use with molten fluoride salts. In preliminary spectral studies, the prototype cell has been entirely compatible with solutions of Pr(III) or U(IV) in molten Li_2BeF_4 maintained at temperatures as high as 550°C. Fluoride melts have not been permitted to solidify between the windows. Instead, prior to the cooling of a sample, the furnace and cell have been inverted so that the salt flows away from the windows; thus, cleaning of the cell is facilitated. The cell is constructed of a graphite body and type II A diamond windows (about 5 × 5 × 1 mm). The windows are not sealed to the cell body; rather, they are merely retained against it. This type of construction exploits the high surface tension and general nonwetting properties of many molten fluoride salts, which prevent these liquids from flowing out of small openings. Since there is no need for elaborate window seals, the design of a windowed cell can be relatively simple. Also, this arrangement decreases the probability of breaking these thin windows during an assembly operation. As presently constructed and used, the cell is not suitable for highly radioactive samples; however, cells of later designs may be.

3.2.g Absorption Spectra of Several 3d Transition-Metal Ions in Molten Fluoride Solution

J. P. Young

Experimental work has been completed on a spectral study of Fe(II), Ni(II), Cr(II), and Cr(III) in various molten fluoride salts. The results have been presented orally[22] and submitted for publication;[23] the abstract from the manuscript follows:

"The spectra of several 3d transition metal ions, Fe(II), Ni(II), Cr(II), and Cr(III) have been studied; these ions were dissolved in several molten fluoride salts which are essentially the stoichiometric mixture Li_2BeF_4 with and without excess F^-, as LiF. The spectra were obtained in the temperature range of 540°C to 650°C; the samples were contained in graphite windowless containers for the study. Fe(II) exhibits a peak and shoulder absorbance envelope with maximum absorbance at 9800 and 5500 cm^{-1}. Respective molar absorptivities are 4.5 and 3. The spectrum appears unaffected by change in solvent composition. The spectrum of Ni(II) at a temperature of 550°C consists of 3 peaks at 23,100, 10,800 and *ca* 6000 cm^{-1}. Respective molar absorptivities are 11, 2, and *ca* 1. The spectrum appears to be likewise unaffected by solvent change. At a temperature of 550°C, Cr(II) appears to exhibit one peak at 14,000 cm^{-1} with a molar absorptivity of 6; Cr(III) exhibits 3 peaks at 33,000, 22,600, and 14,500 cm^{-1}; respective molar absorptivities are *ca* 10, 10, and 7. The spectral results would suggest that these ions are situated in O_h symmetry in these melts with little tendency to change if the "free" fluoride ion concentration is reduced over the range studied. Fe(II) is a possible exception to this generality."

3.2.h Spectrum of MoF_3 in Li_2BeF_4

J. P. Young

In cooperation with L. M. Toth[20] and G. P. Smith,[21] the spectrum of MoF_3 in molten Li_2BeF_4 has been

[19] C. E. Bamberger, J. P. Young, and C. F. Baes, Jr., "Containment of Molten Fluorides in Silica; Effect of Temperature on the Spectrum of U^{4+} in Molten LiF-BeF_2 Mixtures," *J. Inorg. Nucl. Chem.* 30, 1979 (1968).

[20] Reactor Chemistry Division.

[21] Metals and Ceramics Division.

[22] J. P. Young, "Absorption Spectra of Several Transition Metal Ions in Molten Fluoride Solution," presented at the 155th National Meeting, American Chemical Society, San Francisco, Calif., Mar. 31–Apr. 5, 1968.

[23] J. P. Young, "Absorption Spectra of Several 3d Transition Metal Ions in Molten Fluoride Solution," submitted for publication in *Inorganic Chemistry*.

studied. The spectrum of Mo(III) in molten Li_2BeF_4 consists of two peaks at 353 and 472 nm; the ϵ's are ~8 and ~12 liters $mole^{-1}$ cm^{-1} respectively. This spectrum is consistent with the existence of Mo(III) in octahedral symmetry in molten Li_2BeF_4. These studies have been carried out in windowless cells of graphite, platinum, molybdenum, and copper. In all these cells, Mo(III) was lost completely. In the case of the graphite container, a thin film of molybdenum metal (~30 μ thick) was identified by electron-probe analysis. With copper containers it is possible that the Mo(III) is reduced by hydrogen that diffuses out of the copper, since all the copper containers were H_2-fired prior to use.

3.2.i Simultaneous Electrochemical and Spectrophotometric Study of Solutes in Molten Fluoride Salts

F. L. Whiting[24] J. P. Young
Gleb Mamantov[7]

The study of the electrochemical generation and spectral identification of solute species in molten fluoride salts has continued.[21] It has been apparent that melt impurities, primarily hydrolysis products, must be absent if the desired solute species is generated.

Various methods to purify LiF-NaF-KF have been attempted. These have included treatment with HF and with SiF_4 and a vacuum pretreatment at 450°C;[25] the vacuum pretreatment proved to be the most fruitul. The removal of HF from molten LiF-NaF-KF was achieved best by evacuating the melt at 860°C whereas the SiF_4 was removed at 500°C; SiF_4 is highly soluble in molten LiF-NaF-KF, and its dissolution is an endothermic process. An increase in pressure and the forms of the voltammograms of the melt indicated that HF and SiF_4 had been removed at the temperatures cited.

A platinum-rhodium (10%) electrode is apparently passivated by large additions of LiOH and/or H_2O. The passivation results in the elimination of voltammograms that occur anodically with respect to the reduction of OH^-. Thus, with platinum electrodes, it would be difficult to distinguish between a very contaminated and a clean melt.

The pyrolytic-graphite electrode is not passivated by the addition of OH^- or H_2O to molten LiF-NaF-KF. The O^{2-} wave is seen easily at +0.8 V vs a platinum quasi-reference electrode. Hydrogen is liberated from water at −0.45 V. The liberation of H_2 from HF is seen at 0 V in a heavily contaminated melt. The above-mentioned anodic and cathodic processes in molten LiF-NaF-KF correspond well with the results obtained by Pizzini and Morlotti.[26]

The reduction of Ta(V) to Ta(0) in molten LiF-NaF-KF at a platinum electrode appears to be at least a two-step process, as reported by Senderoff, Mellors, and Reinhart.[27] Our attempts to verify the reported oxidation of Ta(V) to Ta(VI) in molten LiF-NaF-KF[28] have been inconclusive.

The anodic dissolution of a copper electrode in molten LiF-NaF-KF produces oxidized copper in at least two oxidation states. An insoluble form of Cu(I) is produced at +1.1 V vs the platinum quasi-reference electrode. A soluble form of Cu(II) is believed to be produced at +1.35 V. The spectrum of Cu(II) in molten LiF-NaF-KF consists of a band at 810 nm and an unresolved charge-transfer band in the ultraviolet region. The spectrum was obtained during the electrochemical generation of Cu(II) and was checked qualitatively by observing the spectrum of molten LiF-NaF-KF to which CuF_2 had been added.

3.3 NUCLEAR SAFETY

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

C. M. Boyd W. F. Peed[29] A. S. Meyer

A General Electric Company's Monopole 600 Residual Gas Analyzer (RGA) was acquired for the in-line analysis of the gases generated by in-reactor tests of fuel elements. To adapt the RGA to the continuous monitoring of the purge gas, a small fraction of the effluent stream will be bled at 1 to 10 torr into an intermediate-pressure system located in a shielded hood at the ORR.

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[24] Student Guest from the Department of Chemistry, University of Tennessee, Knoxville.

[25] S. Pizzini, R. Morlotti, and E. Römer, "Electrode Overvoltages in Molten Fluoride – The Anodic Behavior of Nickel Electrodes in Fluoride Melts," *J. Electrochem. Soc.* **113**, 1305 (1966).

[26] S. Pizzini and R. Morlotti, "Oxygen and Hydrogen Electrodes in Molten Fluorides," *Electrochim. Acta* **10**, 1033 (1965).

[27] S. Senderoff, G. W. Mellors, and W. J. Reinhart, "The Electrodeposition of Coherent Deposits of Refractory Metals. II. The Electrode Reactions in the Deposition of Tantalum," *J. Electrochem. Soc.* **112**, 840 (1965).

[28] G. W. Mellors and S. Senderoff, "Hexavalent Fluorides of Tantalum and Niobium," *J. Electrochem. Soc.* **112**, 642 (1965).

[29] On loan from Thermonuclear Division.

By way of a capillary, a sample will be withdrawn continuously from the intermediate-pressure system and injected into the ionizer section of the RGA to give a pressure in the analyzer of the order of 10^{-6} torr. This system has been assembled in the laboratory for testing and calibration. A thermostatically controlled oven and facilities for heating the ion pump, sample lines, and all parts of the high-vacuum system are included.

Since some of the in-reactor tests will require the exposure of the fuel elements to air, the effect of air samples on the RGA was checked. It was found that exposure to air at 10^{-6} torr adversely affects the analyzer. Some components of the analyzer tube, particularly the electron-accelerating grid, are oxidized. This effect causes poor peak shapes and a loss of adequate resolution. The analyzer tube was returned to the General Electric Company for gold plating of the metal components. This gold plating greatly improved the resistance of the analyzer to the effects of air. Only slight changes in resolution and peak shape were observed after exposure to air at 10^{-6} torr for 2 hr at 200°C.

Tests were made on the pumping speed of the 250-liters/sec ion pump; the speed was found to be ~200 liters/sec for nitrogen and 20 liters/sec for helium. Difficulties were encountered in reducing the background that was caused by moisture and hydrocarbons in the system. The automatic gate valve, which contained Viton O-rings and was located between the analyzer and the ion pump, was removed. This change resulted in a substantial decrease in the background.

Engineers from the General Electric Company are now investigating the cause of an electronic malfunction that produces an interaction between the electron-emission current and the ion-energy adjustment.

A second electronic problem is the loss of the low end of the mass scan range; this loss interferes with the observation of masses 1 through 4. Adequate resolution of the lower-mass ranges depends on the selection of the feedback-rectifier tube (6AL5). Even the more effective tubes are degraded during scans of the higher-mass range (i.e., >200). The Vacuum Products Division of the General Electric Company is attempting to modify the circuit to reduce the loading of this tube.

4. Analytical Biochemistry

Gerald Goldstein

4.1 MACROMOLECULAR SEPARATIONS PROGRAM

4.1.a Determination of Tyrosine-Accepting Transfer Ribonucleic Acid ($tRNA^{tyr}$)

I. B. Rubin

The optimum conditions for the determination of picomole quantities of tyrosine-accepting transfer ribonucleic acid ($tRNA^{tyr}$) have been established. The procedures used for studies of this nature have been described.[1] The conditions that result in maximum aminoacylation are:

pH	8.2
Buffer	Cacodylic acid, 25 micromoles
Tyrosine-^{14}C	375 picomoles
Mg^{2+}	2.5 micromoles
ATP	1 micromole
K^+	1.25 micromoles
Enzyme	10 to 50 μg of protein

The volume of the assay solution is 0.25 ml, and the incubation time is 30 min at 37°C, although aminoacylation is complete in 20 min for this range of protein quantity. A reducing agent is not necessary, but 2.5 micromoles of 2-mercaptoethanol is added with the enzyme. Under these conditions, aminoacylation is linear over the minimum range 4 to 80 picomoles of $tRNA^{tyr}$.

[1] I. B. Rubin, A. D. Kelmers, and G. Goldstein, "The Determination of Transfer Ribonucleic Acid by Aminoacylation. I. Leucine and Phenylalanine Transfer Ribonucleic Acid from *E. Coli* B," *Anal. Biochem.* 20, 533 (1967).

4.1.b Determination of Methionine-Accepting Transfer Ribonucleic Acid ($tRNA^{met}$)

D. W. Hatcher

Partially purified methionine-accepting transfer ribonucleic acid ($tRNA^{met}$) is now being prepared by the Chemical Technology Division. The optimum conditions for its determination were established. The techniques used have been described previously.[1] The following conditions were found to be optimum:

tRNA	0.01 to 0.03 ODU
Methionine-^{14}C	1000 picomoles
pH	8.0
Buffer	HEPES, 50 micromoles
ATP	2 micromoles
Mg^{2+}	5 micromoles
Glutathione or β-mercaptoethanol	5 micromoles
Aminoacyl-tRNA synthetase	90 μg of protein
Incubation time	30 min
Incubation temperature	30°C

These conditions are required for an assay volume of 0.5 ml.

4.1.c A Fractional Factorial Method for Optimizing Conditions for the Determination of Transfer Ribonucleic Acid (tRNA)

I. B. Rubin

A statistical approach for establishing optimum conditions for the determination of specific tRNA's has been designed by T. J. Mitchell.[2] This scheme is the fractional factorial method. It is designed to test simultaneously a maximum number of variables with a minimum number of assays. The first, or screening, step has been carried out on three purified tRNA materials; tyrosine-, lysine-, and glutamic acid-accepting tRNA's ($tRNA^{tyr}$, $tRNA^{lys}$, and $tRNA^{glu}$). The optimum conditions for determining the $tRNA^{tyr}$ have already been established by conventional means, that is, by testing one variable at a time (Sect. 4.1.a). The screening step consists in testing nine variables in only 32 assays. These variables are: (1) time, three incubation periods; (2) two buffers; (3) pH, three values; (4) amino acid-^{14}C, three concentrations; (5) ATP, three concentrations; (6) Mg^{2+}, three concentrations; (7) enzyme, three concentrations; (8) K^+ and NH_4^+, three concentrations each; and (9) two reducing agents, three concentrations each.

[2] Mathematics Division.

The second stage of the program will consist in testing those variables whose effects could not be definitely established from the results of the screening test. This stage for the $tRNA^{tyr}$ study has been planned; it consists in testing five variables in 28 assays. It is estimated that a complete study for a given tRNA will take less than ten man-days of laboratory work as compared with the 30 or more usually needed by the conventional system. The estimated ten days does not include the time needed for analysis of each set of results and for preparation of the next step of the program.

4.1.d Automated Determination of Transfer Ribonucleic Acids (tRNA's)

Gerald Goldstein W. L. Maddox[3] I. B. Rubin

A modified version of the automated tRNA analyzer described last year[4] was designed, constructed, and tested and is now in routine operation. Two major changes were made. By the use of micro tubing on the proportioning-pump manifold, the volume of reagents consumed was reduced by ~30%. The stream-sampling mechanism, which in the previous model was activated by detection of a dye with a flow colorimeter, is now keyed directly to the Technicon Sampler. Further details about the modified analyzer are given in Sect. 1.10. The precision and accuracy of the assays are essentially unchanged.

4.1.e Computer Processing of Liquid-Scintillation-Counting Data in the Analysis of Transfer Ribonucleic Acids (tRNA's)

Gerald Goldstein

With >50,000 tRNA assays per year now being reported, both the Analytical Chemistry and Chemical Technology Divisions have been compelled to spend considerable time in transcribing liquid-scintillation-counting data, making calculations to tRNA concentrations, and plotting. We have now acquired a keypunch adapter and an IBM key punch for one of our liquid-scintillation counters so that the data are recorded directly on punched cards. A computer program has been written that provides two forms of readout:

[3] Analytical Instrumentation Group.

[4] G. Goldstein, W. L. Maddox, and I. B. Rubin, "Automated Determination of Transfer Ribonucleic Acids (tRNA's)," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967,* ORNL-4196, p. 46.

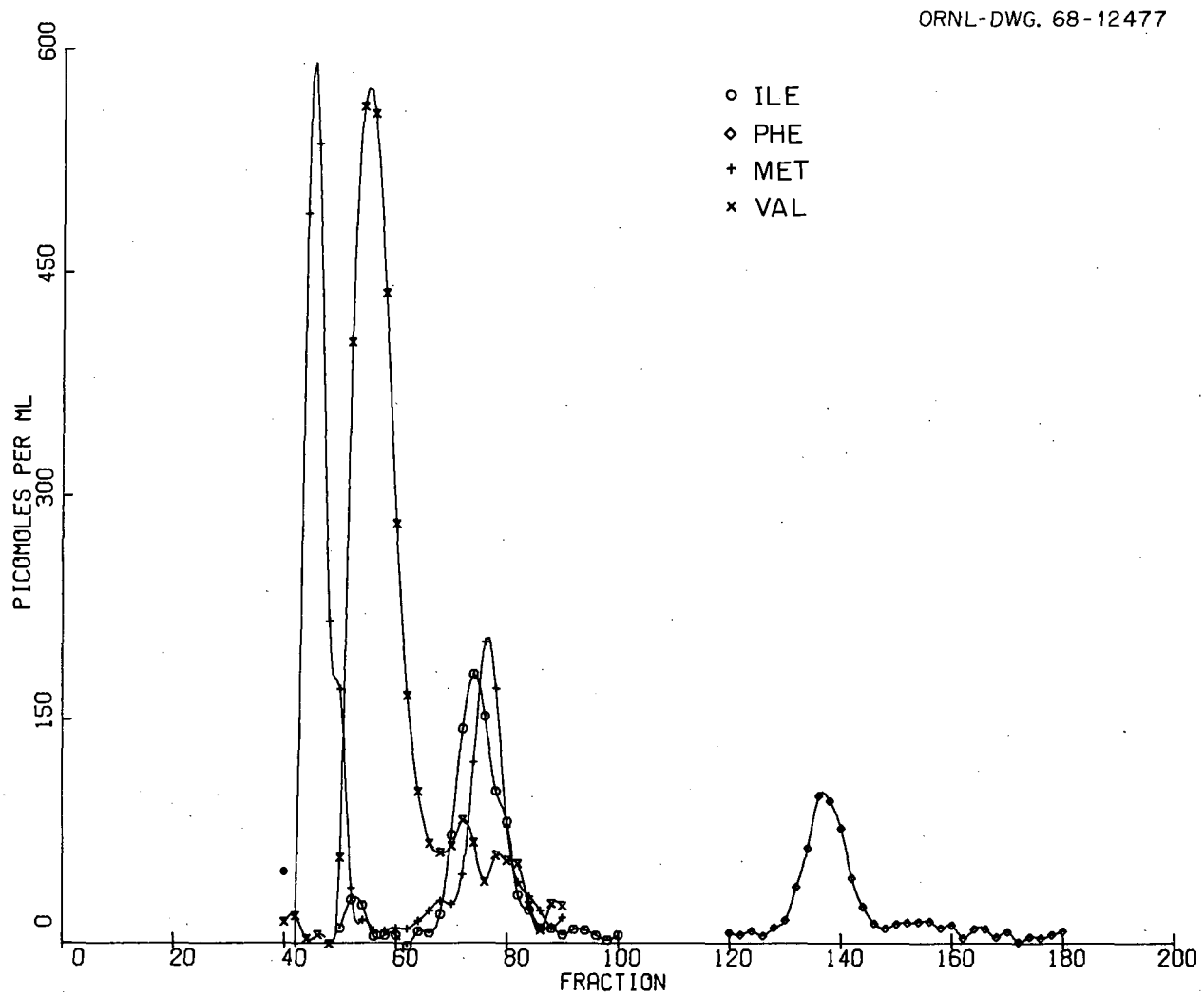

Fig. 4.1. Computer Plot of tRNA Chromatograms.

(1) a printed tabulation of the sample identification, counting data, picomoles of tRNA, and running sum of the total tRNA recovery and (2) a plot of picomoles of tRNA found vs chromatographic fraction number. Chromatograms for as many as ten different tRNA's can be plotted on a single grid; Fig. 4.1 is a typical plot.

We are now in the process of acquiring an adapter and key punch for a second counter. The second key punch will also be capable of converting punched-paper tape to cards. When the Chemical Technology Division installs punched-tape readouts for their spectrophotometer, we will process optical and counting data together.

4.1.f Kinetics of Aminoacylation of Transfer Ribonucleic Acid (tRNA)

D. W. Hatcher

Observed effects of temperature on tRNA assays have suggested to us that the degree of aminoacylation of tRNA, under a given set of conditions, is described best as a steady-state concentration in a dynamic equilibrium. For example, Fig. 4.2 shows the effect of temperature on the aminoacylation of phenylalanine-accepting tRNA ($tRNA^{phe}$). An increase in temperature causes an increase in the initial rate of aminoacylation but not necessarily in the equilibrium

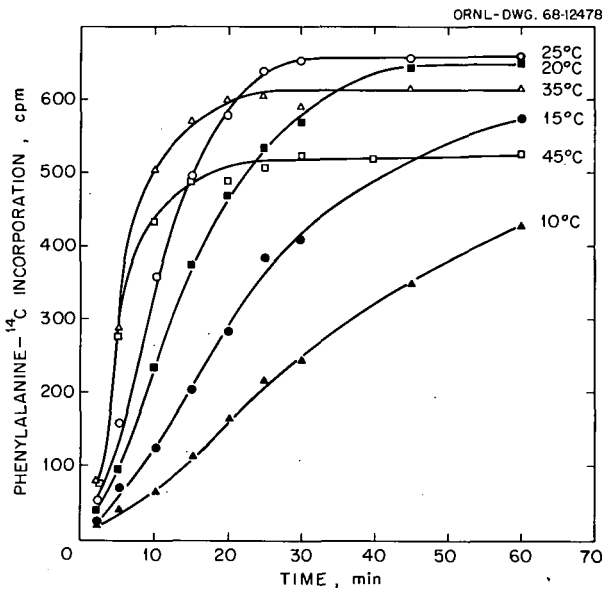

Fig. 4.2. Effect of Temperature on the Aminoacylation of tRNA^{phe}.

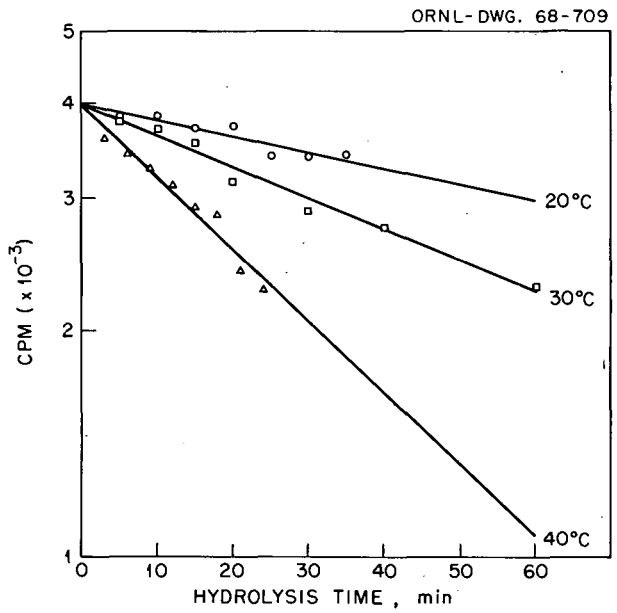

Fig. 4.3. Kinetics of Hydrolysis of Phenylalanine-Accepting (tRNA^{phe}) (pH 8.4 ionic strength = 0.1).

concentration. This temperature effect is rapidly reversible. We postulate a sequence of three reactions, all of which are known to occur, that result in a cyclic process as follows:

$$\text{aa} + \text{E} + \text{ATP} \underset{k_{-1}}{\overset{k_1}{\rightleftharpoons}} (\text{E-aa-AMP}) + \text{PPi}\,,$$

$$(\text{E-aa-AMP}) + \text{tRNA} \underset{k_{-2}}{\overset{k_2}{\rightleftharpoons}} \text{tRNA-aa} + \text{AMP} + \text{E}\,,$$

$$\text{tRNA-aa} + \text{H}_2\text{O} \xrightarrow{k_3} \text{tRNA} + \text{aa}\,.$$

We are attempting to measure the individual reaction rate constants, k_1, k_2, and k_3.

The rate of hydrolysis, k_3, has been determined (Fig. 4.3) and from this the heat of activation of the hydrolysis was calculated to be 13.7 kcal/mole.

4.1.g Reclaiming Amino Acids-^{14}C

D. W. Hatcher

The procedure for determining tRNA's by aminoacylation with ^{14}C-labeled amino acids has been automated (Sect. 4.1.d). A disadvantage of the automated procedure is the increased consumption of amino acids-^{14}C compared with that of manual assay. Therefore, development of an economical procedure for reclaiming the expensive amino acids-^{14}C from their assay mixtures would realize a saving in both the manual and automated procedures and make the automated procedure even more attractive.

Extraction of the amino acid-^{14}C from the assay mixtures by anion- and cation-exchange resin columns and by a liquid cation exchanger has been investigated. The cation-exchange resin worked much better than the anion-exchange resin. However, a large excess of concentrated hydrochloric acid was required to remove the amino acid from the resin column, and no simple means could be found to separate the amino acid from the mineral acid. The liquid cation exchanger did not present this problem.

The liquid cation exchanger di-(2-ethylhexyl)-orthophosphoric acid (HDEHP) dissolved in *n*-hexanol is used to extract the amino acids-^{14}C from the assay mixture. By means of coarse sintered-glass filter tubes, a 10 v/v % HDEHP solution is allowed to percolate as tiny droplets up through the solution that contains the assay mixture and then through three consecutive dilute hydrochloric acid stripping solutions which remove the amino acids from the HDEHP. Figure 4.4 shows the apparatus. The assay mixture must be strongly buffered at pH 3.0 to 3.5 by use of a phosphate buffer, and the stripping stages must be at least 0.1 *M* in HCl. If one recycles the organic solution through the system, only

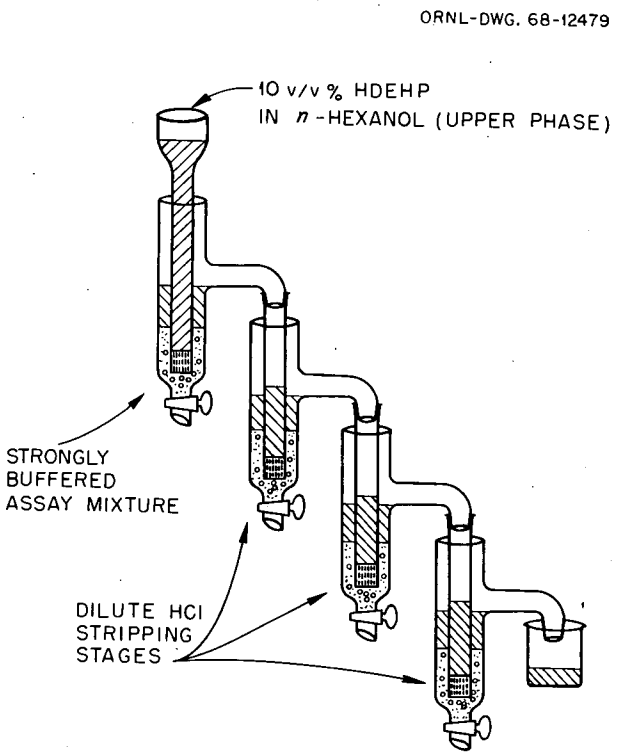

Fig. 4.4. Apparatus for Reclaiming Amino Acids-^{14}C.

small volumes of both the organic and stripping solutions are required.

This procedure has given 93% recovery of phenylalanine-^{14}C from assay mixtures. The recovery of other amino acids looks just as promising. Additional work is necessary to establish the purity and specific activity of the reclaimed material.

4.1.h Chemical Analysis of Transfer Ribonucleic Acids (tRNA's)

D. W. Hatcher

For some time, we have been determining impurities in purified tRNA samples.[5] Recent experience with highly impure materials, however, has indicated that our methods are not adequate and require modification. The following improvements have been made.

Ribonucleic Acid (RNA). – The conventional orcinol spectrophotometric method for RNA[6] was found to be extremely sensitive to the presence of salts, buffers, and other reagents commonly used in the isolation of RNA. By increasing the concentration of HCl in the reaction mixture from 6.0 to 7.2 *N* and adding 0.5 ml of *n*-butanol most of these interferences are eliminated and the sensitivity is increased by 30% (Table 4.1). The recommended composition of the assay mixture is:

Component	Volume (ml)
Sample	1.0
Orcinol (4 g per 100 ml of ethanol)	0.5
Ferric chloride (2.5 g per 100 ml of concd HCl)	0.5
HCl (concd)	2.5
n-Butanol	0.5

From 10 to 800 μg of RNA can be determined with a relative standard deviation of ~5%.

Deoxyribonucleic Acid (DNA). – We find that the diphenylamine spectrophotometric method for DNA[7] is severely influenced by the presence of salts, buffers, and other reagents used to separate RNA from *E. coli* B cells (Table 4.2). The indoleacetic acid procedure for DNA[8] was rejected because it gave poor performance in evaluation tests.

Cadmium(II) quantitatively precipitates DNA and does not interfere in the diphenylamine reaction. We are testing to see whether precipitation by Cd(II) can be used to isolate DNA for its determination. An overnight dialysis of DNA samples does not eliminate alcohol, phenol, or glutathione, which interfere in the diphenylamine reaction.

Polysaccharide. – The enzymatic procedure for determining free glucose[5] can be used to determine polymeric glucose in tRNA samples after hydrolysis of the polysaccharide. Heating for 20 min at 100°C in 2 *N* HCl gives complete hydrolysis of the soluble starch that we use as a polysaccharide standard. The pH of this hydrolyzate is then adjusted to 7.0 ± 0.5 before glucose is determined. No sample interferences have been encountered, and glucose in concentration as small as 5 μg/ml can be measured.

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[5] G. Goldstein, "Determination of Impurities in Transfer Ribonucleic Acids (tRNA)," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1966,* ORNL-4039, p. 40.

[6] G. L. Miller, R. H. Golder, and E. E. Miller, "Determination of Pentoses," *Anal. Chem.* **23,** 903 (1951).

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

[8] P. Schmid, C. Schmid, and D. C. Brodie, "The Determination of the Total Deoxyribose of Deoxyribonucleic Acid," *J. Biol. Chem.* **238,** 1068 (1963).

Table 4.1. Effect of Composition of the Reaction Mixture on Interferences in the Orcinol Spectrophotometric Method for RNA

Interference		RNA Recovered (mg)[b]		
Identity	M^a	6.0 *N* HCl, No Butanol	7.2 *N* HCl, No Butanol	7.2 *N* HCl, Butanol Added
Na acetate	0.10	0.25	0.25	0.26
NaCl	0.10	0.27	0.24	0.25
$MgCl_2$	0.10	0.24	0.26	0.26
$CdCl_2$	0.10	0.20	0.26	0.26
$Na_2S_2O_3$	0.05	(Ppt)	(Ppt)	(Ppt)
EDTA	0.012	0.17	0.26	0.25
Glutathione	0.005	0.14	0.22	0.22
Isopropanol	0.13	0.22	0.27	0.25
Phenol	0.093	0.14	0.15	0.20

[a]Calculated for assay mixture.
[b]0.25 mg added.

Table 4.2. Interferences in the Diphenylamine Spectrophotometric Method for DNA

Substance Tested		Interference (%)
Identity	M^a	
Sodium acetate	0.18	−23
	0.30	−47
Tris buffer	0.01	−15
Glutathione	0.001	−33
β-Mercaptoethanol	0.01	−100
EDTA	0.005	−19
$MgCl_2$	0.01	+14
	0.05	+34
Phenol	0.09	−45
$HClO_4$	0.2	−30
Ethanol	1.7	−70
Glycine	0.4	−73

[a]Calculated for the assay mixture.

Protein. – The spectrophotometric procedure of Groves, Davis, and Sells[9] has proven to be reliable for determining protein concentrations. For nucleic acids, the difference in absorbances at 220 and 240 nm is minimal, whereas for protein the difference is a linear function of concentration. Thus, by use of carefully selected wavelengths at which the absorbances of nucleic acids are equal, we are measuring protein concentrations in the range 5 to 180 μg/ml without interference from nucleic acids.

[9]W. E. Groves, F. C. Davis, Jr., and B. H. Sells, "Spectrophotometric Determination of Microgram Quantities of Protein Without Nucleic Acid Interference," *Anal. Biochem.* 22, 195 (1968).

4.1.i Terminal Nucleoside Assay of Ribonucleic Acid by Ligand-Exchange Chromatography

C. A. Burtis Gerald Goldstein

An article was published[10] that describes the work on the terminal nucleoside assay of ribonucleic acid by ligand-exchange chromatography; the summary from the article follows:

"The nucleosides released from polyribonucleosides and RNA's by alkaline hydrolysis may be identified and quantitated by ligand-exchange chromatography on a copper-loaded chelating ion exchanger. With a suitably sensitive ultraviolet absorbance monitor and recorder, this analysis can be effected on as little as 10 nM of free nucleoisde in 1–2 hr with an accuracy (standard deviation) of 3–4%. Dephosphorylation prior to alkaline hydrolysis allows the assay of nucleosides bearing primary phosphate residues in the 3′- (or 2′-) position. The method is useful in quantitating the nucleoside at the aminoacyl-accepting end of tRNA's."

4.1.j Gel Electrophoresis of Ribonucleic Acid (RNA)

A. L. Travaglini

Preliminary studies on electrophoresis of RNA were begun. The particular RNA used, although thought to be pure, actually contained a large amount of a fast-moving fraction and a small amount of a slow-moving fraction. The staining and fixing solution used was toluidine blue (0.01 w/v %)–trichloroacetic acid (10 v/v %)–lanthanum nitrate (0.1 *M*). Of those

[10]C. A. Burtis and G. Goldstein, "Terminal Nucleoside Assay of Ribonucleic Acid by Ligand-Exchange Chromatography," *Anal. Biochem.* 23, 502 (1968).

studied, this was the only mixture of stain and fixative that is stable to electrophoretic destaining of the background and that provides a medium in which the dyed RNA bands do not diffuse during storage. More study of the effects of buffers and pH on the separation of RNA's and on the sensitivity will be required.

4.1.k Determination of L-Asparaginase

I. B. Rubin

It is anticipated that, in the near future, the Biology and Chemical Technology Divisions will start a program to produce L-asparaginase. Consequently, a study has been initiated to develop a procedure for L-asparaginase (L-asparagine amidohydrolase EC 3.5.1.1) suitable for its routine determination in a large number of chromatographic fractions that would contain various quantities of this enzyme. As a first step, the procedure of Campbell and associates[11] for determining the two L-asparaginases from *E. coli* B has been coupled with Layton's[12] spectrophotometric sodium phenate method for the determination of ammonia. The quantity of ammonia formed by the reaction

$$\text{L-asparagine} + H_2O \xrightarrow{\text{L-asparaginase}} \text{L-aspartate} + NH_3$$

is directly proportional to the amount of enzyme added when the asparagine is present in excess and when the incubation period is <30 min.[11] An aliquot of the assay solution is mixed directly with the chromogenic reagents for the ammonia determination without previous deproteinization of the assay solution. Tests with pure L-asparaginase have indicated that the absorbance of the resulting chromophore varies linearly with the quantity of ammonia released by 0.03 to 0.18 unit of enzyme. A unit is that quantity of L-asparaginase that will release 1.0 micromole of ammonia per minute under the conditions of the assay.[13]

Future work will involve scaling the assay and chromogenic solutions to a smaller volume, studying possible interferences that may be present after the chromatographic separations, and automating the assay.

4.1.l Determination of Subnanomole Quantities of L-Amino Acids by Isotope Dilution–Aminoacylation

I. B. Rubin

A procedure is being developed for the determination of subnanomole quantities of specific L-amino acids. This procedure uses isotope dilution in conjunction with the standard aminoacylation method for the determination of transfer ribonucleic acid (tRNA).[14] The unlabeled amino acid is added to the tRNA assay solution of the same ^{14}C-labeled amino acid. The decrease in the count rate of an aliquot of the solution is directly proportional to the quantity of unlabeled acid present. This method should be very useful for the determination of amino acid concentrations in peptide hydrolysates that result from various biological studies.[15] The hydrolysates need only to be evaporated to remove the hydrochloric acid and then dissolved either in water or in Tris, cacodylate, or acetate buffer. No derivatization of the amino acids is necessary, and only nanomole quantities of sample are needed. This procedure should also be suitable for the determination of the L-component of DL-amino acid mixtures.

To date, calibration data have been obtained for alanine, arginine, asparagine, aspartic acid, cystine, glutamic acid, leucine, lysine, methionine, phenylalanine, serine, tyrosine, and valine. In general, the calibration curves are linear over the range 0 to 3 nanomoles of added unlabeled amino acid. However, the calibration curve for alanine is linear only from 0.2 to 3 nanomoles, and that for cystine only up to 1.5 nanomoles. Cystine and cysteine cannot be distinguished by this procedure. Relatively large quantities (>2 nanomoles) of asparagine and aspartic acid do interfere with the determination of smaller quantities of the other. However, in hydrolysates that contain no asparagine, aspartic acid appears to assay as asparagine. This reaction may be caused by asparagine synthetase activity in the crude aminoacyl synthetase enzyme mixture.

The accuracy of this procedure is good, and there appears to be no bias. The precision also is good ($S \not> $

[11] H. A. Campbell, L. T. Mashburn, E. A. Boyse, and L. J. Old, "Two L-Asparaginases from *Escherichia Coli* B. Their Separation, Purification, and Antitumor Activity," *Biochemistry* 6, 721 (1967).

[12] F. L. Layton, "Ammonia, Spectrophotometric Sodium Phenate Method (Nitrogen)," Method Nos. 1 220020 and 9 00720020 (9-25-62), *ORNL Master Analytical Manual,* TID-7015, Supplement 5 (1963).

[13] 3.5.1.1 Asparaginase (*E. Coli*) (L-asparagine amdohydrolase), Catalog from Worthington Biochemical Corp., Freehold, N.J. (April 1967).

[14] J. C. White, C. A. Horton, and I. B. Rubin, "Analytical Biochemistry," *Anal. Chem. Div. Ann. Progr. Rept. Nov. 15, 1964,* ORNL-3570, p. 29.

[15] J. E. Attrill and R. A. Popp, "Amino-Acid Sequence Analysis," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967,* ORNL-4196, p. 53.

Table 4.3. Comparison of Amino Acid Analyses of a Simulated Peptide Hydrolysate

Amino Acid	Added (micromole/ml)	Found (micromole/ml)	
		This Work[a]	Chromatography[b]
(NH_3)			0.29
Asp	0.30	0.61	0.53
Asn	0.30	0.59	
Leu	0.30	0.31	0.28
Lys	0.24	0.30	0.25
Met	0.30	0.33	0.30
Phe	0.30	0.29	0.27
Val	0.30	0.29	0.32

[a]0.30- and 0.60-nanomole portions taken for analysis.
[b]Analyzed by J. E. Attrill.

10%). The data of Table 4.3 show that the results compare well with those obtained by chromatographic analysis with an amino acid analyzer.

Future work will involve obtaining calibration data for the remaining amino acids, applying this procedure to actual peptide hydrolysates, resolving the anomalous situation with asparagine and aspartic acid, and applying this procedure to racemic mixtures of amino acid.

4.2 LIGAND-EXCHANGE CHROMATOGRAPHY

4.2.a Principles of Ligand-Exchange Chromatography on Copper-Loaded Chelex Resin Columns

Gerald Goldstein

From the chemical equilibria involved and several simplifying assumptions, a simple relationship can be derived between the basicity of a compound and its retention volume on a column of copper-loaded Chelex resin.

Retention volume (V_R) on a column is related to the ratio (K_d) of the distribution of the material between the column and the aqueous phases by the equation

$$V_R = V_0 + K_d W \ , \tag{4.1}$$

where V_0 is the void volume and W is the weight of resin if K_d is independent of the concentration of the material (linear isotherm). It follows that on a column of a given W

$$K_d \propto V_R - V_0 \propto V_R' \ , \tag{4.2}$$

where V_R' is the corrected retention volume.

If it is assumed that the copper on the column is coordinated to only one ammonia molecule[16] which is replaced by one ligand molecule, the equilibrium for the replacement can be written

$$L + -Cu(NH_3) \rightleftharpoons -Cu(L) + NH_3 \ .$$

Also,

$$K_e = \frac{\left(-Cu(L)\right)[NH_3]}{\left(-Cu(NH_3)\right)[L]} = \frac{K_f}{K_{NH_3}} \ ,$$

where K_f and K_{NH_3} refer to the formation constants of $-Cu(L)$ and $-Cu(NH_3)$, respectively. Since

$$K_d \propto \frac{\left(-Cu(L)\right)}{[L]} \ ,$$

then

$$K_d \frac{[NH_3]}{\left(-Cu(NH_3)\right)} \propto \frac{K_f}{K_{NH_3}} \ ,$$

and, if constant terms are left out, then

$$K_d [NH_3] \propto K_f \ . \tag{4.3}$$

It is well known[17] that the formation constants of metal-ion complexes which have a series of structurally similar ligands, in solution, are related to the basicity of the ligand by

$$K_f \propto \frac{1}{K_a} \ . \tag{4.4}$$

If we assume that this trend is true, even when the metal is sorbed on a resin, then by combining Eqs. (4.2), (4.3), and (4.4)

$$V_R' \ [NH_3] \propto \frac{1}{K_a}$$

or

$$\log V_R' \propto pK_a - \log [NH_3] \ . \tag{4.5}$$

[16]K. Shimomura, L. Dickson, and H. F. Walton, "Separation of Amines by Ligand Exchange. Part IV. Ligand Exchange with Chelating Resins and Cellulosic Exchangers," *Anal. Chim. Acta* 37, 102 (1967).

[17]A. E. Martell and M. Calvin, *Chemistry of the Metal Chelate Compounds*, p. 151, Prentice-Hall, Englewood Cliffs, N.J., 1952.

Table 4.4. Retention Volumes and pK_a Values of Some Uracil Derivatives

Compound	pK_a[a]	V_R' (ml)	log V_R'
Pseudouridine	8.97	10.5	1.02
Deoxyuridine	(9.1)	12.3	1.09
Uridine	9.17	12.9	1.11
Uracil	9.45	17.4	1.24
Thymidine	9.65	20.8	1.32
5-Methyluridine	(9.7)	22.1	1.34
Thymine	9.9	26.9	1.43
Dihydrouridine	(10.0)	29.6	1.47

[a]Values in parentheses are not listed in the literature but are estimated from this work.

The relationship of Eq. (4.5) was tested with a series of uracil derivatives on a 60- by 0.9-cm column; the eluent was 10^{-3} N NH_4OH at a flow rate of 0.78 ml/min. Uridylic acid was added as a void-volume marker, because it is not retained on the column. Table 4.4 and Fig. 4.5 show the results. The agreement between the experimental results and the theory is very good; Eq. (4.5) enables us to predict from known pK_a values whether separations are feasible or not.

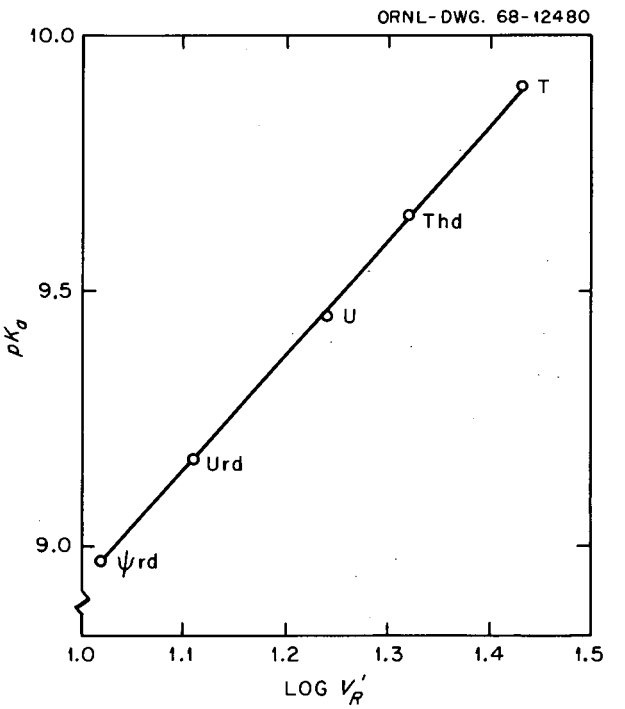

Fig. 4.5. Relationship Between pK_a and Retention Volumes of Uracil Derivatives.

4.2.b Separation of Compounds of Biological Interest by Ligand-Exchange Chromatography

Gerald Goldstein J. C. Wolford[18]

We have shown previously that ribonucleosides can be separated by chromatography on a copper-loaded chelating resin.[19] We find that the deoxyribonucleosides can also be separated and that each except deoxyuridine can be distinguished from the corresponding ribonucleoside (Fig. 4.6).

We are currently testing the separation of xanthine and its methylated derivatives. These compounds are similar in structure to the purine bases found in nucleic acids and are natural products of biological interest. Figure 4.7 shows the separation of caffeine (1,3,7-trimethylxanthine), theobromine (3,7-dimethylxanthine), and theophylline (1,3-dimethylxanthine).

Since sulfur forms strong bonds with copper, we are examining the possibility of separating sulfur-containing compounds on copper-loaded columns. Preliminary tests indicate that the thionucleosides are, indeed, retained on a column and that separations may be possible.

[18]Student Guest from the Department of Chemistry, University of Tennessee, Knoxville.

[19]G. Goldstein, "Ligant-Exchange Chromatography of Nucleotides, Nucleosides, and Nucleic Acid Bases," *Anal. Biochem.* **20**, 477 (1967).

4.3 MOLECULAR ANATOMY PROGRAM

4.3.a Identification of Urinary Constituents Isolated by Anion-Exchange Chromatography

C. A. Burtis Sidney Katz[20]

High-pressure anion-exchange chromatography of urine has resolved more than 100 ultraviolet-absorbing urinary constituents. Two methods have been used to identify them: (1) comparison of the chromatographic properties of reference compounds with those of the urinary constituents (tentative identification) and (2) spectrometric analysis of isolated urinary constituents through identification.

In the first approach, reference solutions of biochemicals known to be in urine were chromatographed by

[20]Chemical Technology Division.

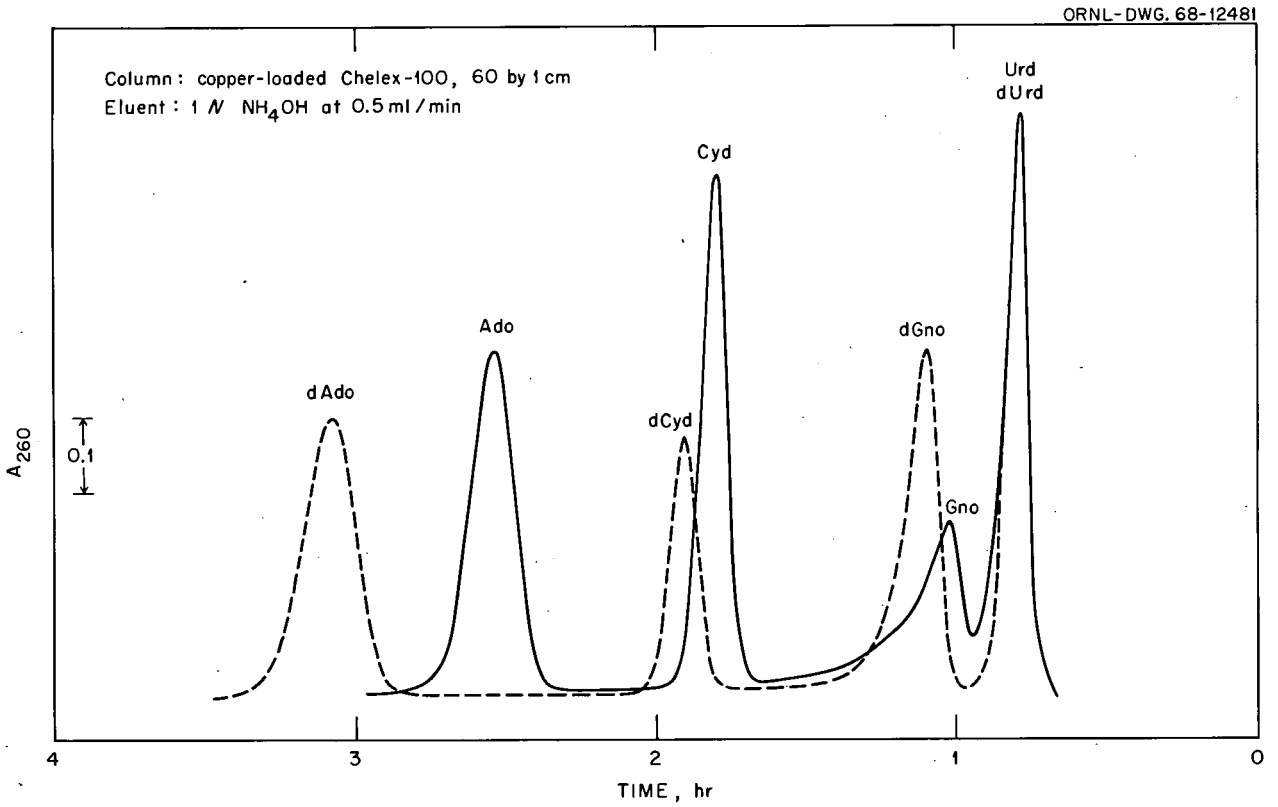

Fig. 4.6. Separation of Ribo- and Deoxyribonucleosides by Ligand-Exchange Chromatography.

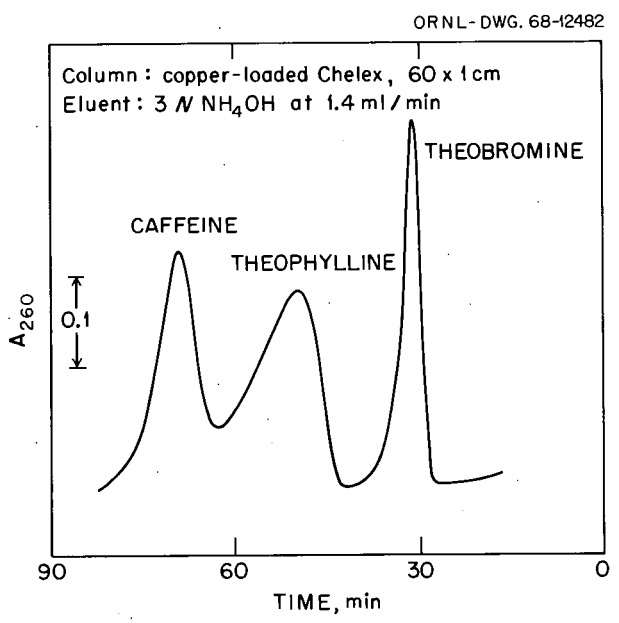

Fig. 4.7. Separation of Xanthine Derivatives by Ligand-Exchange Chromatography.

anion-exchange chromatography.[21,22] The column effluent was collected, and the ultraviolet (UV) spectrum of the effluent associated with each of the separate peaks was determined and compared with the UV spectra of the reference compounds. From either this comparison or absorbance ratios at two or more wavelengths, the chromatographic positions of the reference chemicals were established. In instances where the chemical had little or no UV absorbance, ^{14}C-isomers of the compounds were chromatographed, and their chromatographic positions were determined by monitoring the column effluent for radioactivity. The elution volumes of 112 urinary compounds are given in Table 4.5. Urine samples were chromatographed with and without the above-mentioned chemicals. By comparison, ten of the urine components were tentatively

[21] C. 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).

[22] C. A. Burtis and K. S. Warren, "Identification of Urinary Constituents Isolated by Anion Exchange Chromatography," *Clin. Chem.* **14**, 290 (1968).

Table 4.5. Elution Volumes of Known Urinary Biochemicals Separated by Anion-Exchange Chromatography[a]

Compound	Elution Volume[b] (ml)
Adrenaline	14[c]
Arginine	14
Cyanocobolin	14
Cystine	14
3,4-Dimethoxyphenylethylamine	14
Dopamine	14
Histidine	14
Lysine	14
Metanephrine	14
3-Methoxytyramine	14
Noradrenaline	14
Normetanephrine	14
Thiamine	14
Serotonin	14
p-Tyramine	14
Asparagine	18
Citrulline	18
Creatine	18
Glutamine	18
Glucose	18
Trigonelline	18
Creatinine[d]	19
Cytosine	20
Alanine	21
Ergothioneine[d]	21
Methionine	21
Proline	21
Urea	21
Cytidine	24
Deoxycytidine	25
N-Acetyltryptophan	25
Pyridoxal	25
Pyridoxine	25
Phenylalanine	27
Tryptamine	27
Pseudouridine[d]	29
Uridine	38
Deoxyuridine	39
Theobromine	39
Uracil	42
Nicotinamide	42
Thymidine	45
N-Methylnicotinamide	47
Tyrosine	49
Caffeine	50
Deoxyinosine	62
4-Amino-5-imidocarboxamide	72
Hypoxanthine[d]	85
Riboflavin	92
Deoxyadenosine	94
Adenosine	101
Tryptophan	110
1-Methylguanine	118
7-Methylguanine	120
Theophylline	137
3-Hydroxykynurenine	146
Adenine	152
Xanthosine	163
Xanthine[d]	172
Guanosine	214
Glutamic acid	225
6-Methyladenine	240
Guanine	241
Aspartic acid	270
Urocanic acid[d]	381
Estratriol-16α-glucuronide	428
Uric acid[d]	550
Nicotinuric acid	569
Nicotinic acid	581
Indoleacetamide	598
Phenol	636
Maleuric acid	650
Estratriol-3-glucuronide	727
Testosterone glucuronide	807
Hippuric acid[d]	817
Citric acid	820
p-Aminobenzoic acid	863
3-Methoxy-4-hydroxymandelic acid	871
Estratriol-17α-glucuronide	876
Quinaldic acid	914
Homovanillic acid	916
Syringic acid	975
p-Hydroxyphenyllactic acid	1010
p-Hydroxymandelic acid	1019
p-Hydroxyphenylacetic acid	1020
Anthranilic acid	1064
Benzoic acid[d]	1073
3-Indoleacetic acid	1106
5-Hydroxyindoleacetic acid	1114
m-Hydroxyphenylacetic acid	1125
Vanillic acid	1156
Phloretic acid	1194
Estrone sulfate	1210
Folic acid	1212
Kynurenic acid[d]	1214
o-Hydroxyhippuric acid	1253
3-Indoleglycolic acid	1266
Salicylacetic acid	1272
3-Hydroxyanthranilic acid	1287
Aconitic acid	1297
m-Hydroxybenzoic acid	1343
p-Hydroxybenzoic acid	1349
o-Hydroxybenzoic acid	1350
3-Methoxy-4-hydroxycinnamic acid	1390
Homogentisic acid	1417
α-Resorcylic acid	1430
Indoleacrylic acid	1440
p-Hydroxycinnamic acid	1443
Xanthurenic acid	>1500

[a]Chromatography was performed with a 0.45- by 200-cm stainless-steel column; 5- to 10-μ Dowex 1-X8 resin; temperature, 40°C for 16 hr and 60°C for 16 to 48 hr; operating pressure, 1000 to 2000 psig; eluent, sodium acetate–acetic acid buffer at pH 4.4, varying in concentration from 0.015 to 6 *M*; and average flow rate of eluent, 28 to 30 ml/hr.

[b]Numbers represent the average of three or more determinations with a relative standard error of ±1 to ±3%.

[c]Column breakthrough.

[d]Tentatively identified in a urine chromatogram.

identified. The other compounds were either present at concentrations below our detection limits or, because of chemical conjugation, were eluted at a volume different from that of the reference chemical.

The spectrometric analysis entails separation of the urinary constituent by gel chromatography followed by mass spectrometry (MS), infrared (IR), nuclear magnetic resonance (NMR), and UV spectroscopy and interpretation of the resultant spectra. This approach was used to identify as hippuric acid the component eluting at a column volume of 817 ml. Sodium hippurate was separated from the acetate buffer (Fig. 4.8) and analyzed by MS and NMR. The resultant spectra were compared with the MS (Fig. 4.9) and NMR (Fig. 4.10) spectra of reference hippuric acid. Because the MS and NMR spectra of the urinary component and hippuric acid match, the analyses indicate that hippuric acid was contained in the eluate at 817 ml.

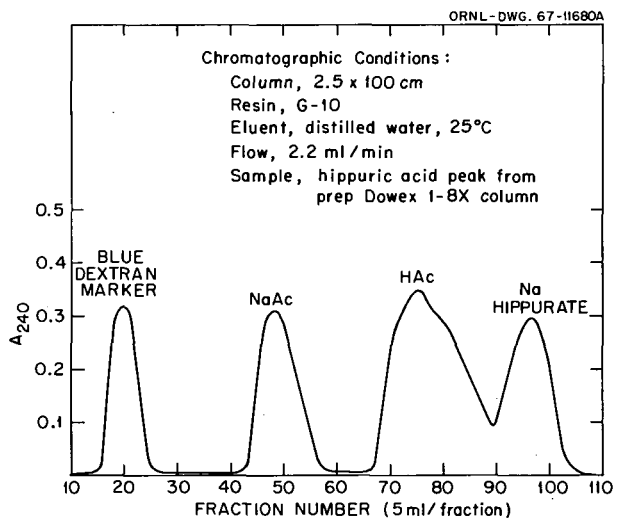

Fig. 4.8. Chromatogram That Shows the Separation of Sodium Hippurate from Sodium Acetate and Acetic Acid by Gel Filtration Chromatography.

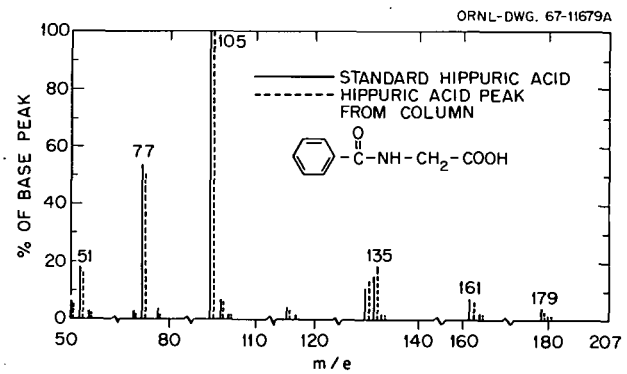

Fig. 4.9. Mass Spectra of Urinary and Reference Hippuric Acid.

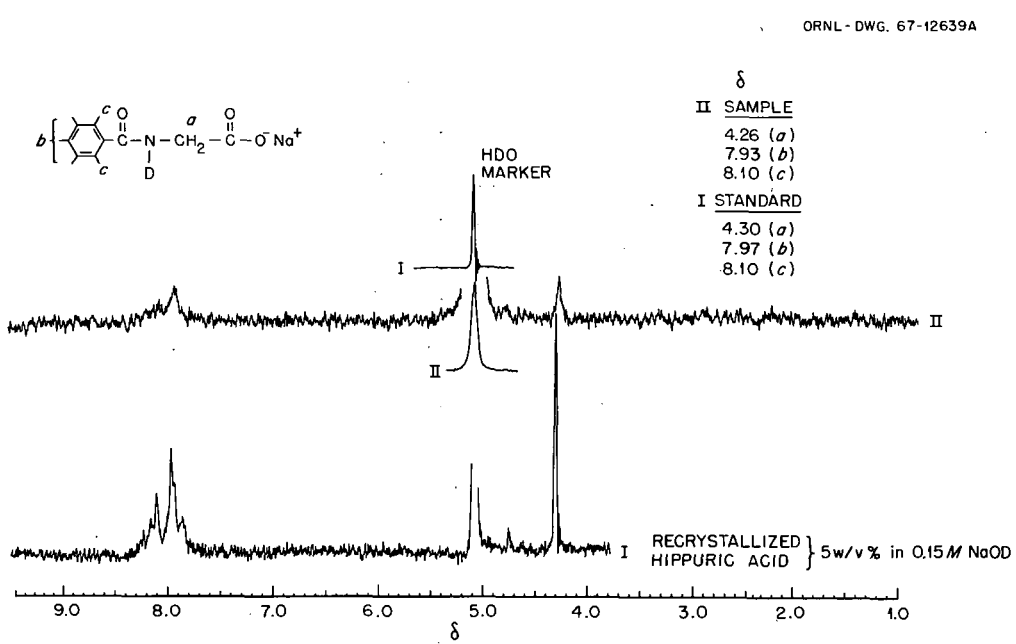

Fig. 4.10. Nuclear Magnetic Resonance Spectra of Urinary and Reference Hippuric Acid.

Likewise, uric acid was found to elute at 550 ml, since the compound isolated from this fraction gave the same MS (Fig. 4.11) and IR (Fig. 4.12) spectra as reference uric acid. The identifications of both the hippuric and uric acid by this method confirmed the tentative identifications of these compounds described earlier.

A large unknown fraction eluting at 40 hr on the preparative system was concentrated and desalted and was successfully identified by MS and UV spectroscopy as *o*-hydroxybenzoic acid (salicylic acid), a hydrolysis product of aspirin.

A paper on this work was presented orally,[23] and a paper was also published.[22]

[23]C. A. Burtis and K. S. Warren, "Identification of Urinary Constituents Isolated by Anion Exchange Chromatography," presented at the Symposium on Urinary Constituents of Low Molecular Weight, Oak Ridge National Laboratory, Oak Ridge, Tenn., Nov. 30–Dec. 1, 1967.

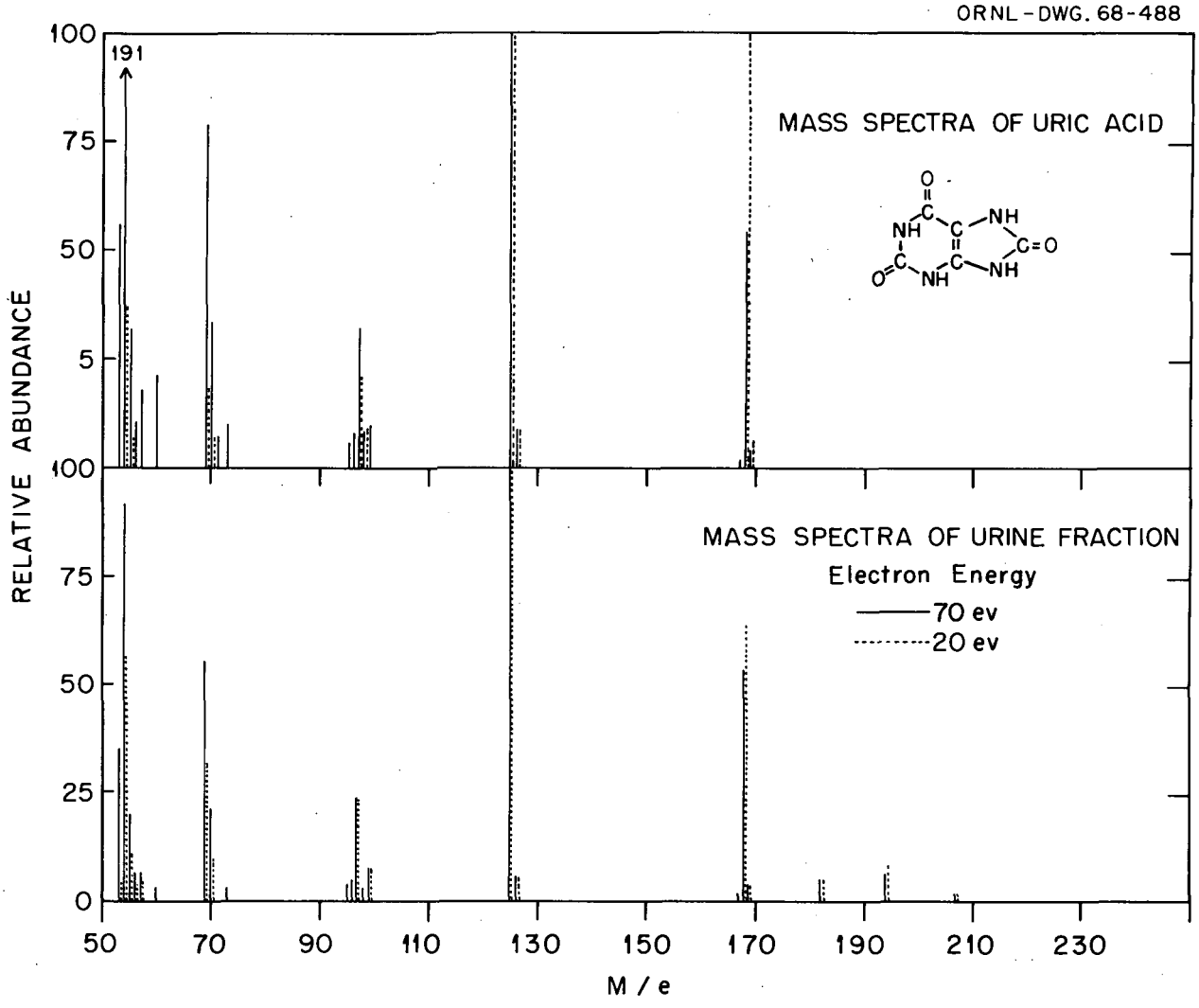

Fig. 4.11. Mass Spectra of Urinary and Reference Uric Acid.

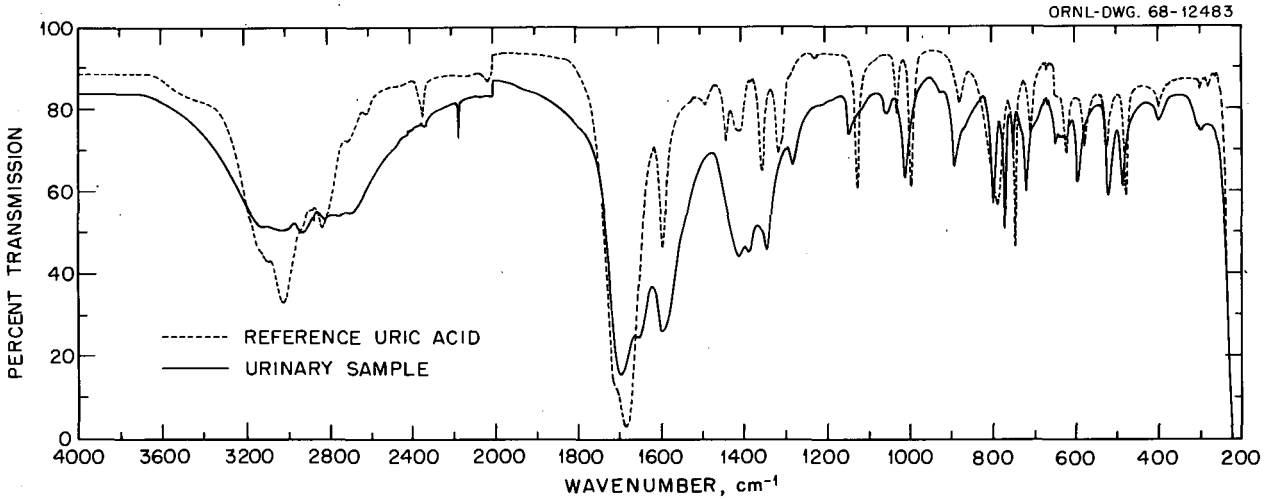

Fig. 4.12. Infrared Spectra of Urinary and Reference Uric Acid.

4.3.b Anion-Exchange Chromatography of Hydrolyzed Urine

C. A. Burtis

Many compounds are excreted in urine as conjugates. The main conjugation reactions that occur in animals are those which involve glucuronic acid, sulfate, glycine, cysteine, methylation, and acetylation. To determine whether any of the compounds associated with the UV-absorbing peaks observed after anion-exchange chromatography of urine are conjugates, urine was enzymatically and chemically hydrolyzed and the resultant hydrolysate was chromatographed by anion-exchange chromatography. By comparison of urine chromatograms before and after acid or enzymatic hydrolysis, the presence or absence of urinary conjugates can be determined. In the enzymatic hydrolyses, a urine blank without the enzyme was carried through each incubation to determine the lability of the urinary constituents to the incubation and hydrolysis conditions. Table 4.6 gives the results of the various hydrolyses. The data presented have been corrected for the gain or loss of chromatographic peaks due to the reaction conditions.

The acid hydrolysis resulted in the loss of 22 chromatographic peaks and the appearance of seven new ones. It was also observed that the hippuric acid (benzoylglycine) peak was unaffected, which indicates that the amide bond contained in this type of conjugation is resistant to the hydrolytic conditions. Conditions known to hydrolyze this type of bond (6 *M* HCl, heat) were too harsh; chromatography of urine hydrolyzed under these conditions resulted in an 80 to 90% loss of chromatographic peaks.

Table 4.6. Results of Anion-Exchange Chromatography[a] of Acid- and Enzyme-Hydrolyzed Urine

Hydrolysis	Number of Peaks in Hydrolyzed Urine	
	Lost	Gained
Acid[b]	22	7
β-Glucuronidase[c]	9	7
Sulfatase[d]	9	5
Pepsin[e]	9	8
β-Glucuronidase and sulfatase mixture[f]	20	18

[a]Chromatography was performed with a 0.45- by 200-cm stainless-steel column; 5- to 10-μ Dowex 1-X8 resin; temperature, 40°C for 16 hr and 60°C for 16 to 48 hr; operating pressure, 1000 to 2000 psig; eluent, sodium acetate–acetic acid buffer at pH 4.4, varying in concentration from 0.015 to 6 *M*; and average flow rate of eluent, 28 to 30 ml/hr.

[b]Urine of normal male adjusted to pH 1.0 with concentrated HCl and incubated at 75°C overnight. The pH was adjusted to 4.4 before chromatography.

[c]Urine from normal male buffered to pH 4.7 with 0.1 *M* acetate and incubated for 48 hr at 37°C with 5000 Fishman units of Worthington Biochemical Corporation's glucuronidase.

[d]Urine from a woman in her eighth month of pregnancy was adjusted to pH 5.0 with 1 *M* acetate and incubated 72 hr at 37°C with 10 mg of sulfatase (Sigma Chemical Company's limpet type 11, 100 units).

[e]Male urine adjusted to pH 2.7 with 0.2 *M* citrate and incubated 36 hr at 37°C with 10 mg of Worthington Biochemical Corporation's pepsin.

[f]Urine from a two-year-old girl, who has a neuroblastoma and has been treated with methylated purines, steroids, and radiation to the cranium was adjusted to pH 4.4 and incubated with 300,000 Fishman units of bovine glucuronidase and 10 mg of limpet sulfatase for 72 hr at 37°C.

Enzymatic hydrolysis is milder and more specific than acid hydrolysis. The conjugation of glucuronic acid with the hydroxyl group of urinary constituents such as steroids and aromatic acids is the type of conjugation most commonly found in urine. Hydrolysis of urine with β-glucuronidase resulted in the loss of nine and the gain of seven chromatographic peaks. The section of the chromatogram between the elution volumes 325 and 351 ml was definitely altered by β-glucuronidase hydrolysis (Fig. 4.13). In this example, two peaks are lost on glucuronidase hydrolysis.

A second type of conjugation that involves steroids and aromatic acids is the conjugation of sulfate with the aromatic hydroxyl groups of such compounds. Since the female hormones (estrogens) are steroids that contain aromatic hydroxyls, the urine of a pregnant female was hydrolyzed with sulfatase. This hydrolysis resulted in the loss of nine and the gain of five chromatographic peaks. To further investigate the effect of glucuronidase and sulfatase hydrolyses on urine, the urine from a two-year-old girl having a neuroblastoma was hydrolyzed with a mixture of the two enzymes. The patient had been administered steroids for therapeutic reasons; therefore, a high concentration of urinary steroids was expected. The chromatography of the resultant hydrolysate resulted in a loss of 20 and a gain of 18 chromatographic peaks. Preliminary investigation indicates that of the components corresponding to the peaks lost several are of a steroidal and several of an aromatic acid nature.

Urine was also hydrolyzed with the enzyme pepsin, a peptidase that cleaves amide bonds involving an aromatic amino acid. Chromatography of this hydrolysate resulted in the loss of nine and the gain of eight chromatographic peaks.

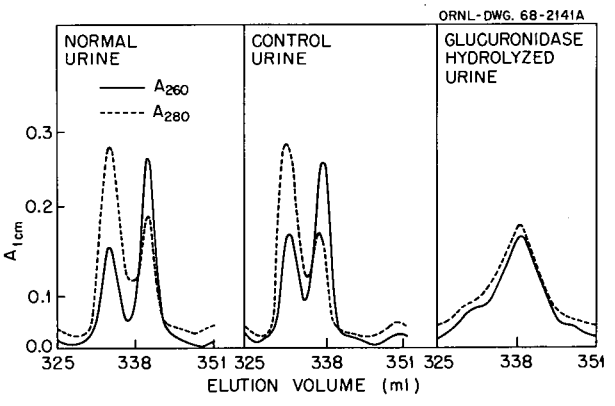

Fig. 4.13. Anion-Exchange Chromatogram of Glucuronidase-Hydrolyzed Urine.

Studies of this nature are useful in ascertaining the type of constituents contained in the column fractions of urine that give the chromatographic peaks. If our assumptions are correct, then separatory procedures can be devised to isolate a particular constituent for spectral analysis and identification.

4.3.c Fractionation of Urine by Gel and Anion-Exchange Chromatography

C. A. Burtis

Prior investigation has indicated the feasibility of using gel chromatography to prefractionate raw urine.[24] Separation of urine components by gel chromatography on Sephadex G-10 yields as many as ten UV-absorbing fractions (Fig. 4.14). The refractive-index monitor indicates four peaks, three of which correspond to UV-absorbing fractions. The fraction that gave the fourth peak was found to be strongly ninhydrin positive and to contain urea.

Because of incomplete resolution of four of the UV-absorbing peaks, the contents of tubes from a chromatographic run were pooled into six fractions. The six fractions were concentrated and chromatographed on a 1.25- by 200-cm anion-exchange column packed with 20-μ Dowex 1-X8. With this system, a typical chromatographic run requires 40 hr and resolves 70 to 80 chromatographic peaks. Figure 4.15 gives the results obtained from the chromatography of these gel fractions. The top chromatogram was obtained from chromatography of 20 ml of whole urine; 83 chromatographic peaks were resolved. The lower chromatograms are those obtained by chromatography of the respective gel fractions in which 18 peaks were observed from fraction I, 30 from II, 13 from III, 63 from IV, 12 from V, and 17 from VI. Several of the peaks that were not well resolved in the chromatogram of a normal urine were found in different gel fractions and appeared in their respective chromatograms as resolved peaks. The increase in resolution is easily seen in Fig. 4.16 in which a peak that was not resolved in the urine chromatogram appears as a single peak in the gel fraction.

These results have confirmed that human urine is a complex biological fluid. The prefractionation of urine by gel chromatography followed by anion-exchange chromatography of the gel fractions has proven useful for increasing resolution and obtaining single peaks for

[24] C. A. Burtis, "Fractionation of Urine by Gel-Permeation Chromatography," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967*, ORNL-4196, p. 50.

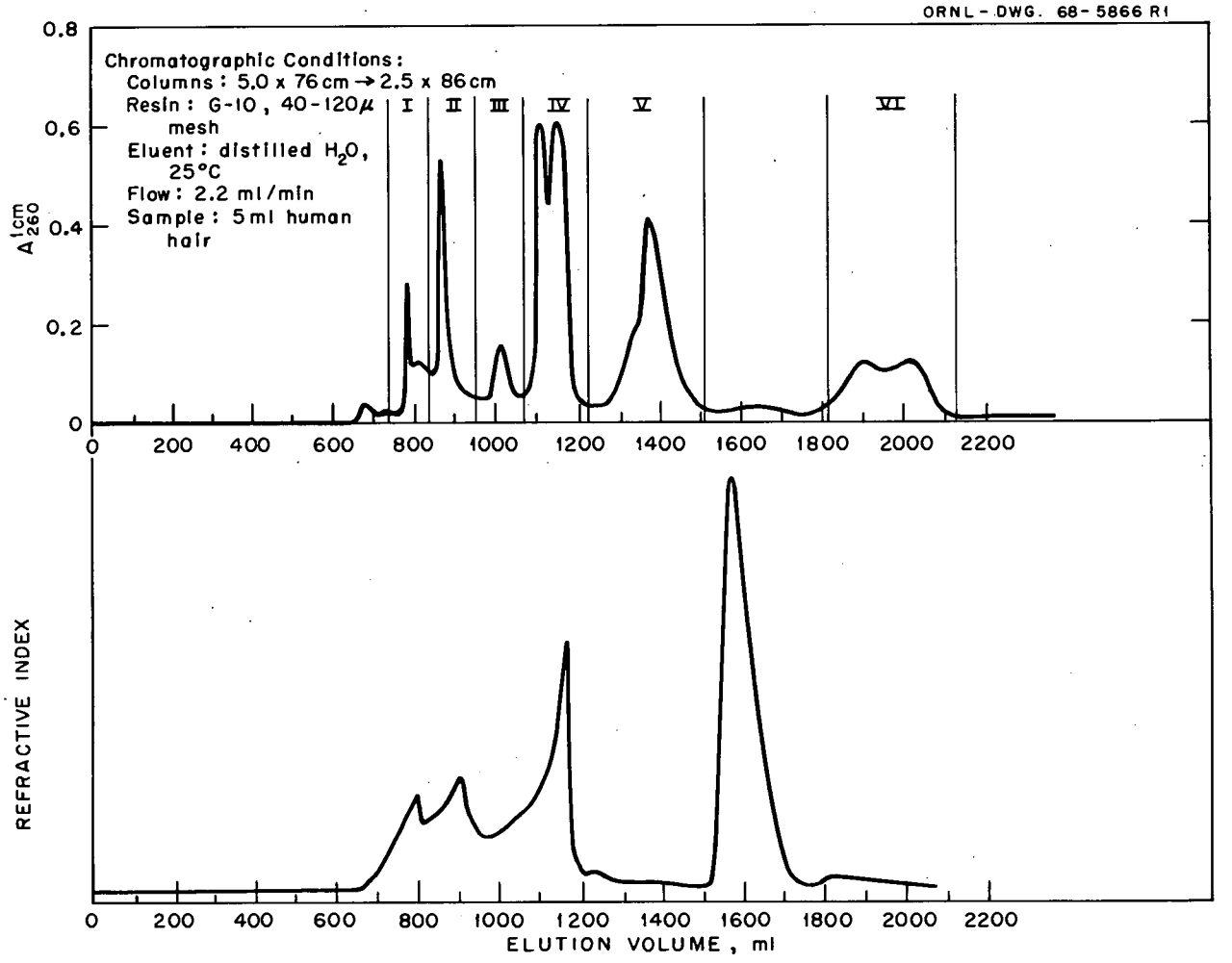

Fig. 4.14. Chromatograms from the Fractionation of Human Urine by Gel Chromatography.

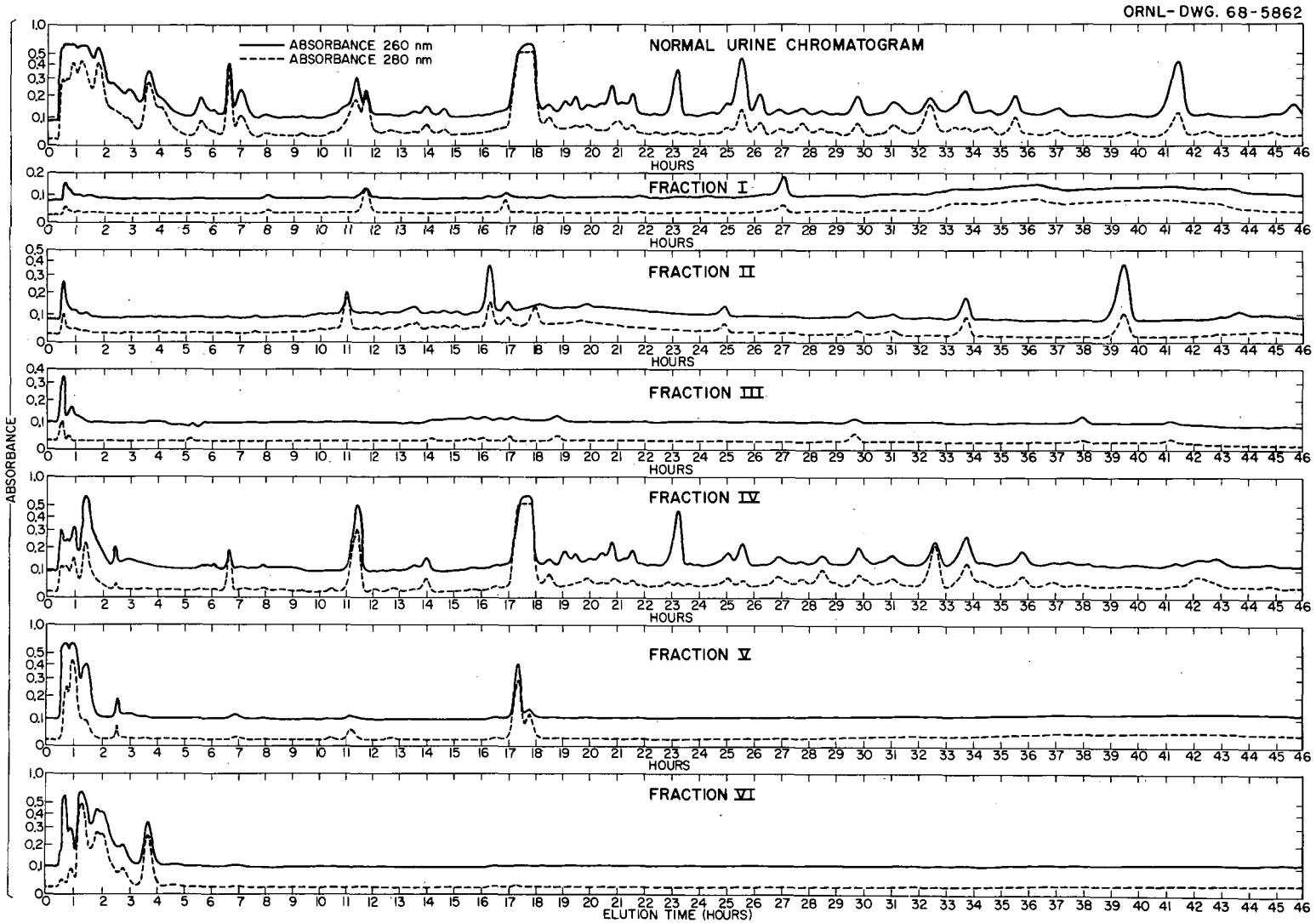

Fig. 4.15. Chromatograms from the Anion-Exchange Chromatography of Urine Prefractionated by Gel Chromatography.

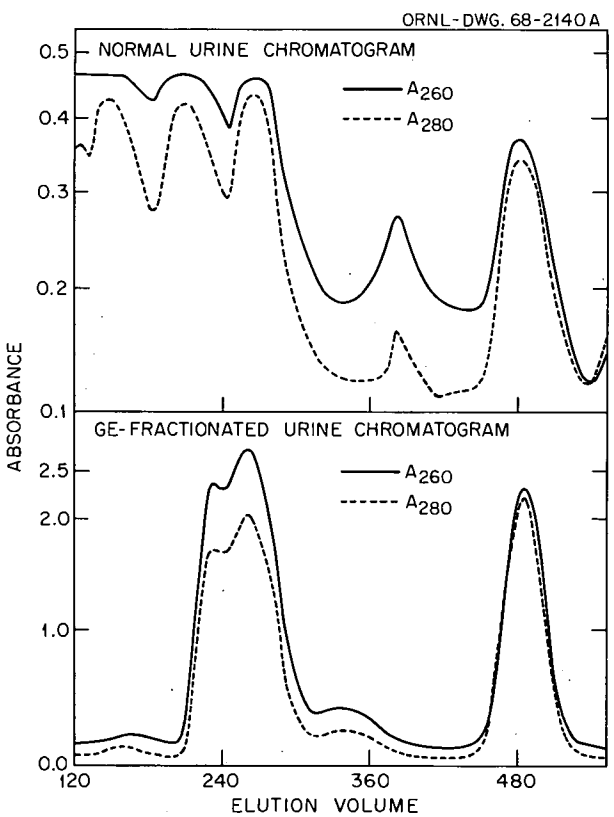

Fig. 4.16. Increased Resolution of a Urine Anion-Exchange Chromatogram by Prefractionation by Gel Chromatography.

spectral analysis. We are presently attempting to analyze these resolved peaks by routine spectrometric analysis.

A paper on this work was presented orally;[25] a manuscript was submitted for publication.[26]

4.3.d Gas Chromatographic Analysis of Fractions from the Anion-Exchange Chromatography of Urine

W. C. Butts

The recently acquired Micro Tek MT 220 gas chromatograph (GC) will be used to identify fractions obtained from the anion-exchange chromatographic separation of urine. A catalog of GC retention data[27] has been compiled from the literature to aid in this identification. The approach to the gas chromatographic analysis of the urine fractions will involve desalting (Sect. 4.3.e), drying, and derivatizing the fraction prior to injecting it into the chromatograph. Fractions eluting from the GC will be collected for mass spectral analysis.

4.3.e Desalting of Fractions from the Anion-Exchange Chromatography of Urine

W. C. Butts

Initial studies indicate that derivatization which renders the urinary constituents suitable for gas chromatographic analysis cannot be performed in the presence of the large amount of sodium acetate present in the acetic acid–sodium acetate buffer used to elute the fractions from the anion-exchange column. Similar amounts of sodium chloride also interfere. With a nonpolar solvent such as hexane, solvent extraction of the aqueous fraction and of the residue left after lyophilization of the fraction gives negligible extraction of the acetate as well as of the urinary component. Polar solvents such as methanol extract both acetate and the urinary constituents. Aromatic and other UV-absorbing solvents are unsuitable, because UV monitoring is used as a method of detection. A compromise solvent, isopropanol, gives the most suitable results.

Lyophilization of the column fraction removes the acetic acid of the buffer and leaves the sodium acetate. Titration of the fraction with hydrochloric acid to convert the sodium acetate to acetic acid followed by lyophilization removes most of the acetate and leaves a residue of sodium chloride and the urinary compound. Most of the urinary compounds can be extracted into isopropanol from the sodium chloride residue. Refinements in this technique are being made.

Preliminary experiments indicate that ammonium acetate can be removed by lyophilization without additional treatment. Further study may warrant the use of ammonium acetate in place of sodium acetate in the anion-exchange buffer.

Several fractions desalted by the HCl titration–lyophilization technique have been submitted for mass spectral analysis. The spectra indicate that the samples are contaminated and/or are mixtures and that additional separation (e.g., by GC) is necessary.

[25] C. A. Burtis, "Fractionation of Urine by Gel Filtration Chromatography," presented at the 20th National Meeting, American Association of Clinical Chemists, Washington, D.C., Aug. 18–23, 1968.

[26] C. A. Burtis, "Fractionation of Human Urine by Gel Chromatography," submitted for publication to *Clinica Chimica Acta.*

[27] W. C. Butts, *Catalog of Gas Chromatographic Retention Data for Urinary Constituents and Related Compounds,* ORNL-TM-2374 (May 13, 1968).

4.3.f Gel Electrophoresis of Proteins

A. L. Travaglini

Study of polyacrylamide gel electrophoresis[28] was continued. Protein samples were separated in both basic and acidic buffers. The effects of the presence and absence of stacking gels, incorporation of samples into gels, and addition of samples in solution were studied. In general, the sharpest separations seemed to occur when the sample was added in a gel on top of a stacking gel. Various stains were studied for detecting the positions of the proteins; nigrosine seems to be the most sensitive. Procion Blue, a new dye, appears to have promise. It stains by reacting with the –NH– group in peptides; therefore, the intensity of the stain color is proportional to the concentration of protein. More study of the effects of buffers, such as citrate, borate, phosphate, etc., on the separation of proteins is needed.

Many samples of papain-solubilized hamster tumor induced by adeno 31 virus were run for E. L. Candler, Jr.,[29] and J. L. Jainchill.[30] The results were used by them to locate the fraction of sample that contained T (or tumor) antigen. They then successfully isolated the tumor antigen.

4.3.g Centriphoresis

A. L. Travaglini

The initial work with centriphoresis was described earlier.[31] Every serum protein sample tried has been separated into three or four fractions, usually four, when centriphoresed. Albumen has been identified as part of the fastest-moving fraction and globulins as part of the slowest-moving fraction. Components of the other fractions have not yet been identified. Centriphoresis appears to be a convenient way to separate albumen from globulins in serum samples.

Before more studies with new buffers are done, a new rotor is needed that has provision for cooling the rotor and its contents and a greatly enlarged compartment for the electrode, buffer, and sample. Heating of the sample and the resulting increase in diffusion has been the only problem encountered during use of this method.

[28] A. L. Travaglini, "Electrophoresis of Proteins," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967,* ORNL-4196, p. 51.

[29] Molecular Anatomy Program.

[30] Loanee to the Molecular Anatomy Program; Staff Associate, U.S. Public Health Service–National Institutes of Health.

[31] N. G. Anderson, A. L. Travaglini, L. C. Patrick, R. L. Jolley, and E. C. Candler, Jr., "Centriphoresis," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967,* ORNL-4196, p. 52.

4.3.h Amino Acid Sequence of Mouse Hemoglobin

J. E. Attrill R. A. Popp[32]

The sequence in which amino acids exist in proteins is of importance in studying biochemical evolution as well as the biochemistry of life. Each amino acid position in each protein can be considered to be a variable trait that has 20 potential levels of distinction. Because of the similarity of all mammalian hemoglobins, work has been undertaken to study genetic factors in hemoglobin with various strains of laboratory mice. The mouse was chosen, because it is easy to raise, grows rapidly, and is available in pure blood lines. Past work[33] has shown

[32] Biology Division.

[33] R. 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).

Table 4.7. Possible Amino-Acid Sequence of the β Chain of C57BL Mouse Hemoglobin

Peptide	Sequence
βT-1	^{1}*Val,* ^{2}*His,* ^{3}*Leu,* ^{4}*Thr,* ^{5}*Ala,* ^{6}*Glu,* ^{7}*Asp,* ^{8}*Lys*
βT-2	^{9}*Ser,* ^{10}*Ala,* ^{11}Val, ^{12}Gly, ^{13}Ala, ^{14}Leu, ^{15}Tyr, ^{16}Gly, ^{17}Lys
βT-3	^{18}*Val, Asn, Ala, Asp, Glu, Val, Gly, Gly, Glu, Ala, Leu, Gly, Arg*
βT-4	Leu, Leu, Val, Val, Tyr, Pro, Tyr, Thr, Glu, Arg
βT-5	*Tyr, Phe, Ser, Ser, Phe,* Gly, Asp, Leu, Ser, Ser, Ala, Asp, Ala, Ile, Met, Gly, Asn, Ala, Lys
βT-6	*Val, Lys*
βT-7	*Ala, His, Gly, Lys*
βT-8	*Lys*
βT-9	*Val, Ile, Thr, Ala, Phe, Ser, Asp, Gly, Leu, Asn, His, Leu, Asp, Ans, Leu, Lys*
βT-10	Gly, Ser, Phe, Ala, Thr, Leu, Ser, Glu, Leu, His, Cys, Asp, Lys
βT-11	Leu, His, Val, Asp, Pro, Glu, Asn, Phe, Arg
βT-12	Leu, Leu, Gly, Asn, Val, Leu, Val, Ile, Gly, Leu, Met, His, His, Ile, Gly, Lys
βT-13	Asp, Phe, Thr, Pro, Ala, Ala, Gly, Ala, Ala, Phe, Glu, Lys
βT-14	*Val, Val, Ala, Gly, Val, Ala, Ala,* ^{140}*Ala,* ^{141}*Leu,* ^{142}*Ala,* ^{143}*His,* ^{144}*Lys*
βT-15	^{145}*Tyr,* ^{146}*His*

the complete sequence of amino acids in the α chain of the hemoglobin of C57 Black, a common pure-bred strain of laboratory mouse. We are still working on the sequence of the β-chain hemoglobin of this same type of mouse; Table 4.7 shows a proposed sequence. The portions in italics indicate a proven sequence.

Procedures for sequencing these chains have been given already.[15] The changes that have been made to improve the procedures include using a longer pH gradient for separating the peptides (obtained by enzymatically splitting the chain) on a Dowex 50-X2 cation-exchange chromatographic column only. Previously, this had been done by means of a run through first a cation- and then an anion-exchange column. Also, improvements in the paper chromatographic separations of both peptides and individual amino acids in micromolar concentrations have been made by K. M. Hilse.[34]

Work with the α chain of another strain (C_3H) of laboratory mouse was completed; an article on this work will be published.

4.3.i Amino Acid Substitutions

J. E. Attrill K. M. Hilse[34]
R. A. Popp[32] W. S. McErwin[32]

A paper is being published[35] which gives evidence that gene duplication rather than translational variations is probably the basis for amino acid ambiguity in the α chain of mouse hemoglobin. The α chain of the hemoglobin of the C_3H type laboratory mouse has three positions that contain alternate forms of amino acids. Position number 25 of the chain may be glycine or valine, position 62 is isoleucine or valine, and position 68 may be serine or asparagine. The chromatographic characteristics of the tryptic-peptide-digestion fragments are altered depending on which amino acid is in each of these positions. Analysis of the two chromatographically separable forms of the tryptic peptide αT4, which include position 25 in the chain, indicates that 70% of this peptide contains valine and 30% contains glycine. Tryptic peptide αT9, when recovered, contained positions 62 and 68 in the α chain. Two forms of the peptide were also separated by paper chromatography. These two forms showed that when valine occurs in position 62, only asparagine is found in position 68 and that when isoleucine occupies position 62, only serine is present at position 68. Analytical results also indicated that the position 62^{Val}-68^{Asn} form is 30% and the 62^{Ile}-68^{Ser} form is 70% of the total αT9 peptide.

The finding of two forms of αT9 in the same ratios as the two forms of αT4 supports the theory that the amino acid duplexity at these three positions results from gene duplication rather than from translational ambiguity.

4.4 ASSISTANCE TO OTHERS

J. E. Attrill

Assistance has frequently been rendered to other groups in the Laboratory. For E. S. Rogers of the Biology Division, samples of protein from papillomas found on two varieties of rabbits were hydrolyzed and checked for amino acid content. These proteins contained the enzyme arginase. It was intended to show that these papillomas contained different types of arginase and to pinpoint these differences. Present results are still inconclusive because of the small amounts of samples available.

Laboratory and technical assistance has also been given to R. H. Stevens of the ORGDP Instruments and Controls Systems Development Department while a micro amino acid analyzer was being designed and built. Moderate success has been achieved on this program. A small high-temperature high-pressure reaction bath, which reduces ninhydrin reaction time from 15 to ~2 min, has been built and tested.

Also, I. B. Rubin of this Division was assisted in procuring and analyzing standards that have been used to develop a new ultrasensitive method for the determination of amino acids (Sect. 4.1.1).

Several types of paper and thin-layer chromatographic (TLC) procedures have been tested for possible use in determining phenylthiohydantoin derivatives of amino acids. This is the form in which amino acids will be removed sequentially from a protein by an automatic sequenator that is being built in connection with the Molecular Anatomy Program. Attempts to reproduce published work[36] in which concentrations of these compounds are determined rapidly by TLC have not been entirely successful.

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[34] Research Fellow in the Biology Division; from the Max-Planck Institute for Biochemistry, Munich, Germany.

[35] K. M. Hilse and R. A. Popp, "Gene Duplication as the Basis for Amino Acid Ambiguity in the α-Chain Polypeptides of Mouse Hemoglobins," accepted for publication in the *Proceedings of the National Academy of Sciences.*

[36] J. O. Jeppsson and J. Sjöquist, "Thin-Layer Chromatography of PTH Amino Acids," *Anal. Biochem.* 18, 264 (1967).

5. X-Ray and Optical Spectrochemistry

Cyrus Feldman

5.1 X-RAY SPECTROSCOPY

H. W. Dunn

Development work is in progress on a computer code for determining crystal lattice spacings and parameters directly from film or strip-chart measurements. It is necessary at present to supply Miller indices to obtain the appropriate lattice parameters, but it should be possible to eliminate this inconvenience.

Electron-probe studies of high-phosphorus (~10 wt % P) organic compounds have shown that sulfur at the 0.1-wt % level can be detected with 95% confidence in such compounds. The limit of detection is now governed by the inhomogeneity of the sample (chemical and/or physical) rather than by the ratio of peak to background counting rates.

5.2 BEAM SPLITTER FOR USE IN CALIBRATING SPECTROGRAPHIC EMULSIONS

Cyrus Feldman

On the basis of previous experience,[1] a beam splitter support was constructed that has only one degree of freedom (the vertical slope of the totally reflecting mirror) and can still accommodate replaceable partially transparent mirrors. Inconel-coated mirrors have been found to be superior to rhodium-coated mirrors with regard to achromaticity of the reflectance/transmittance ratio.

The optimum conditions for use of the beam splitter depend on the type of spectrograph with which it is used (Table 5.1). If the stigmatic position of the spectrograph is not at the slit, the height of the light source and the height of the opening in the Hartmann diaphragm must be limited to the degree necessary to prevent vignetting in or beyond the beam splitter.

5.3 CHEMICAL PRECONCENTRATION OF TRACE ELEMENTS IN BONE ASH

Cyrus Feldman F. S. Jones

5.3.a Reagent Purification

The formic, acetic, and especially the tannic acid used in the modified Mitchell-Scott procedure[2] are potential sources of trace-element contamination and must be

[1] C. Feldman, "Beam Splitter for Use in Emulsion Calibration," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967*, ORNL-4196, p. 55.

[2] C. Feldman and F. S. Jones, "Spectrographic Analysis of Tissue Ash," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967*, ORNL-4196, p. 56.

Table 5.1. Optimum Conditions for Use of Beam Splitter with Fully Stigmatic and Partially Stigmatic Spectrographs

Condition	Optimum Setting for	
	Fully Stigmatic[a] Spectrographs	Partially Stigmatic[b] Spectrographs
Location of beam splitter	Adjacent to slit	Adjacent to slit
Location of image-forming lens	Immediately in front of beam splitter	At stigmatic position of spectrograph[c]
Location of Hartmann diaphragm	At or close to slit	At stigmatic position of spectrograph[c]
Maximum height of opening in Hartmann diaphragm	Half the distance between the centers of the emergent beams	About 2 mm; limit set by onset of vignetting
Location of image of light source	First internal optical element of spectrograph (collimator or grating)	First internal optical element of spectrograph (collimator or grating)

[a] All prism spectrographs; the Ebert and Wadsworth (Jarrell-Ash Corp.) and Quantograph (Applied Research Lab., Inc.) grating mountings.

[b] The Eagle (Baird-Atomic, Inc.) and Abney (Applied Research Lab., Inc.) grating mounting.

[c] Specified by manufacturer.

purified before use. A preliminary precipitation with 8-hydroxyquinoline at pH 4.0, followed by extraction, was effective for removal of copper but would be either less successful or impractical for the removal of other contaminants. Use of a mixed-bed ion-exchange column (hydrogen-form Dowex 50W-X8 and chloride-form Dowex 1-X8) was the most generally effective of the procedures tested. Table 5.2 gives the results of the application of these two procedures to the purification of tannic acid.

The purified material was analyzed spectrographically by a lithium carbonate matrix–dc arc procedure. Since only 0.2 g of tannic acid is used in the Mitchell-Scott procedure, the quantity in micrograms of each impurity contributed by this reagent would be one-fifth the amount shown in Table 5.2. Similar results were obtained for other reagents used in the procedure.

The analysis of a total-process blank, which included all reagents and all manipulations, gave the results shown in Table 5.3.

In future development work, the recovery of trace elements added to the original bone ash will be measured.

Table 5.2. Impurities Remaining in Tannic Acid After Its Purification by the Mixed-Bed Ion-Exchange and the 8-Hydroxyquinoline Extraction Procedures

Impurity	Concentration (μg/g)	
	After Mixed-Bed Ion Exchange	After 8-Hydroxyquinoline Extraction
Ag	0.005	0.005
Al	0.5	5.2
B	0.3	1.6
Ba	<0.004	0.05
Ca	1.6	160
Cr	0.2	0.3
Cu	0.3	0.3
Fe	0.3	0.2
K	10	120
Mg	0.2	16
Mn	0.02	0.2
Mo	<0.03	0.03
Na	20	52
Ni	0.04	0.04
P	<6	12
Pb	0.04	0.26
Pt	0.08	<0.08
Sn	<0.04	0.26
Sr	0.02	0.12
Ti	0.1	0.2
V	<0.01	0.2
Zn	<1.5	12

Table 5.3. Composition of Complete Process Blank for Collection of Trace Impurities from Bone Ash (3-g Sample) by a Modified Mitchell-Scott Procedure

Impurity	Amount Present[a] (μg per gram of bone ash)
Ag	0.07
Ca	0.07
Cr	1.1
Cu	0.1
Fe	4
Mg	0.4
Mo	0.4
Na	14
Ni	0.9
Pb	0.1
Si	15
Ti	0.1
Zn	4
Zr	0.03

[a]The following elements were not detected, with the indicated limits of detection (μg/g):

0.001 to 0.01	Ba, Be, Er, Eu, Ge, Lu, Sc, Sr, V, Y, Yb
0.01 to 0.1	Au, Bi, Co, Dy, Ga, Gd, Hf, Ho, In, Nb, Nd, Pd, Pr, Pt, Rh, Ru, Sm, Sn, Tb, Th, Tm
0.1 to 1	As, Cd, Ce, Ir, Re, Ta, Te, U, W

5.3.b Removal of Phosphate

Tests showed that a two-compartment electrodialysis cell that contains an Ionac MA-3475XL anion-permeable membrane is more efficient than a three-compartment cell in removing phosphate from a sample solution. High currents give fast phosphate removal, but resistance heating of the solution limits the current permissible. Since the use of such a cell involves the possibility of losing trace elements by deposition on the cathode (Pt wire), a way was sought to strip the cathode. Chemical stripping is effective only when aqua regia is used; and the use of this agent inevitably adds platinum to the sample. Graphite cathodes are satisfactory and can be wet-ashed to give complete recovery of trace elements. However, the wet-ashing is slow and considerably increases the danger of accidental contamination. Rhodium metal is more satisfactory than either platinum or graphite, since it functions well as a cathode and is not affected by the aqua regia stripping process. Tracer tests showed that at least 94% of the copper that is plated onto such a cathode is stripped off with aqua regia.

5.3.c Possible Loss of Cesium During Ashing of Bone

To check on the possible loss of cesium during the ashing of bone at 450°C, a sample of bone meal was treated with ^{137}Cs in dilute H_3PO_4 solution and was ashed. The ash was dissolved in nitric acid. Essentially all the ^{137}Cs was recovered.

5.4 MISCELLANEOUS TRACE DETERMINATIONS INVOLVING CHEMICAL PRECONCENTRATION

Cyrus Feldman N. Marion Ferguson

5.4.a Determination of Traces of Rare-Earth Elements in Bismuth (Carrier-Free Procedure)

In the reprocessing of used MSR fuel by molten-bismuth extraction, it is essential to know the degree to which fission-product rare-earth elements are extracted into the bismuth. A method was therefore developed for determining the rare-earth elements in bismuth at the ppm concentration level. The bismuth sample is dissolved in ~2 *M* HNO_3, and the pH of the solution is adjusted to 1.0 with NH_4OH. The solution is then warmed. Quantitative precipitation of coarse-grained $BiO_2(OH)NO_3$ results. The precipitate is centrifuged off and dissolved in 2 *M* HNO_3, and the precipitation is repeated. The supernatant solutions are combined. Tracer studies show that rare-earth elements remain quantitatively in the supernatant solution. These elements can then be determined by flame photometry or emission spectroscopy.

5.4.b Determination of Cadmium in Plutonium

To ensure that nuclear-grade plutonium meets specifications, cadmium must be determined in PuO_2 at the 0- to 1-μg/g level. The procedure consists in fusing the PuO_2 with ammonium bisulfate,[3,4] removing the plutonium from solution by anion exchange, treating the cadmium-containing eluate with ammonium pyrrolidine dithiocarbamate at pH 4.5, and extracting the resulting cadmium complex into methyl isobutyl ketone. The organic phase is evaporated, wet-ashed, and analyzed by atomic absorption spectrometry. In 1-g samples of PuO_2, cadmium in concentrations as small as 0.1 μg/g can be measured routinely.

5.4.c Determination of Traces of Bismuth in LiF-BeF_2 Mixtures

Likewise of interest is the extent of contamination of molten LiF-BeF_2 by bismuth during the molten-bismuth extraction of the rare-earth elements. To determine the trace amounts of bismuth, the LiF-BeF_2 sample is dissolved in perchloric acid, and fluoride is eliminated by evaporating the solution to fumes. The residue is dissolved, and the solution is treated with 10 mg of iron as ferric chloride and brought to pH 4.5. Tracer experiments with ^{207}Bi show that the resulting ferric hydroxide precipitate carried bismuth quantitatively. The precipitate is then redissolved in 6 *N* HCl. The iron is extracted with methyl isobutyl ketone, and the bismuth in the residual solution is determined by atomic absorption spectrometry. The conveniently attainable absolute limit of measurement is 0.5 μg of Bi; for the 20-g samples analyzed, this limit corresponds to 0.025 g of Bi per gram of LiF-BeF_2.

5.5 DETERMINATION OF SILICON IN CURIUM OXIDE ($^{244}Cm_2O_3$)

Anna M. Yoakum Cyrus Feldman

5.5.a Previous Status of Problem

Many trace elements, including silicon, can be determined in curium oxide by the carrier distillation–spectrographic procedure with a reproducibility factor of ~2.[5] It is difficult to estimate the accuracy of this method, since meaningful chemical checks are often impossible, especially in the case of silicon. Additional uncertainty is introduced by the fact that the lack of spectrographically pure curium oxide forces the use of other materials (e.g., neptunium oxide) as the principal component of standards. Since almost any substantial difference in chemical or physical properties among samples (e.g., particle size, thermal history, homogeneity) can introduce bias into this type of analysis,[6] it appeared desirable to develop a method independent of carrier distillation for determining silicon in curium oxide.

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

[4] G. W. C. Milner, A. J. Wood, G. Weldrick, and G. Phillips, "The Determination of Plutonium in Refractory Materials by Electrometric Methods After Dissolution by Fusion with Ammonium Hydrogen Sulphate," *Analyst* 92, 239 (1967).

[5] "Curium Oxide: Trace Impurities, Spectrochemical, Carrier Distillation Method," Savannah River Plant Laboratory Manual Code: Imp/Curium Oxide-1, March 17, 1967.

[6] M. Buffereau, G. Crehange, S. Deniaud, and C. Leclainche, *Spectrochemical Analysis of Impurities in Plutonium and Its Compounds* (in French), Commissariat a l'Energie Atomique, Saclay, France, CEA-R-3051 (January 1967).

5.5.b New Method

The approach decided on was to dissolve the curium oxide in acid, remove curium and ionic impurities by electrodialysis through cation- and anion-permeable membranes, evaporate the residual solution or suspension to dryness, and spectrographically determine the silicon in the residue (dc arc, Li_2CO_3 matrix, argon-oxygen atmosphere). A three-compartment Lucite cell was built that has an Ionac MC-3470XL cation-permeable membrane, which separates the center (sample) compartment from the cathode compartment, and an Ionac MA-3148 anion-permeable membrane, which separates the sample compartment from the anode compartment. The capacity of each chamber is ~125 ml. To check the efficiency of the cell for removing the elements from the sample compartment, tests were then made with tracer ^{244}Cm and ^{152}Eu-^{154}Eu and with stable lithium. The tests showed that only 0.1% of either the ^{244}Cm or the ^{152}Eu-^{154}Eu originally placed in the sample compartment remained after 4.5 hr of electrodialysis at 2 A; Fig. 5.1 gives the rates of removal. Flame photometric tests showed that the 1-μg/ml concentration of lithium originally present had been reduced to <0.0002 μg/ml. Thus, the procedure seems to be effective for cations of various weights and structures.

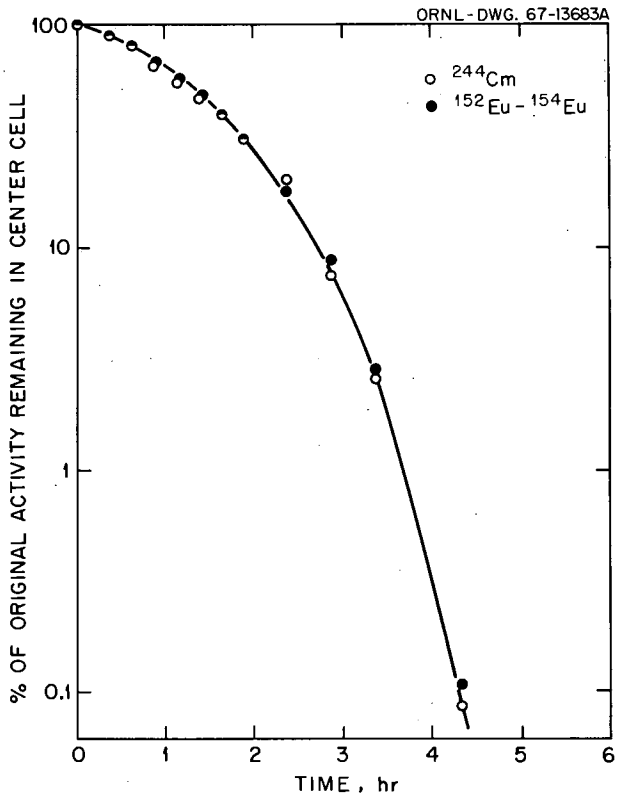

Fig. 5.1. Rates of Removal of ^{244}Cm and ^{152}Eu-^{154}Eu from 1 *N* HNO_3 by Electrodialysis Through Permselective Membranes.

5.5.c Recovery of Silicon

This method is based on the assumption that, regardless of their initial chemical states, all the silicon atoms originally present remain in the sample compartment, that is to say that any silicate radicals (SiO_3^{2-}) present are converted to colloidal particles of hydrated silicon dioxide, which, like the mineral forms of silicon, are incapable of penetrating either membrane. Recovery tests were therefore run on silicon introduced both as sodium silicate (40 μg of Si) and as powdered quartz (45 μg of Si). Recoveries were 101 ± 8% and 101 ± 6%, respectively.

5.5.d Blanks

In a combined chemical enrichment–spectrographic procedure, the useful limit of concentrational sensitivity is determined by the weight of sample represented in a single spectrographic exposure and by the total blank per exposure (from chemical treatment, atmospheric dust, electrode impurities). Test electrodialysis runs made in a conventional fume hood (linear velocity of air at face ~125 ft/min) showed that when the cell was not kept covered during electrodialysis, the absolute blank per exposure was 7.2 μg of Si. When the cell was kept covered, the blank was 1.5 ± 0.1 μg of Si. Thus, for the analysis of a 100-mg sample, the Si blank would be 1.5 μg. It is estimated that, if the absolute blank is reproducible to within ±0.1 μg, 0.3 to 0.5 μg of Si can be detected in a 100-mg sample at the 95% confidence level. The relative standard deviation of measurements of total Si is 5 to 10%.

5.5.e Radioactive Contaminants

Any constituent of the sample that, like silica, forms particles of colloidal size or larger under the experimental conditions or is present as an undissolved particle will accompany the silica to the spectrographic electrode. If this constitutent is radioactive, suitable safety precautions must be taken while making the spectrographic exposures.

6. Mass Spectrometry

A. E. Cameron

6.1 SPARK-SOURCE MASS SPECTROMETRY

J. R. Sites J. A. Carter

The solution spark-source technique described previously[1] was used to make a trace characterization analysis of ^{242}Pu ($t_{1/2}$ = 3.79 X 10^5 y). The sample electrodes were prepared for spark-source analysis in the Transuranium Research Laboratory by drying 5.5 μg of ^{242}Pu, which had been spiked with indium, on two gold electrodes. Twenty-one general-element contaminants in the range 0.001 to 0.6 wt % were found. In addition, transuranium isotopes of masses 240 (0.013 wt %) and 244 (0.009 wt %) were also present.

A set of 30 samples of ashed leaves from many different plants were analyzed to determine the distribution of the rare-earth elements in the leaves. A few milligrams of each sample was mixed with an equal weight of pure bismuth powder, and the mixture was pressed isostatically as enriched ends on bismuth-powder rods. The same was done for a set of 25 samples of ashed wood.

The trace elements in several samples of $CsGd(HFA)_4$ were determined (HFA = hexafluoroacetylacetonate). This compound is unusual in that it is a stable volatile compound of rare-earth elements.

Many samples of materials used to make target foils for cyclotron bombardment were analyzed for trace-element contaminants. These materials included the metallic forms of the following isotopically enriched elements: ^{24}Mg, ^{48}Ca, ^{50}Ti, ^{90}Zr, ^{142}Nd, ^{156}Gd, ^{168}Er, and ^{196}Pt. Metallic terbium and ytterbium were also analyzed.

For the Argonne National Laboratory, the following materials were analyzed for trace elements: a set of (Sc + Gd) samples, UB_2, UC, US_2, Cu-Mg, ZrO_2, Sc, Ho, and Er.

The high-neutron-cross-section contaminants in T-111 alloy (Ta-W-Hf) were analyzed for boron, cadmium, and the rare-earth elements.

6.2 LOW-RESOLUTION MASS SPECTROMETRY OF ORGANIC COMPOUNDS

W. T. Rainey

The low-resolution mass spectrometer, which was built for general-purpose scanning of organic spectra, has been in constant service during the past year. A wide variety of compounds has been run, both to collect reference spectra and to aid in the identification or elucidation of structure. Truxenes, which are polynuclear aromatic hydrocarbons, were investigated for the Y-12 Plant with useful results. The spectra of methyl and butyl phosphates were taken for the Chemical Technology Division. Some fluorinated alkyl phosphates are currently being scanned; attempt is being made to verify the positions of the fluorine atoms in an incompletely fluorinated tripentyl phosphate. These samples were submitted from a university, as were some organometallic compounds. Products from the radiation-induced oxidation of cyclohexane were examined.

Most of the work has been related to the Body Fluids Analyses Program (Sect. 4.3). Spectra have been recorded of about 20 steroids and steroid conjugates and of some 50 other compounds that are known to be constituents of urine. Efforts to identify compounds collected from the high-pressure chromatographic columns were not entirely successful because of the small sizes of the samples, presence of impurities, and difficulty of removing the buffer salt.

Although the low-resolution mass spectrometer was designed to be attached to a gas chromatograph, the only gas chromatographic samples that have been run were trapped as they emerged from a column not connected to the spectrometer. It now appears desirable to connect the two instruments.

6.3 SURFACE IONIZATION STUDIES

G. R. Hertel

6.3.a Surface Ionization of UF_4 at Various Oxygen Pressures

Studies of the emission of uranium and uranium oxide ions from hot tungsten ribbons[2,3] have been

[1] J. R. Sites and J. A. Carter, "Spark-Source Mass Spectrometry," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967*, ORNL-4196, p. 59.

[2] G. R. Hertel, "Surface Ionization. I. Desorption of U^+ Ions from W and Re Surfaces," *J. Chem. Phys.* 47, 133 (1967).

[3] G. R. Hertel, "Effect of Oxygen Contamination on the Surface Ionization of Uranium Tetrafluoride," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1966*, ORNL-4039, p. 48.

continued and extended; the results are interesting. The temperature dependence of the ion emission intensities (I's) and of the mean residence times (τ's) of the ions on the tungsten have been studied at oxygen pressures of 1×10^{-8}, 5×10^{-8}, 1×10^{-7}, and 3×10^{-7} torr. At a filament temperature of ~2500°K, U^+ emission decreased rapidly as the oxygen pressure was increased. The UO^+ and UO_2^+ emissions reached maxima at greater intensities and at higher temperatures than they did prior to the introduction of oxygen. In addition, a second UO_2^+ maximum with an intensity an order of magnitude greater than the first was observed at a lower temperature. Table 6.1 summarizes these data.

A study of τ with respect to temperature indicates the possibility of three distinct evaporation processes. The U^+ ion evaporation occurs at high temperatures with an evaporation energy (E_i) of 7.50 ± 0.09 eV.[2,3]

At temperatures below ~1470°K, the log $(1/\tau)$ vs $1/T$ plot of the data again approximates a straight line and thus indicates a dominant evaporation process with a characteristic energy. A least-squares fit of the data in this temperature region gave $E_i = 6.9 \pm 0.4$ eV. At these temperatures, UO_2^+ is the only ion detected and thus one may consider this E_i of 6.9 eV to be the E_i of UO_2^+ from a tungsten surface.

The log $1/\tau$ vs $1/T$ relationship has one other segment that approximates linearity. This segment is between ~1750 and ~2250°K. At an oxygen pressure of 5×10^{-8} torr, the E_i deduced from this region is 1.6 ± 0.1 eV. The UO_2^+ emission dominates in this temperature region when the atmosphere is oxygen, but no clear-cut assignment of this energy to any simple process can be made.

The behavior of the τ's and the I's in the mid-temperature region implies the existence of a complicated mixture or series of surface reactions. Further study is required.

6.3.b Ionization-Potential Measurements

G. R. Hertel D. H. Smith

The surface-ionization comparison technique[4] is being used to evaluate the first ionization potential of the actinide elements. A tentative value of 7.5 ± 0.3 eV has been determined for thorium.

6.4 MASS SPECTROMETRIC DATA REDUCTION

W. H. Christie H. S. McKown

In June an IBM 1130 computer was delivered and installed. The intention is to have this hard wired to several mass spectrometers to provide immediate calculation of the data, which are presently recorded on punched paper tape and calculated by the computing center at ORNL or ORGDP. Work is under way on the interfacing that is necessary to permit the multichannel analyzers to dump data direct to the computer. The high-resolution mass spectrometer will be connected in a way such as to accumulate data directly in the IBM 1130, since the storage in a 400-channel analyzer will be inadequate. The device has been programmed to handle most of the computational requirements of the digital recording instruments. Thus far, the operation has been very successful.

[4] G. R. Hertel, "Ionization Potential Measurements by Surface Ionization," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1966*, ORNL-4039, p. 49.

Table 6.1. Temperatures and Relative Intensities (I) of Oxide Ion Maxima

O_2 Pressure (torr)	UO^+		UO_2^+			
			1st Maximum		2d Maximum	
	Temperature (°K)	I	Temperature (°K)	I	Temperature (°K)	I
1×10^{-8}	2375	1.0[a]	1830	2.4	1435	3.8
5×10^{-8}	2500	1.5	1860	3.0	1490	~28
1×10^{-7}	2565	1.8	1900	3.6	1505	~26
3×10^{-7}	2625	2.3	1945	3.9	1525	~24

[a] All intensities normalized to this point.

7. Optical and Electron Microscopy

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

7.1 RESEARCH ASSISTANCE IN ELECTRON MICROSCOPY AND ELECTRON DIFFRACTION

Electron microscopy and electron diffraction have been used in a wide variety of fundamental and applied research to study materials of interest in nuclear technology and in other scientific disciplines at ORNL and Y-12.

7.1.a Lanthanide Hydroxide Sols

With both electron microscopy and electron diffraction, the hydroxide sols of the following lanthanide elements were studied: Ce, Dy, Gd, Ho, La, Lu, Sm, Tb, Tm, Yb, and Y. The metamorphosis to the crystalline stage was observed either as the material aged at 25°C or as it aged at 80°C after it had been heated initially to 80°C. It was learned that the lanthanide hydroxides differ greatly among themselves in the time necessary to change from an amorphous to a well-ordered state. Some crystallize after minutes, whereas others are still undergoing changes toward crystallinity after months of aging. Figure 7.1 shows that cerium hydroxide is almost completely crystallized after 70-min aging at 25°C. Figure 7.2 shows that after 99.7-day aging, ytterbium hydroxide appears to have changed from the amorphous state but is only weakly crystalline. The relative crystallinity of the two materials was checked by electron diffraction. It appears that the lanthanide hydroxide sols can be classified into tight subgroups strictly on the basis of aging characteristics. The effect of dilution and pH on the aging characteristics of certain lanthanide hydroxide sols was also studied.

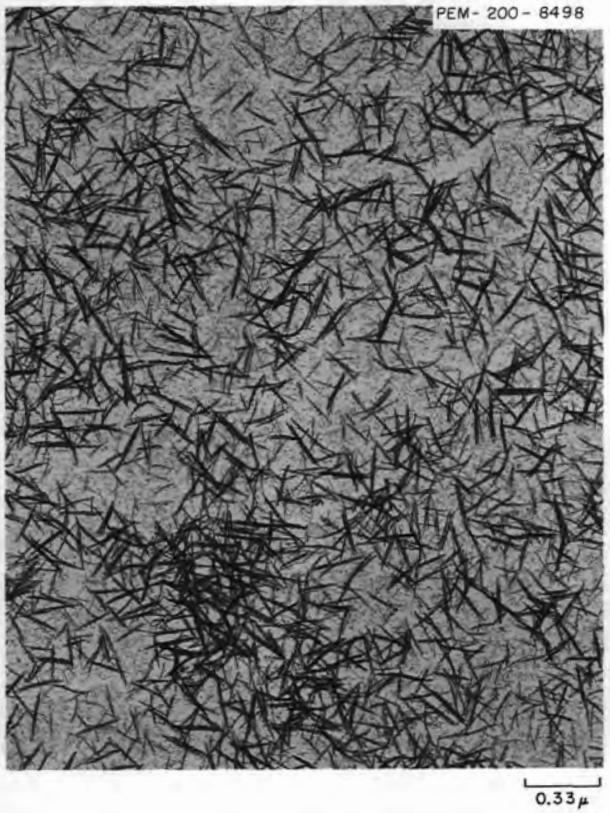

Fig. 7.1. Electron Micrograph of Cerium Hydroxide Aged 70 Minutes.

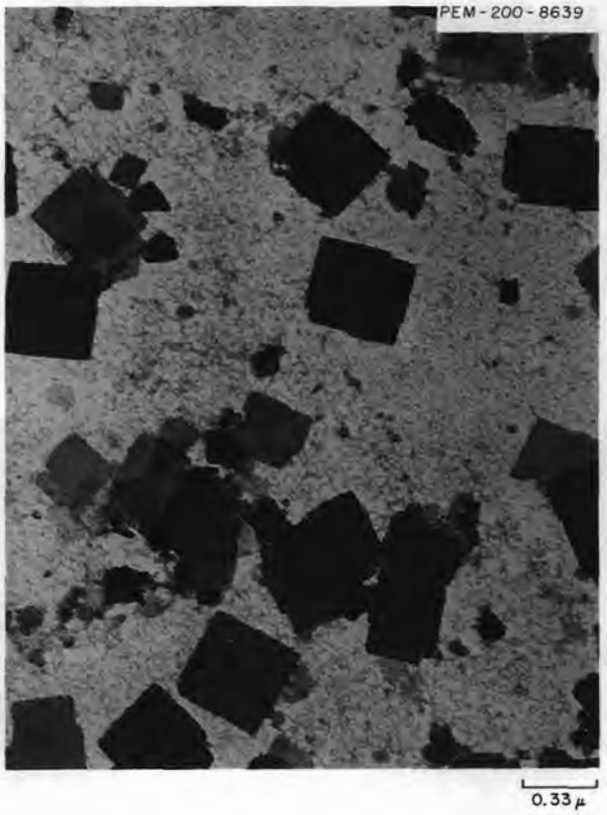

Fig. 7.2. Electron Micrograph of Ytterbium Hydroxide Aged 99.7 Days.

7.1.b Studies of Absolute Filters

An engineering group is studying the deterioration of absolute filters that occur when they are subjected to high temperatures. In cooperation with that group, changes in the filter structures were observed under the Philips EM 200 electron microscope at the extremely low magnification possible with it. Thin layers of filter material were stripped from the test samples by means of cellulose acetate tape and were sandwiched in "hinged" 50- to 100-mesh copper grids for electron microscopy. Figure 7.3 shows a section of the absolute filter before it was heated; Fig. 7.4 was taken after the filter was heated for 15 min at 400°C. The electron micrographs indicate that the physical breakdown of the filters is caused by burnup of the binder material followed by melting of the glass fibers of smallest diameter.

7.1.c Studies of Varcum

Varcum is a viscous derivative of furfuryl alcohol used to fabricate carbon-fiber sponge. Electron microscopy was used to determine what Varcum specifications are required to produce a good sponge. Large drops of the material, which was suspected to contain solids, were diluted with furfuryl alcohol, the solution was broken up in an ultrasonic generator and then diluted further with furfuryl alcohol. The solids were collected by centrifugation, and the remaining furfuryl alcohol was decanted. The solids were then redispersed in ethyl alcohol, and portions of the dispersion were deposited on carbon-substrated 200-mesh copper grids for electron microscopy. Figure 7.5 shows the rosette appearance of typical particulates common to both "good" and "bad" Varcum. Figure 7.6 shows the chains of spherical resinous particles found in "bad" but not in "good" Varcum.

7.1.d Other Research Assistance

Electron diffraction (both optical and electron) and microscopy were used to study many other materials of research interest. Among these were colloids in molten salts; oxide sols of zirconium, hafnium, titanium, and

Fig. 7.3. Electron Micrograph of Absolute Filter Before It Was Heated. *(Binder still present.)*

Fig. 7.4. Electron Micrograph of Absolute Filter After It Was Heated 15 Minutes at 400°C. *(Binder has disappeared.)*

Fig. 7.5. Electron Micrograph of Typical Particulates Found in Both "Good" and "Bad" Varcum.

Fig. 7.6. Electron Micrograph of Resinous Chains Found in "Bad" But Not in "Good" Varcum.

uranium; nitrides of uranium and boron; the oxidation products from Hastelloy N heated to 1800°F in air; tantalum oxide; uranium oxide–tungsten oxide powders; industrial graphite; natural carbon; and vaporized sodium chloride.

7.2 ELECTRON MICROSCOPY OF RADIOACTIVE MATERIALS

Conjointly with the Health Physics Division, we examined particles of $^{238}PuO_2$ in relation to NASA space experiments.

The presence of radioactive particulates in the vapor phase over the molten salts in the MSR was demonstrated. We suggested the method of collecting the particles, which proved to be satisfactory.

7.3 INSTRUMENTATION

An electron-beam device for the vacuum vaporization of metals and oxides has been installed to produce high-purity films for study and for use as substrates and in shadow-casting. A Philips 100 B electron microscope will be moved to Building 3019 to replace the Philips 100 A microscope used for the examination of radioactive materials. The Philips EM 300 electron microscope is being installed in the Transuranium Processing Plant. The purchase of a scanning electron microscope to meet a growing demand for such service has been requested.

8. Nuclear and Radiochemistry

W. S. Lyon
H. H. Ross

8.1 ACTIVATION ANALYSIS

8.1.a Air-Floating Sphere for Homogeneous Photon and Neutron Irradiation in Remote Facilities of Uneven Flux

Enzo Ricci T. H. Handley M. G. Willey[1]

Activation analysis and other nuclear techniques normally require comparative bombardment of two or more samples. For this purpose, irradiation in a homogeneous flux is mandatory. However, photon and neutron fields provided by conventional electron accelerators and neutron generators are uneven. To remedy this situation, a simple device was conceived and was tested in its preliminary stage. The device consists of an aluminum sphere that contains four sample capsules. The sphere can be transferred pneumatically to the bombardment chamber, where it is suspended in an upward stream of air which also causes it to spin around a precessing axis. Even if these rotations are not performed perfectly, the movement of the floating sphere is still sufficiently random to guarantee homogeneous flux. Tests were run in a 14-MeV neutron flux; four fluorine samples were activated for only 1 min by the reaction $^{19}F(n,2n)^{18}F$, 110 min. The S of the ^{18}F count rates of the samples was only 0.83%.

This device is unique in its simplicity of operation; the simple air-floating sphere replaces complicated mechanical equipment. The best previous systems consist of a pneumatic carrier and a dual-axis rotator.[2,3] Two requirements are inherent to their design: (1) transmission of movement to the rotator and (2) alignment of pneumatic pipes with the rotator after each irradiation to ensure safe retrieval of samples. Our system has none of these difficulties, because the sphere itself is the rotator. It is loaded and unloaded in a laboratory hood; it is carried to and from the irradiation chamber through the plumbing of the pneumatic transfer system; and, during bombardment, the same plumbing provides the supporting air cushion that also cools the sphere while imparting the double-axis motion to it. The metallic sphere does not suffer significant radiation damage. One extra advantage of the sphere is that, because of its shape, it can run more easily than the usual cylindrical rabbits through the pipes of the transfer system, thus allowing for sharper bends in the pipes and diminishing the danger of clogging.

The development of this system will continue.

8.1.b Photon and Fast-Neutron Activation Analysis at The Oak Ridge Electron Linear Accelerator (ORELA)

Enzo Ricci M. G. Willey[1]

Progress has been made in the design of the pneumatic rabbit facility for activation analysis to be installed at the ORELA (Building 6010). The preliminary plans for this facility[4] have been changed slightly. The tantalum target designed for physics experiments will also be used to produce photons and fast neutrons for activation analysis. A movable irradiation assembly has been designed that can be positioned at the back of the tantalum target for activation experiments. This same assembly will be used for irradiation with both photons and fast neutrons. When activation experiments are finished, the assembly will be withdrawn to prevent disturbance to physics work. The sample capsules will be located inside an aluminum ball (Sect. 8.1.a), which will travel through the piping to the irradiation site. After bombardment, the ball will be received in a lead enclosure, where it will be opened by remote manipulation to release the sample capsules. The lead enclosure will be installed in a hood to be built in the basement of the ORELA.

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[1] General Engineering Division.

[2] Model A-9104 Dual Axis Rotator, commercially available from Kaman Nuclear Corporation, 1700 Garden of the Gods Road, Colorado Springs, Colo. 80907.

[3] F. A. Lundgren and S. S. Nargolwalla, "Use of a Dual Sample-Biaxial Rotating Assembly with a Pneumatic Tube Transfer System for High Precision 14-MeV Neutron Activation Analysis," *Anal. Chem.* 40, 672 (1968).

[4] E. Ricci, "Photon and Fast Neutron Activation Analysis at the Oak Ridge Electron Linear Accelerator (ORELA)," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967,* ORNL-4196, p. 72.

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

Enzo Ricci T. H. Handley

The hot cell and pneumatic transfer system for use in activation analysis with ^{252}Cf spontaneous-fission neutrons has been completed at the Transuranium Research Laboratory (TRL);[5] also, a source of ^{252}Cf was fabricated by the Transuranium Processing Plant and installed inside the hot cell. Thus, it was possible to carry out the first experiments of this joint program of the Chemistry and Analytical Chemistry Divisions.

A ^{252}Cf neutron source has rather unique characteristics compared with nuclear reactors and neutron generators. It is small and free from power and maintenance requirements; therefore, it is portable. Also, it permits the use of either pure fission neutrons or a thermalized neutron spectrum and thus provides the versatility needed in nondestructive analysis. Consequently, we studied the potential use of ^{252}Cf sources in field (geological or space) exploration for nondestructive analyses at the percent level.

In view of this, our facilities are more versatile and complex than the apparatus that will be required when this technique is finally used in the field. The ^{252}Cf source is in a Teflon block (held by manipulator in Fig. 8.1). It may be transferred either to a dual-axis rotator for irradiations with fast neutrons (foreground) or to a high-density polyethylene cube for thermal- plus fast-neutron bombardments (background). A pneumatic transfer system shown schematically in Fig. 8.2 is used to deliver two samples at a time (unknown and

[5]E. Ricci and T. H. Handley, "Activation Analysis with a ^{252}Cf Source," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967,* ORNL-4196, p. 70.

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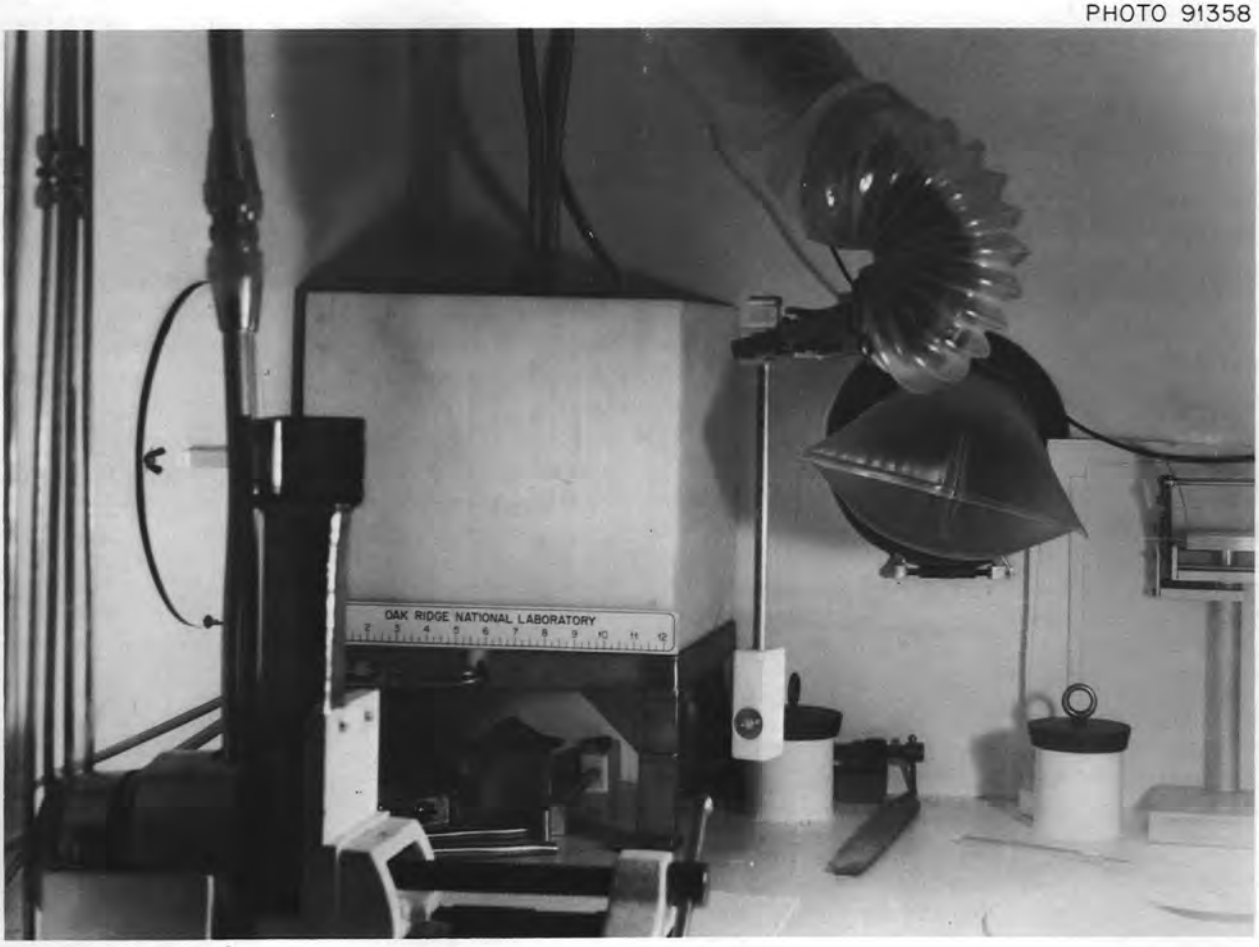

Fig. 8.1. View of the Inside of the ^{252}Cf Cell Through the Zinc Bromide Window.

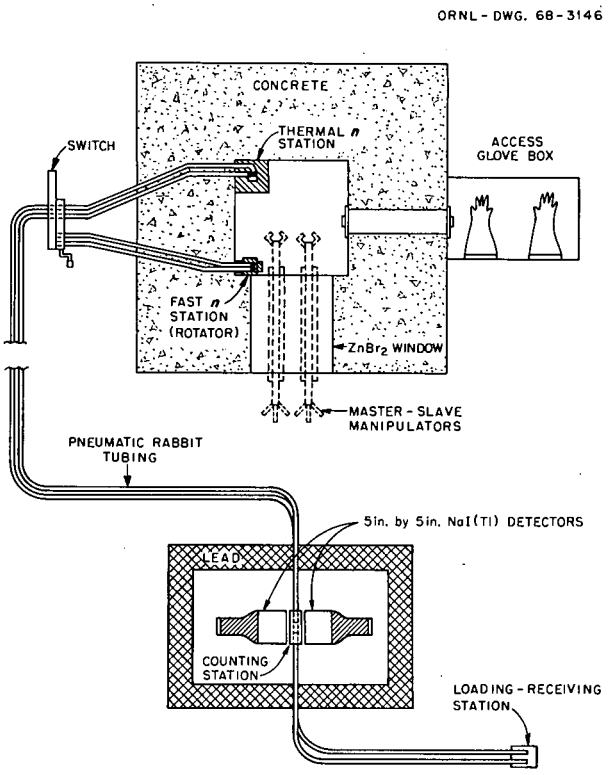

Fig. 8.2. Schematic View of the ^{252}Cf Neutron Activation Analysis Facility.

standard) to the cell; an external switch directs the samples to either of the two irradiation facilities. The same system can take the samples from the cell either to a receiving station or to a counting setup. The latter can hold one sample between two 5 in. by 5 in. NaI(Tl) detectors in an ~4π geometry.

We determined interference-free detection limits for activation analysis with fast and thermal neutrons for 12 elements of interest in geology and mining. By use of the reaction ^{27}Al$(n,\alpha)^{24}$Na, the neutron output of the ^{252}Cf source was found to be 8.6×10^8 neutrons/sec (±10%) on March 10, 1968. This output corresponds to 0.37 mg of ^{252}Cf. A cadmium ratio of 8.3 was obtained for the reaction ^{197}Au$(n,\gamma)^{198}$Au in the high-density polyethylene cube. Detection limits (mg) for a 1-mg ^{252}Cf source follow. Those for thermal neutrons are: Na, 1.2; Al, 0.68; Mn, 0.075; Ni, 170; Cu, 1.1; Zn, 83; Ag, 5.4; Pt, 86; and Au, 0.042. Those for fast neutrons are: Al, 13; Si, 2.3; Fe, 68; Zn, 68; and Pb, 1370. The errors of these results are about ±10 to ±15%.

Interesting conclusions can be readily drawn. First, it is clear that the elements studied can be determined at percent or lower concentrations in 0.1- to 1-g samples. Second, although thermal-neutron activation is generally very sensitive because of its higher cross sections, fast-neutron activation is a better choice in some cases. Therefore, it should be possible by selective activation with fast or thermal neutrons to minimize interferences in nondestructive analysis. Calculations based on the above results show that a 4-ton truck can carry the shielding necessary for a 3-mg ^{252}Cf source. Thus, a mobile unit for field exploration is envisaged that is capable of sensitivities better by a factor of 7 than that at which the above results were obtained.

8.1.d Influence of Channeling in Customary ^{3}He Activation Analysis

Enzo Ricci

The channeling of energetic charged particles in crystals has been known and studied since 1963.[6] This effect can be both beneficial and detrimental to charged-particle activation analysis of crystals. It was suggested recently[7] that a quantitative measure of the fraction of impurities located on different lattice rows or planes can be obtained by bombarding a crystal along different directions. On the other hand, a disadvantage of channeling is that it causes the particle range and the reaction cross section to vary in a rather complex fashion. Thus, when a crystal is irradiated in a channeling direction, the equations that govern charged-particle activation analysis[8,9] do not hold; the mathematical interpretation of the activation experiment then becomes involved and uncertain.

When channeling is used to locate impurities in a crystal lattice, the sample is usually fixed to a rather sophisticated remotely controlled goniometer that

[6] S. Datz, C. Erginsoy, G. Leibfried, and H. O. Lutz, "Motion of Energetic Particles in Crystals," pp. 129–188 in vol. 17 of *Annual Review of Nuclear Science*, ed. by E. Segre, Annual Reviews, Inc., Palo Alto, Calif., 1968.

[7] D. M. Holm, W. L. Briscoe, J. L. Parker, W. M. Sanders, and S. H. Parker, "The Determination of O and C in Ge by ^{3}He Activation," *Proceedings of the 2nd International Meeting on Practical Aspects of Activation Analysis with Charged Particles, Liege, Belgium, 1967*, EUR-3896d, f, e, p. 239.

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

[9] E. Ricci and R. L. Hahn, "Sensitivities for Activation Analysis of 15 Light Elements with 18-MeV Helium-3 Particles," *Anal. Chem.* 39, 794 (1967).

permits finding the channeling directions of the crystal by irradiation. Thus, determination of concentrations as low as ppb is ruled out, because high-intensity bombardments cannot be attempted without cooling the samples. To make cooling possible, the complexity of the goniometer must be increased beyond reasonable limits.

The objective of the ^{3}He activation program at ORNL is to achieve the outstanding potential sensitivity of this method. Therefore, the difficulties that channeling effects may cause in high-intensity customary ^{3}He activation analysis are of great importance to us and deserve investigation. On the other hand, if these effects were apparent in usual charged-particle activation, it might be possible to increase the sensitivity of the lattice-impurity-location techniques. Thus, experiments were carried out to study these points.

A single-crystal ingot of germanium (shaped like a margarine stick) was cut into numerous slices perpendicularly to its (111) axis. Each of these slices was bombarded at a different angle and counted. To ensure the validity of the results over a wide area in charged-particle activation, the sophistication of the experiment was planned to be greater than that of ordinary bombardments and such that observation of channeling effect could be expected. However, since activation and radioactivity measurements are standard in activation analysis, they were preferred to counting of prompt radiation. A simple goniometer accurate to ±0.1° was constructed to hold the samples during bombardment.

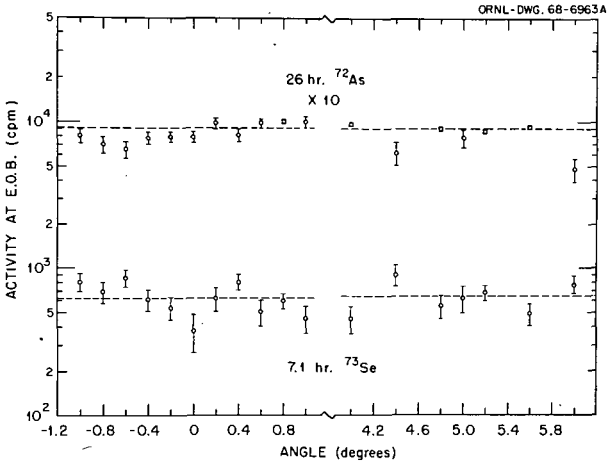

Fig. 8.3. Count-Rates of ^{72}As and ^{73}Se at End of Bombardment (E.O.B.) of Germanium Crystal vs Irradiation Angle. (Conditions: bombardment for 5 min with a 0.1-μA beam of 7.8-MeV ^{3}He particles; counting of gross gamma counts of sample on top of 1.3-cm lucite absorber lying on 3 in. by 3 in. NaI(Tl) detector.)

The alignment of the (111) axis of the sample with the ^{3}He beam was achieved by using a mirror mounted on the goniometer and an external autocollimator. The irradiations were performed at the ORIC with a reasonably parallel ^{3}He beam of 0.15 μA and 7.8 ± 0.2 MeV. The channeling critical angle for bombardment of germanium under these conditions is 0.56 ± 0.01°. Thus, angle intervals smaller than 0.5° were used in the irradiation of samples. A total of 18 samples were bombarded in sequence. Each was etched a short time before irradiation with conc HF-conc HNO_3 (1:2) for 1 to 3 min. The bombardment angles (beam with respect to (111) axis) were: −1.0, −0.8, −0.6, −0.4, −0.2, 0, 0.2, 0.4, 0.6, 0.8, 1.0, 4.0, 4.4, 4.8, 5.0, 5.2, 5.6, and 6.0°. The alignment was checked frequently with the mirror and autocollimator.

The nuclear reactions and products observed were: ^{70}Ge(^{3}He,pn)^{71}As, 62 hr; ^{70}Ge(^{3}He,p)^{72}As, 26 hr; and ^{72}Ge(^{3}He,$2n$)^{73}Se, 7.1 hr. A long-lived residual activity, probably due to 18-d ^{74}As, was also present, as well as 20.5-min ^{11}C and 110-min ^{18}F from carbon and oxygen impurities, respectively.[7] On the basis of these half-lives, least-squares decay-curve analysis of the gross gamma counts (to improve precision) was performed by using the CLSQ program in the IBM 360/75 computer.

Figures 8.3 and 8.4 are plots of germanium product radioactivities vs angle. The error marks correspond to the standard deviations that result from the computer treatment of the data. From the charged-particle activation equation,[8,9] the radioactivity induced in a

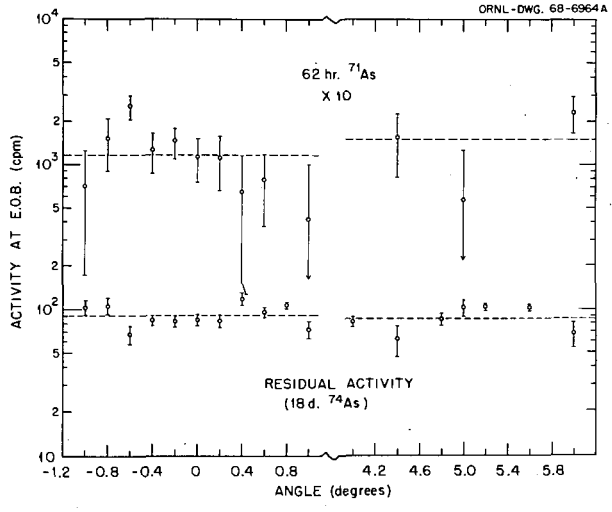

Fig. 8.4. Count-Rates of ^{71}As and Long-Lived Tail (^{74}As) at E.O.B. of Germanium Crystal vs Irradiation Angle. (Conditions: as for Fig. 8.3.)

thick target is proportional to the average cross section of the nuclear reaction involved and to the particle range. If any of these variables were influenced by channeling in these experiments, Figs. 8.3 and 8.4 would show structure, that is, departure from horizontal (constant). However, these graphs show that, within experimental error, the induced radioactivity is independent of irradiation angle. Despite the fact that this study was set up with a much higher degree of optical alignment than is customary in charged-particle activation analysis, its precision is not yet high enough for detecting channeling effects. Thus, we can safely conclude that such effects cannot generally influence the customary methods. This is true particularly because the critical channeling angle of 0.56° in these experiments is relatively broad in comparison with that corresponding to most activation bombardments. Moreover, since channeling effects cannot be observed in an experiment of medium sophistication as this one, it is apparent that fine studies of impurity locations by channeling[7] will have to rely on complex equipment for some time. Thus, no high-sensitivity determinations of this type (requiring high currents) may be feasible in the near future.

During these experiments valuable help was received from T. H. Handley and R. J. Fox.[10]

8.1.e Trace Elements in Drugs

L. C. Bate

A cooperative program is under way with the Bureau of Drug Abuse Control through the Department of Isotopes Development to determine the trace-element content of amphetamine and barbiturate drugs. Samples (115) of the drugs were prepared and irradiated in the Bulk Shielding Reactor for one week. The induced radioactivity was measured by both NaI(Tl) crystal and Ge(Li) diode spectrometry, and the decay was followed. The trace elements identified were Au, Br, Co, Cr, Cu, Fe, K, Na, Sc, Th (tentatively), and Zn. The data are being processed to obtain quantitative results.

8.1.f Neutron Activation Analysis of Brain Tissue for Copper and Manganese

E. D. Bird[11] S. B. Lupica
J. F. Emery W. S. Lyon

Previous studies have shown that the tranquilizing drug phenothiazine causes increase in the manganese content of the basal ganglia of the subhuman primate brain. This increase appears to be associated with an increase in the amount of pigment in the brain. Since this area of the brain has the propensity to concentrate a number of cations, it was considered important to measure other metals in the basal ganglia of primates after they were treated with phenothiazine. Copper as well as manganese is concentrated in biological pigmented tissues, and its clinical effects on the brain are similar to those of manganese. Therefore, it was decided to measure copper in addition to manganese in the various nuclei of the brain of our phenothiazine-treated primates.

Neutron activation analysis was chosen as the method for measurement, because the amount of tissues available for analysis is very small. The high concentrations and good neutron cross section of sodium and chlorine result in the production of 15-hr ^{24}Na and 37-min ^{38}Cl. The gamma-ray spectra of these nuclides obscure the peaks for ^{64}Cu and ^{56}Mn (Fig. 8.5). Girardi and Sabbioni[12] describe a radiochemical separation that uses antimony pentoxide to remove sodium from activated samples. We used the technique and were able to measure simultaneously both copper and manganese in our samples (Fig. 8.6). Table 8.1 summarizes the results.

A paper on this work will be presented orally.[13]

[10] Instrumentation and Controls Division.

[11] Medical Division, Oak Ridge Associated Universities; present address: Bowman Gray Medical School, Winston Salem, N.C.

[12] F. Girardi and E. Sabbioni, "Selective Removal of Radiosodium from Neutron-Activated Materials by Retention on Hydrated Antimony Pentoxide," *J. Radioanal. Chem.* 1, 169 (1968).

[13] E. D. Bird, J. F. Emery, S. B. Lupica, and W. S. Lyon, "Neutron Activation Analysis of Brain for Copper and Manganese," to be presented at the 1968 International Conference, Modern Trends in Activation Analysis, National Bureau of Standards, Gaithersburg, Md., Oct. 7–11, 1968.

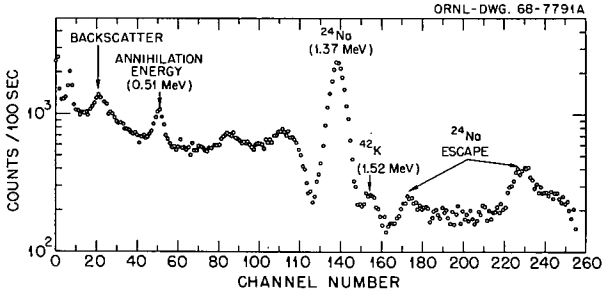

Fig. 8.5. Gamma-Ray Spectrum of Irradiated Brain Tissue from a Phenothiazine-Treated Subhuman Primate.

Table 8.1. Copper and Manganese in Brain Tissue of Phenothiazine-Treated Subhuman Primates

Tissue	Number of Samples	Treated		Control
		Cu[a] (μg/g)	Mn[a] (μg/g)	Mn[a] (μg/g)
Putamen	9	32.4 ± 5.4	2.05 ± 0.24	1.88 ± 0.31
Caudate	7	32.7 ± 3.7	2.05 ± 0.19	1.81 ± 0.08
Globus pallidus	8	25.2 ± 7.5	2.05 ± 0.28	2.09 ± 0.23

[a]Dry-weight basis.

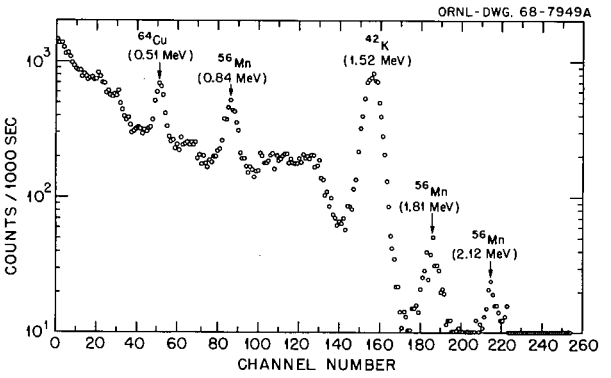

Fig. 8.6. Gamma-Ray Spectrum of Eluate from a Chromatographed (Sb_2O_5 Column) Solution of Sample for Fig. 8.5.

8.2 NEW INSTRUMENTS AND TECHNIQUES

8.2.a Analytical Applications of Cerenkov Radiation

H. H. Ross

Over the last year and a half, a number of papers have appeared in the open literature that describe the use of Cerenkov radiation for radioisotope assay. The technique is used primarily to measure energetic beta emitters. A close study of these papers reveals some divergence of experimental results and the lack of a clear physical model for Cerenkov systems.

To resolve some of these difficulties, a program was initiated to study in detail the Cerenkov process for beta-emitting nuclide assay. The initial phase of the program will involve a study of the experimental and theoretical variables that determine Cerenkov response. After these results have been collected, attempts will be made to rationalize the discrepancies noted in the literature. Also, an experimental physical model will be developed to enable investigators to optimize their measurements and to make reasonable estimates of the responses of nuclides not previously measured.

Preliminary experimentation has been completed. The relatively new Packard liquid-scintillation counter has been successfully interfaced with the Radiation Counter Laboratories multichannel analyzer. The sample-volume response of the detector system has been determined. As expected, the Cerenkov systems are very sensitive to sample volume. For example, ^{14}C samples of volume from 1 to 20 ml exhibit an ~5% variation in detection efficiency; ^{36}Cl Cerenkov detection varies by a factor of ~3. The shape of the experimental volume-response curve has already explained a questionable result in the literature. The shape of this curve can be developed theoretically by consideration of the detector geometry.

8.2.b Photovoltaic Conversion of Radioisotope-Decay Energy

H. H. Ross

The success of electrical power sources that use thermovoltaic conversion of radioisotope-decay energy has already been adequately demonstrated. These power sources have a number of advantages over chemical batteries; stability, reliability, and long life are notable examples. Recently, attention has turned to a consideration of other forms of conversion processes that can be used to exploit the decay energy of radioisotopes. Among the processes being evaluated, photovoltaic conversion appears to offer a reasonable alternate design concept for power sources with modest electrical capabilities.

Photovoltaic generation is, in fact, a two-step energy-conversion process. First, the radioisotope-decay energy must be transformed into photon radiation having an energy usually between 1 and 5 eV. This photon radiation is subsequently converted into electrical power via a secondary converting element (silicon or selenium cells).

We have completed our program to evaluate the primary and secondary conversion processes and to study the emission characteristics of phosphors and the factors that influence the fluorescent output of isotope-phosphor combinations. The results of this program can be used as the design basis for a radioisotopic photovoltaic converter and will permit reasonable calculations to be made on the expected efficiency of many converter systems. An open-literature publication that describes the results is being prepared for submission to the *International Journal of Applied Radiation and Isotopes.*

8.2.c Alpha-Radiation Damage in Inorganic Phosphors

H. H. Ross

A program to study the alpha-radiation damage in inorganic phosphors has been started at the request of an interested government agency. The objective of the program is to determine the intensity and spectral distribution of phosphors under continuous alpha bombardment as a function of time. Phosphor-regeneration techniques will also be evaluated. A specially designed measurement instrument was constructed and tested. Also, an oversize glove-box assembly has been designed and is being fabricated. The box will be used to prepare the isotope-phosphor sources and will house the majority of the detection and measurement equipment.

8.2.d Quantitative Electrodeposition of Actinides from Dimethyl Sulfoxide

T. H. Handley J. H. Cooper[14]

A simple, quantitative, and fast technique was developed for preparing sources for analytical alpha spectrometry by electrodeposition of radionuclides from dimethyl sulfoxide. In the presence of fluoride ion, americium(VI) is electrodeposited preferentially with respect to curium(III). A decontamination factor of several hundred was obtained. It was also observed that solutions of actinides in dimethyl sulfoxide low in mineral acids were stable for at least two months. The results of this work were submitted for publication.[15]

8.2.e A Study of Parameters That Affect Analytical Alpha Spectrometry

T. H. Handley J. H. Cooper[14]

Factors that affect alpha spectra were studied. They included uniformity and reproducibility of sources, counting conditions, and solids in sources. Standardization of these factors will be necessary before computers can be used to resolve alpha spectra. The results of this work will be presented at the forthcoming Gatlinburg Conference.[16]

8.2.f Low-Level Beta Detector

J. F. Emery

The Physics Division was interested in determining the resonance cross section of ^{208}Pb ($E_{\beta^-} = 0.6$ MeV, $t_{1/2} = 3.29$ hr). The expected disintegration rate was a few disintegrations per minute per gram of Pb. To detect beta decay of 0.6-MeV energy in a large mass of lead, the ratio of surface area to mass must be as large as possible. A beta detector was designed with a total surface area of 183 cm^2. Two 5-in.-diam multiplier phototubes were attached to 1-mm-thick plastic scintillators by means of a quartz light pipe. The scintillators were covered with 1.5-mil-thick aluminum foil. The detectors were mounted face to face. Any coincident counts were rejected, since they would be due to cosmic activity. The background of these detectors mounted in this manner is 40 counts/min or 0.22 count min^{-1} cm^{-2} of detector surface. The efficiency for ^{137}Cs is 50%.

8.2.g Liquid-Scintillation Counting of Weak-Beta Emitters in Aqueous Solution by Use of Triton X-100 in an Organic System

S. B. Lupica

The counting efficiency of weak-beta-emitting radionuclides can be improved markedly by the incorporation of Triton X-100, -102, or -114 into the organic scintillator system. Triton X is isooctylphenoxypolyethoxy ethanol that contains ethylene oxide (X-102, 12 to 13 moles; X-100, 10 moles; and X-114, 7 or 8 moles). It is a nonionic surfactant that improves

[14] High-Level Alpha Radiation Laboratory.

[15] T. H. Handley and J. H. Cooper, "Quantitative Electrodeposition of Actinides from Dimethyl Sulfoxide for Use in Analytical Alpha Spectrometry," submitted to *Analytical Chemistry.*

[16] J. H. Cooper and T. H. Handley, "A Study of Parameters Affecting Analytical Alpha Spectrometry," to be presented at the Twelfth Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Oct. 8–10, 1968.

the solubility of water in the scintillator mixture without causing serious quenching (no energy lost to ionization). As a result ^3H can be measured[17] in a 6.55-vol % water solution with 22% counting efficiency and in a 25-vol % water solution with 15% counting efficiency. To compare systems, a figure of merit (FM) is used; it is defined as the product of the volume percent water and the percent counting efficiency (e.g., 21.3 vol % water × 17.7% ^3H counting efficiency = 376 = FM).[17] Table 8.2 gives the compositions of the scintillator systems studied and summarizes the results.

More work will be done with solutions that contain <6.0 vol % water and less Triton X and with others that contain >20.0 vol % water; special attention will be given to the use of Triton X-114. Other systems that incorporate other nonionic surfactants will also be investigated.

8.2.h Low-Level-Radiation Counting Facility for Geochemical and Returned Lunar Samples

J. S. Eldridge P. R. Bell[18] V. A. McKay[10]
G. D. O'Kelley[19] R. E. Wintenberg[10]

Construction was completed on both the prototype low-level-radiation counting system at ORNL and the final underground system in the Lunar Receiving Laboratory (LRL) at the Manned Spacecraft Center, Houston, Texas. The detector assemblies were put together according to the design criteria described previously.[20] Experience gained with the prototype was valuable in making the final installation at the LRL.

Figure 8.7 is a photograph of the final assembly at ORNL. The exterior of the shield, visible in the photograph, is covered by the outer anticoincidence mantel. Figure 8.8 shows the system opened for insertion of a sample; the upper detector and end-plug assembly are visible. The top section of the shield, with the anticoincidence mantel attached, rolls forward on a rail system. That section weighs ~5000 lb and can be opened easily with one hand.

Table 8.2. Liquid-Scintillation Counting Efficiencies for ^3H and ^{14}C Achieved with Scintillator Systems That Contained Triton X

Scintillator systems: toluene (0.7 l)–Triton component (0.3 l)–PPO (5.5 g)–dimethyl POPOP (0.1 g)

Isotope	Amount of Activity (dis/min)	Triton Component	Water (vol %)	Counting Efficiency (%)	FM
^3H	4.79×10^5	X-102	6.55	21.50	143
			9.53	16.67	159
			13.60	18.07	246
		X-100	6.55	21.72	144
			9.53	17.25	164
			13.60	16.68	227
		X-114	6.55	21.43	143
			9.53	15.56	148
			13.60	17.65	240
^{14}C	2.985×10^3	X-102	11.78	79.30	934
		X-100	11.78	75.60	892
		X-114	11.78	78.00	920

PHOTO 90677

Fig. 8.7. Photograph of Exterior of ORNL Low-Level Counting Facility. (The outer anticoincidence mantle that covers all exterior surfaces is visible in this view.)

[17] S. B. Lupica, *Liquid Scintillation Counting of Weak Beta Emitters in Aqueous Solution Using Triton X-100,* Jan. 8, 1968 (unpublished).

[18] Chief of Lunar and Earth Sciences Division and Manager of the Lunar Receiving Laboratory, Manned Spacecraft Center, National Aeronautics and Space Administration, Houston, Tex.

[19] Chemistry Division.

[20] P. R. Bell, J. S. Eldridge, V. A. McKay, G. D. O'Kelley, and R. E. Wintenberg, "Low-Level-Radiation Counting Facility," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967,* ORNL-4196, p. 73.

PHOTO 90680

Fig. 8.8. View of Opened Shield and One of the Principal Detectors in the ORNL Low-Level Counting Facility. (The shield top-upper mantle assembly has been rolled forward for sample changing.)

Background measurements made with the prototype detectors in the ORNL shield were only slightly greater (<10%) than those made with the same detectors in the LRL shield when the integral of 0.1 to 2.0 MeV was chosen for comparison. However, in the LRL measurement, the capture gamma-ray structure at 2.2 MeV decreased significantly. An explanation for this phenomenon is that much of the neutron-producing component of the cosmic flux is removed by the overburden shielding in the LRL assembly.

Experience gained with the prototype detectors resulted in a new design concept for the final version of the detectors. Detector cans of 15-mil wall thickness were fabricated in the ORNL shops by a spinning technique that produces one-piece cans whose contributions to the background are controlled. Two detectors were fabricated from the new spun cans. Preliminary data show that the magnitude of the background from the prototype detectors will be reduced by a factor of ~2. Under less than optimum mantle conditions, both detectors showed integral (0.1 to 2.0 MeV) background count rates of 110 counts/min, or 0.02 count min^{-1} cm^{-3} NaI(Tl).

Final checks of the detector assembly and equipment are scheduled for the remainder of 1968. The initial calibration and the collections of response functions will begin early in 1969. Tentative plans call for the analysis of retrieved lunar samples in the LRL system in the final quarter of 1969.

8.3 NUCLEAR PROPERTIES OF RADIONUCLIDES

8.3.a Interlaboratory Comparison of Measurements of the Radionuclides ^{57}Co, ^{99}Mo-^{99m}Tc, and ^{99m}Tc

J. S. Eldridge

Previous interlaboratory comparisons of radionuclide measurements[21] were extended. The Subcommittee on the Uses of Radioactivity Standards[22] sponsored a test to compare concentration values determined at various laboratories for solutions of the radionuclides ^{57}Co, ^{99}Mo-^{99m}Tc, and ^{99m}Tc. R. C. Koch[23] organized the participation and sample distribution. Laboratories in the United States and Canada known to prepare or use these radionuclides were asked to participate and to report in detail their procedures for obtaining concentration values. Nineteen laboratories accepted the intercomparison samples, but only 13 submitted results. B. Kahn[24] compiled the results and made detailed statistical analyses. Two participants measured radionuclide concentrations absolutely by 4π beta-gamma counting. Kahn took the mean values to be the "best" values.

Both the Nuclear and Radiochemistry Group and the Radioisotopes-Radiochemistry Laboratory measured the samples received by ORNL; routine gamma spectrometric techniques were used. Table 8.3 gives the values reported compared with the best values.

Table 8.3. Results of Interlaboratory Comparison of Radionuclide Measurements – April 1968

Radionuclide	Concentration (μCi/g)		Deviation (%)
	"Best" Value	ORNL Value	
^{57}Co	18.26	17.7 ± 0.9	−3.1
^{99}Mo	24.02	23.6 ± 0.5	−1.7
^{99m}Tc	27.16	27.6 ± 0.8	+1.6

It is interesting to note that every participant measured all three radionuclides within 15% of the best value. However, these radionuclides have been in general use for several years, and standards have been available for them. This agreement is much better than that of the 1966 intercomparison of ^{197}Hg standardizations[25] in which 50% of the participants reported values that differed by >10% from the best values. Mercury-197 standards were not available at that time, and its decay scheme was in some doubt.

8.3.b New Nuclear Data

J. F. Emery

Gamma-Ray Energies. – Variations in the reported values of the 2.16-MeV gamma ray from the decay of ^{38}Cl (ref. 26) and the 2.11-MeV gamma ray from the decay of ^{116}In (ref. 27) prompted the remeasurement of these energies. A high-resolution Ge(Li) gamma-ray spectrometer was used that has an energy resolution of 3.74 keV for the 2.75392-Mev gamma ray of ^{24}Na. The energies of the single escape peak of this gamma ray and of the 2.61447-Mev gamma ray from ^{208}Tl were used as calibration sources; their respective energies are 2.24292 and 2.10347 MeV. These energies were selected so that the gamma-ray energies being measured would be bracketed closely by peaks of known energy. The energies determined for ^{38}Cl and ^{116}In are 2.1668 ± 0.0004 and 2.1122 ± 0.0004 MeV, respectively.

Thermal-Neutron Cross Sections. – The center hole in the east D_2O tank of the Bulk Shielding Reactor has a thermal-neutron flux of 1.2×10^{12} neutrons cm^{-2} sec^{-1}. This facility was used to determine the thermal-neutron cross sections for the ^{64}Ni$(n,\gamma)^{65}$Ni and ^{208}Pb$(n,\gamma)^{209}$Pb reactions. Dilute Au-Al alloy (0.100% Au) was used to measure the thermal-neutron flux.

The disintegration rate of ^{65}Ni was determined by NaI(Tl) gamma-ray spectrometry; the 1.48-MeV gamma ray of ^{65}Ni and a gamma ray per disintegration value of 0.25 were used. The thermal-neutron cross section for ^{64}Ni was determined to be 1.35 ± 0.1 barns.

The disintegration rate of ^{209}Pb was determined by a 4π beta counter. The neutron source on the 4π beta mount was 50 μg/cm^2. The thermal-neutron cross section of ^{208}Pb was determined to be 487 ± 30 microbarns.

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[21] J. S. Eldridge, "Standardization of ^{197}Hg," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967,* ORNL-4196, p. 67.

[22] Subcommittee on the Use of Radioactivity Standards, Committee on Nuclear Sciences, National Academy of Sciences–National Research Council, 2101 Constitution Ave., Washington, D.C. 20418.

[23] Member, Subcommittee on the Use of Radioactivity Standards.

[24] Chairman, Subcommittee on the Use of Radioactivity Standards.

[25] J. 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.

[26] J. Van Klinken, F. Pleiter, and H. T. Dijkstra, "Beta and Gamma-Ray Measurements on the Decay of ^{38}Cl," *Nucl. Phys.* A112, 372 (1968).

[27] C. M. Lederer, J. M. Hollander, and I. Perlman, *Table of Isotopes,* 6th ed., Wiley, New York, 1967.

8.3.c Applications of a Be-Window Ge(Li) Spectrometer System

J. F. Emery

A Be-window Ge(Li) x-ray detector has been found to be a very versatile instrument. This device has been loaned temporarily to us by ORTEC and Bowman Gray Medical School. The efficiency for x-ray energies up to ~100 keV is very good, whereas above ~100 keV the efficiency decreases rapidly. Because of this difference in efficiency, it is possible to measure low-intensity x rays or gamma rays in the presence of high-intensity high-energy gamma radioactivity.

By use of this system, it is possible to determine the highly converted 53-keV gamma ray (11%) of ^{73}As in the presence of ^{72}As, ^{74}As, and ^{76}As. An isotope product of ^{74}As was found to contain ^{73}As in 1-μCi/ml concentration, whereas the ^{72}As, ^{74}As, and ^{76}As contents were 2.1, 10, and 0.6 μCi/ml, respectively. The 53-keV gamma ray of ^{73}As could not be seen on an NaI(Tl) spectrometer.

Because of its high resolution, this detector is very useful for decay-scheme studies. For example, the 80-keV doublet associated with the decay of ^{133}Ba can be resolved (Fig. 8.9). The separation of these two lines is only 1.340 keV, and the intensity of the 79.65-keV gamma is only 8% of the intensity of the 80.99-keV gamma. The resolution of this system at 81 keV is 0.700 keV.

This x-ray spectrometer system was also used to study the gamma and x-ray spectra of ^{129}I (Sect. 8.6.d). The x rays plus gamma ray appear as a single peak on an NaI(Tl) spectrometer, but they are easily resolved on this Ge(Li) x-ray spectrometer.

The calibration of the efficiency of this detector involved a number of sources. Low-energy gamma-ray intensities of ^{241}Am, ^{243}Am, and ^{237}Np can be calculated from these data. These will be determined at a later date.

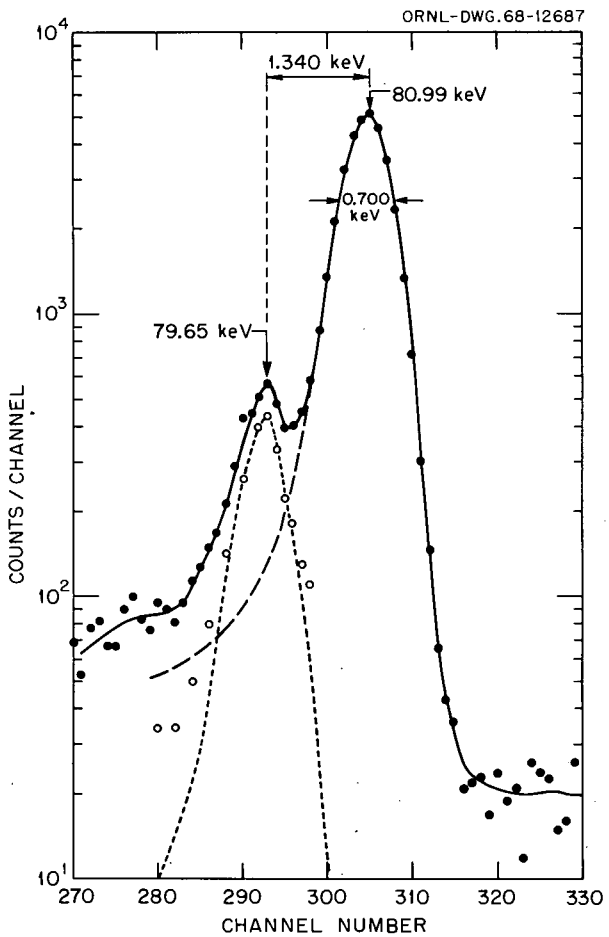

Fig. 8.9. Resolution of the 80-keV Doublet from the Decay of ^{133}Ba with the Be-Window Ge(Li) X-Ray Detector.

8.3.d Nuclear Spectroscopy in the Neutron-Deficient $173 \leqslant A \leqslant 185$ Region

T. H. Handley Ben Harmatz[28]

The internal-conversion spectra of deformed nuclei in the mass interval $173 \leqslant A \leqslant 185$ were investigated with permanent-magnet spectrographs. By combining the conversion results with supplementary data from independent photon studies, partial decay schemes are deduced from energy sums, multipolarities, and intensities of the transitions. The quantum numbers of nuclear-energy levels are associated with intrinsic and collective modes of excitation in the framework of the Nilsson and Bohr-Mottelson models. Parameters relevant to rotational configurations and branching ratios of the de-exciting transitions are evaluated. The energy systematics of proton and neutron particle states in odd-A nuclei is correlated with the particle-coupled spectra in adjacent doubly odd nuclei. More complete conversion-electron measurements permit extension of nuclear-level structure of ^{148}Sm and ^{146}Sm. The results of this work were submitted for publication.[29]

[28] Electronuclear Division.

[29] B. Harmatz and T. H. Handley, "Nuclear Spectroscopy in the Neutron-Deficient $173 \leqslant A \leqslant 185$ Region," submitted to *Nuclear Physics*.

8.3.e Decay of ^{103}Pd-^{103m}Rh

Marcia A. Lepri[30] W. S. Lyon

An article on the decay of ^{103}Pd-^{103m}Rh has been submitted to the *Journal of Nuclear Medicine.* A summary follows:

A radiochemical extraction was used to separate ^{103}Pd from ^{103m}Rh and ^{103}Rh. The decay of ^{103}Pd directly to the ^{103m}Rh state appears to be 100%. A new value was determined for the K conversion coefficient of the 40-keV gamma ray of ^{103m}Rh ($1.8 \pm 0.2 \times 10^2$), and the (K_α/K_β) ratio for rhenium K x rays (4.8 ± 0.2) was redetermined.

8.3.f Gamma-Ray Intensities in the Decay of ^{133}Ba

J. F. Emery Marcia A. Lepri[30]

The large variation (52 to 74%) in the reported values for the intensity of the 356-keV gamma ray from the decay of ^{133}Ba prompted the investigation of the decay scheme of ^{133}Ba. A high-resolution Ge(Li) spectrometer was used to measure the intensities of 100-keV and higher gamma energies; a Be-window Ge(Li) detector was used for the other gamma rays of energy below 100 keV (Sect. 8.3.c).

The relative intensities and the absolute percentage of all known gamma-ray transitions were determined. All the gamma-ray intensities were corrected for internal conversion by use of the values from Notea and Gurfinkel.[31] From the gamma-ray-transition intensities, the electron-capture branching to the 81- and 161-keV levels in ^{133}Cs can be calculated. The absolute intensities of the 356-keV gamma ray is highly dependent on any branching to these levels. The calculated electron-capture branching to these levels is negative; the limits are ~±2%. Since two Ge(Li) detectors were used at different counting geometries, efficiency curves, and calibrations, the errors were somewhat greater than they should be. The ideal system for obtaining the entire gamma-ray spectrum at one time is the Be-window Ge(Li) detector. Use of this detector would require a 4000-channel analyzer; the analyzer available for this work has only 400 channels. Table 8.4 summarizes the work.

[30]ORAU Student Trainee from Mt. Holyoke College, South Hadley, Mass.

[31]A. Notea and Y. Gurfinkel, "Transitions in ^{133}Cs From the Decay of ^{133}Ba," *Nucl. Phys.* A107, 193 (1968).

Table 8.4. Intensities of Gamma Rays from the Decay of ^{133}Ba

E_γ (keV)	Intensity	
	Relative[a]	Absolute[b] (%)
53.4	3.25	2.04
79.6	4.5	2.82
81.0	56.3	35.2
79.6 + 81.0	60.8	38.0
161	0.72	0.45
223	0.63	0.39
277	11.4	7.14
303	29.0	17.8
356	100	62.6
384	14.4	9.0

[a]Relative to the 356-keV gamma ray.

[b]Based on 100 disintegrations.

8.4 APPLICATIONS TO REACTOR PROGRAMS

8.4.a Precise Determination of ^{235}U

J. F. Emery

Since the original study of the determination of ^{235}U by delayed-neutron counting,[32] a need has arisen to improve the accuracy and precision of the method for determining ^{235}U in MSRE fuel salt. This method is also applicable to the determination of ^{233}U.

Three innovations in the technique resulted in the improvement of the precision by a factor of 2. These were increasing: (1) the pressure of the air that propels the rabbit from 10 to 20 psi, a change that resulted in a more reproducible decay time; (2) the duration of the irradiation from 60 to 120 sec; and (3) the decay time from 20 to 25 sec. Under these new conditions, the total count is somewhat less dependent on variations in the decay time. The S for sets of samples was decreased from 0.5 to 0.3%, whereas the S for successive irradiations of a single sample improved from 0.3 to 0.16%.

The lack of capability of the electronics of this system to handle extremely high count rates is still the one single barrier to improving the accuracy of this method. A new current-sensitive preamplifier has been developed by the Instrumentation and Controls Division that holds good promise.

[32]F. F. Dyer, J. F. Emery, and G. W. Ledicotte, *A Comprehensive Study of the Neutron Activation Analysis of Uranium by Delayed-Neutron Counting,* ORNL-3342 (Oct. 2, 1962).

8.4.b Proton Reaction Determination of Lithium and Fluorine in Molten-Salt-Reactor Graphite

T. H. Handley J. H. Gibbons[33]
R. L. Macklin[33] Enzo Ricci
D. R. Cuneo[34]

Prompt radiations from the reactions $^{7}Li(p,n)^{7}Be$ and $^{19}F(p,\alpha\gamma)\ ^{16}O$ have been used to measure concentrations of the target nuclides ^{7}Li and ^{19}F in graphite; the ORNL 3-MeV Van de Graaff accelerator was the proton source. A sample irradiated for nine months in the MSRE shows more than a hundred times the lithium and fluorine penetration found in an unirradiated control sample. The results of this work have been accepted for publication in *Nuclear Applications.*[35]

8.4.c Measurement by Ge(Li) Gamma Spectrometry of Fission Products Deposited in Components of the MSRE

F. F. Dyer Robert Blumberg[34] T. H. Mauney[36]

An effort was made to determine qualitatively and quantitatively the gamma-emitting fission products that deposit on the components (principally the heat exchanger) of the MSRE which came in contact with the fuel salt. This information is needed to estimate the heat generated by the fission products after reactor shutdown and for purposes of remote maintenance. Also, there is interest in the fission-product distribution to determine whether correlations exist between the extent of deposition and such factors as temperature gradients, molten-salt chemical effects, and half-lives of fission-product precursors. In addition, there is a specific interest in determining whether ^{95}Zr deposits out.

The fission products were measured by gamma-ray spectrometry with a highly collimated Ge(Li) diode. The collimator consisted of a lead cylinder (19 in. diam, 33.5 in. length). The diode, in a small Dewar flask, was placed in a cavity in the top of the shield over a collimator hole (1/8 in. diam, 12 in. length). By means of the collimator, the detector could be focused on an area (~3 in. diam) on the reactor components (a source-to-detector distance of 15 ft). Pulse-height spectra were taken with a 400-channel analyzer. Measurements were begun on March 28, 1968 (5 days after reactor shutdown), and were continued at intervals for 12 days.

The fission products found in major amounts in the heat exchanger and fuel-salt-input and -output lines of the heat exchanger were ^{95}Nb, ^{99}Mo, ^{103}Ru, ^{132}I, and ^{132}Te. Minor amounts of ^{106}Ru and ^{140}Ba-^{140}La were found. The fission products detected in the pump bowl and off-gas line included those listed above and also ^{131}I, ^{137}Cs, and ^{141}Ce. No ^{95}Zr was detected. The amount of each fission product deposited on the heat exchanger increased slightly from the fuel-salt entry point over about the first foot of heat exchanger and then decreased over the next 5 ft. Table 8.5 presents the results. The value given in the last column for each fission product was obtained by multiplying the value in the second column by the total metal surface area in the MSRE and dividing the resulting value by the total curies of that fission product in the MSRE at reactor shutdown.

Table 8.5. Major Fission Products Found in MSRE Heat Exchanger

Fission Product	Mean Amount Deposited (Ci/in.^{2})[a]	Fission Product Deposited on All Metal Surfaces of the MSRE (% of total in MSRE)[b]
^{95}Nb	1.9	58
^{99}Mo	4.9	102
^{103}Ru	1.1	64
^{132}Te-^{132}I	2.2	66

[a]Values pertain to heat exchanger only.
[b]Values based on inventories of fission products calculated by E. L. Compere[36] and on total metal surface area in contact with the fuel salt as estimated by R. Thoma.[36]

8.4.d In-Reactor Penetration of Fission Products into MSRE and Pyrolytic Graphite

L. C. Bate F. F. Dyer

The Reactor Chemistry Division is being assisted in a study of in-reactor penetration of fission products into MSRE and pyrolytic graphite. Cylindrical specimens were obtained by core drilling from blocks of graphite removed from the reactor in the spring of 1967 and in March 1968. These specimens were sanded repeatedly to obtain samples on emery paper as a function of depth in the graphite. The thickness of graphite represented by the samples was varied from ~0.001 in.

[33]Physics Division.
[34]Reactor Division.
[35]T. H. Handley, R. L. Macklin, J. H. Gibbons, E. Ricci, and D. R. Cuneo, "Proton Reaction Determination of Lithium and Fluorine in Molten-Salt Reactor Graphite," accepted for publication in *Nuclear Applications.*
[36]Reactor Chemistry Division.

near the surface of the specimen to ~0.015 in. at a depth of ~0.5 in.

The first series of samples was measured by NaI(Tl) spectrometry, the others by Ge(Li) diode spectrometry. The improved resolution of the Ge(Li) diode permitted measurement of ^{95}Nb, ^{95}Zr, ^{103}Ru, ^{106}Ru, ^{131}I, ^{132}Te, ^{137}Cs, ^{140}Ba, ^{140}La, ^{141}Ce, and ^{144}Ce. Resolution of ^{141}Ce and ^{144}Ce, ^{103}Ru and ^{106}Ru, and ^{95}Zr-^{95}Nb was not possible by NaI(Tl) spectrometry. It was observed that the concentrations of fission products decreased rapidly with distance into the graphite. A significant finding was that for most samples the ratio of the disintegration rate of ^{95}Nb to ^{95}Zr was about 10. Since the samples had decayed for several months, this fact indicates that ^{95}Nb penetrates graphite much more readily than does ^{95}Zr. The surface sample of the pyrolytic graphite was very radioactive. However, at greater depths, the pyrolytic graphite contained fission products in considerably lower concentrations than did MSRE graphite. Also, ^{140}Ba-^{140}La was not detected in the pyrolytic graphite.

8.4.e Ge(Li) Diode Gamma-Spectrometry System

F. F. Dyer L. C. Bate M. T. Morgan[36]

A 10-cc-volume Ge(Li) diode was obtained for high-resolution gamma-ray spectrometry. The detector is coupled to a Tennelec 135M low-noise preamplifier, which is coupled to a Tennelec 200 amplifier. A Tennelec 250 biased amplifier is used to establish the energy range of pulse-height spectra accumulated with a 400-channel analyzer. The data output from the analyzer included punched paper tape, printed spectrum, and both logarithmic and linear plots. The system resolution with the Ge(Li) detector is 2.5 keV for the 1.33-MeV gamma ray of ^{60}Co. The photopeak counting efficiency for ^{137}Cs at a source-to-detector distance of 10 cm is 1.1×10^{-4} count per emitted photon compared with an efficiency of 7.0×10^{-3} count per emitted photon for a 3- by 3-in. NaI(Tl) detector. Spectral analysis has been accomplished mainly by means of the data-reduction facilities incorporated in the analyzer. The system has been used primarily for analysis of fission-product mixtures (Sects. 8.4.d and 8.4.f).

8.4.f Fission-Product Release from Reactor-Fuel Microspheres

L. C. Bate F. F. Dyer

The study of fission-product release from reactor-fuel microspheres is a cooperative program with the Reactor Chemistry Division. The microspheres are coated with graphite or silicon carbide. As the result of irradiation and fuel burnup, the coating may crack and release fission products.

To measure the fission-product release under conditions that simulate in-reactor failure, microspheres are irradiated to ~15% burnup and allowed to decay for several months. The ^{95}Zr is then determined with a Ge(Li) diode to measure the percent burnup that occurred during the long irradiation. These data, along with the irradiation history of the sample, are used to calculate the ^{235}U concentration in the original microspheres. The microspheres are then reirradiated for 6 hr to form measurable amounts of ^{131}I and ^{140}Ba-^{140}La; the measured values are also used to calculate the ^{235}U concentration. Next, the coatings of the microspheres are cracked, and the microspheres are placed in an induction-furnace assembly. The furnace temperature for each run is 1100 to 1500°C. After 2, 8, 14, and 20 hr of heating, the microspheres and furnace parts are removed, and the ^{131}I and ^{137}Cs radioactivities are measured to determine the rate and total amount of fission products released. The ^{131}I is determined directly by Ge(Li) diode spectrometry and also indirectly by calculation from the ^{140}Ba-^{140}La (1.60-MeV gamma peak) radioactivity measured by NaI(Tl) gamma spectrometry. The ^{137}Cs is measured by NaI(Tl) spectrometry. The release rates of ^{137}I and ^{137}Cs increase as the temperature is increased; they are much higher above 1400°C.

Several of the microsphere coatings were removed prior to heating, in which cases the coating and the core were heated separately. It appears that the release rate is higher from the coating, although fission-product concentration of the coating is about one hundredth that of the core.

8.5 14-MeV NEUTRON GENERATOR

8.5.a 14-MeV Neutron Generator Studies

J. E. Strain

Determination of Oxygen by 14-MeV Neutron Activation. – The facility designed for the determination of oxygen in alkali and refractory metals has operated routinely during the past year. Samples of lithium, potassium, and calcium have been successfully analyzed for oxygen in concentrations as low as 19 ppm. Interlaboratory samples of lithium have been analyzed to compare results obtained by vacuum distillation, thermal-neutron activation by the $^{16}O(^{3}H,p)^{18}F$ reaction, and 14-MeV neutron activation. Work is con-

tinuing on the comparison of the determination of oxygen in potassium by 14-MeV neutron activation and by amalgamation. The determination of oxygen in sodium by 14-MeV neutron activation is still under investigation as time permits.

A large number of refractory metals (V, Nb, Ta, Nb-1Zr, Ni, stainless steel, etc.) have been analyzed by means of standard additions. Oxygen is added in the form of Mylar tape, of which oxygen is a component. The oxygen content of the tape is known from prior analysis, and the total oxygen added is determined by weighing the tape. After the metal samples are activated and counted, a layer of the tape is applied to the surface of the sample, which is then reanalyzed. This method of standard addition permits the analysis of specimens that have irregular shapes.

Metal coupons used to test the compatibility of refractory metals with alkali metals are of a uniform size. For these samples, standards are prepared by adding oxygen at elevated temperatures to form the oxides. The amount of oxygen added is determined from the weight gain of the specimen. A series of standard coupons of Nb, V, and Ta have been prepared whose oxygen contents range from 40 to >2000 ppm.

8.5.b Determination of Fluorine in Fluorspar by 14-MeV Neutron Activation Analysis

F. F. Dyer L. C. Bate

At the request of the U.S. Bureau of Customs, the fluorine content of 15 samples of fluorspar ore was measured by 14-MeV neutron activation analysis. The method of analysis was described previously.[37] Duplicate samples of each ore were analyzed. The average of the results, converted to percent CaF_2 in the samples, was 1.06% higher than results based on a wet-chemistry method that were reported by the U.S. Bureau of Customs. This difference has not yet been explained.

8.5.c Neutron Generator Target-Development Program

J. E. Strain

In an effort to produce a tritium target for neutron generators that will produce neutrons at a constant rate, a new concept in a self-regenerating target is under study. The target contains a reservoir of gaseous $^3H_2$ that diffuses into the target face as a function of target temperature. A constant amount of available tritium is thereby maintained. This target is now being fabricated and should be available soon.

Conventional targets of titanium tritide and erbium tritide are being analyzed routinely to determine the effects of fabrication techniques on their neutron yield and useful life.

8.5.d 14-MeV Neutron Reaction Research

J. E. Strain

The Texas Nuclear Corporation model 9900 and the Kaman Nuclear Corporation model 1250 neutron generators have been used in a variety of experiments to produce unusual radionuclides via the $(n,2n)$, (n,γ), and (n,p) reactions. Nearly all the experiments have been conducted in cooperation with members of the Physics, Health Physics, and Chemistry Divisions and with ORAU students. The Kaman neutron generator is capable of producing a flux of 14.7-MeV neutrons of 4×10^9 neutrons cm^{-2} sec^{-1}; the flux has a half-life of 2550 μA hr.

8.6 COOPERATIVE ISOTOPES PROGRAM

8.6.a Technical Assistance

S. A. Reynolds

The joint program[38] of the Isotopes and Analytical Chemistry Divisions in analysis and characterization of isotopes has continued. Salient features of the program are discussed either below or in cross-referenced sections of this report. The general program of the Isotopes Division is given in another report.[39]

8.6.b Quality Control

S. A. Reynolds

The quality-control program will take into account standards for radiopharmaceutical raw materials. The standards are under development by the Food and Drug Administration (FDA) with local cooperation. The anticipated FDA requirement for controls on raw radioisotope materials – in addition to those on the radiopharmaceuticals themselves – reflects an interesting general attitude of users that isotopes should not

[37] L. C. Bate and F. F. Dyer, "Fluorine Determination by 14-MeV Neutron Activation Analysis," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967*, ORNL-4196, p. 80.

[38] "Cooperative Isotopes Program," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967*, ORNL-4196, pp. 81–86.

[39] J. H. Gillette, *Review of Radioisotopes Program – 1967*, ORNL-4329 (in press, 1968).

contain *any* impurities. Expectations for radioisotopes are higher than those for chemical reagents, medicinals, and foods, for example. Some radioisotope users want *assurance* of purity rather than the customary guarantee of replacement or adjustment if specifications are not met. Examples of quality-control studies appear in the following paragraphs and in Sect. 8.6.c.

On $Ba^{14}CO_3$ products, a discrepancy developed between isotopic-abundance values by radioactivity measurement and by mass spectrometry. One such occurrence in the past was caused by deterioration of a ^{14}C standard, with resultant low radioassay. Current tests using the standard addition of National Bureau of Standards hexadecane-^{14}C indicated that the radioassays are correct. Another past occurrence was believed to have been caused by $Ba(OH)_2$ coprecipitation, since CO_2 percentages were shown to be low; process changes apparently eliminated the difficulty. The recent difficulty was found by emission spectrography to be caused by the presence of silicate material (glass and possibly barium silicate). Such inert material does not affect normal uses of the product. Sales have been properly based on the measured radioactivity in curies per gram of solid. In connection with this study, radioactivity and mass-spectral data were reviewed. Calculations of the half-life of ^{14}C were made on 19 products that were as old as 18 years. The average half-life was 5776 years, with a standard error of 32 years. The half-life of the four oldest products averaged 5811 ± 70 years; this value was originally reported as ~5800 years.[40] The half-life of four recent products in which carbonate had been determined chemically averaged 5736 ± 56 years. All these values are in reasonable agreement with the accepted "Cambridge average" of 5730 ± 40 years; this agreement indicates that local radiochemical and mass spectral analyses are reliable.

A plan was developed for analyzing solutions of typical ^{170}Tm heat-source wafers. The ^{170}Tm is assayed by end-window beta counting and ^{171}Tm by liquid-scintillation counting. Trace gamma-emitting components such as ^{168}Tm are determined by gamma spectrometry, with the 0.084-MeV gamma of ^{170}Tm removed by an interposed gold or platinum absorber (~1-g/cm^2) or the equivalent, ~3 g/cm^2 of lead. Emission spectrographic analysis is used to estimate ytterbium and other inactive components, and mass spectrometry serves to check the thulium radioassays. The latter is especially useful for ^{171}Tm, since no standard is available.

A SNAP source container was surveyed for possible contamination with sea water or chromate by swabbing the container with dilute HNO_3, followed by activation analysis of the washings of the swabs for chlorine and chromium. No significant amount of chromium was found, and the highest level of chlorine corresponded to 0.003 ml of sea water (it was probably the chlorine from a fingerprint).

To evaluate a soil before heat-transfer studies, classical chemical and petrographic analyses appeared sufficient, as in a previous study.[41]

At the request of the Health Physics Division, a routine method for tritium in water was reviewed. It consists in distillation, followed by liquid-scintillation counting. It was confirmed that the method is reliable in that the presence of other beta emitters would be detected by the instrument and that satisfactory tritiated-water standards are available. Further, in connection with a review for the American Public Health Association of a procedure for tritium in water, doubt was expressed of the necessity of distilling *all* of a water sample as prescribed. A cursory review of the literature gave evidence that if *half* the sample were distilled, the bias of the measurement due to isotopic fractionation would be <2.5%, and probably <1%. The *S* of a radiochemical method for iodine based on solvent extraction (ASTM method D 2334, Part C) was found to be 2% when the ^{131}I activity was sufficient to keep random counting error below 1%.

8.6.c Radioactivity Standards

S. A. Reynolds J. E. Morton[42] W. T. Mullins[42]

Local measurements of radioactivity standards from reliable suppliers have continued and agree adequately with those of the suppliers, that is, usually within their estimates of the uncertainties in calibrations. On the interlaboratory comparison of measurements of ^{57}Co, ^{99}Mo-^{99m}Tc, and ^{99m}Tc (Sect. 8.3.a), the results from the Radioisotopes-Radiochemistry Laboratory were compared with the "best" values. The average results were 0.6, 0.6, and 1.4% lower than the accepted values for ^{99}Mo-^{99m}Tc, ^{99m}Tc, and ^{57}Co, respectively.

Information on radioactivity standards is maintained current by membership on the National Academy of Sciences–National Research Council Subcommittee on the Use of Radioactivity Standards and by receiving

[40] S. A. Reynolds and E. I. Wyatt, *Measurement of Activity and Isotopic Abundance of Carbon-14 in Barium Carbonate,* ORNL CF-53-10-104 (Oct. 16, 1953).

[41] J. J. Dell'Amico, F. K. Captain, and S. H. Chansky, *Characterization and Thermal Conductivities of Some Samples of Conasauga Shale,* ORNL-MIT-20 (Mar. 29, 1967).

[42] Radioisotopes-Radiochemistry Laboratory.

publications and catalogs from other standards organizations. A survey[43] of the availability of and the criteria for standards was published. The criteria are essentially those covered in recent monographs.[44,45]

8.6.d Characterization of Isotope Materials

S. A. Reynolds J. F. Emery

Experimental work on characteristics such as half-lives, decay schemes, and cross sections of isotope materials continues as needed, and our compilation of such properties is constantly updated. Some of the information acquired will appear in the *Isotope Users' Guide,* which is to be published by the Isotopes Information Center. This information has proven useful in local work in the Isotopes Development Center and Analytical and Health Physics Divisions and in our contributions to national organizations such as the ASTM. Some of the work is summarized below and in Sect. 8.6.e.

Work by W. S. Lyon and Marcia A. Lepri (Sect. 8.3.e) and recent publications by others permit the following estimates for radiations from ^{103m}Rh: gamma, 0.07%; K x ray, 8%. The weighted average value for the half-life of ^{59}Fe is 44.6 days, in agreement with our value of 44.3 ± 0.4 days, which we once thought was wrong. The previous estimate of 0.06% for the intensity of the 0.022-MeV gamma ray of ^{151}Sm was confirmed by new data. A mass of discordant data on ^{133}Ba has been published. The local work (Sect. 8.3.f) is definitive and should eliminate the major uncertainty in the assay and application of this nuclide.

Work on the characterization of ^{129}I is almost complete; modern techniques have resulted in the removal of past inconsistencies. The percentage of activity carried by AgI was found to be >99.9%, and the principal chemical components in typical products were determined to be iodine and sodium, as expected. The chemical analyses for total iodine were confirmed by using isotope dilution with known amounts of stable ^{127}I, followed by mass spectrometry. Agreement was within 2 to 5%, and the S of the isotope-dilution analysis was ~2%. The ^{129}I abundance in the products was 85.3 to 85.7%. The spectrum of gamma and x rays (Fig. 8.10) was taken by means of a Be-window Ge(Li) detector (Sect. 8.3.c). The K-conversion coefficient of the 40-keV gamma ray was calculated to be 10.6, and the gamma intensity 7.3%, assuming theoretical ratios of conversion in other shells. Work remains to be done on measurement of specific activity. Preliminary data indicate that the accepted half-life of 1.6×10^7 years will be approximately confirmed.

A typical ^{130}I product was analyzed in detail. Impurities in very low levels (0.2% total) were found; these included ^{126}I, ^{131}I, ^{122}Sb, ^{124}Sb, and ^{197}Hg.

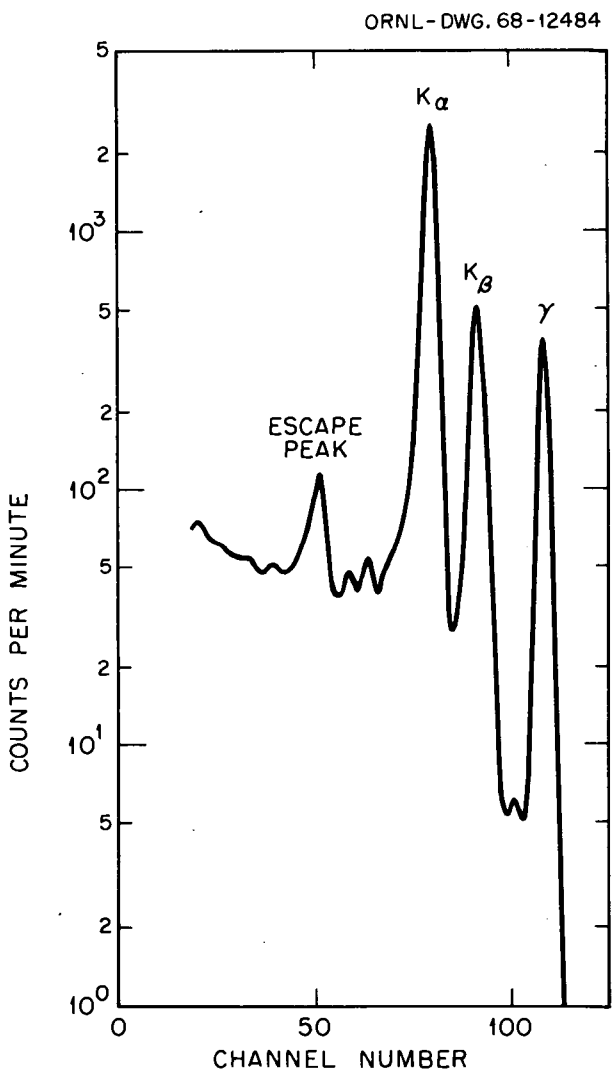

Fig. 8.10. Ge(Li) Spectrum of ^{129}I.

[43] S. A. Reynolds, "Availability of Standards for Common Radionuclides," *Isotopes Radiation Technol.* 52, 118 (1968).

[44] B. Kahn, G. R. Choppin, and J. G. V. Taylor, *Users' Guides for Radioactivity Standards,* NP-17502, National Academy of Sciences–National Research Council, Washington, D.C., 1967.

[45] S. B. Garfinkel, A. P. Baerg, and P. E. Zigman, *Certificates of Radioactivity Standards,* National Academy of Sciences–National Research Council, Washington, D.C., 1966. See also *Certification of Standardized Radioactive Sources,* ICRU-12 (Sept. 15, 1968).

The specific activity and the chemical and radionuclidic purity of ^{91}Y were studied. The specific activity, based on radiochemical and spectrographic analysis, was found to be ~2.3 × 10^4 Ci/g (~95% of theoretical) as expected for fission-product ^{91}Y. Traces of calcium and aluminum were found; the total solids (110°C) and nonvolatile matter were 0.08 and 0.03%, respectively.

Our estimate of 30% 0.54-MeV gamma radiation in ^{140}Ba, which is the same as that of ASTM Committee E-10, was confirmed by measurements of that radiation from freshly separated barium, followed by measurement of the well-known 1.60-MeV gamma in the ^{140}La daughter subsequently formed. In the ^{170}Tm program, the heat output of Tm_2O_3 wafers will be measured calorimetrically. The theoretical heat production was therefore computed. The value was 1.98 W/kCi, derived from the average beta energy, 0.315 MeV, plus 0.020 MeV per disintegration for radiations associated with the gamma transition. A similar calculation was made for ^{147}Pm, based on the firm adoption by the ORNL Nuclear Data Group of 0.0620 MeV as its average energy. The corresponding heat output is 0.367 W/kCi or 0.341 W/g.

Rubidium-84 is produced by reactor irradiation of enriched ^{84}Sr; the reaction is $^{84}Sr(n,p)^{84}Rb$. A typical product was analyzed for ^{84}Rb by NaI(Tl) gamma spectrometry, strontium by radiochemical separation, and others by Ge(Li) spectrometry. The observed intensity of the 1.90-MeV gamma in ^{84}Rb was ~0.86% in agreement with 0.8% in *Nuclear Data Sheets,* but the intensity of the 1.02-MeV gamma was 0.3% vs 0.5%. The ^{86}Rb content was 3%, about as expected from the (n,p) cross sections of ^{84}Sr and ^{86}Sr. Rubidium-83 was 0.7%, over twice as high as expected from the path $^{84}Sr(n,2n)^{83}Sr \xrightarrow[\beta+]{EC} {}^{83}Rb$, probably because of substantial direct yield by $^{84}Sr(n,np)^{83}Rb$. The maximum amount of ^{85}Sr was <0.002%. On the assumption that no separation of rubidium from cesium occurs in chemical processing, the cesium content of the enriched ^{84}Sr target was estimated to be <2 ppm, from the absence of ^{134}Cs in Ge(Li) spectra. The only nonisotopic radionuclidic impurity detected was a trace amount of ^{60}Co.

8.6.e Half-Life Measurements

S. A. Reynolds E. I. Wyatt[42] J. F. Emery

Half-life values for 36 radionuclides were published,[46] and values for ~30 more will be included in a paper to be prepared jointly with G. I. Gleason[47] for submission to *Nuclear Science and Engineering.* Details of the measurements for ^{99m}Tc are given here to illustrate the techniques of determination, data treatment, and selection and use of values from the literature. Portions of the ^{99m}Tc interlaboratory comparison solution (Sect. 8.3.a) were used for a determination of the half-life of the nuclide. Pairs of samples differing in radioactivity by a factor of two were measured repeatedly (1) for 11 half-lives by means of a well-type NaI(Tl) scintillation counter and (2) for eight half-lives by use of a pressurized-argon 4π ionization chamber. Data were analyzed by the CLSQ computer program, and no effects of impurity or other systematic errors were detected. Half-life results were: scintillation counter, 6.030 ± 0.009 and 6.004 ± 0.007 hr; ion chamber, 6.021 ± 0.005 and 6.026 ± 0.004 hr. Uncertainties are standard deviations of least-square fits. The unweighted average was 6.020 ± 0.006 hr, and the weighted average was 6.022 hr. Because of the range of the scintillation-counter results, the value will be published as 6.02 ± 0.01 hr. This new value was combined with six others, and means were computed by use of the AVERAGE program; two additional values were rejected as outliers. The recommended value is the weighted mean, 6.03 hr, and the realistic uncertainty is taken as equal to the standard deviation of the array, ±0.03 hr.

8.6.f Production of ^{123}I for Medical Applications

J. S. Eldridge H. B. Hupf[48] J. E. Beaver[48]

Techniques were developed[49] for the production of ^{123}I in quantity and of quality suitable for distribution as a new radioisotope product. Hundred-millicurie amounts of ^{123}I were produced by the $^{123}Te(p,n)^{123}I$ reaction in a 185-μA proton beam of the ORNL 86-in. cyclotron in a few hours by use of targets isotopically enriched in ^{123}Te. When highly enriched ^{123}Te target material was used, the amount of other longer-lived iodine radioisotopes produced concurrently was 1.5% of the total ^{123}I radioactivity, and the product was suitable for medical diagnostic use. Rapid separation procedures for the product and target materials were

[46] S. A. Reynolds, J. F. Emery, and E. I. Wyatt, "Half-Lives of Radionuclides – III," *Nucl. Sci. Eng.* 32, 46 (1968).

[47] Oak Ridge Associated Universities.

[48] Isotopes Division.

[49] H. B. Hupf, J. S. Eldridge, and J. E. Beaver, "Production of Iodine-123 for Medical Applications," *Intern. J. Appl. Radiation Isotopes* 19, 345 (1968).

developed. Half-life measurements gave a value of 13.02 ± 0.02 hr for this nuclide. Spectral comparisons showed that ^{123m}Te is a convenient and long-lived substitute assay standard for ^{123}I.

8.6.g Special Analytical Assistance for Isotopes Development Center Activities

J. S. Eldridge W. S. Lyon

Numerous assays for a wide variety of developmental products were provided to various members of the Isotopes Development Center. Many of the samples were incompletely processed products that contained radionuclide impurities which would interfere seriously if routine assay procedures were used. Some samples were checked for product purity; others were measured for radionuclide content. The techniques of x- and gamma-ray spectrometry with NaI(Tl) and Ge(Li) detectors were used. Samples that contained the following nuclides were assayed: ^{37}Ar, ^{43}K, ^{67}Cu, ^{74}As, ^{123m}Te, ^{124}I, ^{126}I, ^{129}I, ^{129}Cs, ^{135}Ba, ^{132}Cs, and ^{181}W.

8.6.h Radionuclides in High-Flux Isotope Reactor Coolant Water

S. A. Reynolds

The study of HFIR coolant water[50] was continued in a minor way. At the request of the Operations Division, a method was devised for on-site monitoring of fission-product release from fuel. The chosen method was measurement of the 0.86-MeV gamma radiation of 52-min ^{134}I by gamma spectrometry in samples of the effluent from the cation exchanger. A ^{54}Mn standard was used; it was mounted in the same way as the coolant water samples. The same approach was chosen independently for monitoring fission-product release from reactor-accident experiments.[51] Sample data showed a rapid initial decay (^{101}Mo, ^{104}Tc, etc.). The decrease in activity continued in a way which suggested that ^{134}I was supported in part by its parent 42-min ^{134}Te, as would be the case if the cation exchanger did not remove tellurium. This possibility was verified by determinations of ^{132}Te in cation-exchanger inlet and exit and anion-exchanger exit samples, which show negligible removal of tellurium by the cation exchanger and ~81% removal by the anion exchanger.

The prominence of 0.51-MeV gamma energies in the spectra of the coolant-water samples prompted the identification of the nuclides that emitted this radiation. A decay plot was resolved into a long-lived component (mostly ^{133}I and ^{187}W), 10-min ^{13}N, and 110-min ^{18}F. Interpretation of the ^{13}N data is uncertain because of the lack of knowledge about the behavior of nitrogen in HFIR. The level is inherently reasonable, however, because use of the macroscopic cross section for ^{235}U fission neutrons, 13.5×10^{-10} cm^{-1} (ref. 52), gives $\sim 7 \times 10^{11}$ neutrons cm^{-2} sec^{-1} for the effective flux to which the water was exposed, compared with $\sim 10^{12}$ neutrons cm^{-2} sec^{-1} thermal flux.[50] Because of the removal of F^- by the anion exchanger, the ratio of ^{18}F to ^{13}N was lower than that predicted from their cross sections.[52]

8.7 EFFECTS OF RADIATION ON ANALYTICAL METHODS

8.7.a Radiolysis of Radiation-Safeguard Spray Solutions

H. E. Zittel J. R. Lund[53]

Study of the effects of radiation on radiation-safeguard spray solutions[54] was continued. The results obtained have been presented orally.[55] A summary of the paper follows.

In a reactor accident, volatile fission products are likely to be released to the atmosphere of the reactor containment. Radionuclides of iodine (largely ^{131}I) are possibly the most likely radioisotopes involved and must be removed before the containment air is released to the atmosphere. It has been proposed that spray solutions be used as a means to sequester and fix such

[50] S. A. Reynolds and J. F. Emery, "Study of HFIR Coolant Water," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967*, ORNL-4196, pp. 84–85.

[51] D. R. Wenzel, "Environmental Monitoring of Fission Product Release from the Loss-of-Fluid Test," presented at the Symposium on Environmental Surveillance in the Vicinity of Nuclear Facilities (Health Physics Society), Augusta, Ga., Jan. 24–26, 1968.

[52] L. Hammar and S. Forsen, "Formation of Nitrogen-13, Fluorine-17 and Fluorine-18 in Reactor-Irradiated H_2O and D_2O: Applications to Activation Analysis and Fast Neutron Flux Monitoring," *J. Inorg. Nucl. Chem.* 28, 2111 (1966).

[53] Infrared and N.M.R. Group.

[54] H. E. Zittel and J. R. Lund, "Radiolysis of Radiation-Safeguard Spray Solutions," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967*, ORNL-4196, p. 13.

[55] H. E. Zittel and T. H. Row, "Radiation and Thermal Stability of Sprays," presented at The American Nuclear Society Meeting, Toronto, Canada, June 9–13, 1968.

airborne fission products. Accordingly, in the Nuclear Safety Program at ORNL the radiation and thermal stability of such proposed sprays has been studied.

It is estimated that these spray solutions will be exposed to a maximum gamma-radiation dose of 1×10^8 to 3×10^8 rads accumulated over a period of weeks at temperatures that vary from room temperature to ~150°C. The stability of the spray solutions under these limiting conditions was determined. The solutions (all aqueous) studied were:

Identity	Composition
A-1	1 w/v % $Na_2S_2O_3$, 3000 ppm B, 0.15 *M* NaOH, pH 9.2
A-2	3000 ppm B, 0.15 *M* NaOH, pH 9.3
A-3	H_2O, pH 6.2
A-4	3000 ppm B, pH 4.6
A-5	1 w/v % $Na_2S_2O_3$, 3000 ppm B, pH 4.9
B-1	5 w/v % hydrazine (N_2H_4), pH 9.6
B-2	5 w/v % N_2H_4, 5 w/v % NH_4OH, pH 10.9

Table 8.6 gives the results of the study on the Group A solutions. The acid thiosulfate solution (A-5) is much more susceptible to radiation damage than is the basic thiosulfate solution (A-1). A dose of 1×10^8 rads essentially destroys the I_2 capacity of the former but causes that of the latter to decrease by <50%. The 10^8-rads dose also results in the formation of a fairly large amount of colloidal sulfur in the acid thiosulfate solution, whereas no sulfur was detected in the basic solution. All the solutions studied become more acid on being irradiated.

In all the solutions studied, H_2 was generated radiolytically in amounts sufficient to constitute a possible hazard. In each case, the absolute amount depends on a variety of variables, for example, the amount of O_2 available. The data of Table 8.6 indicate what might occur in the real case, since the experimental conditions were selected to approximate reality. At a dose of 2×10^8 rads, ~1.4 volumes of radiolytic H_2 per volume of solution could be expected to form, because the data indicate that the H_2 generation is essentially a linear function of total dose. Variation of the dose rate from 1×10^3 to 1×10^5 rads/min does not cause a significant change in the amount of H_2 generated per unit of radiation. The possibility of the use of a "scavenger" to decrease the radiolytic generation of H_2 has been investigated.

Thermal stability studies carried out in the absence of radiation indicate that the acid thiosulfate solution (A-5) is completely decomposed at 140°C in less than 72 hr and yields large amounts of sulfur, whereas the basic thiosulfate solution (A-1) shows <10% degradation under the same treatment.

Studies of the B group (hydrazine solutions) indicate that they have far less radiation stability than do the A group.

8.7.b Radiolysis of Nitrate Solutions

Hisashi Kubota

The radiation chemistry of nitrates has been studied exhaustively, and considerable material on this subject exists in the literature. In radiolysis, nitrate reduces the overall yield of molecular hydrogen but does not affect the yield of the OH– radical. Nitrite is a product of the radiolysis. Its formation increases with an increase in either nitrate concentration or pH.

The same radiolytic effects found in pure solutions can be expected to occur also in solutions encountered

Table 8.6. Effects of Gamma Radiation on Group A Solutions[a]

Solution	Gamma Radiation Dose (rads $\times 10^{-7}$)	$-\Delta pH_2$	Change in I_2 Equivalence (%)	Apparent $G(-S_2O_3^{2-})$	H_2 Evolved (cc/ml)[c]
1	1.00	0.1	2		
	1.87	0.2	2		0.12
	2.81	0.3	0		0.24
	4.74	0.4	−13	0.2	0.36
	9.46	0.7	−42	0.3	0.70
2	10.00	0.1			0.69
4	10.00	0.1			0.72
5[b]	10.00	1.7	−97	0.6	0.70

[a]Conditions: Volume ratio of gas phase to liquid phase, 25. Dose rate, 1×10^4 rads/min.

[b]1.65 mg of sulfur per millimeter of test solution present after irradiation.

[c]At STP.

in process and analytical work. The principal products of the radiolysis of concern in chemical systems are hydrogen peroxide and nitrite.

An example of the effect of nitrite is the decrease in the amount of silver chloride formed from the chloride produced by the radiolysis of chloral hydrate. The nitrite formed from nitrate in concentration of 0.1 *M* or more causes a visually detectable reduction of silver chloride to metallic silver. When the nitrate concentration is ~0.01 *M* or less, this reduction cannot be detected visually. No such reduction is observed in perchlorate medium, even at a perchlorate concentration of 0.1 *M* or greater.

8.7.c Radiolysis of Perchloric Acid

Hisashi Kubota

The radiolysis of perchlorate systems is interesting in that there is reported to be very little reaction between the primary products of the radiolysis of water and the perchlorate ion. The effects seen are principally "direct effects" of the radiation on the perchlorate ion itself.[56] The reports of all studies to date claim that chlorate and possibly some chloride, is produced from the perchlorate. Oxygen is another major product.

In the study of the possibility of dissolving some inert metals in irradiated mineral acids, the perchlorate acids were found to be colored yellow after irradiation, even when there had been no interaction with the metal. A spectrophotometric scan revealed that the absorption spectrum of this yellow substance is the same as that of ClO_2. Further work has shown that this product is indeed ClO_2. No formation of Cl^- has been observed thus far.

A post-irradiation effect occurs in irradiated perchloric acid. The amount of ClO_2 in the acid following removal from the source increases with time and doubles in about 12 hr. This growth takes place in the dark and is inhibited by room (fluorescent) light.

It is obvious that any ClO_2 so produced will severely affect certain chemical systems. A study is now under way to determine the extent of this interference as a function of solution composition and dose of irradiation.

8.7.d Stability of Some Inert Metals in Irradiated Mineral Acids

Hisashi Kubota

The dissolution of the metals Au, Nb, Pt, Ta, W, and Zr in irradiated HCl was reported previously.[57] This study was extended to the mineral acids in general, and silver was also included, because in most acids it forms a passive surface that renders it immune to further attack.

Foils of the respective metals were cleaned and weighed before being sealed into tubes with the acids. The acids were essentially air-saturated at the start. The air-to-liquid volume ratio for each tube was roughly 1:1. All samples were given gamma radiation doses of 3×10^7 to 5×10^7 rads. The foil was washed, dried, and weighed after the irradiation to determine the amount dissolved. The acid was analyzed for the metal ion. The weight criterion of dissolution was set arbitrarily at 0.1 mg/cm^2. Table 8.7 gives the results together with previously reported results of irradiations in HCl. The slow attack on niobium by irradiated H_2SO_4 was unexpected. The mechanism by which this dissolution occurs has not yet been ascertained. Zirconium tends to react with most oxygenated acids except HNO_3. This immunity may be due to the formation of a passive film that is not attacked by the radiolytically active species present in this system; zirconium is even attacked slightly by H_3PO_4 to form a black surface film.

Table 8.7. Solubility of Metals in Irradiated Mineral Acids[a]

Metal	Solubility In[b]				
	HCl	HNO_3	H_2SO_4	H_3PO_4	$HClO_4$
Au	+	–	–	–	+
Ag	+	*	+	+	+
Nb	+	–	+	–	–
Ta	–	–	–	–	–
W	+	–	–	–	–
Zr	+	–	*	**	+
Pt	+	–	–	–	–

[a]Conditions: foil weight, 0.1 to 0.4 g; foil area, 10 cm^2; gamma radiation dose, 3×10^7 to 5×10^7 rads.

[b]+ = dissolution >0.1 mg/cm^2. – = dissolution <0.1 mg/cm^2. * = dissolves without irradiation. ** = foil surface darkened no appreciable weight change.

Metallic silver is stable in these acids except HNO_3; however, it dissolves in each acid when irradiated. Silver placed in concentrated HCl and irradiated forms a dense coating. When this coating is treated with NH_4OH, the amorphous components are removed and crystals of AgCl remain attached to the metal surface; Fig. 8.11 shows these crystals.

[56]D. Katakis and A. O. Allen, "The Radiolysis of Aqueous Perchloric Acid Solutions," *J. Phys. Chem.* 68, 3107 (1964).

[57]H. Kubota, "Dissolution of Some Inert Metals in Irradiated Acid Chloride Solutions," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1966*, ORNL-4039, p. 10.

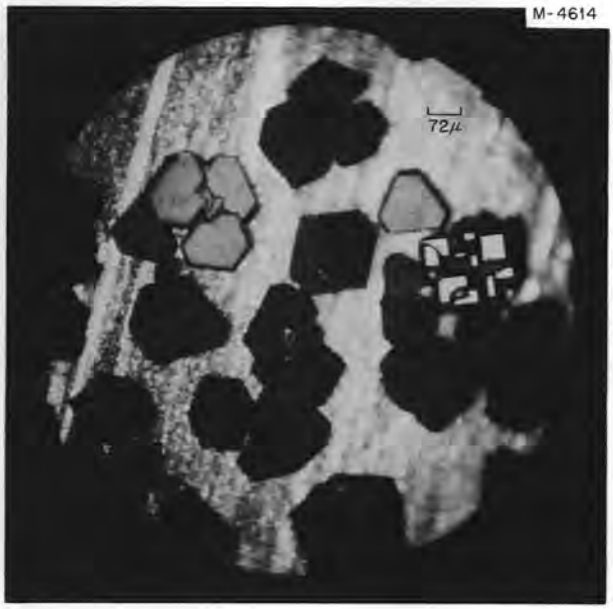

Fig. 8.11. Photomicrograph of AgCl Crystals Formed on Silver Foil by Gamma Irradiation in Concentrated Hydrochloric Acid.

8.7.e Effect of Radiation on Common Analytical Reagents

H. E. Zittel

A chapter titled "Effect of Radiation on Common Analytical Reagents" was prepared for inclusion in the forthcoming book, Vol. 10 of SERIES IX, in **Progress in Nuclear Energy.**[58]

8.7.f Effect of Gamma Radiation on Spectrophotometric Methods for Iron

H. E. Zittel

A study was completed to determine the effect of gamma radiation in the more widely used spectrophotometric methods for determining trace amounts of iron. The thiocyanate, α,α'-bipyridyl, and 1,10-phenanthroline methods were evaluated to determine which is preferable under conditions of high radiation.

Gamma radiation introduces a positive error in the iron-thiocyanate method. Invariably, the absorbance increases on radiation. In nonirradiated solutions, fading occurs as a result of the reduction of Fe(III) by the acid thiocyanate; gamma radiation reverses this effect. At a dose of $\sim 10^4$ rads, the absorbance of the complex reaches a constant value; additional radiation up to 10^6 rads does not increase the effect. In a sense, therefore, the radiation effect is desirable.

The iron–α,α'-bipyridyl complex is fairly stable against gamma radiation. A dose of 2×10^6 rads causes an error <1% in the determination of 2 ppm iron. At higher doses (10^6 rads), an increase in absorbance causes significant error. The increase is due to the products of the radiolytic decomposition of hydroxylamine which is present in large excess.

The iron–1,10-phenanthroline complex is extremely stable against gamma radiation. If both blank and sample are exposed to the same radiation, very little error is caused by a dose as large as 10^6 rads.

This study indicates that, on the basis of effect of gamma radiation only, the 1,10-phenanthroline method is preferred for use under conditions of high radiation.

The results of this study were presented orally[59] and are being prepared for publication.

8.7.g Studies Related to Project Salt Vault

Hisashi Kubota

Project Salt Vault has been completed except for a few post-demonstration studies. One item of interest is the residual moisture in the salt, both vertically and horizontally from the disposal hole.

During the course of the demonstration Project Salt Vault, an abnormal amount of moisture came out of each disposal hole. The amount was much greater than was expected from the moisture content of the salt at the periphery of the hole. It was postulated that, as a result of a temperature gradient, moisture would move within the salt toward the region of higher temperature. This postulation was made from calculations based on theory.[60]

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[58] H. E. Zittel, "Effect of Radiation on Common Analytical Reagents," 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.

[59] H. E. Zittel, "The Effects of Radiation on Some Analytical Methods," presented at the Second Society of Analytical Chemistry Conference, Nottingham, England, July 15–19, 1968.

[60] R. 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., August 1967.

After the removal of the radioactive elements from the demonstration holes, salt samples were taken at various depths and distances away from the disposal holes and were analyzed for moisture. It was shown definitely that moisture moved from the cold toward the hot zones. There was some evidence of a steady-state moisture concentration in the salt near the periphery of the holes. This concentration was ~0.1% near the region of maximum temperature (~200°C). The salt samples taken close to the hottest part of the heated holes did not shatter when heated up to >600°C, although they did lose some moisture. This behavior indicated that all moisture initially present in "negative crystals" had migrated and that the only moisture present was the transient moisture in the intercrystalline voids.

9. Inorganic Preparations

D. E. LaValle
R. B. Quincy

In a reduction of the fused-salts program, the preparation of anhydrous rare-earth-metal halides for the former High Temperature and Structural Chemistry Group of the Chemistry Division was terminated, the last salt prepared in this series being YbF_2. For the Ion Spectroscopy Group of the Metals and Ceramics Division, however, work continued with the preparation of such anhydrous materials as $Ni(NO_3)_2$, $NiCl_2$, NiI_2, $NiSO_4$, $MgCl_2$, $TiCl_3$, $CuCl_2$, $LiNO_3$, $NaNO_3$, KNO_3, LiCl, LiI, CsCl, and the eutectics KCl-LiCl and KI-LiI.

A series of ferrites was prepared for the Neutron Spectrometry Group of the Solid State Division. The ferrites are compounds of the structure Fe_3O_4 in which part of the iron is replaced by other metals. Examples in this series are

$$Fe_{2.25}Mn_{0.25}{}^{7}Li_{0.50}O_4,\ Fe_{2.3}Ti_{0.3}{}^{7}Li_{0.4}O_4,$$

$$Fe_{2.3}Ni_{0.7}O_4,\ \text{and}\ Fe_{1.7}Mn_{0.9}Cu_{0.4}O_4.$$

For another section of the same group, samples of TiO and VO were prepared, and several attempts, only partially successful, were made to grow single crystals of ^{7}Li.

In other work for the Solid State Division, high-purity ZnF_2 and MgF_2 were prepared for the Lattice Defects in Halides Group, fused AgCl and sublimed LiI for the Low Temperature Physics Group, and high-purity MnF_2 for the Lattice Defects in Oxides Group. In a continuation of work for the Neutron Diffraction Group, numerous rare-earth-metal alloys were heat-treated, and the project to produce 50 g of ^{160}Gd by the reduction of GdF_3 with calcium metal was successfully concluded. The preparation of high-purity KCl for the Research and Development of Pure Materials Group has been concluded. Work is now in progress to prepare high-purity MgO, about 20 lb of which has thus far been produced.

For the Low Temperature, Nuclear, and Solid State Physics Group of the Physics Division, further AuLi alloys were prepared in the range 0.25 to 0.50 at. % Au, and, in addition, the alloy AuCa (0.7 at. % Au). For another section of the same group, several intermetallic compounds were prepared: GeSe and GeTe, with both normal Ge and ^{73}Ge, and the isotopic compounds $^{64}NiAl$ and $^{61}NiFe$. Also, some compounds were prepared that contained nickel in various valence states: $[(C_6H_5O)_3P]_4Ni$ (0), $K_4[Ni_2(CN)_6]$ (I), $K_3[Ni(CN)_4]$ (II), and $KNiIO_6 \cdot \frac{1}{2}H_2O$ (IV). The methods of preparation are described in the standard treatise *Inorganic Syntheses.*

Other preparations of interest were ammonium azide (NH_4N_3) and beryllium nitride (Be_3N_2) for the Neutron Scattering Cross Section Studies Group of the Neutron Physics Division. Also, the decontamination of ~7 kg of platinum was undertaken for the Stores Department. This project is about half completed.

Part B. Service Analyses

A summary of the service analyses made by the laboratories of the Analytical Chemistry Division is given in the table below.

Summary of Analytical Service Work

Group Making Analyses	Average Number in Group		Number of Results Reported																							
			For ORNL Divisions															For Others								
	Scientists	Technicians	Analytical Chemistry	Biology	Chemical Technology	Chemistry	Health	Health Physics	Inspection Engineering	Isotopes	Metals and Ceramics	Neutron Physics	Operations	Physics	Reactor	Reactor Chemistry	Solid State	Argonne National Laboratory	General Biochemicals, Inc.	General Electric Co.	Miscellaneous	Nuclear Fuel Services, Inc.	Other Plants	Westinghouse Atomic Power Development Division	Y-12	Total
Mass Spectrometry Service Laboratory	2	4	370	20	425	166		549		9,245	3,858	25	121	25	2,791	2,528	1536				6940		1540		1484	31,623
Optical and Electron Microscopy	3	1	23		1,051	21			15	15	75				69	213	6								90	1,578
Process Analyses																										
General Analyses Laboratory	11	22	312	615	88,238	652		1595		175	11,254	175	508	83	2,418	2,922	1000	374	349						953	111,623
General Hot-Analyses Laboratory	10	8			5,613			104		434	2,104	63	7,161		2,117	1,995		180		28		35				19,834
High-Level Alpha Radiation Laboratory	9	10			21,272	13		17		421	150	3			8	1,006						9				22,899
Radioisotopes-Radiochemistry Laboratory	9	14			9,836	61		2534		6,821	1,492	41	5,648		3,903	18,586	626			1348	61					50,957
Spectrochemistry (Y-12)	2	1	160		2,149	446	18,786	30		12,469	5,389	212	163	31	1,524	3,236	1716				211				450	46,972
Transuranium Mass Spectrometry	1	2	554		809	188				1,020	626	11	70	15	388	145					21			289		4,136
X-Ray and Spectrochemistry	8	4	231	682	8,016	1382		2943		2,347	10,909	480	366	115	1,196	2,717	1767				501					33,652
Total	55	66	1650	1317	137,409	2929	18,786	7772	15	32,947	35,857	1010	14,037	269	14,414	33,348	6651	554	349	1376	7734	44	1540	289	2977	323,274

10. Methods Development and Evaluation

L. T. Corbin
P. F. Thomason

10.1 NEW METHOD FOR THE RAPID SEPARATION OF BERKELIUM(IV) FROM CERIUM(IV) BY ANION EXCHANGE

F. L. Moore

A new method was developed for the rapid separation of berkelium from cerium. The simple method is based on the marked difference in the sorption of the anionic nitrate complexes of Bk(IV) and Ce(IV) on anion-exchange resins. Berkelium is separated quantitatively from cerium on small columns. The method is valuable, because it functions in nitric acid solutions at room temperature and provides practical flow rates in glove-box or hot-cell facilities. The special case of an anion-resin column loaded with lead dioxide is particularly attractive, because it eliminates a preoxidation step and introduces no solids into the solution. The method has several useful analytical and process applications.

An article was published[1] that describes this work.

10.2 CHROMATOGRAPHIC–SOLVENT EXTRACTION ISOLATION OF ^{249}Bk FROM HIGHLY RADIOACTIVE SOLUTIONS AND ITS DETERMINATION BY BETA COUNTING

L. G. Farrar[2] J. H. Cooper[2] F. L. Moore

A method was developed for the chromatographic–solvent extraction isolation of ^{249}Bk from highly radioactive solutions and its subsequent determination by beta counting. A paper on this work was published;[3] the abstract follows:

"A small high-pressure ion-exchange system has been used together with liquid-liquid extraction to achieve decontamination sufficient to permit the determination by beta counting of ^{249}Bk isolated from highly radioactive solutions. A high-pressure pump forces solutions through a column of Dowex 50 resin (20- to 40-μ-diam particles) at pressures from 1800 to 2500 psi. The eluent is a dilute aqueous solution of α-hydroxyisobutyric acid of pH 4.2. The eluted ^{249}Bk is purified by extraction into 2-thenoyltrifluoroacetone–xylene. In this way the ^{249}Bk is separated sharply from many elements including the actinides, fission products, and corrosion products. Separations can be effected at eluent flow rates as fast as 18 ml cm^{-2} min^{-1}, thereby minimizing loading time, elution time, and radiation damage to the resin. High-pressure operation makes possible the use of a long column (~2 ft) having a small free column volume (~1 ml)."

10.3 NEW EXTRACTION CHROMATOGRAPHIC METHOD FOR THE RAPID SEPARATION OF AMERICIUM FROM OTHER TRANSURANIUM ELEMENTS

F. L. Moore

For the rapid separation of americium from other transuranium elements, a new extraction chromatographic method was developed. The work is described in an article to be published,[4] the abstract from which follows:

"A simple, rapid method is described for the separation of americium from curium and other actinide elements. The method exploits a new oxidation technique for the preparation of americium(V) in dilute nitric acid solution with ammonium persulfate. Isolation of the americium is achieved by extraction chromatography with small Teflon columns containing di(2-ethylhexyl) phosphoric acid as the stationary phase. The method considerably simplifies the purification and determination of ^{243}Am in the presence of high levels of ^{244}Cm. Several useful applications for both analytical and purification purposes are discussed."

10.4 THERMOGRAVIMETRIC ANALYSIS OF THORIUM IODATE

D. C. Canada

Solid thorium iodate, $Th(IO_3)_4$, frequently contains a large amount of absorbed moisture. The determinations of thorium, iodate, and water in this material were requested. Since $Th(IO_3)_4$ can be converted to the oxide according to the equation

[1] F. L. Moore, "New Method for Rapid Separation of Berkelium(IV) from Cerium(IV) by Anion Exchange," *Anal. Chem.* 39, 1874 (1967).

[2] High-Level Alpha Radiation Laboratory.

[3] L. G. Farrar, J. H. Cooper, and F. L. Moore, "Chromatographic-Solvent Extraction Isolation of Berkelium-249 from Highly Radioactive Solutions and Its Determination by Beta Counting," *Anal. Chem.* 40, 1602 (1968).

[4] F. L. Moore, "New Extraction Chromatographic Method for the Rapid Separation of Americium from Other Transuranium Elements," to be published in the December 1968 issue of *Analytical Chemistry*.

$$Th(IO_3)_4 \xrightarrow{\sim 550^\circ C} ThO_2 + 2I_2 + 5O_2 ,$$

the complete analysis can be done thermogravimetrically. The strip-chart recording obtained in the thermogravimetric analysis shows sharp breaks at 100 and 550°C. Water is calculated from the weight loss at 100°C, iodate from that at 550°C ($2I_2 + 5O_2$), and thorium from the total weight loss. A confirmatory iodate determination[5] may be done volumetrically.

10.5 GRAVIMETRIC DETERMINATION OF TETRAFLUOROBORATE WITH NITRON

D. C. Canada

A sample of solid sodium tetrafluoroborate was analyzed for tetrafluoroborate by a gravimetric nitron ($C_{20}H_{10}N_4$) procedure.[6] Nitron was purified by fractional crystallization from absolute ethyl alcohol. An alternate method[7] that consisted in the partition titration of tetrafluoroborate with tetraphenylarsonium chloride and amperometric end-point detection lacked the required precision and accuracy.

10.6 DETERMINATION OF FLUORIDE IN A MIXTURE OF MOLYBDENUM FLUORIDES

D. C. Canada

A mixture of solid molybdenum fluorides was analyzed for fluoride content. The best results were obtained by pyrolysis followed by titration of the acid. An alternate null-point potentiometric method[8] proved to be somewhat inconsistent.

10.7 DETERMINATION OF HAFNIUM AND ZIRCONIUM IN HAFNIUM OXIDE–ZIRCONIUM OXIDE MIXTURES

D. C. Canada

Determinations of hafnium and zirconium were requested on hafnium oxide–zirconium oxide mixtures. Total milliequivalents of hafnium and zirconium were measured by back-titration with EDTA. From this known value for a given weight of hafnium oxide plus zirconium oxide, simultaneous equations can be set up and solved for percent hafnium and percent zirconium.

10.8 BIOCHEMICAL ANALYSES

D. C. Canada

The Analytical Biochemistry Group was assisted in the separation, identification, and determination of ribonucleic acids, deoxyribonucleic acids, proteins, lipids, and polysaccharides. The results are discussed in Sects. 4.1.b, -f, -g, and -h.

10.9 RAPID-SCAN POLAROGRAPHIC DETERMINATION OF URANIUM

P. F. Thomason

The polarographic single-cell procedure and apparatus previously described by W. L. Belew and co-workers[9] was studied for the determination of uranium in MSRE fuel as a possible supplementary method to the controlled-potential coulometric titration method now in use. With the ORNL model Q-2792 controlled-potential dc polarograph-voltammeter, repetitive scans were obtained of standard solutions of uranium (5×10^{-4} *M*) in various supporting electrolytes: 1.0 *M* hydroxylamine sulfate, 1.0 *M* H_2SO_4, and 0.1 *M* HNO_3 that contained 0.001 vol % Triton X-100 as a maximum suppressor. Twenty five scans, each from +0.1 to −0.8 V vs S.C.E., were obtained in ~20 min. The peak heights of the uranium first-derivative wave were measured by means of a digital voltmeter readout, and the average diffusion currents were corrected by subtracting the average residual current. An unexplained decrease in the peak heights with time was obtained. This decrease was ~1% more than the calculated electrolytic reduction of the UO_2^{2+} ion.

The first results were encouraging, because an *S* of 0.1% was obtained for all the electrolytes. However, when synthetic MSRE fuel samples that contained known amounts of uranium were analyzed by the standard-addition technique, a 1% negative bias in accuracy was obtained; also, the *S* was higher (~0.5%).

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[5] R. Z. Bachman and C. V. Banks, pp. 331–32 in "Thorium," chap. 3 of *Analysis of Essential Nuclear Reactor Materials*, ed. by C. J. Rodden, Division of Technical Information, U.S. Atomic Energy Commission, 1964.

[6] C. A. Lucchesi and D. D. DeFord, "Gravimetric Determination of Boron. Precipitation as Nitron Tetrafluoborate," *Anal. Chem.* 29, 1169 (1957).

[7] K. Behrends, "Die Verteilungstitration von Tetrafluoroborat," *Z. Anal. Chem.* 216, 13 (1966).

[8] T. A. O'Donnell and D. F. Stewart, "Null-Point Potentiometric Determination of Fluoride," *Anal. Chem.* 33, 337 (1961).

[9] W. L. Belew, M. T. Kelley, D. J. Fisher, R. W. Stelzner, and E. S. Wolfe, "Investigation of Precision Attainable in Single-Cell First Derivative DC Polarography," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967*, ORNL-4196, p. 7.

The polarographic peak height of uranium is hydrogen-ion dependent; the rate of disproportionation of U(V) to U(IV) and U(VI) is influenced greatly by the hydrogen ion. However, Verdingh and Lauer[10] found that uranium peak heights become constant at an H_2SO_4 concentration of 1.8 *N*. Therefore, these low results probably could not be due to the disproportionation. W. L. Maddox[11] and D. R. Martin[12] recently found that a D.M.E. controlled by a drop-knocker to 0.5-sec drop-time had an ac pickup from some source. This same type of trouble could have occurred while this study was in progress and accounted for some of the erratic behavior encountered with the MSRE synthetic fuel samples.

When the ac pickup difficulty is eliminated and the apparatus becomes available, this study will be continued.

10.10 THIN-FILM MERCURY ELECTRODE VOLTAMMETRY OF BISMUTH

P. F. Thomason

A sensitive method is needed to determine bismuth in MSRE fuel. A thin-film mercury electrode was prepared by amalgamation of the platinum disk in an inlay style Beckman No. 1273 platinum electrode. This mercury electrode was used to study the voltammetric determination of bismuth with the ORNL model 2792 controlled-potential polarograph-voltammeter.

For the polarographic determination of bismuth in urine, Lanza, Concialini, and Benati[13] recommend 1.0 *M* EDTA–0.1 *M* NaOH as supporting electrolyte. It was found that in this medium bismuth gives a well-defined wave at $E_{1/2} = -0.65$ V vs S.C.E. and that the diffusion current is proportional to bismuth concentration over the range 0.5 to 10 μg/ml. Interferences of materials in the MSRE fuel will be studied.

Also, the stripping technique (inverse polarography) will be studied as a way to increase the sensitivity of the method. A Metrohm Micro-Mercury-Feeder E-410 was obtained to deliver uniformly and reproducibly a small hanging drop of mercury. This unit will be used in the stripping technique of analysis.

10.11 CHARACTERIZATION OF TOBACCO SMOKE AND SMOKE CONDENSATES

H. L. Holsopple

In cooperation with the Lung Cancer Task Force of the National Cancer Institute of the National Institutes of Health, the Analytical Chemistry Division has initiated a program of research in the field of smoking and health (Sect. 2.3).

10.11.a Literature Survey

As an initial step, a survey was made of the literature, and appropriate articles were collected and indexed for use by researchers in the program. It was found that a broad experimental and clinical approach to the subject has been manifest for the past few decades, the most extensive and definitive studies being undertaken since 1955.

Although our chief interest is the analysis of tobacco smoke, it was thought advisable to also obtain during the literature survey additional articles on the use of tobacco and the resulting effects. It was hoped that this information would give the analytical researcher a broader view of the overall problem and, consequently, a better idea of areas that need his attention. However, since the scope of the problem is so large and includes such a great number of disciplines (this survey alone gleaned 1200 articles from >200 journals), there is no pretense that the collection is complete.

The literature is assembled alphabetically and chronologically by journal in over 50 volumes. The journals themselves are color-coded in five classifications: (1) chemistry of tobacco and tobacco smoke, (2) medical – general, (3) medical – neoplastic and cardiovascular, (4) sociological and psychological, and (5) general interest or popular literature. For the chemist or biologist starting research in this area, the Analytical Chemistry Division's collection of tobacco-related articles and indexes should prove useful.

10.11.b ORNL Smoking Machine

The first step in any smoke-characterization program is to provide a device for collecting samples under standard reproducible conditions. Such a device (Fig. 10.1) was constructed to smoke simultaneously six cigarettes at a rate of one puff per minute – a 35-ml intake with a puff duration of 2 sec per cigarette. The

[10] V. Verdingh and K. F. Lauer, "Accurate Determination of Microgram Quantities of Uranium by Direct and Differential Polarography," *Z. Anal. Chem.* 235, 311 (1968).

[11] W. L. Maddox, personal communication to P. F. Thomason, July 1968.

[12] ORAU Student Trainee from Sacramento State College, Sacramento, Calif.

[13] P. Lanza, V. Concialini, and A. Benati, "Determination of Bismuth Content of Urine. Polarographic Method," *J. Electroanal. Chem.* 17, 395 (1968).

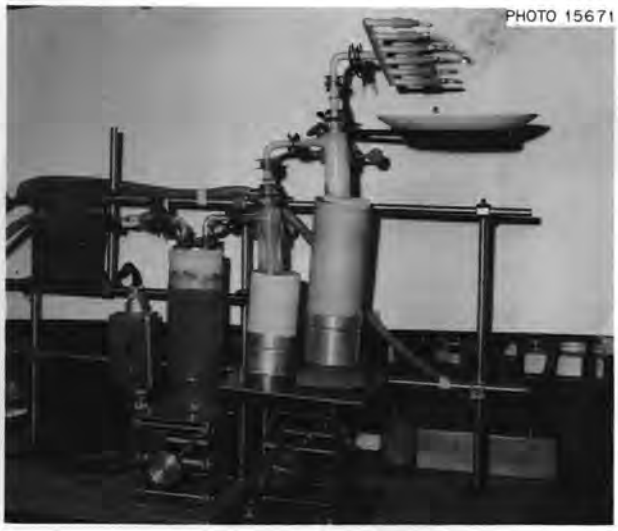

Fig. 10.1. ORNL Smoking Machine.

system has three traps for collecting the smoke condensates: one in an ice-salt bath at 0 to 10°C, another in isopropanol–liquid nitrogen at –40 to 50°C, and a third in liquid nitrogen at –195°C. This method of collection provides an initial fractionation before the separations are made that are based on the solubility of the condensates in various solvents.

To collect appreciable quantities of smoke aerosols for gas-liquid chromatographic (GLC) analysis, 500 cigarettes were smoked; 35 g of pyrolysis products was produced. The results of GLC analysis are described in Sect. 2.3.a.

10.11.c Gel-Permeation Chromatography as a Separations Technique

Components of tobacco extracts were separated on columns of Sephadex gels in both organic and aqueous mediums. In the organic system, isopropanol was the solvent and LH-20 Sephadex was used as gel in a 2½- by 100-cm column. At a flow rate of 50 ml/hr, components of both cigar- and cigarette-tobacco extracts were separated on the basis of molecular size.

In the aqueous system, a G-10 Sephadex column (1- by 100-cm) was used. Three different brands of cigars were agitated for 1 hr in water, and portions of the filtered extracts were passed through the column. A comparison of the elution volumes of each of the resulting chromatographic peaks (256 mμ) of the three brands showed that each brand has essentially the same five major components.

The acid and basic components of tobacco fractions were separated, and the test solutions were passed through gel-permeation columns. Further differentiation was thus accomplished. It is concluded tentatively that a gel-permeation technique for separating tobacco components should be a useful adjunct to gas chromatographic and mass spectrometric methods for the analysis of these fractions.

10.12 ABSORPTION SPECTROPHOTOMETRY OF ALPHA-EMITTING MATERIALS

D. A. Costanzo

The glove box and absorption-cell compartment designed for a Cary model 14 spectrophotometer[14] have been used satisfactorily for the absorption spectrophotometry of alpha-emitting materials. The spectrophotometric method was used to determine americium in the presence of curium, plutonium in the presence of zirconium, and ionic plutonium in the presence of polymeric plutonium.

10.13 MODIFIED WILLARD-WINTER DISTILLATION METHOD FOR FLUORIDE

D. A. Costanzo

The modified Willard-Winter distillation method[15] was evaluated for the separation of microgram amounts of fluoride in acid nitrate media that contain moderate amounts of nickel, thorium, and bismuth. Over the fluoride range 5 to 50 μg, the recovery was not less than 95%. When present in the distillate, sulfate interferes in the spectrophotometric determination with alizarin complexone.[16] The sulfate interference can be minimized by the addition of sodium nitrate to the distillate prior to the addition of the chromogenic reagent.

10.14 PRECISE DETERMINATION OF URANIUM IN MSRE FUEL

Gerald Goldberg

The current method to determine uranium in MSRE fuel, that is, remote measurement by controlled-poten-

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[14] D. A. Costanzo and L. T. Corbin, "A Glove Box System for a Cary Model 14 Spectrophotometer," accepted for publication in *Nuclear Applications.*

[15] H. Kubota, "Rapid Distillation Separation of Microgram Quantities of Fluoride," *Microchem. J.* **12**, 525 (1967).

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

tial coulometric titration in a solution of the fuel, has a relative standard deviation (S) of ~1%. For best control of the reactor by chemical analysis, S should be no greater than ~0.2%. Accordingly, a method is being developed that consists in the preliminary separation of the uranium from the remainder of the fuel by fluorination at 600°C and collection of the resulting UF_6 in a trap of sodium fluoride. The contents of the traps is dissolved, and the fluorine is removed. The uranium, now free from interfering ions, is determined with a high-sensitivity controlled-potential coulometric titrator.

To apply this method to highly radioactive samples that must be processed remotely in a hot-cell facility, special apparatus was designed and constructed (Fig. 10.2). The sample is melted from the ladle (seen hanging at the left) into a nickel liner within the reaction pot. The ladle and supporting flange are then replaced by a flange that contains the fluorine-inlet tube and gas-transfer line. The UF_6 flows from the reaction pot through a pretrap of sodium fluoride at 400°C and then to the quartz collection trap. The excess fluorine is absorbed in the soda-lime-filled pipe. Nitrogen is used as a purge gas.

To achieve high precision in the analytical procedure, it is necessary to remove the quartz collection trap from the hot cell. Since the fission-product radioactivity within the filled sample ladles exceeds 1000 rem, the pretrap was incorporated to trap the bulk of the fission products evolved during fluorination, for example, niobium, tellurium, and molybdenum. The UF_6 is not adsorbed at 400°C. If the quartz trap is maintained at 100°C, the radioactive iodine passes completely through the system. An S >0.5% would be very difficult to achieve if it were necessary to dissolve, pipet, and titrate the sample within the hot cell.

The apparatus has been installed in a hot cell. All gas and temperature controls are located outside the cell except the full-open and full-closed valves (Fig. 10.2). In preliminary tests, the recovery of uranium from standard samples has been not less than 97.5%. Coulometric titrations have been carried out with an S between replicates of 0.1%. It is expected that uranium recovery >99.5% will be achieved by adjustment of the fluorine gas flow and flush rates.

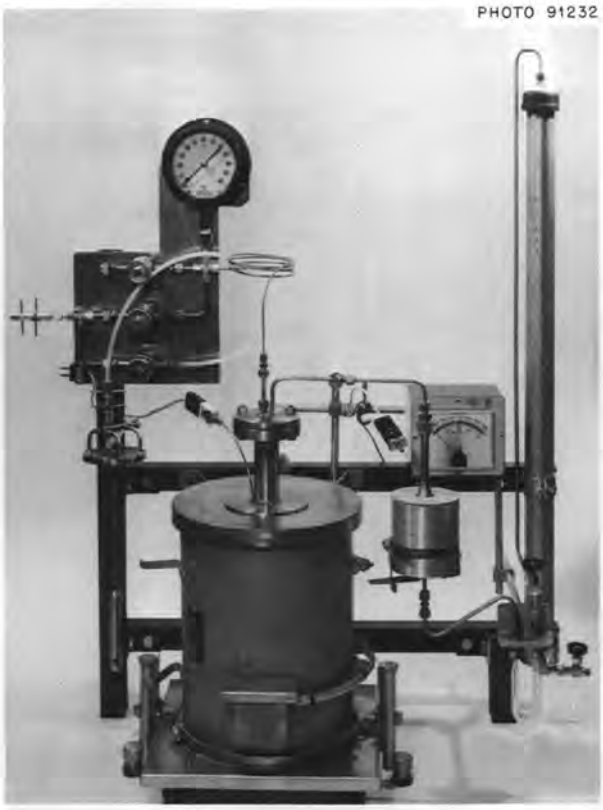

Fig. 10.2. High-Temperature Fluorination Apparatus for the Precise Determination of Uranium in MSRE Fuel.

Several MSRE samples have been analyzed for uranium. The gamma radioactivity within the quartz trap after fluorination is <20 millirem; therefore the trap may be removed from the hot cell. A gamma scan of the purified uranium sample showed that niobium is the major source of the radioactivity collected in the trap. The total amount of niobium was <1 ng.

11. Spectrometry

A. E. Cameron

11.1 MASS SPECTROMETRY

11.1.a Transuranium Mass Spectrometry

R. E. Eby

The Transuranium Mass Spectrometry Laboratory reported 4136 results, about the same number as in the previous year. Because of the addition of an analyst, the cost per analysis increased slightly, from $12.86 in FY-1967 to $13.80 in FY-1968. No one customer accounted for as much as a quarter of the analyses. The Isotopes and Chemical Technology Divisions continued to be the principal customers.

In collaboration with J. Halperin[1] and C. E. Bemis, Jr.,[1] the partial alpha-decay half-lives of ^{242}Pu and ^{244}Pu were determined. The ^{242}Pu alpha half-life was determined by use of samples that were mixtures of ^{239}Pu and ^{242}Pu. From mass and alpha spectral analyses and by use of the value 24,401.5 ± 21.2 years for the ^{239}Pu alpha half-life, the value 3.869 ± 0.016 X 10^5 years was obtained for the alpha half-life of ^{242}Pu. Similarly, mixtures of ^{244}Pu and ^{242}Pu were made, and the value for the ^{244}Pu alpha half-life was determined relative to the adopted value 3.869 ± 0.016 X 10^5 years for ^{242}Pu. The value 8.28 ± 0.10 X 10^7 years was determined for the ^{244}Pu partial alpha half-life. The results of this work were submitted for publication.[2]

A similar program in which americium was used was conducted with C. E. Bemis, Jr.[1] Isotopic ratios of various mixed standards of ^{241}Am and ^{243}Am were determined by mass spectrometry, and the data were combined with counting measurements to determine the relative half-lives. The data have not yet been analyzed.

Other samples of interest include one from D. N. Metta of Argonne National Laboratory. The plutonium collected from the decay of curium was analyzed to aid in the determination of the half-lives of curium radionuclides. Californium samples were analyzed for J. Halperin;[1] the results were used to calculate the cross sections of the nuclides of that element. The laboratory participated in an interlaboratory check on a proposed revision to ASTM method E-267, which is used to measure uranium and plutonium concentrations and isotopic abundances in reactor fuels. The analysis of samples from the ^{171}Tm and ^{153}Gd programs[3] was continued.

Seven samples of einsteinium were analyzed. No difficulties were encountered except that each sample, because of its radioactivity, ruined the electron multiplier in the spectrometer. Some preliminary work has been done to determine the relative values for the half-lives of the einsteinium nuclides.

11.1.b Mass Spectrometry Services

J. R. Sites

E. J. Spitzer

The number of analyses reported by the Mass Spectrometry Service Laboratory increased 20% from last year. This increase includes a 5% increase in analyses for the Isotopes Division of isotopically enriched samples of 46 elements.

The relative isotopic abundances of several radioactive solid samples were determined; the samples included those enriched in ^{36}Cl, ^{40}K, ^{41}Ca, ^{129}I, and ^{131}I.

In gaseous-sample mass spectrometry, certification was continued of argon and helium contained in cylinders and trailers, of nitrogen contained in cylinders, and of the fission-product krypton and xenon that are prepared for sale.

Many sets of samples were analyzed for experimenters who are studying such things as gaseous cleanup, contamination buildup in dry boxes, diffusion of gas mixtures through membranes, and off-gases from the baking of various fuel pellets.

11.2 EMISSION SPECTROMETRY (Y-12)

J. A. Carter

S. A. MacIntyre

The Spectrochemistry Laboratory reported about 47,000 results on 2500 samples, which represent

[1] Chemistry Division.

[2] C. E. Bemis, Jr., and J. Halperin, "The Alpha Decay Half Lives of Plutonium-242 and Plutonium-244, submitted to the *Journal of Inorganic and Nuclear Chemistry.*

[3] R. E. Eby, "Transuranium Mass Spectrometry," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967,* ORNL-4196, p. 94.

increases of 12 and 19%, respectively. The Metals and Ceramics Division was the largest supplier of samples (26%). Many of these samples were experimental alloys submitted for composition analysis as well as for trace characterization. The Isotopes and Health Divisions submitted about the same number of samples as last year.

Stable isotopes of the following 46 elements were analyzed for trace impurities: Ag, B, Ba, Br, Ca, Cd, Ce, Cr, Cu, Dy, Er, Eu, Ga, Gd, Ge, Hf, Hg, In, Ir, K, La, Lu, Mg, Mo, Nd, Ni, Os, Pb, Pd, Pt, Rb, Ru, S, Sb, Se, Si, Sm, Sn, Sr, Te, Ti, Tl, W, Yb, Zn, and Zr. More than 300 bismuth samples from the MSRE salt cleanup program were analyzed for thorium, lithium, and beryllium; the analyses were primarily for the Reactor Chemistry and Chemical Technology Divisions.

11.3 INFRARED SPECTROMETRY

M. M. Murray

The Infrared Laboratory has become increasingly involved with the Body Fluids Analyses Program. Preliminary investigation of a few of the materials known to occur in urine indicates that some are not amenable to the treatment involved in the standard alkali-halide-disk technique. Since the major portion of the energy applied to the sample is transmitted during the grinding of the sample together with the alkali halide, it was considered advisable to eliminate this step if possible. Of the alternative methods considered, lyophilization was the most attractive. Accordingly, a small "freeze-dry head" that uses liquid nitrogen as the refrigerant was designed, fabricated, and installed. Shop test of the instrument indicates that it can maintain a 5-μ pressure under pumping. Laboratory test indicates that it can reduce five 2-ml samples to satisfactory dryness in a short time (1 to 2 hr). The apparatus has seven ports fitted with commercially produced rubber valves. These valves fit a 16-mm-diam tube. Small samples can be lyophilized by freezing the sample in a small tube and inserting it in a 16-mm-diam tube; large samples, by using a flask having a 16-mm-diam neck.

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.

Table 11.1. Compounds Synthesized at ORNL That Were Examined by Infrared Spectroscopy

Barium-*p*-ethylbenzenesulfonate
Bicyclobutane
Boron nitride (vapor-deposited)
Bromo-3-chlorocyclobutane
3-Chlorocyclobutane carboxylic acid
Dimethylmonobutyl phosphate
Methyldibutyl phosphate
1-Methyl-4-thiouracil
1-Methyluracil
Silver 1-methylpyrimidine-4-sulfenate
Sodium 1-methyl-2-oxo-pyrimidine-4-sulfenate
Trimethyl phosphate

Fractions from compounds synthesized at ORNL were examined for structure and purity (Table 11.1). Materials produced by irradiation, chemical reaction, or other processes were identified. A summary of the results follows: irradiations of tall oil (a by-product of paper production) and some of its fractions (oleic acid and a sesquiterpene called caryophyllene); reaction mixtures from attempted syntheses of Na_2LiF_6; motor oils; pump oils; materials leached from the charcoal filters of the Transuranium Processing Plant and of the U.S.S. Savannah; SiF_4; $SiOF_2$; plastic tubing; an air sample from the Eagle Bend area near Clinton, Tenn.; numerous column chromatographic samples from the Body Fluids Analyses Program; and mixtures of Span 80 and di-2-ethylhexanol.

Future work includes continued cooperation with L. C. Brown[1] in his investigation of the bromate-perbromate system; acquisition of spectra of biological compounds; investigation of a possible method of removing water from the alkali halide disk during the pressing procedure, and investigation of the possible use of symmetrical difluorotetrachloroethane as an infrared solvent and/or mulling agent.

11.4 NUCLEAR MAGNETIC RESONANCE SPECTROMETRY

J. R. Lund

Much of the nuclear magnetic resonance (NMR) spectrometry has dealt with the elucidation of chemical structure and configuration of organic compounds that resulted from studies by the Y-12 Organic Preparations Group. The NMR spectra, along with the mass (MA) and infrared (IR) spectra, were used to determine the

structure of such synthesis products as cinnamylidenindene, diphenylbenzofulvene, dibromofluorene, and some isomers of truxene (Sect. 6.2). The spectra of the simpler derivatives of indene, fluorene, and fulvene were very helpful in the elucidation of their structures. However, the truxenes are only slightly soluble in solvents suitable for NMR spectrometry. The spectra of compounds of limited solubility (<5%) were improved by use of a computer of average transients (CAT); Fig. 11.1 shows the improvement in the spectrum of a sample of truxene. Not only was the background noise reduced significantly, but the signals that result from proton absorption were enhanced considerably.

Various polymers such as polymethylenepolyphenylisocyanate (PAPI), Varcums (polymers of furfuryl alcohol), organophosphorus polymers, and polymers of furan aldehyde were examined. Their NMR spectra indicate that most of them are mixtures; however, such groups as >P(O)H, >P(O)CH_2P(O)<, and ordinary alkyl groups were identified in the organophosphorus polymers. The spectra of the PAPI samples consisted of only two broad single peaks and indicated a ratio of aromatic to aliphatic protons of the order of 3 to 1. Much more information was available from the spectra of the Varcums; they were found to be mixtures of furfuryl alcohol (I) and 5-furfurylfurfuryl alcohol (II) or of higher polymers of (I) and/or (II). Since the polymer of furfural is relatively insoluble in such usual NMR solvents as $CDCl_3$ or CCl_4, attempt was made to measure the spectra of these polymers in such solvents as deuterodimethyl sulfoxide, $(CD_3)_2SO$, and hexamethylphosphoric triamide, $((CH_3)_2N)_3PO$ at ambient and elevated temperatures. The attempts were unsuccessful, because depolymerization occurred and the spectrum of the furfural molecule was observed instead.

The NMR spectra of numerous materials were recorded for the purpose of identification or comparison. Among these materials were hippuric acid; urea; caryophyllene; barium *p*-ethylbenzenesulfonate; and the four lipids estrololactone, 2β-methyl-19-nortestololactone, and 2α- and 2β-methyl-estra-4-ene-3,17-dione.) The chemical shift of the signals due to angular methyl groups in lipids reveals a great deal about the nature of the substituents and the stereochemistry of the rings.

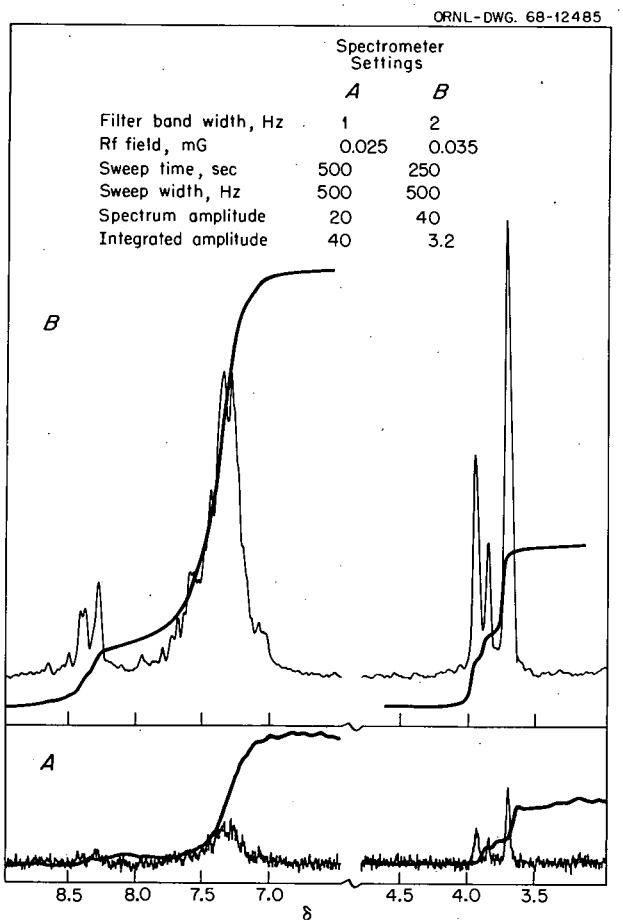

Fig. 11.1. NMR Spectra of a Mixture of Truxene Isomers. (Sample; saturated solution of truxene in CS_2. NMR spectrometer; Varian A60; —— NMR spectra; ▬▬ integrated spectra.) *A* – one scan. *B* – 25 scans accumulated with the C-1024 computer of average transients (CAT); courtesy of Varian Associates, Instrument Division, Palo Alto, Calif.

12. Process Analyses

L. T. Corbin

12.1 HIGH-LEVEL ALPHA RADIATION LABORATORY

J. H. Cooper

Over 23,000 results were reported by the High-Level Alpha Radiation Laboratory. Most of these were for the Chemical Technology Division. Although the total personnel was decreased by 10%, the number of results reported increased 17%.

A method for determining ^{249}Bk was developed (Sect. 10.2) and used routinely on samples from the Transuranium Processing Plant. This work is described in a published article.[1]

A method for determination of the oxygen-to-metal ratio in PuO_2-UO_2 pellets is now being used that depends on changing the oxygen-to-metal ratio to 2.000. A weighed portion of the dry sample is oxidized in air at 850°C and then is reduced in a hydrogen-argon mixture at 850°C for 24 hr. The reduced sample is assumed to be stoichiometric dioxide, and the change from the initial weight can be used to calculate the original oxygen-to-metal ratio.

Samples of high-fired plutonia, urania, and mixtures of these with zirconia have been dissolved in sulfuric acid after a fusion in ammonium bisulfate. The fusion can be done easily on a hot plate in a glove box. The sulfuric acid solution is in a form suitable for determination of plutonium and/or uranium by coulometric titration.

Samples of LiF-BeF_2-ThF_4 salt have been submitted for the determination of Th, Li, Be, Pa, Ni, Bi, and U. Samples of bismuth that had been in contact with the molten salt were submitted for the same analyses. The salt samples are dissolved in nitric acid–boric acid and are fumed in perchloric acid to remove the fluoride. The bismuth samples are dissolved in nitric acid.

12.2 GENERAL ANALYSES LABORATORY

W. R. Laing

The work load in analytical biochemistry increased 48%; the total number of determinations of tRNA was 69,996 compared with 46,464 last year. For this analysis, the average cost per analysis was reduced from $1.89 to $1.62.

A method was developed to determine bulk density of large (about 1 × 4 × 8 in.) porous graphite blocks. The densities ranged from 0.8 to 1.6 g/cc. No liquid could be allowed to enter the pores, nor could the samples be cut. Thin latex bags were made to hold the samples. The samples were placed in the bags, and the bags were evacuated and sealed by a clamp. A sample volume was determined by immersing the bagged sample in a cylinder filled with water and catching and weighing the overflow. For samples of density <1, a small piece of lead of known volume was placed in the bag with the sample. The S of the density measurements was 0.1%.

A small cylindrical pycnometer was constructed to measure the density of sections cut from HFIR fuel plates. The volume of the pycnometer was 0.9 cc; that of the samples was 0.1 cc. The S of this method was 0.5%.

The arsenazo III colorimetric method[2] for thorium was used to determine thorium in bismuth metal. Thorium contents as low as 5 ppm were measured without separation of the bismuth. The S was 3% at the 80-ppm level and 2% at the 1600-ppm level.

The American Public Health Association method for cyanide[3] was used to determine cyanide in KCl crystals. No interference from the KCl was encountered at cyanide concentrations of 0.2 ppm.

The sealed-tube method was adopted for the dissolution[4] of samples of uranium nitrides submitted for the determination of nitrogen. This technique gave good

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[1] L. G. Farrar, J. H. Cooper, and F. L. Moore, "Chromatographic-Solvent Extraction Isolation of Berkelium-249 from Highly Radioactive Solutions and Its Determination by Beta Counting," *Anal. Chem.* 40, 1602 (1968).

[2] S. B. Savvin, "Analytical Use of Arsenazo III, Determination of Thorium, Zirconium, Uranium and Rare Earth Elements," *Talanta* 8, 673 (1961).

[3] *Standard Methods for the Examination of Water and Wastewater Including Bottom Sediments and Sludges,* 11th ed., ed. by L. Farber, pp. 356–58, American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 1960.

[4] G. W. C. Milner and I. G. Jones, *Improvements in the Determination of Nitrogen in Plutonium Mononitride and Uranium Higher Nitrides by Chemical Methods,* AERE-R-5329 (November 1966).

nitrogen results, but when the samples were heated at 195°C, as specified, dissolution sometimes required several days. A recently translated Japanese article[5] describes the effects of the additions of oxidizing agents on the dissolution of boron nitride. Forty milligrams of $KClO_4$ was added with the 9 *N* HCl to dissolve a 0.2-g sample aliquot. Dissolution time was reduced from several days to 4 to 16 hr at 195°C. When $KClO_4$ was used, it was necessary to add Devarda's alloy for the ammonia distillation to recover all the nitrogen.

A Laboratory Equipment Corporation Nitrox-6 analyzer was modified for the determination of oxygen in uranium nitride. A column of hot copper oxide was added to oxidize the carbon monoxide in the furnace exit gas to carbon dioxide. The molecular-sieve gas-chromatographic column was replaced by a column that contains silica gel. The molecular-sieve trap was operated at room temperature. These modifications allow the nitrogen released from the sample to pass through the analyzer with the carrier gas. The small amount of oxygen present can be measured without interference from the large amounts of nitrogen gas.

Moist-oxygen pyrolysis with $Na_2WO_4 \cdot WO_3$ flux was used to determine fluoride in MoF_3 and MoF_5.

The total number of results reported increased from 99,125 to 111,623.

12.3 GENERAL HOT-ANALYSES LABORATORY

C. E. Lamb

Interest continued for essentially the same analytical services required last year. A total of 19,834 reported results reflects a 4% increase.

The recently installed glove-box facility was used extensively in the measurement of solid samples for gas release, density, carbon, surface area, and oxygen. The samples analyzed were received mainly from the Chemical Technology and Metals and Ceramics Divisions but also from customers outside ORNL, namely, Argonne National Laboratory, Vallecitos Nuclear Center, and Nuclear Fuel Services. The gas-release equipment is being modified for use up to 1800°C from the previous limit of 1200°C.

A Kollmorgen periscope with 2X, 4X, 10X, and 20X magnifications was installed through the wall for viewing within the storage cell. With the periscope, samples or unique materials can be examined visually. Those items of interest can be photographed with a camera especially fabricated for attachment to the periscope.

Equipment for fluorinating the uranium from 50-g samples of MSRE fuel salt was installed and tested in a work cell. The fluorinated products will be further treated and then measured for uranium isotopes present. The apparatus will also be used to improve the measurement of the uranium concentration in fuel-salt samples from the MSRE.

A chapter titled "Sampling, Sample Dissolution, Evaporation, and Combustion" was completed for inclusion in Vol. 10 of SERIES IX, **Progress in Nuclear Energy.**[6]

12.4 RADIOISOTOPES-RADIOCHEMISTRY LABORATORY

E. I. Wyatt

The types of work performed in the Radioisotopes-Radiochemistry Laboratory have remained essentially unchanged for the past two years. The total volume of analyses was only about 75% of that of 1967. This decrease was due primarily to a reduction of the funds available to the Chemical Technology and Reactor Chemistry Divisions.

New equipment put into service includes an IBM card-punch machine, a 4096-channel pulse-height analyzer, and a flame spectrophotometer. Cell 5 of the HRLAF (Building 3019) was cleaned and put into service, and cell 7 was cleaned and stripped and is out of service.

The largest part of our radiometric analyses were performed in support of the Fast Breeder Reactor Fuel Processing Program of the Chemical Technology Division. Almost all these analyses were done by computer resolution of gamma-ray spectrometric data. Another large block of work consisted in the dissolution and subsequent radiometric and radiochemical separation of various samples from the core and pump bowl of the MSRE.

The half-lives of 36 radionuclides were reported (Sect. 8.6.e).[7] Decay-scheme data were collected for all radionuclides that have a half-life of 1 min or longer

[5] S. Nakamura, N. Azuma, and Z. Arai, "Decomposition of Boron Nitride with Acid and the Effects of Some Oxidizing Agents," *Kogyo Kagaku Zasshi* 63, 903 (1960); ORNL-tr-1242.

[6] C. E. Lamb, "Sampling, Sample Dissolution, Evaporation, and Combustion," 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.

[7] S. A. Reynolds and J. F. Emery, "Half-Lives of Radionuclides-III," *Nucl. Sci. Eng.* 32, 46 (1968).

and also gamma radiations of 50 keV or greater. A computer program was written to list the data in readily accessible form for qualitative Ge(Li) spectrometry. Also, a computer program for the quantitative resolution of Ge(Li) spectra is almost completed.

Many $LiF\text{-}BeF_2$ salt samples were analyzed for trace quantities of europium, lanthanum, and neodymium by neutron activation analysis. These samples were submitted in aqueous solutions and were irradiated in the air-cooled HB-5 facility in the LITR at a flux of 5×10^{11} neutrons cm^{-2} sec^{-1}. Under the conditions of the experiment, the detection limits were 1, 10, and 6 ppb for europium, lanthanum, and neodymium, respectively. A similar technique was used to determine ^{129}I to a limit of 20 ppb by use of a flux monitor instead of a standard.

12.5 RADIATION CONTROL AND SAFETY

C. L. Burros

The absolute filters in the off-gas system at the HRLAF (Building 3019) were replaced and tested. The containment liner in cell 5 was dismantled and placed in a plastic-lined coffin. After the lid was secured, the coffin was wrapped with plastic and then moved to the burial ground. Cell 5 was decontaminated and painted. The 8-ft hood in the southwest zone was moved to the burial ground, and the air conditioner was relocated to permit the installation of an air lock. These jobs were completed with minimum spread of contamination and exposure to personnel.

On July 15, construction of the West Annex to Building 2026 (HRLAL) was begun.

The annual safety inspection of all work areas in the Division was made with a safety engineer July 9–12. This inspection included checks for such items as poor housekeeping, defective electrical equipment, unsecured gas cylinders, unguarded mechanical or experimental equipment, improper storage and use of flammable liquids, lack of use of protective personal equipment, or any unsafe conditions. A written report on the inspection was made to the Division Director. Twenty-one medical-treatment injuries were reported for the past 12-month period. Most of these were hand injuries due to glass breakage. On several occasions, safety films were shown at Buildings 2026 and 4500S for employees of the Division.

12.6 QUALITY CONTROL

G. R. Wilson

The fixed $2S$ values of the control programs that have been in operation for over a year remained at the same level. The 2.8% decrease in the quality level for the past year is due to the newly initiated control programs whose $2S$ limits are not yet established.

Several new control programs were established. The General Hot Analyses Laboratory now has a control program for the determination of plutonium in the presence of uranium by coulometric titration. Control programs for the following determinations are now in operation in the General Analyses Laboratory: flame photometric lithium, dibenzylmethane colorimetric uranium, and arsenazo III colorimetric thorium; the last three determinations are done in the presence of bismuth. The High-Level Alpha Radiation Laboratory has new control programs for coulometric titration of plutonium, dibenzylmethane colorimetric uranium, and arsenazo III colorimetric thorium; the last three are run in the presence of bismuth.

The decrease in the total number of control results is due to fewer samples from the MSRE. The quality level by laboratory is shown in Table 12.1.

Table 12.1. Distribution by Laboratories of Control Tests for July 1967–June 1968

Laboratory	Number of Control Results		Quality Level (%)[a]	
	Total	Outside Fixed Limits	1967	1968
General Hot-Analyses	1235	121	93.89	90.20
General Analyses	2022	131	95.75	93.52
High-Level Alpha Radiation	29	11		62.07
Total	3286	263	94.82	92.00

[a]Control results within prescribed $2S$ limits.

The number of control results and their specific characteristics are given in Table 12.2.

Table 12.2. Distribution by Methods of Control Results for July 1967–June 1968

Type of Method	Constituent	Number of Control Programs		Number of Control Results	
Amperometric	Chromium	1		147	
	Zirconium	1	2	119	266
Colorimetric (Spectrophotometric)	Aluminum	1		53	
	Chromium	1		118	
	Iron	2		357	
	Molybdenum	1		38	
	Nickel	2		281	
	Thorium	4		219	
	Uranium	4	15	223	1289
Coulometric titration	Plutonium	2		41	
	Uranium	3	5	950	991
Flame photometric	Lithium	1	1	72	72
Fluorometric	Uranium	2	2	283	283
Gravimetric	Carbon	2	2	202	202
Photoneutron	Beryllium	1	1	80	80
Polarographic	Uranium	1	1	83	83
Volumetric	Aluminum	1	1	20	20
Total			30		3286

Part C. ORNL Master Analytical Manual

13. ORNL Master Analytical Manual

M. T. Kelley
Helen P. Raaen

13.1 CUMULATIVE INDEXES TO THE *ORNL MASTER ANALYTICAL MANUAL*

The cumulative indexes to the *ORNL Master Analytical Manual* were not updated in 1967. However, they will be in 1968 to indicate changes made in the content of the *Manual* during both 1967 and 1968.

13.2 MAINTENANCE OF THE *ORNL MASTER ANALYTICAL MANUAL*

No supplement to the reprinted form of the *ORNL Master Analytical Manual* was published in 1967. The tenth supplement will be published in 1968 and will contain the new methods and revisions issued in both 1967 and 1968.

Altogether, five new methods were added to the *Manual*; one of these was for the purpose of record only. Revisions were made to six methods. (See "Presentation of Research Results.") The status of nine methods for the Transuranium Process was changed from tentative to formal.

A survey was made to determine what methods now contained in the *Manual* are no longer used, what revisions to existing methods are needed, and what new methods are required for current or anticipated analytical work. On the basis of the survey, method-writing assignments were made to persons in the Analytical Chemistry Division.

Part D. Activities Related to Educational Institutions

14. 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 125 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.

14.1 ADVISORY COMMITTEE MEMBERS AND CONSULTANTS

The Advisory Committee members and 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.

14.2 ORAU RESEARCH PARTICIPANTS

Two ORAU Research Participants were with us in the summer of 1968.

J. C. Guyon, Professor of Chemistry, University of Missouri, Columbia, worked with *W. D. Shults*, Methodology Group, to investigate the voltammetric and coulometric behavior of plutonium, neptunium, and americium in their various oxidation states in several aqueous media (Sect. 2.7.d).

C. R. Spell, Chairman, Department of Chemistry, Emory and Henry College, Emory, Va., worked with *D. J. Fisher*, Analytical Instrumentation Group, to survey the dc polarographic characteristics of urinary-constituent biochemicals that are chromatographically separated in the ORNL Body Fluids Analyses Program and to assess the feasibilities of using dc polarography for the qualitative and quantitative monitoring of column effluent streams and for laboratory identification and concentration analyses of biochemicals in column fractions (Sect. 1.8).

An S Contract Holder under the ORAU Participation Agreement worked with us from July 21 through August 3.

T. T. Davies, Assistant Professor of Geology, University of South Carolina, Columbia, worked with *H. W. Dunn* in the X-Ray and Spectrochemistry Group to evaluate the electron microprobe for use in studying the concentration of stable elements in skeletal material of benthic invertebrates collected on the West Coast of Puerto Rico. This work was a continuation of work he began in the summer of 1967 at the Puerto Rico Nuclear Center.

14.3 GRADUATE THESIS RESEARCH PROGRAMS

14.3.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, Knoxville, 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 (Sect. 3.2.c).

14.4 ORNL LOANEES

R. W. Morrow, a graduate student in the Department of Chemistry with *Professor J. A. Dean* at the University of Tennessee, Knoxville, is conducting research toward the Ph.D. degree. In the Analytical Methodology Group with *W. D. Shults* and *M. R. Guerin,* he is studying flame-emission and atomic-absorption techniques as gas chromatographic detectors and numerous uses of these specific detectors in the gas chromatography of silicon derivatives.

L. P. Turner III, a Visiting Professor from the Department of Chemistry, University of Tennessee, Knoxville, continued the pyrolytic gas chromatography of macromolecules with *A. S. Meyer*, Reactor Projects Group. He has submitted an article on this work for publication.

F. L. Whiting, a graduate student in the Department of Chemistry with *Professor Gleb Mamantov* at the University of Tennessee, Knoxville, 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 (Sect. 3.2.*i*).

J. C. Wolford, a first-year graduate student in the Department of Chemistry with *Professor J. A. Dean* at the University of Tennessee, Knoxville, is doing his doctoral thesis research in the Analytical Biochemistry Group with *Gerald Goldstein*. He is studying the separation of compounds of biological interest by ligand-exchange chromatography.

14.5 SUMMER STUDENT PROGRAMS

In the summer of 1968, five students participated in summer student programs.

14.5.a ORAU Student Trainee Program

Linda J. Crist, now a senior at Arkansas State University, Jonesboro, worked with *Helen P. Raaen*, Methodology Group, to investigate the thin-layer chromatography of steroids (Sect. 2.2.e) and the application of mass spectrometry (Sect. 2.2.f) and infrared spectrometry (Sect. 2.2.g) to the identification and measurement of steroids so isolated.

Marcia A. Lepri, now a senior at Mount Holyoke College, South Hadley, Mass., worked in the Nuclear and Radiochemical Group with *W. S. Lyon* on the decay scheme of ^{103}Pd-^{103}Rh (Sect. 8.3.e) and with *J. F. Emery* on a number of special activation-analysis problems including the calibration of Ge(Li) detectors (Sect. 8.3.f).

D. R. Martin, now a senior at Sacramento State College, Sacramento, Calif., worked with *W. L. Maddox* and *D. J. Fisher,* Analytical Instrumentation Group, to examine the performance of D.M.E.'s with a digital instrument computer and to explore the utilities of digital data acquisition and ensemble averaging in polarography (Sect. 1.21).

14.5.b ORNL Undergraduate Program

J. D. Lodmell, a graduate of Concord College, Athens, W.Va., now a first-year graduate student at the University of Tennessee, Knoxville, worked with the Reactor Projects Group. With *A. D. Horton* he performed special gas-chromatographic analyses and used gas chromatography as a preparative technique (Sect. 2.1.g). With *R. F. Apple* and *A. S. Meyer* he assisted in the development of analytical methods for radioactive fluoride salts (Sects. 3.1.b and 3.1.c).

F. V. Newsome, a graduate of Harvard University, Cambridge, Mass., now a first-year student at the University of West Virginia Medical School, Morgantown, worked with *I. B. Rubin*, Analytical Biochemistry Group. He assisted in the development of an isotope-dilution technique for the determination of amino acids.

14.6 ALIEN GUESTS IN RESIDENCY

One scientist from another country is now a guest in the Analytical Chemistry Division.

Guest	Sponsor	Division Group
Young Kuk Kim Atomic Energy Research Institute Chyungryang-ri Sequl, Korea	International Atomic Energy Agency	Nuclear and Radiochemistry 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.

W. L. Belew

W. L. Belew is on temporary assignment to the Analytical Chemistry Group at the Australian Atomic Energy Commission Research Establishment at Lucas Heights, N.S.W., Australia 2232, under the USAEC/AAEC exchange of technical personnel. At the Second Australian Conference on Electrochemistry, University of Melbourne, February 19–23, 1968, he presented a paper on an ORNL cell design for minimizing iR error in controlled-potential polarography. He has assisted in the check-out of the ORNL model Q-2792 polarograph and the model Q-2942 D.M.E. drop-time controller fabricated there and has demonstrated the satisfactory performance of these instruments compared with that of the Davis A-1660 polarograph. Now he is doing electroanalytical research with T. M. Florence under the direction of Dr. Lloyd Smythe (Sects. 1.7 and 1.11).

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

ASTM Committee E-21 on Fuel Fabrication

J. M. Dale

Chairman: General Session
Analytical Chemistry Division
155th National American Chemical Society Meeting
San Francisco, California

Secretary-Treasurer:
Analytical Group
East Tennessee Section
American Chemical Society

J. S. Eldridge

Secretary: Subcommittee on the Use of Radioactivity Standards
Committee on Nuclear Sciences
National Academy of Sciences–National Research Council (NAS-NRC)

C. Feldman

Chairman: Task Group on Photographic Photometry
Committee E-2 on Emission Spectroscopy
American Society for Testing and Materials (ASTM)

Member: Editorial Board
Optics and Spectroscopy

D. J. Fisher

Member: Editorial Board
Chemical Instrumentation

A. D. Horton

Member: Subcommittee III, Research
ASTM Committee E-19 on Chromatography

C. A. Horton

C. A. Horton is spending a second year at the Seibersdorf Laboratory of the International Atomic Energy Agency, Vienna, Austria. He is there as head of the analytical chemistry work.

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

Member: 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 (1967–1968)
(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 N43-3, Equipment for Non-Medical Radiation Applications
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)

T. R. Mueller

Session Chairman:
21st Annual Summer Symposium
"Computer Assisted Analytical Chemistry"
The Pennsylvania State University, June 19–21, 1968

S. A. Reynolds

Vice-Chairman:
Subcommittee 4, Methods of Radiochemical Analysis
ASTM Committee D-19

Member: ASTM Committee D-19 on Water

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)

Task Group on Nuclear Data and Radiation Safety (E-10)

Subcommittee on Use of Radioactivity Standards, National Research Council

Task Group 1 of Scientific Committee 18 (Standards) of National Council on Radiation Protection and Measurements (NCRP)

Member: Subcommittee on Radiological Methods for Biological and Environmental Samples
American Public Health Association (APHA)

E. Ricci

Member: Executive Committee
Radiations and Isotopes Division
American Nuclear Society (ANS)

W. D. Shults

Member: Program Committee
Analytical Group
East Tennessee Section
American Chemical Society

Committee E-19 (Chromatography)
American Society for Testing and Materials

P. F. Thomason

Participant: Cloud Gap-34 Project, October 16–23, 1967

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)

Chairman: Subcommittee on Reference Materials
NAS-NRC Committee on Analytical Chemistry

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

BOOK

AUTHORS	TITLE	PUBLISHER
1 Raaen, V. F.,* G. A. Ropp,[1] Helen P. Raaen	*Carbon-14*	McGraw-Hill, New York, 1968

CONTRIBUTIONS TO BOOKS

AUTHOR(S)	TITLE	PUBLISHER
2 Bate, L. C., F. F. Dyer	"Forensic Application of Trace Elements in Hair," pp. 247–259 in *Proceedings of the First International Conference on Forensic Activation Analysis* held September 19–21, 1966, in San Diego, Calif. (V. P. Guinn, ed.), GA-8171 (1967)	Gulf General Atomic, Inc., San Diego, Calif.
3 Goldstein, G., W. L. Maddox, I. B. Rubin	"A Semi-Automated Filter Paper Disk Technique for the Determination of Transfer Ribonucleic Acid," pp. 47–49 in *Technicon Symposia 1967, Automation in Analytical Chemistry,* New York, October 2–4, 1967	Mediad, New York, 1968
4 Lyon, W. S.	"Physical Techniques of Activation," pp. 13–30 in *Nuclear Activation Techniques in the Life Sciences* (Proceedings of the Symposium on Nuclear Activation Techniques in the Life Sciences, IAEA, Amsterdam, May 8–12, 1967)	International Atomic Energy Agency, Vienna, 1967

THESIS

AUTHOR	TITLE	PUBLISHER
5 Lyon, W. S.	*Validity of Some Tests of Intellectual Ability for Predicting Grades in Related Craft Training* (M.S. thesis)	University of Tennessee, Knoxville, August 1968

[1] Professor of Chemistry, Coker College, Hartsville, S.C.

ARTICLES

	AUTHOR(S)	TITLE	PUBLISHER
6	Bamberger, C. E.,* J. P. Young, C. F. Baes, Jr.*	"Containment of Molten Fluorides in Silica. Part I. Effects of Temperatures on the Spectrum of U^{4+} in Molten BeF_2-LiF Mixtures"	*J. Inorg. Nucl. Chem.* 30, 1979 (1968)
7	Bate, L. C., F. F. Dyer	"Particle Size Distribution of Particles from 10 to 2000 Microns by Sedimentation Analysis"	*Anal. Chem.* 40, 468 (1968)
8	Bell, J. T.,* R. E. Biggers[2]	"Absorption of the Uranyl Ion in Perchlorate Media. III. Resolution of the Ultraviolet Band Structure; Some Conclusions Concerning the Excited State of UO_2^{2+}"	*J. Mol. Spectry.* 25, 312 (1968)
9	Burtis, C. A., G. Goldstein	"Terminal Nucleoside Assay of Ribonucleic Acid by Ligand-Exchange Chromatography"	*Anal. Biochem.* 23, 502 (1968)
10	Burtis, C. A., K. S. Warren*	"Identification of Urinary Constituents Isolated by Anion Exchange Chromatography"	*Clin. Chem.* 14, 290 (1968)
11	Dyer, F. F., L. C. Bate, L. H. Thacker*	"A Film Recording Radiation-Event Monitor for Pulsed X and Gamma Radiation"	*Nucl. Instr. Methods* 58, 157 (1968)
12	Dyer, F. F., L. C. Bate, J. E. Strain	"Three-Dimensionally Rotating Sample Holder for 14-Million Electron Volt Neutron Irradiations"	*Anal. Chem.* 39, 1907 (1967)
13	Farrar, L. G., J. H. Cooper, F. L. Moore	"Chromatographic-Solvent Extraction Isolation of Berkelium-249 from Highly Radioactive Solutions and Its Determination by Beta Counting"	*Anal. Chem.* 40, 1602 (1968)
14	Hahn, R. L.*	"The Recoil Technique and Its Possible Use in Activation Analysis"	*Anal. Chem.* 40, 219 (1968)
15	Hahn, R. L.,* E. Ricci	"Interactions of ^3He Particles with B, N, Na and Be"	*Nucl. Phys.* A101, 353 (1967)
16	Hertel, G. R.	"Surface Ionization. III. The First Ionization Potentials of the Lanthanides"	*J. Chem. Phys.* 48, 2053 (1968)
17	Hupf, H. B.,* J. S. Eldridge, J. E. Beaver*	"Production of Iodine-123 for Medical Applications"	*Intern. J. Appl. Radiation Isotopes* 19, 345 (1968)
18	Kubota, H.	"Rapid Distillation Separation of Microgram Quantities of Fluoride"	*Microchem. J.* 12, 525 (1967)
19		"Chlorine Production by Gamma Radiolysis of Acid Chloride Solutions and Its Effect on Analytical Procedures"	*Anal. Chem.* 40, 271 (1968)
20	Lyon, W. S., E. Ricci, H. H. Ross	"Nucleonics"	*Anal. Chem.* 40(5), 168R (1968)
21	Maddox, W. L., M. T. Kelley	"An Automatic Sampler for Obtaining Discrete Samples from the Output of a Continuous-Flow Analyzer"	*Chem. Instr.* 1, 105 (1968)
22	Mamantov, G.,[3] D. L. Manning	"Electrochemical Reduction of U(IV) in Molten LiF-BeF_2-ZrF_4 (65.6–29.4–5.0 mole %). Evidence for Adsorption of U(IV)"	*J. Electroanal. Chem.* 18, 309 (1968)
23	Manning, D. L., G. Mamantov[3]	"Disproportionation of Electrochemically-Generated Uranium(V) in Molten LiF-BeF_2-ZrF_4 at 500°C"	*J. Electroanal. Chem.* 18, 137 (1968)

[2]Present address: Coca Cola Research Laboratory, Atlanta, Ga.

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

24 Moore, F. L.	"New Method for the Rapid Separation of Berkelium(IV) from Cerium(IV) by Anion Exchange"	*Anal. Chem.* **39**, 1874 (1967)
25 Mueller, T. R.	"On the Separation of Half-Wave Potentials Required for Resolution of Simple, Reversible, Overlapping Waves in Stationary Electrode Voltammetry"	*Chem. Instr.* **1**, 113 (1968)
26 Potterton, Susan S.,[4] W. D. Shults	"An Evaluation of the Performance of the Nitrate-Selective Electrode"	*Anal. Letters* **1**, 11 (1967)
27 Raaen, Helen P., Frances E. Kraus[5]	"Resolution of Complex Mixtures of Nucleic Acid Bases, Nucleosides, and Nucleotides by Two-Dimensional Thin-Layer Chromatography on Polyethyleneimine-Cellulose"	*J. Chromatog.* **35**, 531 (1968)
28 Reynolds, S. A.	"Availability of Standards for Common Radionuclides"	*Isotopes Radiation Technol.* **5**, 118 (1968)
29 Reynolds, S. A., J. F. Emery, E. I. Wyatt	"Half-Lives of Radionuclides – III"	*Nucl. Sci. Eng.* **32**, 46 (1968)
30 Ricci, E., R. L. Hahn*	"Rapid Calculation of Sensitivities, Interferences, and Optimum Bombarding Energies in ^{3}He Activation Analysis"	*Anal. Chem.* **40**, 54 (1968)
31 Ross, H. H.	"Analytical Applications of the Secondary Effects of Radiation: Precision Photometry Using a Radioisotopic Light Source"	*Record Chem. Progr.* **28**, 261 (1967)
32 Shults, W. D., H. H. Ross, E. Ricci	"Synopsis of Eleventh Conference on Analytical Chemistry in Nuclear Technology"	*Anal. Chem.* **40**(3), 71A (1968)
33 Shults, W. D., D. J. Fisher, W. B. Schaap[6]	"Controlled-Potential Differential DC Polarography. V. Subtractive Polarography"	*Chem. Instr.* **1**, 7 (1968)
34 White, J. C. (Chairman), M. D. Cooper,[7] H. Fischbach,[7] C. Merrit, Jr.,[7] G. H. Morrison,[7] A. Thomas,[7] S. M. Tuthill,[7] S. Vigo[7]	"Standard Reference Materials as Viewed by the Laboratory Supervisor – A Status Report"	*Anal. Chem.* **40**(3), 24A (1968)
35 White, J. C.	Book Review of: *Analytical Chemistry of Plutonium*, P. N. Palei, ed., Daniel Davey & Co., New York, 1967	*Anal. Chem.* **40**(1), 96A (1968)
36 Zittel, H. E., T. M. Florence[8]	"A Voltammetric Method for Fluoride"	*Anal. Chim. Acta* **40**, 27 (1968)

[4]ORAU Student Trainee from Randolph-Macon Woman's College, Lynchburg, Va.; summer 1967.

[5]ORAU Student Trainee from Nazareth College, Nazareth, Ky.; summer 1967.

[6]Professor of Chemistry, Indiana University, Bloomington.

[7]Member of the Subcommittee on Reference Materials, Committee on Analytical Chemistry, Division of Chemistry and Chemical Technology, National Academy of Sciences–National Research Council.

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

REPORTS

AUTHOR(S)	TITLE	REPORT NO. AND DATE
37 Butts, W. C.	*Catalog of Gas Chromatographic Retention Data for Urinary Constituents and Related Compounds*	ORNL-TM-2374 (May 13, 1968)
38 Guyon, J. C.,[9] J. R. Stokely, W. D. Shults	*Spectrophotometric Determination of Total Plutonium: Observations on the Reactions Between Arsenazo and Pu(III), Pu(IV) and Pu(VI)*	ORNL-TM-2108 (Jan. 2, 1968)
39 Kelley, M. T.	*Statistical Quality Control Report, Analytical Chemistry Division, July Through September 1967*	Oct. 20, 1967 (unpublished)
40	*Statistical Quality Control Report, Analytical Chemistry Division, October Through December 1967*	Jan. 12, 1968 (unpublished)
41	*Statistical Quality Control Report, Analytical Chemistry Division, January Through March 1968*	Apr. 18, 1968 (unpublished)
42	*Statistical Quality Control Report, Analytical Chemistry Division, April Through June 1968*	Oct. 14, 1968 (unpublished)
43	*Report of Foreign Travel to Czechoslovakia, Yugoslavia, Austria, Greece, August 29, 1967, Through October 8, 1967*	Dec. 7, 1967 (unpublished)
44	*Report of Foreign Travel to Australia, February 1, 1968, Through March 7, 1968*	May 7, 1968 (unpublished)
45 Lupica, S. B.	*Liquid Scintillation Counting of Weak Beta Emitters in Aqueous Solution Using Triton X-100*	Jan. 8, 1968 (unpublished)
46 Macklin, R. L.,* J. H. Gibbons,* T. H. Handley	*Proton Reaction Analysis for Lithium and Fluorine in Graphite, Using a Slit Scanning Technique*	ORNL-TM-2238 (July 1968)
47 Meyer, A. S., Jr.	*Report of Foreign Travel to Czechoslovakia, Italy, Switzerland, Germany, West Germany, Norway, and England, September 2, 1967, Through September 30, 1967*	Nov. 2, 1967 (unpublished)
48 Mueller, T. R., H. C. Jones, D. J. Fisher, R. W. Stelzner	*Modification of the ORNL Model Q-2564 High-Sensitivity Coulometric Titrator*	ORNL-TM-2175 (Mar. 18, 1968)
49 Raaen, Helen P., ed.	*Analytical Chemistry Division Annual Progress Report for Period Ending October 31, 1967*	ORNL-4196 (January 1968)
50 Raaen, Helen P., Ann S. Klein,* eds.	*Cumulative Indexes to the Analytical Chemistry Division Annual Progress Reports 1964–1967*	ORNL-3904, rev. 2 (January 1968)
51 Reed, R. E.,* C. W. Dean,* R. E. McDonald,* J. F. Emery	*Sources of Contamination During Electron-Beam Melting*	ORNL-TM-2208 (July 1968)
52 Ricci, E.	*Report of Foreign Travel to France, Italy, Germany, The Netherlands, and Harwell, England, September 5, 1967, Through September 26, 1967*	Nov. 2, 1967 (unpublished)
53 Strain, J. E., W. J. Ross, G. A. West,* J. W. Landry*	*The Design and Evaluation of a Delayed-Neutron Leached-Hull Monitor*	ORNL-4135 (February 1968)

[9]ORAU Research Participant; Associate Professor of Chemistry, University of Missouri, Columbia.

54	White, J. C.	*Analytical Chemistry Research and Development Monthly Summary – November 1967*	Nov. 29, 1967 (unpublished)
55		*Analytical Chemistry Research and Development Monthly Summary – December 1967*	Jan. 4, 1968 (unpublished)
56		*Analytical Chemistry Research and Development Monthly Summary – January 1968*	Jan. 26, 1968 (unpublished)
57		*Analytical Chemistry Research and Development Monthly Summary – February 1968*	Feb. 28, 1968 (unpublished)
58		*Analytical Chemistry Research and Development Monthly Summary – March 1968*	Mar. 26, 1968 (unpublished)
59		*Analytical Chemistry Research and Development Monthly Summary – April 1968*	Apr. 29, 1968 (unpublished)
60		*Analytical Chemistry Research and Development Monthly Summary – May 1968*	May 29, 1968 (unpublished)
61		*Analytical Chemistry Research and Development Monthly Summary – June 1968*	June 28, 1968 (unpublished)
62		*Analytical Chemistry Research and Development Monthly Summary – July 1968*	July 26, 1968 (unpublished)
63		*Analytical Chemistry Research and Development Monthly Summary – August 1968*	Aug. 28, 1968 (unpublished)
64		*Analytical Chemistry Research and Development Monthly Summary – September 1968*	Sept. 30, 1968 (unpublished)
65	Zittel, H. E.	*Report of Foreign Travel to England, Switzerland, Italy, Germany, and The Netherlands, July 10, 1968, Through July 31, 1968*	Aug. 23, 1968 (unpublished)
66	Anonymous	"Polarographic Drop Time Controller/Q-2942/" in *Engineering Materials List*, TID-4100	CAPE-1651
67		"Controlled-Potential D. C. Polarograph-Voltammeter/Q-2792" in *Engineering Materials List*, TID-4100	CAPE-1652
68		"Controlled-Potential and Controlled-Current Cyclic Voltammeter" in *Engineering Materials List*, TID-4100	CAPE-1653
69		"Automatic Potentiometric Titrator" in *Engineering Materials List*, TID-4100	CAPE-368 (revised)
70		"High-Sensitivity Coulometric Titrator/Q-2564/" in *Engineering Materials List*, TID-4100	CAPE-1196 (revised)
71		"Servo-Controlled Pipetters" in *Engineering Materials List*, TID-4100	CAPE-1220 (revised)

METHODS ISSUED TO THE *ORNL MASTER ANALYTICAL MANUAL*

New Methods

FORMAL ISSUES

	AUTHOR	TITLE	NUMBER	DATE
72	Mueller, T. R.	"Cyclic Voltammeter, Controlled-Potential, Controlled-Current"	1 003045 9 003045	9-1-68
73	Maddox, W. L.	"Filter Photometers, Remotely Operated, ORNL Models Q-1734 and 64-3171"	1 003092 9 003092	3-1-68

74 Wyatt, E. I.	"Strontium-92, Product Analysis Guide"	9 0733805	1-27-67
75	"Strontium-90, Product Analysis Guide"	9 0733806	1-27-67

RECORD-COPY ISSUES

AUTHORS	TITLE	NUMBER	DATE
76 Crist, Linda J.,[10] Helen P. Raaen	"Separation of Six Corticosteroids by Thin-Layer Chromatography on Gelman ITLC-SA Medium"	1 00714	8-12-68

Revised Methods

AUTHOR(S)	TITLE	NUMBER	DATE
77 Jones, H. C.	"Coulometric Titrator, Controlled-Potential, High-Sensitivity, ORNL Model Q-2564"	1 003025 9 003025	R.3-1-68
78 Wagner, E. B., W. L. Maddox	"Automatic Potentiometric Titrator, Velocity-Servo, ORNL Models Q-1728 and Q-1728A"	1 003028 9 003028	R.3-15-68
79 Wyatt, E. I.	"Proportional Beta Counter, Windowless"	2 00352 5 00352 9 00352	R.3-1-68
80 Rickard, R. R.	"Ruthenium Radioactivity in Aqueous Solutions, Pyridine Extraction–Gamma Counting Method"	2 21734 9 062310	R.3-15-68
81 Apple, R. F.	"Fluoride in MSRE Fuel, Pyrolysis Method"	9 021203	R.3-1-68
82 McDowell, Betty L., D. C. Canada	"Niobium in Mixtures of Fluoride Salts, Spectrophotometric Thiocyanate Method"	9 0922550	R.3-15-68

Oral Presentations

AT MEETINGS OF PROFESSIONAL SOCIETIES, CONFERENCES, AND THE LIKE

AUTHOR(S)	TITLE	PRESENTED AT
83 Belew, W. L.,[11] D. J. Fisher, M. T. Kelley, J. A. Dean[12]	"A Cell Design for Minimizing iR Error in Controlled-Potential Polarography"	Second Australian Conference on Electrochemistry, Royal Australian Chemical Institute, Melbourne, Australia, Feb. 19–23, 1968

[10] ORAU Student Trainee from Arkansas State University, Jonesboro.

[11] Speaker.

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

84	Burtis, C. A.	"Fractionation of Human Urine by Gel Chromatography"	Annual Meeting, American Association of Clinical Chemists, Washington, D.C., Aug. 9–23, 1968
85	Burtis, C. A.,[11] G. Goldstein	"Assay of 3′-Terminal Nucleosides of Transfer Ribonucleic Acids by Ligand Exchange Chromatography"	Southeastern Regional Meeting, American Chemical Society, Atlanta, Ga., Nov. 1–3, 1967
86	Burtis, C. A.,[11] K. S. Warren*	"Identification of Urinary Constituents Isolated by Anion-Exchange Chromatography"	Symposium on Urinary Constituents of Low Molecular Weight, Oak Ridge National Laboratory, Oak Ridge, Tenn., Nov. 30–Dec. 1, 1967
87	Dale, J. M.,[11] R. F. Apple, A. S. Meyer	"A Transpiration Method for the Determination of U(III) in Radioactive Molten Salt Reactor Fuel"	155th Meeting, American Chemical Society, San Francisco, Calif., Mar. 31–Apr. 5, 1968
88	Dean, C. W.,*[11] R. E. McDonald,* R. E. Reed,* J. F. Emery	"Sources of Contamination During Electron Beam Melting"	Third International Conference on Electron and Ion Beam Science and Technology, Boston, Mass., May 6–9, 1968
89	Feldman, C.	"Beam-Splitter for Use in Calibrating Spectrographic Emulsions"	7th National Meeting, Society for Applied Spectroscopy, Chicago, Ill., May 13–17, 1968
90	Kelley, M. T.	"What Industry Expects of the Graduate in Chemistry or Chemical Engineering"	19th Southeastern Regional Meeting, American Chemical Society, Atlanta, Ga., Nov. 1–3, 1967
91		"Some Approaches to More Rapid Analysis"	1967 Eastern Analytical Symposium, New York, Nov. 8–10, 1967
92		"Analysis for Nuclear Reactors"	Seminar, AAEC Research Establishment, Lucas Heights, New South Wales, Australia, Feb. 15, 1968
93		"Electroanalytical Research at Oak Ridge National Laboratory"	Seminar, University of New South Wales, Sydney, Australia, Feb. 16, 1968
94		"Electronic Instrumentation" (Invited Lecture)	Second Australian Conference on Electrochemistry, Royal Australian Chemical Institute, Melbourne, Victoria, Australia, Feb. 21, 1968
95		"Electroanalytical Research at Oak Ridge National Laboratory"	Analytical Chemistry Group, Perth, Australia, Feb. 23, 1968
96		"Electroanalytical Research at Oak Ridge National Laboratory"	Analytical Chemistry Group, Adelaide, Australia, Feb. 26, 1968
97		"The Analytical Chemistry of the Actinide Elements" (Invited Paper)	Royal Australian Chemical Institute, Canberra, Australia, Feb. 28, 1968
98		"Electroanalytical Research at Oak Ridge National Laboratory"	Analytical Chemistry Group, Brisbane, Australia, Mar. 1, 1968
99		"The Analytical Chemistry of the Actinide Elements" (Invited Paper)	Royal Australian Chemical Institute, Newcastle, Australia, Mar. 4, 1968
100		"The Role of the Large National Research Laboratories in the USA"	Royal Australian Chemical Institute, University of New South Wales, Australia, Mar. 6, 1968
101		"Operational Amplifier Systems in Electroanalytical Chemistry at Oak Ridge National Laboratory" (Invited Paper)	Central Regional Meeting, American Chemical Society, Akron, Ohio, May 9–10, 1968
102		"A Comparison of Methods of Analytical Chemistry"	ORAU Neutron Activation Course, Special Training Division, Oak Ridge, Tenn., Aug. 12–30, 1968
103	Kubota, H.	"Homogeneous Precipitations Using Radiolytically Generated Hydrochloric Acid"	155th Meeting, American Chemical Society, San Francisco, Calif., Mar. 31–Apr. 5, 1968
104	Lyon, W. S.	"Prospects and Problems for Activation Analysis of Ocean Sediments"	D.I.D. Roundtable on Development of Nuclear Techniques for Rapid Analysis of Ocean Elements, Washington, D.C., Oct. 16, 1967

105		"Activation Analysis Interferences"	ORAU Neutron Activation Analysis Course, Special Training Division, Oak Ridge, Tenn., Aug. 28, 1968
106		"Recent Advances in Activation Analysis"	Midwest Regional Meeting, American Chemical Society, Columbia, Mo., Nov. 2–3, 1967
107		"Basic Concepts of Activation Analysis"	Radioisotopes in Medicine in Vitro Studies, ORAU, Oak Ridge, Tenn., Nov. 13–16, 1967
108	Manning, D. L., M. T. Kelley,[11] G. Mamantov[3]	"Electro-reduction of Uranium(IV) in Molten Lithium Fluoride-Beryllium Fluoride-Zirconium Fluoride at Fast Scan Rates and Short Transition Times"	Second Australian Electrochemistry Conference of Royal Australian Chemical Institute, Melbourne, Australia, Feb. 21, 1968
109	Manning, D. L.,[11] G. Mamantov[3]	"Disproportionation of Electrochemically Generated Uranium(V) in Molten $LiF-BeF_2-ZrF_4$ at 500°C"	155th Meeting, American Chemical Society, San Francisco, Calif., Mar. 31–Apr. 4, 1968
110	Manning, D. L.,[11] J. M. Dale	"Voltammetric Studies of Cr(II) in Molten Lithium Fluoride-Beryllium Fluoride-Zirconium Fluoride at 500°C"	156th Meeting, American Chemical Society, Atlantic City, N.J., Sept. 8–13, 1968
111	Ricci, E.	"Experience and Future Outlook on ^{3}He-Activation Analysis" (Invited Lecture)	National Bureau of Standards, Washington, D.C., Oct. 23, 1967
112		"Activation Analysis with ^{252}Cf Neutrons"	ORNL Transuranium Research Laboratory, Oak Ridge, Tenn., Jan. 11, 1968 (Lecture to visiting ORAU Group)
113	Ricci, E.,[11] T. H. Handley	"Neutron Activation Analysis with ^{252}Cf Sources"	Transplutonium Program Committee Meeting. The Transplutonium Elements: Availability and Applications, AEC Headquarters, Germantown, Md., Apr. 18, 1968
114	Ricci, E.	"New Developments in Activation Analysis" (in Spanish)	Argentinian Atomic Energy Commission, Buenos Aires, Argentina, July 29, 1968
115	Roche, M. F.,*[11] K. S. Toth,* R. L. Hahn,* C. R. Bemis,* T. H. Handley	"Investigation of γ-Rays in the Decay Chain $^{172}Hf \rightarrow {}^{172}Lu \rightarrow {}^{172}Yb$"	Spring Meeting, American Physical Society, Washington, D.C., Apr. 22–25, 1968
116	Ross, H. H.	"Nuclear Methods for Extra Terrestrial Analysis"	ORAU Special Seminar, ORAU, Oak Ridge, Tenn., Jan. 9, 1968
117		"Theory and Practice of Liquid Scintillation Counting"	Isotope Technology Training Course, ORAU, Apr. 23, 1968
118		"Modern Nuclear Instrumentation"	ORAU Special Seminar, ORAU, Oak Ridge, Tenn., July 31, 1968
119	Shults, W. D.	"Recent Advances in Differential Polarography"	University of Georgia, Athens, Mar. 14, 1968
120		"Performance Characteristics of Ion-Selective Electrodes"	Union Carbide Corporation Analytical Specialist Meeting, Bound Brook, N.J., May 20, 1968
121	Strain, J. E.	"Standard Gamma-Ray Spectra of the Elements Following 14-MeV Neutron Irradiations"	Conference, Small Accelerators for Teaching and Research, Oak Ridge Associated Universities, Oak Ridge, Tenn., Apr. 8–10, 1968
122	White, J. C.	"Analytical Chemistry"	Engineering Training Seminar, Oak Ridge National Laboratory, Oak Ridge, Tenn., Mar. 15, 1968
123	Young, J. P.	"Absorption Spectra of Several Transition Metal Ions in Molten Fluoride Solution"	155th Meeting, American Chemical Society, San Francisco, Calif., Mar. 31–Apr. 5, 1968
124		"Spectrophotometric Studies of Solute Species in Molten Fluoride Media" (Invited Paper)	156th Meeting, American Chemical Society, Atlantic City, N.J., Sept. 8–13, 1968
125	Zittel, H. E.,[11] T. H. Row*	"Radiation and Thermal Stability of Sprays"	American Nuclear Society Meeting, Toronto, Canada, June 9–13, 1968
126	Zittel, H. E.	"The Effect of Radiation on Some Analytical Methods"	Second Conference, Society for Analytical Chemistry, Nottingham, England, July 15–19, 1968

UNDER THE TRAVELING LECTURE PROGRAM

LECTURER	TITLE	PRESENTED AT
127 Cameron, A. E.	"Geological Age Determination by Isotopic Measurements"	Indiana Central College, Indianapolis, Nov. 28, 1967
128	"Geological Age Determination by Isotopic Measurements"	North Carolina State University, Raleigh, Nov. 30, 1967
129 Ricci, E.	"Recent Developments in Activation Analysis"	Western Kentucky University, Bowling Green, Apr. 5, 1968

Patents

AUTHOR	TITLE	PATENT NO.	DATE ISSUED
130 Moore, F. L.	"Method for Separating Members of Actinide and Lanthanide Groups"	British Patent No. 1,098,350	Jan. 10, 1968

Translations

Translator	ORIGINAL REFERENCE			TRANSLATION		
	Author (Transliterated)	Source	Language	Language	Translated Title	ORNL-tr Number
1 Kubota, H.	K. Hamada, T. Hayashi, and I. Oguchi	*Hitachi Hyoron,* 48, 812–816, 1966	Japanese	English	"Stress Analysis in Tapered Transition Joints in Reactor Pressure Vessel"	1769
2 Kubota, H.	T. Kamiyama	*Genshiryoku Kogyo,* 13(2), 5–6, 1957	Japanese	English	"Trend in Atomic Power Generation and Uranium Resources"	1825

Articles Reviewed or Refereed for Periodicals

Reviewer or Referee	Number of Articles Reviewed or Refereed for Indicated Periodical																
	Anal. Chem.	*Anal. Chim. Acta*	*Anal. Letters*	*Appl. Optics*	*Appl. Spectry.*	*Chem. Instr.*	*Intern. J. Appl. Radiation Isotopes*	*Isotopes Radiation Technol.*	*J. Electroanal. Chem.*	*J. Phys. Chem.*	*Nuclear Data*	*Nucl. Appl.*	*Nucl. Phys.*	*Nucl. Sci. Eng.*	*Science*	*Talanta*	Total
Bate, L. C.	1																1
Cameron, A. E.	1																1
Canada, D. C.	1																1
Dyer, F. F.	3																3
Emery, J. F.	4																4
Feldman, Cyrus	2	1		1	2												6
Fisher, D. J.	1					5											6
Goldberg, Gerald	1																1
Handley, T. H.	4															2	6
Horton, A. D.	1																1
Lyon, W. S.	3							1			2	1			1		8
Moore, F. L.	3	1															4
Mueller, T. R.									1								1
Reynolds, S. A.	1						1	3					2	1			8
Ricci, Enzo												2					2
Ross, H. H.	6																6
Ross, W. J.	2																2
Shults, W. D.	2																2
Stelzner, R. W.	3																3
Stokely, J. R.	1																1
White, J. C.	3		1														4
Young, J. P.	1									1							2
Zittel, H. E.	1																1
Total	45	2	1	1	2	5	1	4	1	1	2	3	2	1	1	2	74

Indexes

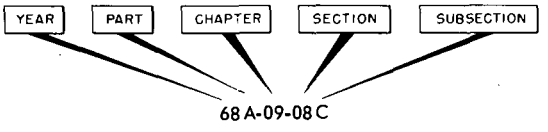
Helen P. Raaen Ann S. Klein[1]

Indexes are a part of the Analytical Chemistry Division annual report for the fifth 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 1964–1968 are bound separately and are designated ORNL-3904 (Rev. 3).

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 sections. 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.

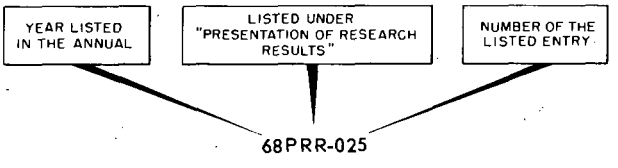
The Author Index is an alphabetical listing of authors, together with number entries that specify the material to which each author contributed. This index includes the authors of material in the body of the report, the authors of the entries listed in the Presentation of Research Results, and the translators 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 68A-09-08C, where 68 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 68PRR-025, where 68 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.

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Machine limitations necessitate that some entries be in an unusual form. Because special type cannot be used, entries that otherwise would require it are written out; for example, chemical symbols, chemical formulas, Greek letters, *o-*, *m-*, *p-*, etc. Superscript and subscript numbers cannot be printed; hence, valence is designated by a Roman numeral following the name of the element or ion, and an isotope mass number is indicated by a hyphen and an Arabic number following the name of

[1] Technical Information Division.

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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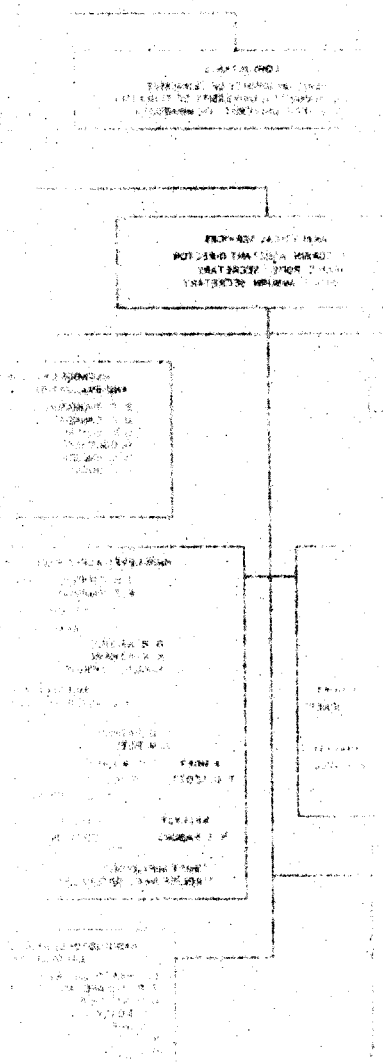

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