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ANALYTICAL CHEMISTRY DIVISION

ANNUAL PROGRESS REPORT

FOR PERIOD ENDING OCTOBER 31, 1969

OAK RIDGE NATIONAL LABORATORY

operated by

UNION CARBIDE CORPORATION

for the

U.S. ATOMIC ENERGY COMMISSION

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ANALYTICAL CHEMISTRY DIVISION

ANNUAL PROGRESS REPORT

For Period Ending October 31, 1969

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J. C. White, Associate Director

Compiled and Edited by
Helen P. Raaen

JANUARY 1970

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 four small dedicated computers have been acquired and commitments have been made to purchase two more such devices. Initially, these will be used primarily to obtain data in digital form, but at least three of these will be used to explore the feasibility of computer-controlled analytical instrumentation. One of these computers is being used routinely to acquire and process mass spectrometric data, and another has been used to demonstrate the feasibility of computer acquisition of voltammetric data from molten fluoride salt systems.

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 chemistry directed towards the life sciences 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 the life sciences in the research efforts of ORNL and of the entire scientific community.

Summary

PART A. ANALYTICAL RESEARCH AND DEVELOPMENT

1. Analytical Instrumentation

The research and design work of the Analytical Instrumentation Group has continued to produce results of demonstrated value in the Division and elsewhere. Instruments and scientific information have been generated that are used as high-quality working and research tools. Part of the work was done specifically for use in service and in other groups in the Division, and part was done in cooperation with members of other groups; much of it is also of particular interest to the broader chemistry community. Some of the prior limitations of instruments and of instrumental methods have been overcome or circumvented so that better data can be obtained, problems previously unsolvable can be attacked successfully, or new scientific information can be acquired. Progress has been made in the use of digital integrated-circuit-logic modules as well as analog modules and toward computer-aided and -automated analyses. We have developed and demonstrated digital interfacing, digital programming, and digital data-acquisition and processing systems. The results of this work were disseminated as invited oral presentations and publications.

Dividends have accrued in several research projects that are of general interest as well as of value in the missions of the Division. With a direct-displacement pump of new design, precise control of the average flow rate of mercury through a D.M.E. has been achieved. Moreover, now that this variable is also controllable, the long-term (several days) relative standard deviations (single observation, 28 trials) of polarographic averaged diffusion current and of first-derivative peak height are reduced to 0.15%. This achievement constitutes a significant advance toward a simple, reliable, and practical one-cell method of high-precision polarography. We have placed in operation a Digital Equipment Corporation model PDP-8/I computer for use in studying its applicability in analytical chemistry where on-line data acquisition and rapid computation can be of benefit. Computer-assisted analytical procedures can increase accuracy, precision, and throughput. A number of general-purpose algorithms have been written. Additional peripheral equipment and a second computer are on order. We will evaluate these computer systems in remotely controlled analyses in hot cells, polarography, and programs that exploit the GeMSAEC rapid analyzer. Future programs at ORNL are expected to produce many samples that will require multielement, automatic, flame-spectrophotometric analysis in a short burn time. To meet this need, we are investigating the usefulness of a digital flame-spectrophotometric computer system to provide monochromator control and rapid data acquisition and processing (mainly statistical); an ORAU Research Participant has assisted us. Analog components of the system were assembled, a PDP-8/I computer was incorporated, several programs were written and tested, several modes of data acquisition were automated, and convolution following ensemble averaging was provided. Computer automation has greatly facilitated the optimization of procedural and instrumental conditions. Earlier work which showed that the flame noise is predominantly white random noise has been confirmed. We demonstrated that a computer-assisted and -controlled system is useful for acquiring and processing data from a flame-photometric experiment.

A method (a philosophy) has evolved for the design of instrument systems for measurements of highest sensitivity. This philosophy is presented in a published paper and is illustrated through descriptions of its applications for flame spectrophotometric, dc polarographic, and other measurements. The paper also includes an explanation and survey of topics that are particularly pertinent to the design of such systems. Another paper, which is in press, describes several of the mathematical design techniques that have led to the electronic circuitry of the ORNL model Q-2792 polarograph. An ar-

ticle prepared for publication discusses a cell of simple design that minimizes the *iR* error in controlled-potential polarography of solutions having high specific resistance. By invitation, a manuscript on the advances in instrumentation for dc polarography and for coulometry was submitted for inclusion in a forthcoming volume of the series *Advances in Analytical Chemistry and Instrumentation* (Interscience). Four papers were published that present the design features and performances of our ORNL model Q-2792 polarograph, model Q-2942 D.M.E. drop-time controller, and controlled-potential controlled-current cyclic voltammeter. In still another paper, the use of ensemble averaging to increase the sensitivity of measurements with flame spectrophotometers was described. In a practical problem, enhancement of the signal-to-noise ratio by ensemble averaging may be attractive because of the achievement of sufficiently high sensitivity (and low detection limits) with the use of a relatively simple procedure. Other advantages of ensemble averaging are also described.

Two cooperative investigations performed at the Australian Atomic Energy Commission Research Establishment, Lucas Heights, were concluded. One showed the superiority of 2,2-dimethoxypropane as an agent for dehydrating nonaqueous solutions under study by electrochemistry. In the other, the polarographic behavior of metal-solochrome violet RS complexes was elucidated; it is described in a published paper.

As in the past years, a substantial part of the Group effort was in direct and specific support of needs of other Division groups and missions. Several of our ORNL model Q-4010 coulometric titrators are being fabricated (two completed, three nearly completed) and checked out for use in the Analytical Services Laboratories (General Analyses Laboratory, General Hot-Analyses Laboratory, High-Level Alpha Radiation Laboratory) and for further research in coulometry and coulometric instrumentation (an integrated system) in the Analytical Instrumentation Group. Our ORNL model Q-3119 voltage comparator was developed to calibrate the coulometer (integrator) in the Q-4010. Diode-transistor-logic integrated circuits simplify construction. In addition to being used as a laboratory test instrument, the Fabri-Tek model 1064 Instrument Computer will be made available for ensemble averaging during overnight NMR runs by the Infrared and N.M.R. Group. This use of the instrument is expected to increase the sensitivity of NMR analysis so that analysis of certain biochemical samples can be offered as a service. The controlled-potential controlled-current cyclic voltammeter being constructed for the Reactor Chemistry Division (in addition to one previously built and checked for them) is to be checked out and calibrated. A GeMSAEC fast analyzer of modified design was built and is being checked out for the Methodology Group. A graphite-tube furnace for atomic absorption spectroscopy was designed and fabricated for the X-Ray and Spectrochemistry Group. Several ORNL model Q-2792 polarographs and Q-2942 drop-time controllers are in use in the Division; another one of each is being built for the Methodology Group and will be checked out for them. A model IX-A flame spectrophotometer was designed and fabricated for its present use in the General Analyses Laboratory. A task force has been organized within the Division to provide instruments and equipment for several kinds of analyses of irradiated LMFBR fuels in the HRLAL for the Metals and Ceramics Division. As the first part of this program, in cooperation with the Methods Development and Evaluation Laboratory, a Leco rapid oxygen analyzer was modified for remote operation and is to be installed in an HRLAL cell and tested. Computer programs were written in FOCAL for demonstrations to the General Analyses Laboratory, High-Level Alpha Radiation Laboratory, Radioisotopes-Radiochemistry Laboratory, and Reactor Projects Group. In work with the Reactor Projects Group, we have interfaced a PDP-8/I with our voltammeter to control a molten-salt voltammetric experiment for the determination of U(IV)/U(III) ratios as a function of time. The results of the analyses are obtained and printed out automatically. The system is well-suited for kinetic studies and should be readily adaptable to continuous monitoring of analyses. Because this experiment was a success, a third PDP-8/I computer is on order for use in the Reactor Projects Group.

2. Analytical Methodology

The activities of the Analytical Methodology Group are extremely diverse. We have worked conjointly this year with other groups and other divisions, as well as with educational institutions; the scope of this interaction appears to be increasing.

Gas chromatography (GC) continues to be a powerful technique for many of our problems and is receiving a great deal of attention. Gas chromatography has clearly become the technique-of-choice, for example, in our tobacco-smoke characterization program. The chromatographic behavior of numerous compounds and classes of compounds pertinent to tobacco-smoke analysis has been determined by using generally applicable chromatographic conditions of several types. This "fin-

ger-printing" effort is continuing, and we have begun to seek specialized conditions to resolve those compounds that are not resolvable under the general conditions. A new chromatograph was acquired, and a smoking machine has been ordered; these will enable us to initiate a program of study into analysis of the vapor-phase components of cigarette smoke. A silicon-specific detector has been developed that is uniquely suited for studying and using silylation reactions in gas chromatography. We hope to study soon the "on-column silylation" technique with this silicon-specific detector. We have explored further the possibility of rapid elemental analysis ("profiling") by high-temperature hydrogenation and GC determination, with positive results. Several short-range applications of GC have been made, and a number of compounds have been highly purified with our preparative GC equipment.

In thin-layer chromatography (TLC), too, we have made several short-range applications. Our research effort in TLC has been devoted largely to the development and application of polytetrafluoroethylene (Teflon) layers. We are now preparing to investigate temperature effects in TLC.

Our program in electrochemistry has increased significantly. Polarographic work with Teflon D.M.E.'s has been concluded. We are now studying so-called "conducting glass" electrodes (C.G.E.'s) for their electroanalytical usefulness, and we have just begun to investigate thin-layer electrochemical techniques. A new concept in coulometric instrumentation – programmed-current coulometry – is under study. Our electroanalytical work has been concerned primarily with inorganic (particularly transuranium) elements in the past. Interest in this area continued, and significant work has been accomplished. For example, we have determined directly the formal potential of the Bk(III)/Bk(IV) couple in several media and intend to use this knowledge (and this experience) to develop the first ionic method for determining berkelium. We have used, and will continue to use, electrochemical techniques to seek higher and lower oxidation states of the transuranium elements. Whereas past efforts have emphasized inorganic systems, we have begun to consider electrochemical techniques for analysis of organic systems; we did some preliminary work in this field.

A new project comprises a study of decentralized abstracting and indexing of reports for *Nuclear Science Abstracts.* Our assignment is to provide on-site abstracting and indexing of all ORNL reports. This project is being carried out jointly with the Division of Technical Information, the purpose being to delineate the idiosyncrasies of the decentralized abstracting concept.

We have continued to support a number of groups within ORNL, including Ecology, Office of Saline Waters, Housing and Urban Development, and HFIR. By and large, our work comprises technical support, but we continue to try to upgrade and improve methodology. Of particular import is the pyrolysis-GC analytical system that the Health Physics Ecology Section purchased. We are attempting to make this system give meaningful results, and we anticipate operating the equipment for ecology. With some modification and development, this system should provide a real advance in analytical methodology for ecological materials. We are convinced that a vigorous program of analytical research in the area of environmental science is warranted and is rapidly becoming imperative.

Finally, we have committed ourselves to a program of study into nonclinical applications of the ORNL GeMSAEC Fast Analyzer. An instrument for this project is being constructed.

3. Analytical Chemistry for Reactor Projects

The first sample of the ^{233}U fuel FP-17-5 was analyzed for oxide by the hydrofluorination procedure, and a concentration of 61 ppm was determined. New parts are being fabricated for the renovation of the oxide apparatus, because the NaF trap is restricting the gas flow in the system and must be replaced.

The off-gas of the circulating fluoroborate loop has been found to be contaminated with a dense corrosive liquid, tentatively identified as HBF_3OH contaminated with corrosion products and equilibrium quantities of other BF_3 hydrates. It is planned to generate these hydrates within a laboratory system, to study their transfer, and to develop methods for removing them from off-gas streams. A program has also been undertaken within the Metals and Ceramics Division to study methods for the purification of the $NaBF_4$ coolant.

A sealed-tube dissolution technique was applied to the determination of the total reducing power of radioactive MSRE fuel. The method was originally developed to determine free metals in the reprocessed solvent salt, but an attempt was also made to apply it to the determination of U(III). It became evident that the method was not suitable for U(III) determination, but that the technique offered high sensitivity for total-reducing-power measurements. An apparatus was assembled to simulate the hydrogenation of radioactive salt samples for the determination of U(III) by a hydrogen-reduction method. It was found that small amounts of the HF formed in the reduction process were

chemisorbed on the apparatus. This chemisorption would complicate batch analyses of the fuel but would not interfere with in-line methods. An apparatus was designed for the electrochemical determination of U(III) in radioactive MSRE fuel. Voltammetric measurements on an MSRE fuel sample indicated that ~0.4% of the uranium was present as U(III). An experimental voltammetric setup at the Y-12 site is being used to follow the change in U(IV)/U(III) ratio in a simulated MSRE fuel as either the U(IV) is reduced or the U(III) is reoxidized.

A standard potential of the U(IV)/U(III) couple has been calculated with respect to a hypothetical unit-mole-fraction Ni/Ni(II) electrode from emf measurements on the galvanic cell Pt|U(IV),U(III)||Ni(II)/Ni in molten $LiF-BeF_2-ZrF_4$. A voltammetric method was also developed for the determination of the U(IV)/U(III) ratios. Spectrophotometric and voltammetric results were in good agreement on samples that had various U(IV)/U(III) ratios. To gain experience in adapting small on-line computers to electroanalytical methods, a PDP-8/I computer was programmed to operate a cyclic voltammeter. The PDP-8/I furnished all command signals to the voltammeter and computed U(IV)/U(III) ratios. In other electroanalytical studies, kinetic parameters were determined for the Ni/Ni(II) couple in molten $LiF-BeF_2-ZrF_4$, and standard electrode potentials were measured for several other electrode couples.

The design and fabrication of the optical components of the hot-cell spectrophotometer have been completed by the vendor. The optical components were received and are undergoing the final acceptance tests here at ORNL. It is planned that the entire system will be in operation within 6 months. The possibility now exists for the fabrication of a windowless fixed-pathlength spectrophotometric cell from porous metal for studies of molten fluoride salts. The developer, Mallory Metallurgical Company, has agreed to fabricate experimental batches of porous metal from Hastelloy N, which is compatible with molten fluoride salts. Spectrophotometric study of the 3d transition-metal ions in molten fluoride solution was continued, and the results were summarized graphically. Other spectrophotometric studies have shown that the superoxide ion O_2^- exists in molten fluoride solutions.

The system for the analysis of the gases from the in-pile tests of GCR fuel-element samples was subjected to extensive testing and modification. The General Electric Monopole 600 Residual Gas Analyzer, which is used to measure concentrations of H_2, CO, CO_2, CH_4, water vapor, and atmospheric contaminants, is now being installed at the ORR where it will be given additional calibration tests before it is used to analyze off-gas samples. An apparatus is being constructed for use in the analysis of natural-gas samples from wells in which nuclear explosives have been used to enhance the production of natural gas. The measurement of the concentration of the long-lived radioactive gases ^{85}Kr and ^{3}H and the determination of the distribution of ^{3}H among various chemical constituents of the samples (H_2, H_2O, CH_4, and higher hydrocarbons) is the main interest.

4. Analytical Biochemistry

Analytical assistance to the Biology and Chemical Technology Divisions was continued in support of the Macromolecular Separations and Molecular Anatomy (MAN) Programs. In the analysis of transfer ribonucleic acids (tRNA's), a statistically designed experimental plan is now being used to optimize assay conditions and has been applied to lysine-, arginine-, and glutamic acid-accepting tRNA's. Studies have been completed on the assay of formylmethionine-accepting tRNA by formyl-group acceptance. The effects of various organic compounds on the assay of tRNA's are being examined. Polyacrylamide-gel electrophoresis has proved to be a powerful tool. Two types of gels are used; a 10% gel for examining low-molecular-weight RNA's and a gradient (2.5 to 12%) gel for high-molecular-weight RNA's. In both cases, a Pyronin Y–chromium sulfate–acetic acid mixture is used to fix and stain the RNA bands in the gel. Excellent resolution of RNA's of different molecular weight has been achieved. We intend to determine the chemical composition of RNA's with a recently acquired High-Pressure UV Analyzer equipped with an anion-exchange column. So far, the elution positions of over 40 bases, nucleosides, and nucleotides have been established. Another potentially useful method for RNA analysis is gas chromatography. Separation of trimethylsilyl (TMS) derivatives of nucleic acid bases and most nucleosides is excellent. We are attempting to isolate from tRNA by gel filtration minor nucleosides that absorb at wavelengths from about 300 to 360 nm. Improved methods for total RNA and DNA analysis of crude bacterial-cell extracts were developed. Work has been completed on an ultrasensitive method for L-amino acids by an isotope-dilution technique based on the aminoacylation reaction.

We have continued to identify for the Body Fluids Analyses Group those urinary constituents isolated by high-pressure anion-exchange chromatography and by a carbohydrate analyzer. Techniques now used involve

removal of the elution buffer, gas chromatography after formation of TMS derivatives, and mass spectrometry. A total of 18 compounds have been identified positively, and an additional 41 tentatively. The vast amount of information that is accumulating on the nature of the components in human urine, their elution volumes in the analyzer systems, and gas chromatographic retention data is being transferred to punched cards, and computer programs are being written to retrieve this data in various ways. Construction of the automatic protein sequenator is almost completed, and studies of gas chromatographic separations of various kinds of amino acid derivatives are in progress with a view towards incorporating an on-line gas chromatograph into the sequenator system for identification purposes. Development of the GeMSAEC Fast Analyzer in the MAN Program has proceeded at a rapid rate. Analytical contributions to this program include methods for total serum protein and cholesterol and development of an optical-density thermometer for measuring the temperature of a spinning rotor.

We are now engaged in the new activity of characterizing and determining the purity of organic compounds. We are helping to establish standard materials for clinical laboratories, authenticating compounds used in chemical mutagenesis studies, and testing commercially available biochemicals to determine whether they meet specifications. In the latter case, gas chromatography has been very useful.

5. Nuclear and Radiochemistry

The preliminary study of the determination of trace elements in drugs by neutron activation analysis was completed, and the delayed-neutron method for determining fissionable materials has been extended to ^{233}U and ^{239}Pu. In charged-particle activation work, the influence of channeling in ^{3}He activation analysis has been shown to be negligible, and proton-reaction analysis has been studied for ^{12}C, ^{13}C, and ^{15}N.

The 14-MeV neutron generator has operated smoothly for determining oxygen in a variety of materials, but the generator target program has been suspended temporarily. An ^{241}Am-B neutron source has been used to monitor oil contamination in the organic coolant of a machining operation. A ^{252}Cf neutron source was calibrated and studied for activation analysis. The new High-Flux Activation Analysis Laboratory at the HFIR is under construction as is the smaller facility at the ORELA.

Specimens from the Pueblito de Allende meteorite fall were examined in the Lunar Receiving Laboratory (LRL), Manned Spacecraft Center, Houston, Texas, for induced radioactivity and were analyzed by activation analysis at ORNL for fourteen elements. Two lunar-sampling devices were evaluated by using radiotracers. The Radiation Counting Facility at the LRL was equipped, checked out, and used to measure induced radioactivities in lunar samples received from the Apollo 11 mission.

The Cooperative Isotopes Program continued; the major projects were technical assistance (writing, reviewing, etc.) and a program sponsored by the National Academy of Sciences to survey the status of and needs for radioactivity standards. Neutron cross sections for ^{109}Ag and $^{40-45}Ca$ were determined, and the use of ^{237}Np to measure reactor fast-neutron flux was investigated. The nuclear properties of a number of radioactive rare-gas isotopes were determined, and over 40 new measurements of half-lives were made. The quality-control program for the radioisotope program continued, activation by radioisotope gamma radiation was attempted, and a number of special applied problems were handled; these included customer complaints, assembly of requested information, assay of special products, and separation and identification of new isotope products.

Applications of Cerenkov radiation have been suggested as a result of a comprehensive study of the phenomenon; one of these is the measurement of ^{14}C by Cerenkov counting. A new liquid-scintillation technique for the assay of ^{129}I was developed, and a number of nonionic surfactants were evaluated for liquid-scintillation counting of tritium in aqueous samples. The nuclear properties of erbium radionuclides with $158 \leqslant A \leqslant 161$ were studied, a search was made for alpha decay in ^{172}Hf, and a number of fast-neutron cross sections were measured.

The ^{232}U content of calutron-separated ^{233}U products was determined by gamma-ray spectrometry. A collimated scanner was used to determine distribution of ^{134}Cs and of ^{244}Cm inside tubes. Improvement in fission-product gas analysis by use of Ge(Li) detectors was made, and the study of fission-product release from reactor-fuel microspheres was continued. MSRE graphite samples were analyzed for fission products, and an expanded program on remote gamma spectrometry at the MSRE was begun.

Gamma radiolysis of iodide solutions, radiation stability of electrodes, and radiation stabilities of standard pH-buffer solutions were measured. A better method to determine hydrogen peroxide at the small concentrations found in radiolytic studies was developed, and a method to ensure homogeneous precipitations with

radiolytically generated reagents was improved. The Spray and Pool Absorption Technology Program continued with investigation into the radiation, thermal, and chemical stability of radiation-safeguard spray solution. A new dynamic radiation facility was designed and put into use. Study of the alpha radiation damage produced in a number of inorganic phosphors is just beginning. The nature of zirconium complexes of alizarin red S, alizarin, and hystazarin has been studied.

6. Methods Development and Evaluation

A specially enclosed Cary 14 spectrophotometer has been used to determine valence states of plutonium to study the self-oxidation of ^{238}Pu(IV) in concentrated solutions of nitric acid and to determine 1 to 2 μg of ^{243}Am in the presence of a 100-fold excess of ^{244}Cm.

A liquid-liquid extraction method was developed to separate Ce(IV) from Bk(IV). Fluorination of MSRE fuel has proved to be a useful way to separate uranium from radioactive fission products and salts in the fuel. The decontaminated uranium then can be analyzed for isotopic distribution and also determined quantitatively by regular methods. Thermogravimetric equipment has been installed in a double glove box to permit the analysis of alpha-emitting materials. Three electrolysis cells were evaluated for the controlled-potential coulometric determination of uranium and plutonium; also, new solid-state controlled-potential coulometric titrators were evaluated in this study. An inverse polarographic method was developed to determine ppb concentrations of bismuth. New equipment for the analysis of proposed reactor fuels was obtained and is being evaluated. We have analyzed these fuels by chemical, thermogravimetric, and x-ray methods to characterize their composition.

7. Mass Spectrometry

The Associated Electrical Industries, Ltd. (AEI) MS-702 spark-source mass spectrometer was installed in the new addition to Building 2026 for the analysis of alpha-emitting radioactive and transuranium elements. The resolution of the instrument is 4000 at an *m/e* ratio of 208. The instrument has been used to determine impurities in samples of the following transuranium isotopes: ^{241}Am, ^{243}Am, ^{246}Cm, ^{248}Cm, ^{249}Bk, ^{249}Cf, and a ^{232}Th-^{241}Am (24:1) alloy.

The MS-7 spark-source mass spectrometer continues to be used mainly to determine trace elements in special alloys and pure metal oxides.

Low-resolution mass spectrometry was used to determine structures of organic compounds as well as to develop further a library of spectra of known compounds. In many cases, gas chromatography and mass spectrometry, as separate techniques, and, when possible, infrared and nuclear magnetic resonance spectrometry, have been used conjointly to solve structural problems. Many samples were investigated for the Body Fluids Analyses, MAN, and NAS-NRC Biochemical Standards Programs.

The design of the direct-inlet probe was improved. Several types of helium separators were studied, and a sintered stainless-steel type separator was installed. Field-emission sources are being developed, in particular, a combination field-emission–electron-bombardment source for general use.

Although computer-interfacing equipment that is on order has not been delivered, all calculations in the organic program are now being made by the IBM-1130 computer.

Surface-ionization studies were continued; the first ionization potentials of Th, Pu, and Np were measured.

8. X-Ray and Optical Spectrochemistry

Computer codes were improved for calculating and plotting x-ray fluorescence and electron-probe data. It was found possible to determine S/P ratios with the electron probe at the 1/1000 level; the determination was tested with a mixture of mercaptoguanosine and guanosine. A platinum-mirror beam-splitter for calibrating spectrographic emulsions gave satisfactory reproducibility on a stigmatic Ebert spectrograph. Although electrodialysis through anion-permeable membranes successfully removes phosphate from bone-ash solutions, certain trace elements are lost. We are now attempting to collect trace elements from bone by separating Ca from phosphate by oxalate precipitation and separately recovering the trace elements from the two fractions. A technique was devised to measure the moisture and fat contents of bone. Procedures were developed to determine traces of Bi and Gd in MSRE fuel and to dissolve partly glassy GeO_2 in water and Hastelloy N in 1% H_2SO_4. Preliminary studies were carried out toward developing a carrier-distillation procedure for determining impurities in UO_2-PuO_2 fuels.

9. Optical and Electron Microscopy

Electron microscopy and electron diffraction have been used to study various materials of interest in a

number of applied and fundamental research problems. This work included studies of lanthanide hydroxide sols in relationship to the preparation of actinide compounds and aid in the development of a method to produce concentrated crystalline urania sols by solvent extraction (CUSP). Investigations of radioactive materials done at the Transuranium Processing Plant dealt chiefly with preparations of urania, plutonia, and urania-plutonia sols but also included observations for evidence of compounds of californium, berkelium, and einsteinium. Also, at the 3019 Facility, the Mound Laboratory and the Nuclear Fuel Services were assisted in studies of radioactive materials. A cooperative program with the Physics Division has been established to evaluate electron spectroscopy for chemical analysis (ESCA); thus far, three tRNA nucleotides have been studied. A scanning electron microscope was inspected that can use Auger electrons for surface analysis. The Philips 300 electron microscope was installed and is operating at the Transuranium Processing Plant. Equipment was fabricated and set up to prepare thin specimens of metallographic and other solid materials for electron microscopy, and, with it, a method for thinning graphite was developed.

10. Inorganic Preparations

The major work of the year was for the Solid State Division. The high-purity MgO program was completed with the accumulation of 15 lb of material. In the ferrite program, the preparation of substituted magnetites is being continued. Some high-purity fluorides were prepared, and the heat-treatment of numerous alloys was continued. The production of 50 g of ^{163}Dy and 50 g of ^{170}Er was undertaken. Some large crystals of NH_4Cl and ND_4Cl were grown. For the Physics Division, the preparation of small quantities of alloys that contained isotopic materials was continued, several other alloys were prepared, and a series of mixed disulfides of Fe and Co was made. The fused-salts program was at a minimum, only a few preparations having been made for the Chemistry Division and for the Metals and Ceramics Division.

PART B. SERVICE ANALYSES

11. Spectrometry

The Transuranium Mass Spectrometry Laboratory continued to make isotopic analyses for a diverse group of customers. Isotopic determinations were made on 20 different elements and on nine isotopes of purity >99.99%. The isotopic analysis of all elements from uranium through einsteinium was done routinely.

The Mass Spectrometry Services Laboratory made 10% more analyses than last year on separated stable isotopes and continued to certify argon and helium and to analyze fission-product xenon and krypton. Also, they determined ^{15}N in ecological samples.

The Spectrochemistry Laboratory analyzed 2700 samples, an increase of 8%, and reported ~49,000 results. The Reactor Chemistry Division was the largest supplier of samples. Methods were developed that used the Paschen direct-reading spectrometer to analyze many of the samples for Th, Li, Be, and other elements of interest.

The Infrared Spectrometry Laboratory became increasingly involved in the analysis of biological compounds and developed a mulling technique for use in preparing solid biological compounds for infrared spectrometry. In this way, numerous purine and pyrimidine derivatives were examined. A file of reference spectra was established. Other materials studied included a variety of biologically related substances and many inorganic and organic materials from various sources.

Use of the services of the Nuclear Magnetic Resonance Spectrometry Laboratory continued to increase. The NMR spectra taken were used primarily to gain information about preparative and separation processes and to identify and determine structures of organic compounds. The technique was useful in a study of the effects of curing agents and resins and their separation by molecular distillation, in the specification analysis of nucleotides and related compounds, and in the determination of the structures of mutagens.

12. Process Analyses

The laboratories doing process analyses performed some 205,000 analyses, about the same as last year, but we had a 6% decrease in personnel. Brief statements about the work and new developments in each laboratory follow.

The High-Level Alpha Radiation Laboratory determined plutonium in samples that contain high levels of radiozirconium and curium. Refractory plutonia-zirconia was dissolved in a Teflon-lined bomb.

The work load of the General Analyses Laboratory in analytical biochemistry continued to increase; the average cost per analysis of this type was reduced from $1.62 to $1.33.

Two methods for the determination of bismuth in fluoride salts were developed – an iodide spectrophotometric method for levels >10 ppm and a dithizone

extraction–spectrographic method for concentrations as low as 0.1 ppm. An apparatus was constructed for the measurement of helium formed in metals by fast-neutron irradiation. Improved methods to protect samples from extraneous oxygen were used in the determination of oxygen in uranium mononitride.

The General Hot-Analyses Laboratory continued to analyze MSR samples and samples from HTGR and LMFBR. Three new laboratories were added to the group. An MS-702 mass spectrograph was installed in one of the new laboratories and a glove-box facility in another. The apparatus for determining gas release on mixed-oxide fuels was modified to operate at temperatures up to 1800°C.

The Radioisotopes-Radiochemistry Laboratory continued to use computer resolution of gamma-ray spectrometric data primarily in support of the Fast Breeder Reactor Fuel Processing programs. Neutron activation analyses consisted primarily in determining rare-earth elements in $LiF\text{-}BeF_2$ salts and iodine in gas-liquid systems and in irradiated fuel from "melt-down" experiments. Some tracer-level experiments were done in support of the TRU processing program.

The Radiation and Safety Officers made two safety inspections during the year. Also, they replaced the oil in the storage-cell window in the HRLAF to improve visibility and reviewed and approved the handling of certain radioactive materials.

The number of control samples included in the Statistical Quality Control Program decreased 12.0%; the overall quality of the work decreased somewhat, primarily because of decreases in the concentrations of iron, nickel, and chromium in MSRE samples.

PART C. ORNL MASTER ANALYTICAL MANUAL

13. *ORNL Master Analytical Manual*

Two new methods were added to the *Manual.* A number of methods were discontinued; they were previously used in the analysis of radioisotopes that are no longer produced at ORNL.

Part A. Analytical Research and Development

The research and development programs of the Analytical Chemistry Division reflect 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 POLAROGRAPHIC MERCURY-FLOW-RATE CONTROLLER

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

We have demonstrated the feasibility of controlling directly and precisely the average flow rate of mercury ($\bar{m}$) through a D.M.E. with a piston pump of new design. The prototype mentioned earlier[1] was unacceptable primarily because of the large size of the displacement piston of the Gilmont micrometer buret. After consultation with C. R. Rickard[2] and D. L. Holcomb,[2] we decided that a satisfactory buret with a piston diameter of ~1 mm could be constructed. R. S. Jones[2] and W. A. Walker,[2] working from rough sketches, produced an excellent finished product.

Figure 1.1 shows the pump (at right) attached to the rear of an ORNL model Q-2942 polarographic drop-time controller.[3] In the model shown, a stepping motor powered by an oscillator locked to line frequency drives the piston by means of a high-quality Tesa micrometer screw and a spline mechanism. The piston is a 0.040-in.-diam steel drill rod selected from among several for its fine finish and uniform diameter. The body of the pump was fabricated from a glass-ball check valve to permit connection to a reservoir for refilling with mercury. The valve (Pyrex) was joined to the D.M.E. capillary (leaded glass) with a graded seal.

[1] D. J. Fisher and W. L. Maddox, "Apparatus for Precise Control of Mercury Flow Rate of Dropping-Mercury Electrodes (D.M.E.'s)," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968*, ORNL-4343, p. 2.

[2] Plant and Equipment Division.

[3] W. L. Belew, D. J. Fisher, H. C. Jones, and M. T. Kelley, "Apparatus for Precision Control of Drop Time of Dropping Mercury Electrode in Polarography," *Anal. Chem.* 41, 779 (1969).

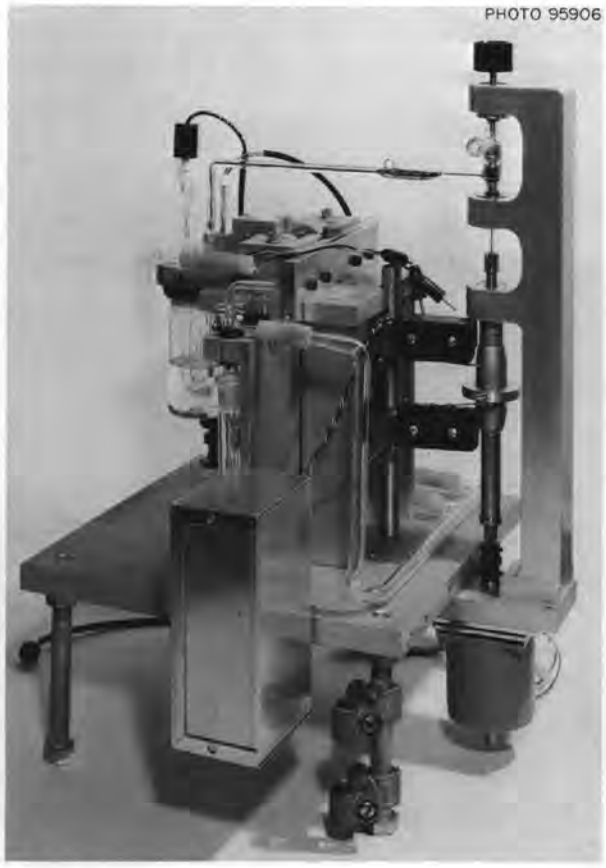

Fig. 1.1. Photograph of Assembly of Mercury-Flow-Rate Controller and ORNL Model Q-2942 Drop-Time Controller.

The apparatus was housed in a controlled-temperature glove box. Temperature control of the atmosphere in the glove box was adequate, but solutions did not quickly reach the temperature of their surroundings when they were moved about within the box. We therefore resorted to using a thermistor that dipped directly into the polarographic cell solution as a final check to ensure that temperature equilibrium had been attained after any manipulations were made and before any critical tests were run. The $\overline{m}$ obtained with the assembly shown was 1.624 mg/sec. The relative standard deviation of a single observation (S) was 0.1% for 18 trials of 20 drops each at a drop time of 0.500 sec. Over the range 1.000 to 0.125 sec, $\overline{m}$ is independent of drop time with the mercury-flow-rate controller, whereas $\overline{m}$ decreased ~20% when the conventional mercury head provided the impetus.

The mercury-flow-rate controller was used in polarographic experiments performed with the ORNL model Q-2792 polarograph,[4] Q-2942 drop-time controller,[3] and 90° vertical-orifice Smoler capillary on a solution 5×10^{-3} M in Cd^{2+}, 10^{-3} M in HCl, 1 M in KCl, and 0.001% in Triton X-100. The results indicate that, over a period of several days, the polarographic calibration factor (measured by either first-derivative peak height or averaged diffusion current on the plateau of a wave) is sufficiently constant (S = 0.15% for 28 trials).

The development of an effective mercury-flow-rate controller constitutes a significant advance toward achieving a simple, reliable, and practical one-cell method of high-precision polarography.[5] Control of $\overline{m}$ has resulted in excellent long-term precision.

1.2 COULOMETRIC TITRATOR (ORNL MODEL Q-4010): PROTOTYPE AND INSTRUMENTS FOR ANALYTICAL SERVICES

T. R. Mueller

On March 12, the Instrumentation and Controls Division delivered the prototype of the ORNL model Q-4010 solid-state coulometric titrator[6] (Fig. 1.2) for evaluation. After minor construction faults were corrected, its performance was tested extensively. Several changes in the front-panel layout and a few circuitry refinements were made. Preliminary test results are encouraging and indicate that the design specifications have been met or exceeded.

The titrator is built in the modular concept of Nuclear Instrument Module (NIM) systems. Modules are 8¾ in. high. The "bin" fits a standard 19-in. relay rack; also, it can be used on the desk top. The composite instrument is designated "ORNL Model Q-4010 Coulometric Titrator." According to the Q-number-assignment system used by the Instrumentation and Controls Division, individual modules are numbered in the 3000 series. Thus, the potentiostat module is designated Q-3091; the current-amplifier module, Q-3092; the meter module, Q-3093; and an analog-integrator module, Q-3094. Digital integration may be

[4] H. C. Jones, W. L. Belew, R. W. Stelzner, T. R. Mueller, and D. J. Fisher, "A Solid-State Controlled-Potential DC Polarograph with Cyclic Scanning and Calibrated First- and Second-Derivative Scales," *Anal. Chem.* **41**, 772 (1969).

[5] 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.

[6] T. R. Mueller and H. C. Jones, "Solid-State Controlled-Potential Coulometric Titrator," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968*, ORNL-4343, p. 1.

offered as an alternative, but the digital equipment will not be housed in the bin with the other modules.

The current-amplifier and the meter modules are necessarily used together, but separate modules were created to facilitate troubleshooting should a circuit fail. The potentiostat may be used, as such, independently of the other modules. The analog-integrator module has a separate input, which makes it also available for other uses.

The bin provides ±15-V dc at 1 amp, ±24-V dc at 0.75 amp, and two auxiliary 120-V ac outlets; both dc supplies have ±0.01% line and load regulation. The four modules normally used for the titrator require less than half the available current from either supply. Thus, an ample reserve is available for powering other modules (four widths are vacant in the bin) or for expanding the current-handling capability of existing circuits. Individual modules will be calibrated in a maintenance bin and can then be used interchangeably without recalibration in any instrument bin. This means, in practice, that all integrators will have the same "electrical factor" predetermined in the maintenance shop to within ±0.01%, and that, should a failure arise in a module, another module can be substituted without loss of laboratory time for recalibration. Integrator-calibration time is reduced by use of the Q-3119 voltage comparator (Sect. 1.3).

Two titrators have been delivered to the General Analyses Laboratory. Delay in procuring parts has prevented delivery of two additional titrators, one each to the General Hot-Analyses Laboratory and the High-Level Alpha Radiation Laboratory. The rebuilding of the prototype and the delivery of the latter two instruments are scheduled to be completed in November 1969. The study of the design of titration cells is continuing.

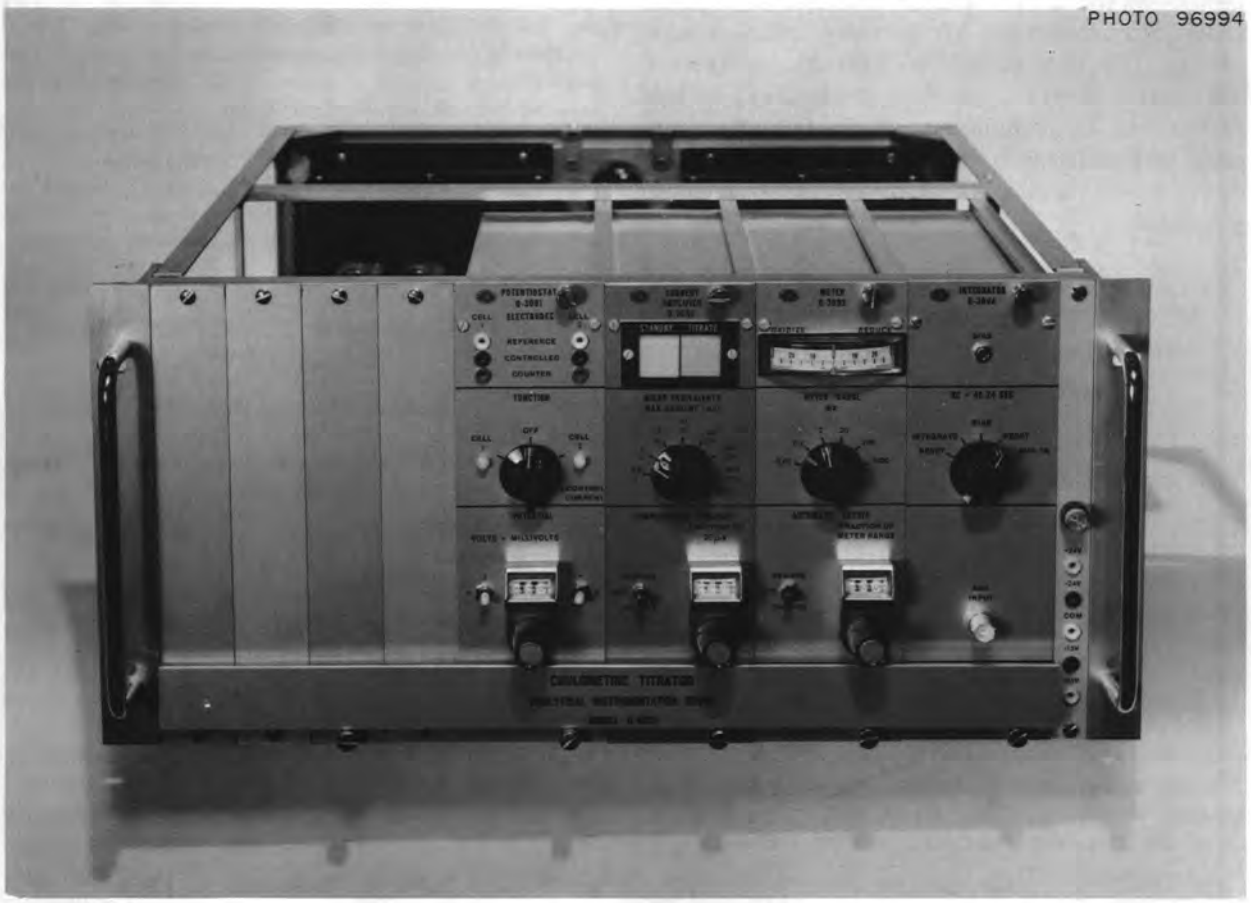

Fig. 1.2. Photograph of ORNL Model Q-4010 Coulometric Titrator.

1.3 LOW-FREQUENCY DUAL-SET-POINT VOLTAGE COMPARATOR (ORNL MODEL Q-3119)

T. R. Mueller

The ORNL model Q-3118 (formerly Q-2950) voltage comparator[1,7] was developed for sweep-rate calibration of the controlled-potential controlled-current cyclic voltammeter.[8] For this application, its ±0.01% stability was adequate. During the development of the ORNL model Q-4010 coulometric titrator (Sect. 1.2), the requirement for calibration of the Q-3094 integrator modules to ±0.01% became necessary. Therefore, the Q-3119 comparator was developed. By use of a chopper-stabilized amplifier and a fixed voltage interval and by replacement of the commutating transistor with a relay, the long-term stability of the comparator was increased. Also, reduction in the number of discrete components by the use of diode-transistor-logic integrated circuits simplifies construction. The comparator is constructed in the NIM system and can be used in the Q-4010 bin (Sect. 1.2). Speed of response, not required in this latter application, has been sacrificed in the new design. It is now possible to calibrate the Q-3094 integrator modules to ±0.01% in a reasonable time and to compare the performances of the Q-3094's fabricated with different types of components. A detailed description of the new comparator is being prepared for publication.

1.4 SWEEP-CONTROL MODULE FOR NMR MEASUREMENTS BY USE OF THE SIGNAL-AVERAGING TECHNIQUE FOR INFRARED AND N.M.R. GROUP

M. T. Kelley R. W. Stelzner

An SW-34 sweep-control module for the Fabri-Tek model 1064 Instrument Computer (signal-averager) is on order to be attached to a Varian model A60 NMR spectrometer. After installation and check-out, the signal-averager will be applied in long-term (overnight) NMR runs by the Infrared and N.M.R. Group. The objective is to increase the sensitivity of NMR measurements for compounds having biochemical significance. The instrument computer system will be available to the Analytical Instrumentation Group for continued daytime use as a test facility.

1.5 CONTROLLED-POTENTIAL CONTROLLED-CURRENT CYCLIC VOLTAMMETER

T. R. Mueller H. C. Jones[9]

With the appearance of a method[10] and an article[8] that describe the controlled-potential controlled-current cyclic voltammeter, formal development work on its basic circuitry is considered to be complete. The Instrumentation and Controls Division is fabricating a second unit for the Reactor Chemistry Division. We have been asked to perform the check-out and calibration of the unit. The summary from the article follows.

"An instrument has been designed and constructed to be used primarily for electrochemical investigations in molten salt systems. It operates in either a controlled-potential or controlled-current mode. In the controlled-potential mode it may be used for linear potential sweep voltammetry or chronoamperometry; in the controlled-current mode, for chronopotentiometry with control of the initial potential. Fast electronic switching circuitry permits reversal of the sweep direction or current polarity, or termination of the experiment at preselected electrode potentials. Easily implemented extensions of the basic operating modes are discussed. The potentiostat is designed to deliver 100 mA ±10 V, but larger transient currents can be accommodated. Accurate, calibrated sweep rates of 0.005 to 500 V/sec make the instrument suitable for kinetics studies. The rapid sweep rates also are used to advantage to minimize the effects of convective mass transport."

1.6 MODIFIED GeMSAEC FAST ANALYZER FOR METHODOLOGY GROUP

W. L. Maddox W. D. Shults[11]

The GeMSAEC fast analyzer[12] is a multiple-cuvette, centrifugally operated filter photometer developed in the ORNL Molecular Anatomy Program. We obtained drawings of the latest version (G-IIC) of this instrument from the ORGDP Development Division and had one

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[7] H. C. Jones and T. R. Mueller, "Modification of Dual Set-Point Voltage Comparator (ORNL Model Q-2950)," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968,* ORNL-4343, p. 7.

[8] T. R. Mueller and H. C. Jones, "A Controlled-Potential and Controlled-Current Cyclic Voltammeter," *Chem. Instrum.* **2**, 65 (1969).

[9] Present address: Instrumentation Section, Isotopic Analysis Department, Laboratory Division, Oak Ridge Gaseous Diffusion Plant.

[10] T. R. Mueller, "Cyclic Voltammeter, Controlled-Potential, Controlled-Current," Method Nos. 1 003045 and 9 003045 (9-1-68), *ORNL Master Analytical Manual,* TID-7015, suppl. 10 (to be published).

[11] Methodology Group.

[12] N. G. Anderson, "Analytical Techniques for Cell Fractions XII. A Multiple-Cuvet Rotor for a New Microanalytical System," *Anal. Biochem.* **28**, 545 (1969).

assembly constructed in the ORNL shops for use by the Methodology Group (Sect. 2.6). The design was modified to use a Heath monochromator and light source instead of filters and a tungsten lamp.

1.7 HIGH-TEMPERATURE GRAPHITE-TUBE RESISTANCE FURNACE FOR ATOMIC ABSORPTION SPECTROSCOPY FOR X-RAY AND SPECTROCHEMISTRY GROUP

W. L. Maddox

The design and fabrication of a graphite-tube furnace (L'vov furnace) requested by C. Feldman[13] were completed, and check-out of the apparatus has been started.

1.8 SOLID-STATE CONTROLLED-POTENTIAL DC POLAROGRAPH WITH CYCLIC SCANNING AND CALIBRATED FIRST- AND SECOND-DERIVATIVE SCALES (ORNL MODEL Q-2792)

H. C. Jones[9] W. L. Belew R. W. Stelzner
T. R. Mueller D. J. Fisher

A paper was published[4] that describes a solid-state controlled-potential dc polarograph with cyclic scanning and calibrated first- and second-derivative scales (ORNL model Q-2792); the abstract follows.

"This instrument is a versatile, reliable polarograph incorporating desirable design features of previous ORNL DC polarographs and having additional functional features and operating conveniences. Precision scan rates (less than 0.1% relative standard deviation) from 0.1 to 10 volts/minute in a 1, 2, 3, 5, sequence are provided. Current ranges from 0.02 μA to 10 mA in a 1, 2, 5 sequence are available. Efficient filtering of the current signal from the polarographic cell makes possible the use of active first- and second-derivative networks giving accurate derivative waves on precisely calibrated scales. The gain of each derivative network is changed automatically as the scan rate is changed to make the recorded derivative peak heights essentially independent of scan rate, thus eliminating the need for a range search. When the ORNL Polarographic Drop Time Controller is used with this polarograph at a drop time of 0.5 second and a scan rate of 1 volt/minute, polarograms can be recorded in approximately 30 seconds with a relative standard deviation of 0.2% at a concentration of 10^{-3} *M*."

1.9 APPARATUS FOR PRECISION CONTROL OF DROP TIME OF A DROPPING-MERCURY ELECTRODE (ORNL MODEL Q-2942)

W. L. Belew H. C. Jones[9]
D. J. Fisher M. T. Kelley

A paper was published[3] that describes an apparatus for precision control of drop time of a dropping-mercury electrode (ORNL model Q-2942). The abstract follows.

"An apparatus is described for the precise electromechanical control of the drop time of a dropping mercury electrode (DME) at values from 0.25 to 10 sec. A constant drop time is obtained over the entire potential range with capillaries having their orifice in the horizontal or the vertical plane. Excellent performance is most easily obtained with a Smoler 90° vertical orifice DME. Corresponding portions of controlled drop time current-time curves are essentially unchanged from uncontrolled drop time curves. The constant drop time simplifies the efficient filtering of the current fluctuations from the DME. Increased sensitivity and precision are obtained. At a 0.5-sec controlled drop time, a scan rate of 1 V/min, and with the ORNL model Q-2792 polarograph, regular or first- or second-derivative polarograms can be recorded in ~30 sec with a relative standard deviation of 0.2% at the $10^{-3}M$ concentration level. Satisfactory first- or second-derivative dc polarograms are obtained at concentrations as low as $10^{-7}M$."

1.10 INTERPRETATION OF THE POLAROGRAPHIC BEHAVIOR OF METAL–SOLOCHROME VIOLET RS COMPLEXES

T. M. Florence[14] W. L. Belew

A paper on the interpretation of the polarographic behavior of metal–solochrome violet RS complexes was published;[15] the summary follows.

"A detailed investigation has been carried out to elucidate the electrode and solution phenomena that produce two discrete polarographic reduction waves when the azo dye, solochrome violet RS (5-sulfo-2-hydroxybenzene-azo-2-naphthol), is complexed with metals. Previous explanations put forward to explain the polarographic activity of di-*o*-hydroxyazo compounds towards certain metals were shown to be invalid. It is proposed that the half-wave potential shift ($\Delta E_{1/2}$) results from the metal remaining bonded to the reduced form of the dye (hydrazo derivative), the potential of the DME being controlled by the metal-azo/metal-hydrazo redox couple. The $\Delta E_{1/2}$-values are then independent of pH and metal concentration, and depend only on the relevant stability constants and acid dissociation constants.

"The rates of reaction of Al^{3+} and Fe^{3+} with solochrome violet RS have been measured, and both reactions were shown to proceed *via* metal hydroxy complexes."

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[13] X-Ray and Spectrochemistry Group.

[14] Chemistry Division, Australian Atomic Energy Commission Research Establishment, Lucas Heights, New South Wales, Australia.

[15] T. M. Florence and W. L. Belew, "Interpretation of the Polarographic Behavior of Metal–Solochrome Violet RS Complexes," *J. Electroanal. Chem.* **21**, 157 (1969).

1.11 PHILOSOPHY OF THE DESIGN OF INSTRUMENT SYSTEMS FOR MEASUREMENTS OF HIGHEST SENSITIVITY

D. J. Fisher

A method (design philosophy) has evolved for the design of instrument systems for measurements of highest sensitivity. The method is described in a paper,[16] the summary from which follows.

"The over-all purpose of this paper is to present an explanation and survey of topics that are particularly pertinent to the design of instrument systems for measurements of highest sensitivity. The paper necessarily includes a wide range of subject matter. To concisely describe the scope of the paper and to provide a means for quick access to the specific topics as well, a table of contents is located immediately after the summary. The headings and subheadings in the text are keyed to this table. The first specific purpose of this paper is to present a method, a design philosophy, for the design of instrumentation for highly sensitive measurements. Two premises of the method, the significance of signal-to-noise ratio, a definition of a measurement system and the identification of the system elements, and the procedure for the optimal design of the system and of its elements are given in Section I. The second specific purpose of this paper is to present information essential to the implementation of this method: namely, meanings, significances, implications, comparisons, illustrations, and conclusions regarding the pertinent topics. These topics are discussed in Sections II-VII. The third specific purpose of this paper is to further clarify and illustrate the method itself by means of examples of its application in two measurement areas: dc polarography (Section VIII. A) and flame photometry (Section VIII. B). The fourth is to provide illustrations of the design and uses of circuit elements and systems to optimize and enhance S/N, with examples of performance capabilities in exemplary applications (Section IX). The fifth is to furnish a critical bibliography of sources of details about underlying theory and principles, and to include a selected bibliography of application information for biological, physical, chemical, and technical measurements."

1.12 USE OF ENSEMBLE AVERAGING TO INCREASE THE SENSITIVITY OF MEASUREMENTS WITH FLAME SPECTROPHOTOMETERS

D. J. Fisher R. W. Stelzner H. C. Jones[9]

A paper on the use of ensemble averaging to increase the sensitivity of measurements with flame spectrophotometers was published;[17] the summary from the paper follows.

"A number of steps that optimize the signal-to-noise ratio, S/N, were provided in the flame spectrophotometer design, including beam chopping followed by narrow-band-tuned amplification. It has been shown that the relative effectiveness of three methods of optimizing or enhancing S/N to increase the sensitivity and decrease the lower limit of detection of flame photometric measurements increases in this order: linear detection followed by RC filter averaging; linear detection followed by RC filter averaging followed by active integration averaging; ensemble averaging of output of tuned amplifiers. Correlation in both the frequency and time domains is provided, respectively, by tuned amplification and by ensemble averaging. Other advantages of the use of ensemble averagers in flame photometry are pointed out, including correction for background, calibration in the sample matrix, data transformations, faster wavelength scanning without S distortion, testing of design steps to optimize S/N, digital data acquisition, and the attainment of a greater advantage from the dependence of multiplier phototube gain upon the spectral sensitivity of the photocathode and upon the voltage applied to the tube. To obtain measurements of highest sensitivity, all portions of the measurement system must be designed to optimize S/N wherever economically and technically feasible in order to obtain a system output that has the highest S/N. The enhancement of the optimized S/N by ensemble averaging can provide a further increase in the sensitivity of measurement, or, in a practical problem, enhancement of S/N by ensemble averaging may be attractive because of the achievement of sufficiently high sensitivity (and low detection limits) with the use of relatively simple procedural steps."

1.13 DESIGN OF MEASURING CIRCUITS FOR A DC POLAROGRAPH

R. W. Stelzner

A paper was accepted for publication[18] that describes the design of measuring circuits for a dc polarograph; the summary follows.

"The purpose of this paper is to describe several of the mathematical design techniques that have led to the electronic circuitry of the ORNL model Q-2792 Controlled-Potential DC Polarograph. Primary emphasis is placed upon the time-averaging and time-derivative circuits of that instrument. The mathematical techniques range from simple algebra and arithmetic through the calculus, Fourier and Laplace transformations, matrix algebra and the theory of the complex variable."

1.14 CELL DESIGN FOR MINIMIZING *iR* ERROR IN CONTROLLED-POTENTIAL POLAROGRAPHY OF HIGH-SPECIFIC-RESISTANCE SOLUTIONS

W. L. Belew M. T. Kelley
D. J. Fisher J. A. Dean[19]

A paper was prepared that describes a cell of simple design which minimizes the *iR* error in controlled-

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[16] D. J. Fisher, "A Method for the Design of Instrument Systems for Measurements of Highest Sensitivity," *Chem. Instrum.* **2**, 1 (1969).

[17] D. J. Fisher, R. W. Stelzner, and H. C. Jones, "The Use of Ensemble Averaging to Increase the Sensitivity of Measurements with Flame Photometers," *Chem. Instrum.* **2**, 51 (1969).

[18] R. W. Stelzner, "The Design of Measuring Circuits for a DC Polarograph," to be published as *Chem. Instrum.* **2**, 213 (1970).

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

potential polarography of solutions having high specific resistance. During the entire lifetime of each drop, the tip of a micro reference electrode (probe) is very close to the surface of the mercury drop. Thus, only a small part (which can be negligible) of the iR_{inner} voltage drop is picked up by the probe and remains as an uncompensated *iR* error. Polarograms that have almost no distortion due to *iR* error can be obtained when potentiostatic electrolysis is used in conjunction with this cell. Solutions up to millimolar in concentration of the species of interest and having specific resistances as high as 22,000 ohm-cm can be analyzed satisfactorily.

1.15 ADVANCES IN INSTRUMENTATION FOR DC POLAROGRAPHY AND COULOMETRY

D. J. Fisher

By invitation from Priv.-Doz. Dr. H. W. Nürnberg,[20] a manuscript, "Advances in Instrumentation for DC Polarography and Coulometry" was written to be included in a forthcoming volume (edited by H. W. Nürnberg) *Advances in Electroanalytical Chemistry*, of the series (edited by C. N. Reilley) **Advances in Analytical Chemistry and Instrumentation**, Interscience, New York. The first draft has been submitted.

1.16 USE OF 2,2-DIMETHOXYPROPANE AS A DEHYDRATING AGENT IN POLAROGRAPHY OF NONAQUEOUS SOLUTIONS

W. L. Belew T. M. Florence[14]

The conventional ways to dry solvents for the polarography or electrochemical investigation of nonaqueous solutions are distillation and the use of drying agents such as calcium hydride or molecular sieves. These methods satisfactorily reduce the water content to as little as 5×10^{-4} *M*. However, it is extremely difficult to transfer a solution to a polarographic cell and sparge the cell and solution of oxygen without introducing water to a concentration level of at least several millimolar. A new method that uses 2,2-dimethoxypropane to remove traces of water from a solvent was developed. The method permits the water concentration of a nonaqueous test solution in a polarographic cell to be maintained at a very low level for a long time. The reagent has been used as a dehydrating agent for infrared spectrometry.[21] At pH 6 and lower, it reacts quantitatively with water to form methyl alcohol and acetone. When it is added to dimethyl sulfoxide that has been made slightly acid (pH 6 or lower) with H_2SO_4, the water concentration is reduced to a level ($<5 \times 10^{-4}$ *M*) not detectable by titration with Karl Fischer reagent. The acetone and methyl alcohol are removed by sparging with argon, and an essentially anhydrous medium can be maintained for ~30 min. This technique should be applicable to other nonaqueous solvents and could be useful in the study of electrochemical reactions in nonaqueous media.

1.17 MODEL IX-A FLAME SPECTROPHOTOMETER FOR THE GENERAL ANALYSES LABORATORY

T. R. Mueller

A flame spectrophotometer was designed and fabricated for use in the General Analyses Laboratory. It is similar electronically to the model IX[7] instrument but has a different optical arrangement. In the model IX configuration, the flame image was focused on the entrance slit to the monochromator by a quartz lens in line between the flame and the slit. In the model IX-A, a spherical mirror is used to flood the entrance slit with light from the flame, which is housed in a Jarrell-Ash model 82-500 flame attachment.

The model IX-A is equipped with a Salient Electronics Company model SEI-15/15-100 low-voltage power supply instead of the more expensive Philbrick/Nexus Research model PR-30C supply; it has a variable attenuator on the output of the amplifier to replace the fixed divider of the model IX; it has provisions for checking the electrical "zero" of the recorder or for off-setting the recorder zero; and it is supplied with a Brown 10-mV recorder instead of the Heath model EUW 20A recorder.

The model IX-A was placed in service in May 1969 as a substitute for a model VII[22] instrument. The model VII instrument had been in service ~6 yr and was in need of extensive mechanical repairs. The performance of the model IX-A is being followed, and the need to rebuild and to reinstall the model VII is being assessed.

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[20] Director, Zentrallabor für Chemische Analyse der Kernforschungsanlage, Jülich GMBH.

[21] D. S. Erley, "2,2-Dimethoxypropane as a Drying Agent for Preparation of Infrared Samples," *Anal. Chem.* **29**, 1564 (1957).

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

1.18 INSTRUMENTS AND EQUIPMENT FOR ANALYSIS OF IRRADIATED LMFBR FUELS IN THE HRLAL

W. L. Belew J. L. Botts[23]

In the near future it will be necessary to analyze irradiated samples for the Metals and Ceramics Division's LMFBR fuels-irradiation program. The program will include analysis of oxide and nitride fuel samples for oxygen, nitrogen, carbon, and total gas release. Members of several groups in the Division have formed a task force to design or modify several instruments to permit these analyses to be done remotely in the work cells at the HRLAL facility.

1.18.a Modification of a Leco Rapid Oxygen Analyzer

A Leco rapid oxygen analyzer is being modified to permit its use in the work cells at the HRLAL (Sect. 6.2.d.a). The gas train has been removed from the analyzer and placed in a separate instrument cabinet, which will be located in the work cell. Thus, all radioactive gases will be contained in the cell. The induction-furnace controls are being designed to be operated either remotely or with master-slave manipulators. A preliminary evaluation of the instrument will be made in the mock-up facility at the HRLAL; further modifications will be made if necessary.

1.19 ANALYTICAL COMPUTER SYSTEMS

M. T. Kelley R. W. Stelzner

We have placed in operation a Digital Equipment Corporation (DEC) model PDP-8/I computer (4K-word core memory, 1.5-μsec cycle time) for use in studying its applicability in analytical chemistry where on-line data acquisition and rapid computation can be of benefit. The incorporation of computer assistance into analytical procedures can greatly increase the accuracy, precision, and throughput of the procedures. The peripheral equipment acquired with the basic computer consists of a multiplexed 32-channel analog-to-digital converter (ADC) that has selectable 6- to 12-bit accuracy for corresponding conversion times from 9 to 35 μsec; a three-channel digital-to-analog converter with a 12-bit accuracy; and a Teletype model ASR-33 printer.

[23]Methods Development and Evaluation Laboratory.

After the computer was installed by the DEC field engineer, it was tested with factory-designed diagnostic programs. We devoted an initial period to learning the high-level conversational language (FOCAL), a mnemonic assembly language, and the machine language (octal code). We attended courses in programming at the factory in Maynard, Mass., (RWS) and at Oak Ridge (RWS and MTK); the latter course was sponsored by the ORNL Instrumentation and Controls Division.

We wrote some general-purpose algorithms, for example, square root, cube root, exponential, sin, cos, and a random-number generator, as well as some general-purpose programs for least-squares fitting of a straight line and a parabola and for numerical integration by use of the trapezoidal method and Simpson's rule.

Computer programs were written in FOCAL for the following groups within the Division to make the indicated computations:

Group	Computation
General Analyses Laboratory	Concentrations from spectrophotometric data
	Total anions, total cations, and material balance in molten fluoride salts
High-Level Alpha Radiation Laboratory	Radioactivity (counts min^{-1} ml^{-1} and counts min^{-1} g^{-1}) corrected for background
Radioisotopes-Radiochemistry Laboratory	Radioactivity (counts min^{-1} ml^{-1} and counts min^{-1} g^{-1}) corrected for background
Reactor Projects Group	Gas chromatographic results

The purpose of these programs was to demonstrate to each group PDP-8/I computer calculation of a large number of analytical results.

The computer has been used as a component of systems for voltammetry (Sects. 1.20 and 3.2.b) and flame spectrophotometry (Sect. 1.21).

The PDP-8/I computer system was upgraded to date by DEC field engineers. Also, a high-speed reader (Digitronics model 2540) was installed by the Instrumentation and Controls Division, and a DF32 magnetic disk (32K-words) was added. A crystal-controlled real-time clock and a second PDP-8/I computer system that will be compatible with the first is on order. The second computer will consist of the basic 4K-word memory with an additional 4K of fast core, an AA01A digital-to-analog converter, an AF01A analog-to-digital con-

verter, a DF32 magnetic disk, and a line-frequency real-time clock.

In the future, analytical computer systems will be evaluated for applicability in remotely controlled analyses in hot cells, a program that exploits the GeMSAEC Analyzer, and polarography.

1.20 DETERMINATION OF U(IV)/U(III) RATIOS IN MOLTEN FLUORIDE SALT FOR THE REACTOR PROJECTS GROUP

M. T. Kelley R. W. Stelzner D. L. Manning[24]

The PDP-8/I was interfaced to a molten-salt voltammetric experiment for the determination of U(IV)/U(III) ratios as a function of time (Fig. 1.3). These ratios were determined at a temperature of 500°C by application of a controlled-potential sweep to a three-electrode cell. The first derivative of cell current as a function of time (and therefore of potential, since $E = kt$) is the analog signal that furnishes the input to the analog-to-digital converter of the computer system (Fig. 1.4). The signal is secured by an analog derivative module and the controlled-potential controlled-current cyclic voltammeter.[8] The voltammetric phase of the work is discussed in Sect. 3.2.b.

Two successful programs were written in FOCAL for the automatic operation of the analysis. The programs differ mainly in the manner in which the cell potential that corresponds to the minimum of the di/dt vs E curve is detected. One program uses zero-slope measurement; the other employs computer search.

A typical printout follows.

```
OFFSET (MV)      :300 TEMP      :500      TIME:1022

U4/U3  = 75.5 W = 3.2 MV/DP E = 0.288 V START = 0.112V
U4/U3  = 75.4 W = 3.2 MV/DP E = 0.288 V START = 0.112V
U4/U3  = 76.1 W = 3.2 MV/DP E = 0.289 V START = 0.113V
U4/U3  = 75.7 W = 3.2 MV/DP E = 0.288 V START = 0.112V
U4/U3  = 79.4 W = 3.2 MV/DP E = 0.292 V START = 0.112V

S = 1.685
MEAN = 76.432
E(AVE) = 0.289
```

[24] Reactor Projects Group.

ORNL-DWG. 69-9641B

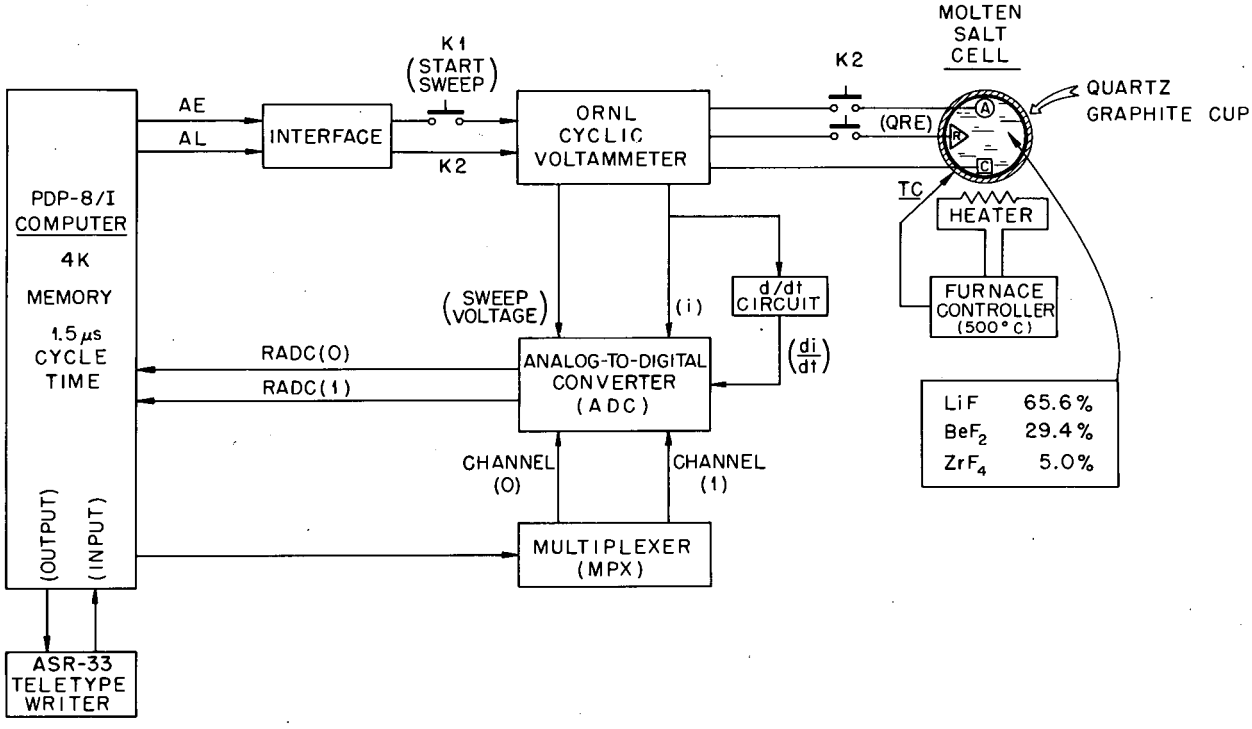

Fig. 1.3. Block Diagram of Instrumentation for Computer-Controlled Voltammetric U(IV)/U(III) Ratio Determination.

ORNL-DWG. 69-10274A

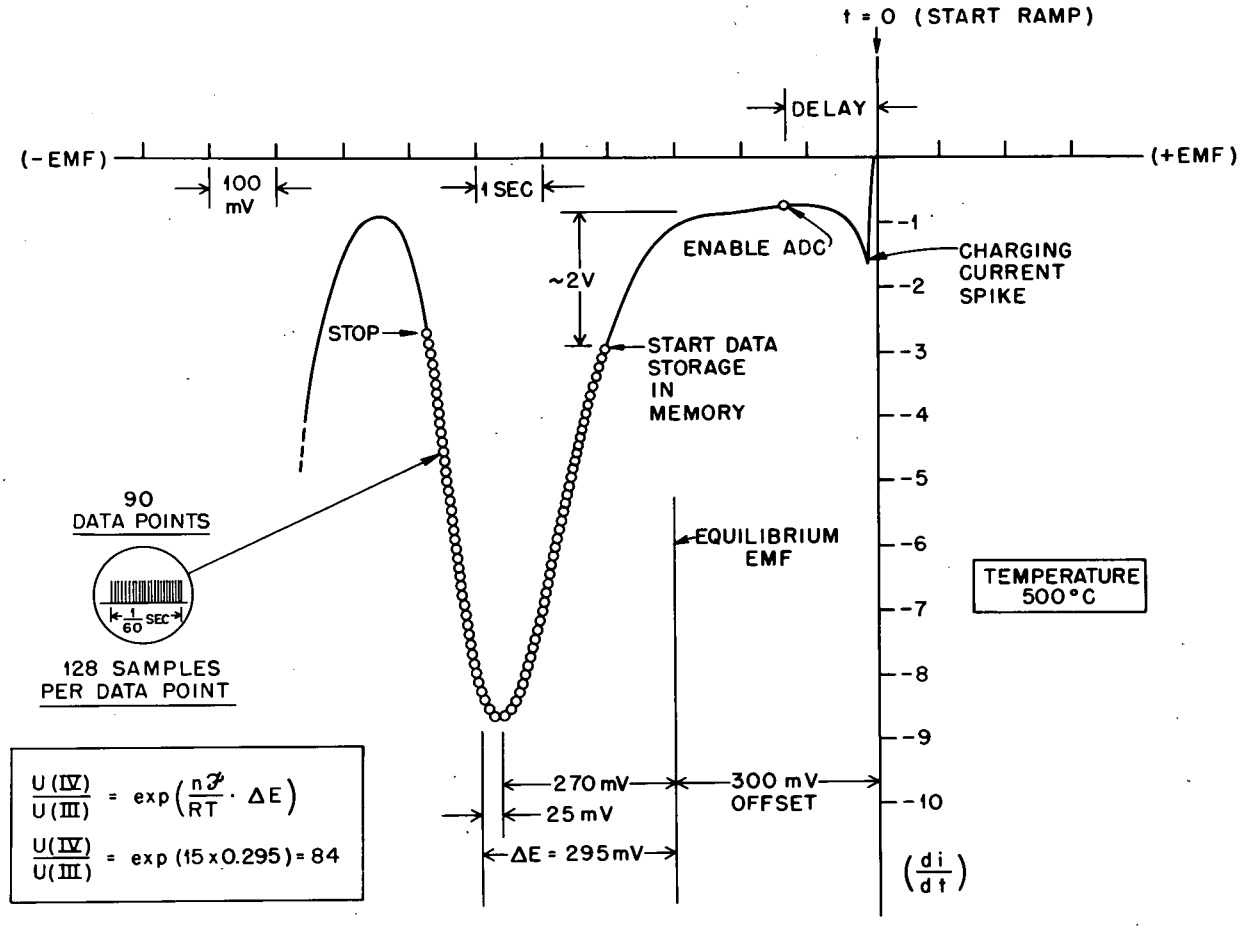

Fig. 1.4. Computer-Controlled Voltammetric U(IV)/U(III) Ratio Determination (First Derivative).

The U(IV)/U(III) ratio can be monitored for long periods of time. The computer system is well-suited for kinetic studies and should be readily adaptable to continuous analysis.

Because the FOCAL compiler uses ~75% of the 4K-core, only ~1000 words remain for the computer program and data storage. Since each stored data point requires five words, a severe limitation is imposed on the number of data points. This limitation has been overcome by writing essentially the same voltammeter programs in machine language. The octal machine-language programs have provision for storing and processing 256 data points.

1.21 DIGITAL FLAME SPECTROPHOTOMETRY

M. T. Kelley T. R. Mueller
C. R. Spell[25] R. W. Stelzner

Future programs at ORNL are expected to produce many samples for which multielement automatic flame spectrophotometric analyses must be done with a very short burn time. A computer system that provides monochromator control and rapid digital data acquisi-

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

tion and processing (mainly statistical) should meet this need. Work to develop such a system is progressing. The system will include analog and digital components and computer software.

The analog portion was assembled from the following units: a Beckman model 4020 O_2-H_2 atomizer-burner with a model 9220 gas regulator, a front-surface plastic-coated spherical mirror, a Heath model EU-700 monochromator unit with digitally controlled wavelength-drive unit, a combination dark-slide and phototube-mounting plate, an ORNL model IX-B flame-spectrophotometer amplifier system, and a Fluke model 415B high-voltage power supply. The burner was mounted in line with the mirror and entrance slit to the monochromator. Positioning adjustments were provided. The phototube-mounting plate, the amplifier system, and an inexpensive housing for the flame and mirror were constructed locally. The mounting plate was used with a 1P28 multiplier phototube and with an Electro Optics Associates model PM-103 housing that contains an EMI 9558 multiplier phototube. The PM-103 housing permits the phototube to be cooled to −78°C. For some of the work, nitrogen gas from the evaporation of liquid nitrogen can be used for cooling. The amplifier system is an extension of the ORNL model IX[7] system. The only significant difference between the model IX-B and the model IX is the addition of an inverting amplifier, which permits signal amplification, and of selectable damping factors. The option of minimum internal damping is available. The inverting amplifier also provides the negative-going signal required for the analog-to-digital converter (ADC).

For the digital portion of the system, the PDP-8/I computer is used. In earlier work, the Fabri-Tek model 1064 Instrument Computer (signal-averager) was used. Although the data could be acquired in reasonable time, data processing of the 4096 channels required instrument readout and manual selection and reading of an 80-sample group followed by manual entry into a desk calculator for statistical evaluation. The PDP-8/I with its automatic operations demonstrated clear-cut superiority.

A computer program was written in machine language for acquiring 1000_8 data points, each of which is the average of 1 to 4000_8 samples. From these data, the mean, variance, and relative standard deviation are computer calculated. The results have been of help in optimizing conditions (H_2 and O_2 pressures, slit width, multiplier phototube types, and high voltage) for a burn to produce the most reliable results with minimum noise. The program has been expanded to calculate, print, and plot a frequency distribution of the data in the range $\pm 200_8$ addresses from the mean.

For a scan λ_1 to λ_2, the computer operates the stepping motor of the monochromator via software-generated pulses at a rate up to 2 mm/sec. Digitized data obtained from the ADC are stored in memory for later readout on an *X-Y* recorder. A plot of multiplier phototube current (flame intensity) vs wavelength is secured by using the digital-to-analog converter commanded by the DAL1 and DAL4 functions. The stored data are smoothed before readout by the application of convolution techniques (choice of a 5-point cubic, 7-point quintic, or 9-point quintic equation). This powerful mathematical smoothing procedure, coupled with the ensemble averaging obtained in the original data acquisition, produces very smooth emission curves.

Another mode of operation involves computer-operated rapid sweeping of the monochromator over a narrow band width that encompasses the emission peak. An effective modulation occurs at the sweep frequency; thus, advantages associated with a lock-in amplifier are achieved.

The system was used to study noise characteristics in the flame source; the test solutions contained sodium or calcium. Earlier work which showed that the band-limited flame noise is predominantly white random noise[17] was confirmed. Also, no essential difference in noise characteristics was detected between the emission band and the emission line of calcium.

This preliminary work demonstrates the usefulness of a computer-controlled system for the rapid acquisition and processing of data from a flame-photometric experiment.

2. Analytical Methodology

W. D. Shults

2.1 CHARACTERIZATION OF TOBACCO SMOKE AND SMOKE CONDENSATES

M. R. Guerin

The program "Collection, Separation, and Elucidation of the Components of Cigarette Smoke" is under way to develop methods that definitively characterize tobacco smokes. The Subgroup on Chemical Analysis of Cigarette Smoke (Subgroup III), Lung Cancer Task Force, National Cancer Institute, has established "protocols" to define specific chemical compounds as indicators of the stability and biological potency of smoke preparations. We are developing analytical methods to monitor a large number of individual smoke constituents rather than a selected few and will emphasize those classes of constituents that contain indicator compounds.

2.1.a Preliminary Activities

Dr. H. L. Falk[1] of the Lung Cancer Task Force expressed interest in those papers in the Holsopple Collection[2] that deal with the determination of smoke constituents. That collection and several other sources were used to prepare a bibliography on the application of gas chromatography to this problem; the bibliography was forwarded to Dr. Falk. These compilations of information have been updated and expanded by computer searches of *Gas Chromatography Abstracts* and *Chemical Titles;* they now contain ~300 publications, abstracts, and references. Data from the compilations are being put into a table that lists gas chromatographic methods and the results of their use to analyze tobaccos and tobacco smokes. Both the table and the file of publications are extremely useful in determining the direction and emphasis of our experimental work.

We have established a library of tobacco-related reference chemicals. It now includes more than 200 compounds: ~70 bases (pyridines, anilines, alkaloids, indoles, acridines, and others), 60 phenols (monohydric, dihydric, and phenolic acids), 25 aliphatic carboxylic acids, 40 aromatic hydrocarbons (primarily dicyclic through pentacyclic), and selected inorganic and organic gases. The availability of these chemicals greatly accelerates the development of gas-chromatographic (GC) procedures and simplifies fractionation-concentration studies.

Gas chromatograms (component profiles) of the fractions and subfractions generated in the exploratory[3] work and in a subsequent study were invaluable in defining shortcomings of existing techniques. In the procedures used initially to condense the smoke and to concentrate extracts, substantial amounts of the more volatile smoke constituents were lost. The very large number and great size variation of profile peaks demonstrated the need for greater specificity in fractionation and in chromatographic techniques.

2.1.b Smoke Generation and Collection

To date at ORNL, cigarette smoke has been generated with the Holsopple machine.[4] It was presumed that passing the smoke through a series of cold traps at progressively lower temperatures would effect both efficient collection of all the smoke constituents and their prefractionation according to volatility. In fact, neither was achieved with the original trap arrangement. Component profiles of the unfractionated contents of the various traps were much more alike than different, only a very minor excess of high-boiling components being in the first trap (ice-salt bath) and a very minor excess of low-boiling components being in the third trap (liquid-nitrogen bath). As a result of the slow condensation of smoke that stood between puffs, the dead volume within the system (lines from the cigarettes to the first trap and between the traps) collected a substantial amount of condensate of essentially the same composition as that in the traps. The facts that standing smoke was always present in the system and that the solenoid (downstream of the liquid-nitrogen

[1] Chairman, Subgroup III, Chemical Analyses, of the Tobacco Working Group, Lung Cancer Task Force, National Cancer Institute; and Associate Director for Laboratory Research, National Environmental Health Services Center, Research Triangle Park, N.C.

[2] H. L. Holsopple, "Characterization of Tobacco Smoke and Smoke Condensates. A Literature Survey," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968,* ORNL-4343, p. 88.

[3] M. R. Guerin, "Characterization of Tobacco Smoke and Smoke Condensates. Exploratory Studies," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968,* ORNL-4343, p. 16.

[4] H. L. Holsopple, "Characterization of Tobacco Smoke and Smoke Condensates. ORNL Smoking Machine," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968,* ORNL-4343, p. 88.

trap) was greatly contaminated with condensate showed that the overall trapping efficiency of the apparatus is very poor. The notable lack of evidence from the component profiles for the presence of low-boiling smoke constituents in the various traps likewise indicated poor trapping efficiency. Also, the geometries of the traps and of the flow lines prevent complete removal of the condensate.

A puff taken by the Holsopple smoking machine is of standard duration (2 sec) and frequency (one/min) but only approximates the standard puff volume (35 ml). Puff volume depends greatly on the resistance-to-draw of the cigarette smoked and on the degree of vacuum at both the source and time of puffing. Our experimental efforts have reached the point where approximation of a standard smoke sample is no longer always sufficient. Accordingly, we are acquiring a machine that will generate standard total particulate matter and smoke condensate; it is a modified version of the machine used by the Federal Trade Commission. We will further modify the machine to generate whole smokes and gas-phase fractions in forms convenient for analysis.

In spite of the serious shortcomings of the Holsopple smoking machine, it was very useful in producing whole condensate for qualitative work. We have recently used a more simple, and apparently more efficient, way to generate and collect cigarette smoke. Cigarettes are smoked directly into a solvent contained in a gas-washing bottle. Although complications from standing smoke and removal of trapped constituents still exist, they are decreased substantially. When the smoke from 20 or less cigarettes is required, this procedure is especially superior to the cold-trap system.

2.1.c Fractionation and Concentration

Most procedures for the fractionation of tobacco and tobacco-smoke constituents begin with classical solvent extractions at various acidities. Batch extractions are commonly used to obtain fractions enriched in acid, basic, or phenolic components. Such techniques are well established and will continue to be relied upon heavily.

For certain cases, continuous liquid-liquid extraction is preferable to batch extraction. We have achieved 75 to 95% recovery of mixed isomeric phenols from synthetic samples after only several hours of ether extraction. A properly assembled continuous-extraction apparatus would require a minimum of operator attention and would avoid those sample losses that accompany manual transfers of solvent to and from separatory funnels. The possibility of continuous extraction directly from the smoke trap is of particular interest.

To increase the specificity of fractionating smoke constituents, column chromatographic procedures will be required. Lipophilic resins such as Amberlite XAD-2 hold promise for removing less polar from very polar compounds and for concentrating neutral constituents. We have not yet explored the general usefulness of such procedures.

Methods of concentrating extracts and column effluents are as critical as the extraction or chromatographic procedure itself. The most gentle of concentration procedures can result in intolerable losses of solutes. In a very gentle blow-down of etheral solutions of phenols, a loss as great as 90% occurs when the solution is taken to dryness. If blow-down is stopped with 1 to 2 ml of residual solution, the loss is decreased to 10 to 30%. Preliminary experiments indicate that the loss can be reduced to 0 to 10% by including a partly miscible solvent of low vapor pressure (e.g., dimethyl sulfoxide) in the ethereal extract. Thus, as much care must be taken in the final step of fractionation as in any part of the entire analytical scheme.

It seems feasible to construct an apparatus that can semiautomatically generate concentrates of selected smoke constituents. Its design would be based on the combined use of solvent collection of smoke, continuous liquid-liquid extraction, and continuous concentration of the generated extract. Such an apparatus should improve substantially the quantitative reproducibility of the generation of given smoke-constituent fractions.

2.1.d Gas Chromatography

By trimethylsilylation of smoke-related phenols, we were able to extend the applicability of gas chromatography to polyhydric phenols and phenolic acids and to resolve 17 components of a 22-component synthetic sample in less than 20 min. Of 27 components of a synthetic mixture of aliphatic and aromatic carboxylic acids, 27 were resolved in 20 min as their trimethylsilyl esters. Although silylation substantially extends the number of compounds of many classes to which gas chromatography can be applied, it causes complications in quantitative measurements. We are giving considerable attention to the quantitative aspects of silylation.

The ability to chromatograph samples without prior derivatization simplifies analytical procedures and yields inherently superior quantitative results. Thus, un-derivatized species have been used primarily. We have studied the elution behaviors of some 70 free

bases, phenols, and acids in four column systems at various column temperatures. As the liquid phase of a chromatographic column, polyglycols are generally superior to polyesters for the separation of both basic and acid compounds. Differences in the elution order of certain compounds on liquid phases of the two types can prove useful in identifying sample components. Methyl silicone liquid phases are excellent for the separation of important neutral constituents of smoke; at least 20 polycyclic aromatic hydrocarbons can be resolved easily in one programmed temperature run. We are assessing further the usefulness of methyl silicone columns for the separation of free bases and phenols.

The results of our gas-chromatographic studies assure us of our ability to chromatograph any smoke-related compounds now considered to be of biological significance. However, it is clear that no conventional chromatographic system is capable of the discrimination required for the total analysis of whole smoke or of major subfractions of whole smoke. The combined use of high-resolution techniques, multiple-column gas chromatography, high-specificity–high-sensitivity detection, and subambient to high-temperature programming offers the only hope of progressing toward that goal. We are exploring all these possibilities.

2.2 GAS CHROMATOGRAPHY (GC)

2.2.a Silicon-Specific Flame-Photometric Detector

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

The development and study of a silicon-specific detector for gas-liquid chromatography (GLC)[7] has continued. The detector is based on flame-photometric measurement of the silicon present in the GC effluent. Both atomic absorption and flame emission were examined as a means of detection. Each technique is feasible, but flame emission is preferable, mainly because of the availability and low cost of acetylene relative to nitrous oxide. Also, the calibration curve is linear for flame emission but nonlinear for atomic absorption.

The system consists of a Beckman model DU flame photometer interfaced with a suitable GC unit. The GC effluent is fed directly to the oxygen-acetylene burner in such a manner that the flame-photometric detector (FPD) can be operated simultaneously with another detector; thus, it is possible to detect both silylated and nonsilylated compounds. The chromatograms in Fig. 2.1 are exemplary; they were obtained with a Beckman model GC-2A gas chromatograph, which has thermal-conductivity (TC) detection. Subsequently, we have used a Barber-Colman model 20 gas chromatograph, which is equipped with glass columns and has programmable temperature capability and a flame-ionization detector (FID). For simultaneous FPD and FID, an outlet splitter having a ratio of 6:1 was installed.

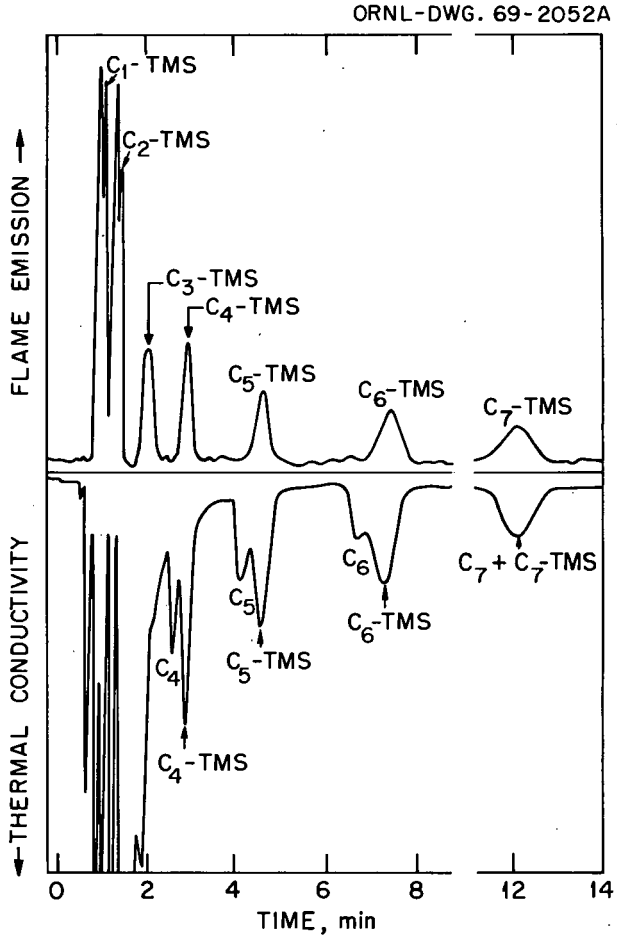

Fig. 2.1. Gas Chromatograms of Partly Silylated *n*-Alcohols in Pyridine Solution Recorded Simultaneously by Flame-Photometric and Thermal-Conductivity Detectors. [Conditions: column, 6-ft by ¼-in. stainless steel packed with SE-30 (20%) on Chromosorb W; column temperature, 130°C; helium flow, 100 cc/min.]

[5]Predoctoral Student Guest from the Graduate School of Chemistry, University of Tennessee, Knoxville.

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

[7]R. W. Morrow, J. A. Dean, M. R. Guerin, and W. D. Shults, "Derivative Gas Chromatography," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968*, ORNL-4343, p. 13.

The silicon-specific FPD gives linear response from ~3 μg to at least 100 μg of silicon. The detection limit of the FPD, expressed as the amount of silicon necessary to produce a signal equal to twice the peak-to-peak noise level, is 0.44 μg. The response of the silicon-specific detector is independent of the type of chemical bonding of the silicon atom within the compound. Equal weights of silicon present in compounds that contain Si–O, Si–C, and Si–N bonds give equal peak areas.

We used the silicon-specific FPD to estimate the number of silicon atoms per molecule of silylated compound. This estimate was made by comparing the peak areas obtained simultaneously by FPD and TC detection. Once this peak-area ratio is obtained for a standard compound, silicon-containing unknowns can be analyzed, and the number of silicon atoms per molecule can be computed.

Silicon-specific detection provides a unique method to study silylation reactions. Since the detector response depends only on the atoms of silicon per molecule of compound, any suitable organosilicon compound can be used as an internal standard. Silicon-specific detection offers a rapid way to determine directly the percent trimethylsilylation of an active-hydrogen-containing compound. The percent derivatization of a compound is given by the equation

$$\text{derivatization, \%} = 100\left(\frac{\omega_x}{\omega_{100}}\right) = 100\left(\frac{A_x}{A_s}\cdot\frac{\omega_s}{\omega_{100}}\right),$$

where

ω_x = weight of Si found in the test compound

ω_{100} = weight of Si in test compound if 100% derivatization occurs

ω_s = weight of Si in standard compound

A_x = peak area of test compound

A_s = peak area of standard compound.

Table 2.1 gives the percent trimethylsilylation determined, by this method, of some organic compounds; alkyl and aryl silanes were used as internal standards. Since organosilicon compounds suitable as standards may not be available, we studied the feasibility of using *n*-aliphatic trimethylsilyl ethers of some long-chain alcohols and found them to be suitable standards for use in determining the percent conversion of some high-boiling acids.

We believe that the silicon-specific FPD is especially suitable for studying the technique of "on-column silylation." This technique has many practical GC applications that we plan to explore by using the silicon-specific FPD. Other work with this detector that is planned or is in progress includes the silylation of multicomponent solutions, study of the stability of silyl derivatives with time, and the selective silylation of hydroxy compounds with trimethylsilylimidizole. The initial work was done at the University of Tennessee; during the past year the work was moved to ORNL. The results of these studies were presented orally[8] and have been published.[9]

Table 2.1. Percent Trimethylsilylation of Some Organic Compounds as Determined by Silicon-Specific Detection

Compound	Trimethylsilylation (%)[a]
Phenolics	
Phenol	101.5
4-Ethylphenol	98.0
2,6-Dimethylphenol	97.0
Hydroquinone	102.9
3,4-Dihydroxybenzaldehyde	101.9
3,4-Dihydroxybenzoic acid	92.9
Acids	
Valeric	101.5
Nicotinic	93.0
Decanoic	94.1
Adipic	95.1
Salicylic	101.6
n-Aliphatic Alcohols	
Hexanol	100.5
Octanol	97.4
Dodecanol	95.0
Tetradecanol	102.0
Amines	
N-methylaniline	40.8 (48.6)[b]
Indole	56.9 (90.7)[b]

[a]Compound was allowed to react for 1 hr at room temperature with a 2 to 5 molar excess of bis-trimethylsilyltrifluoroacetamide (BSTFA).

[b]Second value obtained after heating the reaction mixture 45 min at 70°C.

[8]J. A. Dean, R. W. Morrow, and W. D. Shults, "A Silicon-Specific Detector Based on Interfacing a Gas Chromatograph with a Flame Emission or Atomic Absorption Spectrometer," 20th Mid-America Symposium, Society for Applied Spectroscopy, Chicago, Ill., May 12–15, 1969.

[9]R. W. Morrow, J. A. Dean, W. D. Shults, and M. R. Guerin, "A Silicon-Specific Detector Based on Interfacing a Gas Chromatograph and a Flame Emission or Atomic-Absorption Spectrometer," *J. Chromatogr. Sci.* 7, 572 (1969).

2.2.b Elemental Analysis

J. L. Bowling[10] W. D. Shults
M. R. Guerin

The possibility was suggested[11] of developing a GC system to determine simultaneously carbon, sulfur, phosphorus, nitrogen, and perhaps the halogens or their relative amounts (profiles). Work was begun to develop such a system.

First, a Coulson Conducting Detector was evaluated in its normal mode, that is, as a nitrogen detector. When both a nickel catalyst and a $Sr(OH)_2$ filter are in the reduction chamber, the system is selective for nitrogen. Without the filter, nitrogen and chlorine are detected well and sulfur poorly. When the catalyst is platinum, chlorine, nitrogen, and sulfur are detected.

The reduction furnace of the Coulson Detector was interfaced with a gas chromatograph. This arrangement permitted separation and detection of the reduction products. Various Poropak column packings and chromatographic conditions were evaluated. With a 4-ft by $^1/_4$-in.-OD glass column packed with Porapak-T and operated at 100°C, the products CH_4, NH_3, H_2S, HCl, and H_2O were eluted in that order and were detected by thermal conductivity. This preliminary work indicates that the simultaneous separation and determination of total nitrogen, sulfur, carbon, oxygen, phosphorus, and the halogens is possible by GC. We envision numerous applications of this method.

2.2.c Applications

2.2.c.a Fatty Acids (M. R. Guerin). – Studies involving the GC of fatty acids were completed.[12] Fatty acids (C_{14}–C_{20}) were detected in extracts submitted by Dr. J. Souto.[13] Gas chromatography of the trimethylsilyl esters of the acids on silicone columns was more satisfactory than the chromatography of the free acids on FFAP (modified Carbowax 20M) columns.

2.2.c.b Mixture of Formic Acid, Acetic Acid, Methanol, and Ethyl Alcohol (A. D. Horton[14]). – When a mixture of acids and alcohols is injected into a gas chromatograph, the heat of the injection port causes the acids to react with the alcohols and to produce the corresponding esters. Of the compounds formic acid, acetic acid, methanol, and ethyl alcohol and their reaction products, all except methyl acetate and ethyl formate were separated on a 6-ft by $^3/_{16}$-in. stainless steel column filled with Porapak Q–Porapak T (3:1). The column was operated at 110°C for 4 min and then was temperature programmed at 5°C/min to 170°C.

The boiling points and structures of methyl acetate and ethyl formate are too closely related to permit their separation with these mixed Porapaks. Columns of Carbowax 20M and of FFAP were also tested for this separation, but no improvement was obtained.

2.2.c.c Phenylthiohydantoin Derivatives of Amino Acids (M. R. Guerin, W. D. Shults). – A paper[15] on the chromatography and sulfur-specific photometric detection of silylated phenylthiohydantoin derivatives of amino acids was accepted for publication; the abstract follows.

"The gas chromatographic behavior of twenty-six trimethylsilylated phenylthiohydantoin amino acids is described. The potential utility of the combined use of trimethylsilylation, sulfur-specific photometric detection, and retention indices for the identification of phenylthiohydantoin amino acids is discussed. Retention indices of the trimethylsilylated phenylthiohydantoin amino acids are tabulated."

Other work with derivatives of amino acids is discussed in Sects. 4.2.d and 4.2.e.

2.2.c.d Determination of Dimethylcholanthrene in a Serum Preparation (M. R. Guerin). – Dr. P. Nettesheim[16] asked that a preparation of dimethylcholanthrene in serum be assayed for its dimethylcholanthrene content. The expected large concentration (~2 mg/ml) and the vapor-phase chromatographic characteristics of polycyclic aromatic hydrocarbons indicated that gas chromatography should be a suitable assay method. It was found possible to analyze for dimethylcholanthrene by directly injecting 5-μl samples of the serum into the chromatograph. A glass "demister" inlet sleeve was used to protect the chromatographic system from contamination by the serum. The only other precaution was the use of a glass rather than a metal chromatographic column to decrease the thermal decomposition of the dimethylcholanthrene. The result agreed with that expected. A brief study

[10] ORNL Summer Employee from Concord College, Athens, W.Va.

[11] M. R. Guerin, W. J. Ross, and W. D. Shults, "Analysis of Water Systems. Aqueous-Injection Gas Chromatography," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968,* ORNL-4343, p. 17.

[12] M. R. Guerin, "Biomedical Applications," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968,* ORNL-4343, p. 12.

[13] Professor, University of Tennessee–A.E.C. Agricultural Research Laboratory, Oak Ridge, Tenn.

[14] Reactor Projects Group.

[15] M. R. Guerin and W. D. Shults, "Gas Chromatography of Silylated Phenylthiohydantoin Amino Acids. Utility of Sulfur-Specific Detection," *J. Chromatogr. Sci.,* in press.

[16] Biology Division.

showed that half the dimethylcholanthrene settled out of the suspension in 10 min.

2.2.c.e Purines and Pyrimidines (M. R. Guerin). – The feasibility of applying gas chromatography to the analysis of mixtures of purines and pyrimidines was demonstrated with the successful separation of six trimethylsilylated nucleoside-derived bases.

2.2.c.f Determination of Span-80 and Ethomeen (M. R. Guerin, W. D. Shults, J. A. Ealy[17]). – The surfactants Span-80 (sorbitan monooleate, mol. wt ≅420) and Ethomeen (polyoxyethylene tallow amine, mol. wt ≅470) are important in the Sol-Gel Process. Whereas the quantity of either, when free of the other, can be determined volumetrically, Span-80 heretofore could not be determined in the presence of Ethomeen. Span-80 and Ethomeen are much too polar for direct determination by gas chromatography. Each, however, can be chromatographed as a trimethylsilyl ether. Chromatograms of silylated Span-80 show several minor components in addition to sorbitan monooleate. Although these components may have resulted from the derivatization, very likely at least some are true impurities. Chromatograms of silylated Ethomeen indicate at least eight minor components; they emerge from the column in a pattern which indicates that they are homologs of polyoxyethylene tallow amine.

Chromatograms of mixtures of silylated Span-80 and silylated Ethomeen show substantial overlap of the primary Span-80 peak by an Ethomeen peak. Efforts to increase the peak resolution were abandoned, because the separation was adequate for quantitative determinations of the Span-80 and Ethomeen. Analysis of samples that contained 0 to 0.5% Span 80 and 0 to 0.5% Ethomeen consisted in evaporation of the 2-ethylhexanol solvent, silylation with bis(trimethylsilyl) acetamide, and gas chromatography at 290°C on a column of SE-30 (5%) on Chromoport XXX.

2.2.c.g Preparative Gas Chromatography (A. D. Horton[14]). – The following organic compounds were purified by preparative gas chromatography to the purity indicated: toluene, 99.99%; mesitylene, 99.99%; ethylbenzene, 99.99%; 1-bromopropane, 99.00%; 1-bromobutane, 99.88%; 1-bromopentane, 99.98%; *n*-tetracosane, 99.0%; and α- and β-naphthalene, 99.5% and 99.6% respectively. The purification of these compounds except α- and β-naphthalene was straightforward. Attempts were made to separate these closely boiling isomers on columns of silicone SE-30, Carbowax 20M, and FFAP. A 30-ft column of FFAP operated isothermally at 225°C with a carrier-gas flow of 125 cc/min gave the best separation. The crude starting mixture contained 69.5% α-naphthalene, 30% β-naphthalene, and 0.5% other compounds. To obtain a clean base-line separation of α- and β-naphthalene, the volume injected could not exceed 0.15 ml.

It was difficult to collect fractions of compounds that gave chromatographic elution peaks with very broad half-band widths. The recorder pen contacts a preset microswitch upscale on the recorder to advance the collector turntable and to place the sample-collecting bottle under the exit tip of the column. When the peak is broad, the pen travels very slowly, and, frequently, a backlash due to a pressure surge is encountered as the silicone seal of the collector bottle contacts the exit tip. The backlash may cause the microswitch to again move the turntable to the standby position and to simultaneously inject a new sample aliquot. This behavior throws the program cycle out of phase, thus causing loss of the desired material and collection of impurities. This situation was corrected by introducing a time-delay relay into the tripping circuit. The relay has an adjustable range of 5 to 60 sec and is actuated by the upscale or downscale contact of the pen with the microswitch. The delay period is set to prevent premature tripping, yet is short enough to allow the desired downscale tripping to occur.

2.3 THIN-LAYER CHROMATOGRAPHY (TLC)

2.3.a Polytetrafluoroethylene as an Adsorbent

Helen P. Raaen

Our development of polytetrafluoroethylene layers for TLC was described in a paper presented at the IUPAC International Symposium on Analytical Chemistry[18] and also in an article accepted for publication in the *Journal of Chromatography.*[19] The summary from the article follows:

"Polytetrafluoroethylene layers have been evolved whose properties are satisfactory for thin-layer chromatography (TLC). These properties include versatility of composition, rate of drying, uniformity, adherence to a support without binder,

[17] General Analyses Laboratory; present address, Graduate School of Chemistry, University of Tennessee, Knoxville.

[18] Helen P. Raaen, "Teflon Layers for Thin-Layers Chromatography," presented at the IUPAC International Symposium on Analytical Chemistry, The University of Birmingham, Birmingham, England, July 21–25, 1969.

[19] Helen P. Raaen, "Polytetrafluoroethylene Layers for Thin-Layer Chromatography," accepted for publication in the *Journal of Chromatography.*

wettability, rate of development, and resolution of test-mixture components. The layers have been used to separate both inorganic and organic mixtures. Polytetrafluoroethylene is compatible in thin layers with numerous TLC adsorbents and thus can be a component of mixed-adsorbent or gradient-adsorbent layers or an inert diluent for other TLC adsorbents. The inertness of polytetrafluoroethylene layers is an advantage in their deposition from, development by, or visualization with highly corrosive liquids. Polytetrafluoroethylene layers are especially suitable for extraction chromatography; the extractant may be included in either the layer or the developer. A thin layer of polytetrafluoroethylene is a suitable solid support for liquid ion exchangers. Therefore, by TLC on polytetrafluoroethylene, it should be possible to study the mechanisms of liquid ion exchange without the need to consider the reactivity of the liquid phases with the solid support."

To determine whether irradiation of polytetrafluoroethylene affects its behavior as a TLC layer material, samples of polytetrafluoroethylene were subjected to three different doses of absorbed gamma radiation and then were used to prepare thin layers for chromatography. A solution of $^{55}Fe^{3+}$ was chromatographed on the layers by development with 0.5 *M* di-2-(ethylhexyl)orthophosphoric acid (HDEHP) in methylisobutyl ketone. For each of the three chromatograms, the autoradiogram was identical with that of a chromatogram on unirradiated polytetrafluoroethylene.

The polytetrafluoroethylene layers have been used in studies of the separations of numerous substances including amides, amino acids, cacodylic acid and sodium cacodylate (Sect. 2.3.c.f), aromatic and polynuclear hydrocarbons (Sect. 2.3.c.b), inorganic ions (Sect. 2.3.c.e), and selected organic carcinogens (Sect. 2.3.c.c). Manuscripts on the use of the layers for the separations of metal ions[20] and of hydrocarbons[21] are being submitted for publication.

2.3.b Temperature Effects

R. L. Coleman[22] J. A. Dean[6]
W. D. Shults

A program was initiated to establish – at least semiquantitatively – the influence of temperature in thin-layer chromatography. We hope to study sub-ambient-, superambient-, and programmed-temperature modes of operation. Equipment for these studies is being fabricated. Meanwhile, factorial experiments are being conducted to determine statistical limits about TLC data. The Joyce-Loebl TLC Scanner is being used for these latter experiments, and it will be used in subsequent experiments.

2.3.c Applications

2.3.c.a. Amanita Toxins (Helen P. Raaen). – The improvement[23] made in an existing TLC separation of amanita toxins is described in a note published in the *Journal of Chromatography.*[24]

2.3.c.b Hydrocarbons (Helen P. Raaen). – The first experiments to separate organic compounds on polytetrafluoroethylene layers were made. The separation selected for study was that of aromatic and heterocyclic hydrocarbons based on their differences in solubility in a single solvent (*n*-propanol) in a system requiring no corrosive agent or wetting agent. The hydrocarbon separation is of particular interest, because it is directly applicable to the work on cigarette-smoke condensates and environmental contaminants. The following hydrocarbons were used: *o*-hydroxydiphenyl, naphthalene, fluorene, phenanthrene, pyrene, chrysene, anthracene, fluoranthene, benzo-e-pyrene (1,2-benzopyrene), and benzo-ghi-perylene (1,12-benzoperylene). Layers were prepared from 100% polytetrafluorethylene and from polytetrafluoroethylene-polyamide mixtures. The organic liquids used to slurry the adsorbents included acetone, *n*-propanol, methylisobutyl ketone, perfluorodiethylpropylamine, and perfluoro-2-butyltetrahydrofuran (the 3M Co.'s product FC-75). Of these, the fluorocarbons and *n*-propanol give the best resolutions. On a 100%-polytetrafluoroethylene layer, the fluorocarbons show considerable promise as developers and have the advantage of being chemically inert. With *n*-propanol as a developer, the polytetrafluoroethylene-polyamide mixed layers give best resolution. Also when used in a horizontal-gradient arrangement and spotted repetitively at close intervals with the test solution, a polytetrafluoroethylene-polyamide gradient layer gives distinctive "curves" for the migrating hydrocarbons. It is possible to use such curves as a means, more exact than R_f values, to identify resolved components.

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[20] Helen P. Raaen, "Chromatography of Metal Ions on Thin Layers of Polytetrafluoroethylene with Di-(2-ethylhexyl) Orthophosphoric Acid," to be submitted for publication in the *Journal of Chromatography.*

[21] Helen P. Raaen, "Chromatography and 77° Luminescence of Some Hydrocarbons on Thin Layers of Aviamide-6–Fluoroglide 200," to be submitted for publication in the *Journal of Chromatography.*

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

[23] Helen P. Raaen, "Amanita Toxins," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968,* ORNL-4343, p. 14.

[24] Helen P. Raaen, "Improved Thin-Layer Chromatographic Separation of Amanita Toxins on Silica Gel G Chromatoplates," *J. Chromatog.* 38, 403 (1968).

A new form of polyamide, Aviamide-6, which is a microcrystalline nylon being developed by the FMC Corporation, appears to be superior to Woelm "polyamide for TLC." Aviamide-6 is available as both a powder and a paste (42% solids in water); the powder performs best. A mixture of the ten hydrocarbons was resolved on Aviamide-6 (powder)–polytetrafluoroethylene (4:1) layers developed with *n*-propanol. The distinctive fluorescent and phosphorescent properties of the resolved hydrocarbons can be the basis of their exact identification and possibly quantitation. Color photographs of the chromatograms were made that show the fluorescence at room and liquid-nitrogen (77°K) temperatures under 254- and 366-nm ultraviolet light and the phosphorescence at 77°K after excitation with 254- and 366-nm ultraviolet light. It appears worthwhile to extend the study of hydrocarbons in this TLC system because of the specificity and sensitivity it provides.

The excellent separation of hydrocarbons on mixed polytetrafluoroethylene-polyamide layers suggested that it might be possible to separate hydrocarbons on 100% polytetrafluoroethylene layers if amides are used as developers. Attempts were made to separate the ten hydrocarbons on polytetrafluoroethylene layers that contained enough wetting agent to cause the amide developer to migrate. The developers evaluated were formamide, *N,N*-dimethylformamide, *N*-methylacetamide, and *n*-propanol–dimethylformamide (1:1). Some separation occurred, the best being with *N,N*-dimethylformamide, but the resolution was very much poorer than that achieved with the polytetrafluoroethylene layers.

It was of interest to determine whether the presence of HF in an organic solvent in which hydrocarbons migrate at the solvent front on 100%-polytetrafluoroethylene layers (*e.g.*, *n*-propanol) might inhibit the migration and result in some resolution. Therefore, chromatograms were run on layers of 100% polytetrafluoroethylene developed with *n*-propanol–48% HF (2:1 v/v) and with methylisobutyl ketone saturated with 48% HF. In each case, some resolution of the hydrocarbons occurred, but separations were not satisfactory.

The separation of hydrocarbons on 100%-polytetrafluoroethylene layers by development with FC-75 equilibrated with 48% HF was attempted. Some of the hydrocarbons migrated, but the resolution was not discrete.

2.3.c.c Organic Carcinogens (Helen P. Raaen, R. L. Coleman[22]). – Chemical carcinogenesis is of particular interest to biologists. For example, evidence exists that *N*-acetoxy-acetylaminofluorene (*N*-acetoxy-AAF) binds to DNA and RNA and thus alters the transfer of cellular information. The compound thereby acts as an initiating carcinogen, because it causes misreading of the genetic code, which misreading results in somatic mutation. A TLC method of detecting chemical carcinogens and impurities in them was studied with five organic compounds. This work is described in a technical memorandum,[25] the abstract from which follows:

"The purities of five organic compounds of interest in carcinogenesis were investigated by thin-layer chromatography. Four of the compounds are related to the carcinogen 2-acetylaminofluorene (AAF); they are: 2-aminofluorene, 2-acetamidofluorene, 2-diacetylaminofluorene, and *N*-acetoxy-2-acetylaminofluorene (*N*-acetoxy-AAF). The fifth compound was 6-chloro-9-(3′-diethylamino-2′-hydroxypropylamino)-2-methoxyacridine dihydrochloride, whose molecular structure is very similar to that of the nitrogen-mustard compound 6-chloro-9-(3′-ethyl,3′-β-chloroethylaminopropylamino)-2-methoxyacridine dihydrochloride, a carcinogen known as ICR-170.

"The compounds were chromatographed on Gelman ITLC-SA medium and on Teflon layers. The developer used in most of the work was carbon tetrachloride–ethyl alcohol (19.8:0.2 v/v). To detect resolved compounds, the chromatograms were observed under 254- and 366-nm ultraviolet light for fluorescence at room temperature and for both fluorescence and phosphorescence under liquid nitrogen (77°K).

"From each of the compounds except 2-acetamidofluorene, more than one substance was resolved. The discreteness and relative areas of the spots of the resolved substances indicate the presence of impurities in the other four compounds chromatographed. However, thin-layer chromatographic procedures more elaborate than that used, together with identification tests and quantitative measurements, are needed to determine whether the extraneous substances are impurities in the particular compound chromatographed or result either from its decomposition in the selected TLC system during chromatography or its inherent lability. Two-dimensional thin-layer chromatography of the *N*-acetoxy-AAF demonstrated that additional information is gained from the more elaborate technique."

2.3.c.d Steroids (Linda J. Crist,[26] Helen P. Raaen). – In the work done earlier[27] on the TLC separation of steroids on Silica Gel G layers, it was observed that androsterone, 11-dehydrocorticosterone, 5β-pregnane-3-α,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 ultra-

[25] Helen P. Raaen and R. L. Coleman, *Thin-Layer Chromatography of Five Organic Compounds of Interest in Carcinogenesis*, ORNL-TM-2637 (July 18, 1969).

[26] ORAU Student Trainee from the University of Arkansas, Jonesboro; summer, 1968.

[27] Linda J. Crist and Helen P. Raaen, "Steroids," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968*, ORNL-4343, p. 14.

violet light. It was believed that this characteristic of these steroids had not been reported before. Therefore, a note on this observation was published in the *Journal of Chromatography.*[28] Color photographs of the fluorescing and phosphorescing chromatograms are included with the note.

2.3.c.e Inorganic Ions (Helen P. Raaen). – With radioisotopes and autoradiography, a study was made of the TLC behavior of some 37 inorganic ions on layers of polytetrafluoroethylene developed with 0.5 *M* di-2-(ethylhexyl)orthophosphoric acid in methylisobutyl ketone that contained a surfactant and nitric acid. The results indicate that, with this TLC system, numerous separations are possible among the ions studied.

2.3.c.f Cacodylic Acid and Sodium Cacodylate (Helen P. Raaen, W. D. Shults). – Some exploratory work was done with cacodylic acid, $(CH_3)_2AsO_2H$, and sodium cacodylate, $(CH_3)_2AsO_2Na$, which are compounds of interest in ecology. Aqueous solutions of each (0.1 *M*) were chromatographed in several systems. Numerous developers and both Gelman ITLC-SA medium and 100% polytetrafluoroethylene layers were used. The most promising system was a 100%-polytetrafluoroethylene layer developed first with *n*-hexane-acetone (5:1 v/v) and then with methanol. Both cacodylic acid and sodium cacodylate migrate with the solvent front. After the development, both these compounds fluoresce blue-violet on the layer at room temperature when viewed under either 254- or 366-nm ultraviolet light, thus providing a sensitive means of detection. The fluorescence under 366-nm ultraviolet light is observed on the polytetrafluoroethylene layer but not on the Gelman ITLC-SA medium. With water as a developer, neither compound migrates. By adding some water to the developer, it may be possible to resolve the two compounds.

2.4 ELECTROANALYTICAL CHEMISTRY

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

Helen P. Raaen

The polarographic studies with the Teflon dropping-mercury electrode (D.M.E.) were completed. The results of these studies are presented in two published articles and one to be published; summaries from the manuscripts were given earlier.[29]

2.4.a.a Evaluation of Vertical-Orifice Rapid Teflon D.M.E.'s for Obtaining Fundamental Polarographic Data. – An article on the evaluation of vertical-orifice Teflon D.M.E.'s was published in *Chemical Instrumentation.*[30]

2.4.a.b Polarography of Sulfur Dioxide (Sulfite) in Aqueous Hydrofluoric Acid. – An article on the polarography of sulfur dioxide (sulfite) in aqueous hydrofluoric acid was published in *Analytica Chimica Acta.*[31]

2.4.a.c Rapid Polarography of Uranium in 1 *M* Hydrofluoric Acid with a Vertical-Orifice Teflon Dropping-Mercury Electrode. – An article that describes the rapid polarography of uranium in 1 *M* HF with a vertical-orifice Teflon D.M.E. is in press.[32]

2.4.b Voltammetry and Coulometry with the "Conducting Glass" Electrode

J. R. Stokely Beverly A. Brown[33]
W. D. Shults

Exploratory studies of the electrochemical properties and possible uses of the "conducting glass" electrode (C.G.E.)[34] were initiated. "Conducting glass" designates a glass surface onto which a semiconducting layer of stannic oxide–antimony oxide has been fired. The oxide film has a low specific resistance. The C.G.E. is reported[34] to have a high oxygen overvoltage. We think that this electrode may be applicable in our studies of the transuranium elements and in organic analysis.

The electrode design being studied first is a cylindrical cell coated on the inside with stannic oxide(97)–

[28]Linda J. Crist and Helen P. Raaen, "Fluorescence and Phosphorescence Response of Steroids to Sulfuric Acid in Thin-Layer Chromatography at 77°K," *J. Chromatog.* 39, 515 (1969).

[29]Helen P. Raaen, "Polarographic Studies with the Teflon Dropping-Mercury Electrode (D.M.E.)," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968,* ORNL-4343, p. 22.

[30]Helen P. Raaen, "Vertical-Orifice Dropping Mercury Electrode of Polytetrafluoroethylene for Rapid Polarography in Glass-Corroding Media," *Chem. Instrum.* 1, 287 (1969).

[31]Helen P. Raaen, "Polarography of Sulfur Dioxide (Sulfite) in Aqueous Hydrofluoric Acid," *Anal. Chim. Acta* 44, 205 (1969).

[32]Helen P. Raaen, "Rapid Polarography of Uranium in 1 *M* Hydrofluoric Acid with a Vertical-Orifice Polytetrafluoroethylene Dropping-Mercury Electrode," *Anal. Chim. Acta* 48, in press (1969).

[33]ORAU Student Trainee from Evangel College, Springfield, Mo., summer, 1969.

[34]R. C. Propst, Savannah River Laboratory, private communication to J. R. Stokely, Dec. 13, 1968.

antimony oxide(3). Electrical contact to the film is through a platinum wire sealed into the side of the cell. The dc resistance of the cell is low enough to be negligible for controlled-potential techniques in which small currents (<500 μA) are involved.

An electrode property of interest is the potential at which electrolytic oxidation of water begins. Linear potential scans on a 21-cm^2 C.G.E. showed that decomposition of water begins at +1.3 V vs S.C.E. in neutral salt (KNO_3) solution and at +1.7 V in 0.5 *M* H_2SO_4. In 10^{-3} *M* NaOH, decomposition begins at +0.9 V vs S.C.E. The negative potential limit for the electrode in 0.5 *M* H_2SO_4 is –0.4 V vs S.C.E. It is not now known whether the negative limit is imposed by hydrogen evolution or by reduction of the oxide film. The high positive potential limit, particularly with sulfuric acid, will permit studies in aqueous media that have not been possible with other electrodes.

In exploratory work, a number of organic compounds were studied voltammetrically to determine the response of the electrode to various organic functional groups. Voltage scans from 0 to +1.7 V vs S.C.E. were made on 10^{-3} *M* solutions of the compounds in 0.1 *M* KNO_3. No significant response was observed for azobenzene, glycine, benzoic acid, nitrobenzene, benzene, acetone, and acetaldehyde. For benzidine hydrochloride, a nearly reversible peak was observed at +0.4 V vs S.C.E. in an anodic scan and +0.2 V vs S.C.E. in a cathodic scan. Also for aniline, peaks were observed that appear to be due to adsorption on the electrode. Further work is planned on the use of the electrode for organic analysis.

Exploratory coulometric studies were made with the C.G.E. to determine its possible uses in transuranium-element chemistry. These studies showed that the electrode can be used in the potential region –0.4 to +1.7 V vs S.C.E. with very little contribution from the continuous faradaic current. Several possible uses for the electrode were demonstrated. One is to determine Pu(IV) and U(VI) in the same solution with the same electrode. Conventionally, Pu(IV) is determined with a platinum electrode, and U(VI) with a mercury electrode. With the C.G.E., Pu(IV) and U(VI) can be determined by coulometric reduction at +0.30 V and –0.32 V vs S.C.E. respectively. Another possibility is to determine Bk(III). Experiments with Ce(III) as a stand-in for Bk(III) have shown that it should be possible to coulometrically determine berkelium by either oxidation or reduction. Work is in progress to develop a way to determine berkelium with the C.G.E.

We hope to explore other practical applications of this electrode.

2.4.c Electrochemistry of Thin-Layers of Solution

J. R. Stokely W. D. Shults

An investigation of thin-layer electrochemical techniques[35,36] has been initiated to assess their real usefulness. Our preliminary studies were made with the sandwich thin-layer cell of the type described by Yildiz, Kissinger, and Reilley[37] except that a C.G.E. (Sect. 2.4.b) was used instead of a platinum or gold electrode. These studies showed that the problem of edge effect – diffusion of electroactive species into and out of the thin-layer – must be solved if the cell is to be used easily for analysis. A cell of new design is now being fabricated in an attempt to minimize this edge effect.

2.4.d Programmed-Current Coulometry

J. E. McCracken[38] J. C. Guyon[39]
W. D. Shults

We have initiated a study in programmed-current coulometry. This study will comprise part of the doctoral research for J. E. McCracken. The first objective has been to develop a programmed-current coulometric titrator for quantitative analysis that has accuracy and versatility superior to those of conventional constant-current coulometric instruments. Three models have been built and tested; the performance of the latest, "Coulotrator III," is very satisfactory. We are now using various selected electrochemical systems to determine the capabilities of the instrument. The circuitry development was done largely at the University of Missouri, Columbia campus; subsequent work will be done within the Division laboratories.

2.4.e Determination of Plutonium by Controlled-Current Coulometry

J. R. Stokely W. D. Shults

Work was completed to determine plutonium by controlled-current coulometry, and the method was

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[35] C. R. Christensen and F. C. Anson, "Chronopotentiometry in Thin-Layers of Solution," *Anal. Chem.* 35, 205 (1963).

[36] C. N. Reilley, "Electrochemistry Using Thin-Layer Cells," *Rev. Pure Appl. Chem.* 18, 137 (1968).

[37] A. Yildiz, P. T. Kissinger, and C. N. Reilley, "Evaluation of an Improved Thin-Layer Electrode," *Anal. Chem.* 40, 1018 (1968).

[38] ORAU Predoctoral Fellow from the Graduate School of Chemistry, University of Missouri, Columbia.

[39] Professor of Chemistry and Chairman of the Department of Chemistry, University of Missouri, Columbia.

published.[40] Compared with the controlled-potential coulometric method, which is used extensively to determine plutonium, the controlled-current coulometric method gives similar precision and accuracy, uses much of the same instrumentation, can be faster for large numbers of samples, and is insensitive to relatively large amounts of iron(II) or (III) and sulfate in the sample.

2.4.f Elimination of Iron Interference in the Controlled-Potential Coulometric Method for Plutonium

J. R. Stokely W. D. Shults

In the presence of sulfate, Fe(II) and (III) interfere seriously in the controlled-potential coulometric determination of plutonium. A method was devised to eliminate this interference. The method is based on complexation of Fe(II) with the disodium salt of bathophenanthroline disulfonic acid. The complexation shifts the potential of the Fe(II)-Fe(III) couple from +0.47 to +0.85 V vs S.C.E. without affecting the potential of the Pu(III)-Pu(IV) couple (+0.47 V vs S.C.E.). The feasibility of this method has been demonstrated, but final testing is not completed.

2.5 RESEARCH AND SUPPORT ACTIVITIES FOR THE TRANSURANIUM RESEARCH LABORATORY

2.5.a Potentiometric Determination of the Formal Potential of the Bk(III)/Bk(IV) Couple in Sulfuric Acid

J. R. Stokely R. D. Baybarz[41]
W. D. Shults

Microgram quantities of berkelium have been available recently. Since all previous estimates of the formal potential of the Bk(III)/Bk(IV) couple have been obtained with submicrogram quantities of berkelium and by indirect techniques, we have measured the potential by direct potentiometry in aqueous solutions of sulfuric acid. Potentiostatic electrolysis and coulometry were used to prepare solutions that contained Bk(III) and Bk(IV) in known ratios. The potentials of these solutions were measured with a platinum electrode. The formal reduction potentials found for the couple in 0.5, 1.1, and 2.0 *F* H_2SO_4 are +1.38, +1.37, and +1.36 V vs S.C.E., respectively, with an uncertainty of ±20 mV in the measurement of the potentials. Radiolytic reduction of Bk(IV) to Bk(III) was observed; from potential measurements, the rate is estimated to be 1%/min. The results of radiochemical and absolute coulometric assays of berkelium agree well; this agreement indicates that berkelium can be determined by potentiostatic coulometry.

An article on this work was published.[42]

2.5.b Absorption Spectra of Berkelium(IV)

R. D. Baybarz[41] J. R. Stokely
J. R. Peterson[43]

The absorption spectra were obtained of Bk(IV) in sulfuric and perchloric acid media. To avoid adding chemical oxidants that might obscure the spectra, the Bk(IV) was prepared by electrolytic oxidation with a platinum electrode. About 400 μg of berkelium was available for this work. A micro coulometric cell for controlled-potential electrolysis was fabricated to accommodate 300 to 400 μl of a solution that contained the berkelium.

Electrolytic oxidation of Bk(III) at a controlled-potential of +1.7 V vs S.C.E. gave an intense yellow-green solution. Potentiostatic reduction at +0.9 V vs S.C.E. caused the color to disappear gradually as the electrolysis proceeded to completion. The solution of Bk(IV) exhibited intense absorption in the ultraviolet region, the absorption maximum being at 285 nm and the molar extinction coefficient being 1900. This intense ultraviolet absorption is due to electron charge transfer from water to Bk(IV). In the wavelength region 450 to 700 nm, absorption due to Bk(IV) was small. Over a period of a few hours, the ultraviolet absorbance decreased linearly because of radiolytic reduction of Bk(IV) to Bk(III). After complete radiolytic or electrolytic reduction of Bk(IV), three new absorption peaks of Bk(III) appear in the region 222 to 250 nm.

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[40] J. R. Stokely, Jr., and W. D. Shults, "Determination of Plutonium by Controlled-Current Coulometry." *Anal. Chim. Acta* 45, 528 (1969).

[41] Chemical Technology Division.

[42] J. R. Stokely, Jr., R. D. Baybarz, and W. D. Shults, "Direct Potentiometric Determination of the Formal Potential of the Bk(III)-Bk(IV) Couple in Sulfuric Acid," *Inorg. Nucl. Chem. Letters*, in press (1969).

[43] Assistant Professor of Chemistry, University of Tennessee, Knoxville.

2.5.c Investigation of the Divalent Oxidation State of Mendelevium

J. R. Stokely R. J. Silva[44]

Recent studies[45] have shown that Md(III) can be reduced to Md(II), which is stable in solution, and that the reduction potential of the Md(II)/Md(III) couple is near –0.2 V vs N.H.E. Additional chemical experiments were performed to confirm the existence of Md(II) and to obtain a better estimate of the potential of the Md(II)/Md(III) couple.

For chemical experiments, ^{256}Md(III) was produced by bombardment of ^{253}Es with alpha particles from the Oak Ridge Isochronous Cyclotron (ORIC). Experiments consisted in reducing Md(III) to Md(II) with various chemical reductants and separating the Md(II) from Md(III) and ^{256}Fm(III) on small extraction chromatographic columns of Kel-F powder coated with di-(2-ethylhexyl)orthophosphoric acid. When 0.02 *M* Cr(II) or V(II) solution was the reductant, at least part of the Md(III) was reduced. With hydrazine, Sn(II), and hydroxylamine, very little or no reduction occurred. The most effective reductant was Cr(II). Fission counting of the ^{256}Md(II) fraction showed the growth of the ^{256}Fm daughter of ^{256}Md and very efficient initial separation of ^{256}Md from ^{256}Fm. Decay data showed that from 2- to 3-μg/cm^2 ^{253}Es targets about 10^4 atoms of ^{256}Md were made in each bombardment; the maximum count rates were of the order of 50 fission counts per minute. The results of these experiments confirm the existence of Md(II) and suggest that the potential of the Md(II)/Md(III) couple is between –0.3 and 0.0 V vs N.H.E. The experiment is being refined to obtain a better value for the potential. This work has been reported.[46]

2.5.d Carbon-Hydrogen-Nitrogen Analysis with the Perkin-Elmer Elemental Analyzer

J. R. Stokely

The glove-boxed Perkin-Elmer Elemental Analyzer (model 240) has been used to determine carbon, hydrogen, and nitrogen in a number of organic and organometallic samples for members of the Chemistry and Analytical Chemistry Divisions.

2.6 GeMSAEC FAST ANALYZER SYSTEM FOR NONCLINICAL APPLICATIONS

W. L. Maddox[47] W. D. Shults

Construction is nearing completion of a GeMSAEC Fast Analyzer system (Sect. 4.2.f) to be used to develop nonclinical applications of the system. Our unit will be similar to the clinical analyzer except that it will incorporate Heath light-source and monochromator modules.

2.7 WATER ANALYSES

2.7.a Deaeration Studies

W. J. Ross[48] W. D. Shults

For the Office of Saline Water (OSW), we have continued to provide technical support for ORNL performance tests of various desalination facilities. As before,[49] this support consists primarily in determining dissolved oxygen, carbon dioxide, and occasionally corrosion products in sea water at the test site. Tests at the Wrightsville Beach Facility enabled us to evaluate the performances of several oxygen probes and of the Orion sulfide-selective electrode.[50] The performance of the electrode was satisfactory, and it is now used routinely at several OSW facilities. Further tests of this nature are scheduled.

2.7.b HFIR Coolant Water

H. W. Wright W. D. Shults

As part of studies of the corrosion of the HFIR coolant system, we performed a number of analyses for dissolved oxygen, hydrogen peroxide, nitrate, and nitrite by standard ASTM methods.

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[44] Chemistry Division.

[45] E. K. Hulet, R. W. Lougheed, J. D. Brady, R. E. Stone, and M. S. Coops, "Mendelevium: Divalency and Other Chemical Properties," *Science* 158, 486 (1967).

[46] R. J. Silva, R. L. Hahn, M. L. Mallory, C. E. Bemis, P. F. Dittner, O. L. Keller, J. R. Stokely, and K. S. Toth, "Nuclear Chemistry with the Oak Ridge Isochronous Cyclotron," presented at the International Conference on the Use of Cyclotrons in Chemistry, Metallurgy, and Biology, Oxford, England, Sept. 22–23, 1969.

[47] Analytical Instrumentation Group.

[48] On leave of absence to Instituto Venezolano de Investigaciones Cientificas, Caracas, Venezuela.

[49] W. J. Ross, "Analysis of Water Systems. Deaeration Systems," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968,* ORNL-4343, p. 17.

[50] W. D. Shults, "Evaluation of the Performance of Ion-Selective Electrodes," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968,* ORNL-4343, p. 23.

2.7.c Rapid Fluorometric Methods for Determination of Phosphate

J. C. Guyon[39] W. D. Shults

The rapid fluorometric methods for determination of phosphate in waters were reported previously[51] and have now been published.[52]

2.8 RESEARCH AND SUPPORT ACTIVITIES FOR ECOLOGY

2.8.a Eutrophic Water Systems

H. W. Wright W. J. Ross[48]
W. D. Shults

In the continuing cooperative work with the Ecology Section of the Health Physics Division, ~1500 samples of water collected from controlled watersheds were analyzed for orthophosphate by the AutoAnalyzer methods mentioned previously.[49] The demand for nitrate determination has decreased. We are currently exploring means to distinguish and determine organic and inorganic species that contain phosphorus or sulfur in samples of ecological interest.

2.8.b Pyrolysis–Gas Chromatographic System for Determining Phosphorus, Sulfur, Nitrogen, and Carbon

A. D. Horton[14] H. W. Wright

Elemental gas chromatographic analysis using element-specific detectors is being done on a cooperative arrangement between the Analytical Chemistry Division and the Ecology Section of the Health Physics Division. A Micro Tek model 220 gas chromatograph with dual flame-photometric detectors (FPD),[53] a Coulson electrolytic-conductivity detector (CECD)[54] and a flame-ionization detector (FID)[53] has been set up in the laboratory of the Ecology Section, and each of these detectors has been evaluated. Exploratory analyses have been made, and a program is under way to establish the accuracy of the analyses and to develop analytical ecological standards by correlating data obtained from these detectors with data obtained by conventional methods.

The gas chromatographic procedure is as follows. Dried organic specimens ground to <40-mesh size are pyrolyzed in helium at 490°C. The pyrolyzate passes into a 3% OV-1 silicone column, and the effluent from the column passes into the desired detector. The dual detector response for phosphorus and for sulfur is compared with a standard calibration curve prepared by injecting parathion into the column. The detector response for sulfur is proportional to the square of the sulfur concentration; hence, the calibration data are plotted on log-log paper. The curve is linear from 2 to 30 ng of sulfur. The phosphorus response curve is linear from 20 to 200 ng of phosphorus. The nitrogen response curve of the CECD is linear from 30 to 200 ng of nitrogen. The FID detector response to carbon is linear; the manufacturer states that it is linear over the full range of the electrometer.

The phosphorus content of the materials thus far analyzed is lower than the sulfur content. No nitrogen or carbon determination has been made. The limiting parameters in the simultaneous determination of phosphorus and sulfur are the limited linear range of the sulfur calibration curve and the fact that the sulfur content usually exceeds that of phosphorus by several hundred percent. Therefore, the dry sample weights are usually limited to <0.150 mg, and phosphorus response must be read on the portion of the calibration curve from 0 to 20 ng.

Table 2.2 gives preliminary results of analyses of leaf litter, grass, and insects.

Table 2.2. Phosphorus and Sulfur in Ecological Specimens

Specimen	Phosphorus (ppm)	Sulfur (ppm)
Leaf litter (Walker Branch Water Shed)	3.6[a]	380
Kentucky 31 fescue:		
Live	2.3	218
Standing dead	5.4	304
Grasshopper:		
Melanoplus	4.3	550
Conocephalus	5.3	520

[a]1000 ppm by wet-ashing and colorimetric analysis.

[51] J. C. Guyon and W. D. Shults, "Fluorometric Determination of Phosphate," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968*, ORNL-4343, p. 18.

[52] J. C. Guyon and W. D. Shults, "Rapid Phosphate Determination by Fluorimetry," *J. Am. Water Works Assoc.* 61, 403 (1969).

[53] S. S. Brody and J. E. Chaney, "The Application of a Specific Detector for Phosphorus and for Sulfur Compounds – Sensitive to Subnanogram Quantities," *J. Gas Chromatog.* 4, 42 (1966).

[54] D. M. Coulson, "Selective Detection of Nitrogen Compounds in Electrolytic Conductivity–Gas Chromatography," *Ibid.* 285.

2.9 RESEARCH AND SUPPORT ACTIVITIES FOR HOUSING AND URBAN DEVELOPMENT

H. W. Wright W. J. Ross[48]
A. D. Horton[14] W. D. Shults

The Housing and Urban Development Group at ORNL has been studying means to treat wastes of various types. Among these studies was a wet-oxidation treatment for paper products. We assisted in these studies by performing chemical analyses on the solutions, residues, and gaseous phases generated by the wet-oxidation treatment. Standard (ASTM) procedures were used for chemical- and biological-oxygen-demand (COD and BOD) analyses. Gas chromatography was used to determine the nature of the gaseous products. These latter samples were analyzed semiquantitatively on a 16-ft by $\frac{1}{8}$-in. stainless steel column that contained equal amounts of Porapaks Q and R. It was found that the concentration of hydrocarbons (mostly *n*-paraffins) increased as the concentration of CO_2 increased. When the concentration of CO_2 was 2.8 vol % (minimum), the total hydrocarbon concentration was 335 vol ppm; when CO_2 was 58.8 vol % (maximum), total hydrocarbons were 8.5 vol %. A maximum of nine different hydrocarbons were found in the head gas.

Means for in-depth characterization of samples of these types are being considered.

2.10 DECENTRALIZED PREPARATION OF ABSTRACTS FOR *NUCLEAR SCIENCE ABSTRACTS*

C. A. Horton

The decentralized preparation of abstracts for *Nuclear Science Abstracts (NSA)*, along with indexes for *NSA* and for the Euratom Key Word System, has been compared with centralized preparation of the same material by the regular *NSA* staff. Unclassified ORNL reports in all fields were abstracted and indexed by this minimally trained staff member.

The work of the first six months was evaluated;[55] a summary of the evaluation follows. Items are prepared about two weeks sooner on-site at ORNL than at DTIE. The average time required to complete an abstract and indexes for a short report is ~60 min compared with 49 min for all types of items handled by the *NSA* staff. Parts of long progress reports, however, required 2.5 hr per part at ORNL. The ORNL cost is about twice the cost at *NSA*, mainly because of overhead charges. The ORNL-generated index contains ~50% of the total index terms used by the *NSA* staff for both *NSA* subject entries and Euratom key words. The *NSA*-index modifier file-words generated by ORNL and the *NSA* staff do not agree well (<5%); also, only ~15% of the Cosati[56] categories agree. The author considers most of these findings to be unsatisfactory. Indexing consistency does not correlate with the ORNL indexer's knowledge of the subject matter, but it seemed that errors in judgment on inclusion in *NSA* do. The descriptive cataloging and abstracting of ORNL reports show only minor faults except for excessive length for the latter. Comments from the *NSA* staff on indexing were returned to ORNL too late to be of value in advancing the proficiency of the ORNL indexer; moreover, the written guides provided for the work are inadequate.

It is recommended that a second indexer, preferably with training in other topics, be available, to maintain timeliness should decentralized abstracting become permanent. The possibility of on-site training of an extra or alternate indexer deserves study during the remainder of this project. Some alternate projects have been suggested for future work on information-science problems during the next fiscal year.

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[55] C. A. Horton, *ORNL Decentralized NUCLEAR SCIENCE ABSTRACTS Preparation. First Semiannual Report, Period Ending July 1, 1969,* ORNL-TM-2653, in press, 1969.

[56] Committee on Scientific and Technical Information.

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 J. M. Dale A. S. Meyer

The first sample of the ^{233}U fuel (FP 17-5) was analyzed for oxide by the hydrofluorination procedure[1] and was found to contain 61 ppm, which value does not differ significantly from that found during the operation of the reactor with ^{235}U (54 ± 8 ppm). A satisfactory recovery of oxide from an SnO_2 standard indicated that the apparatus was functioning reliably.

Two samples of $LiF\text{-}BeF_2$ submitted by E. L. Compere[2] were analyzed for oxide. They were taken from a larger quantity of salt that had been exposed in the molten state to the atmosphere of a helium-filled dry box for ~72 hr. The oxide contents of the samples of the surface and bulk salt were 113 and 92 ppm respectively.

During the last few analyses, a potential source of failure of the oxide apparatus was noted. Increasingly high pressures are required to maintain the flow of gas through the system during the hydrofluorination. Apparently, this increase occurs across the NaF trap used to separate HF from water in the effluent gas. It is assumed that during the repeated absorption and regeneration steps the absorbent particles degrade to form a more tightly packed bed, particularly at the trap entrance. As these more closely packed particles swell during HF absorption, the flow is restricted. Preparations are now under way to replace the absorbent component of the hydrofluorination system. If this technique is applied to the in-line analysis of MSR salt streams, it will be necessary to provide remotely replaceable traps.

[1] R. F. Apple, A. S. Meyer, and J. M. Dale, "Determination of Oxide in MSRE Salts by Hydrofluorination," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968,* ORNL-4343, p. 23.

[2] Reactor Chemistry Division.

3.1.b Analysis of Blanket Gas from $NaBF_4$ Tests

A. S. Meyer R. F. Apple

The off-gas of the circulating fluoroborate loop (BF_3 in He) has been found to be contaminated with a dense corrosive liquid and with small amounts of a white solid. Both these materials were deposited, at least in part, at points remote from the pump bowl and could introduce maintenance problems when $NaBF_4$ is used as an MSR coolant salt.

From its physical properties and F/B ratios, the liquid has been identified tentatively as HBF_3OH that is contaminated with corrosion products and equilibrium quantities of other BF_3 hydrates. The solid was found to be primarily $NaBF_4$ and to contain iron and titanium in concentrations of ~600 and ~100 ppm respectively.

Since the liquid is no longer being collected in the off-gas system, it will not be possible to characterize it unambiguously. In view of the above information, we plan to assume that it consists of BF_3 hydrates, to generate such hydrates within a laboratory system, to study their transfer, and to develop methods for removing them from off-gas streams. A gas train that consists of fluorothene traps connected by Teflon tubing has been assembled for use in these tests. The first trap was charged with water and sparged with an equimolar mixture of BF_3 and helium. When no further increase in the volume of the solution was noted and BF_3 passed freely through the system, the delivery tube was withdrawn to a point just above the solution, and the effluent gas was passed through two traps cooled in an acetone–Dry Ice bath. The bath temperature is not low enough to condense BF_3; liquid was observed to be transferred at a significant rate. After a 40-hr sparge at 100 cc/min, ~0.5 cc was collected. The trapping was obviously of low efficiency, since comparable quantities were collected in both traps. The traps have now been packed with Teflon Tee Six (Analabs, Hamden, Conn.), and the experiment will be repeated to try to obtain valid values for the transfer rates. When reliable values have been established, various adsorbents will be interposed between the source and collection traps. We plan

first to test molecular sieves because of their high capacity and convenience in handling. A large-pore (13X) sieve will be tested as a total adsorbent of the hydrate. A 3A sieve also will be evaluated to determine whether these compounds can be dehydrated.

3.1.c Removal of Oxide from $NaBF_4$

R. F. Apple A. S. Meyer

Because studies of the compatibility of $NaBF_4$ coolant with structural materials indicate some correlation of corrosion rates with oxide and water contamination, we have undertaken a program with the Metallurgy Division to study ways to purify the coolant. Since BF_3 is used as a blanket gas for the salt, an attempt was made to remove oxide as a hydrate of BF_3 by passing a He-BF_3 gas mixture through the $NaBF_4$. When the water in the effluent gas was titrated with Karl Fischer reagent, it was found that the $NaBF_4$ actually removed traces of moisture from the BF_3. If a He-HF gas mixture is passed through the salt at 950°F, oxide can be removed from the melt according to the hydrofluorination reaction

$$2HF + O^{2-} \rightleftharpoons H_2O + 2F^- .$$

Optimum conditions for oxide removal by this reaction are now being established.

3.1.d Determination of Total Reducing Power of MSRE Salts

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

Measurement of the redox condition of the ^{233}U fuel is currently the most important analytical development problem remaining for the MSRE. The oxidation state of the fuel is believed to influence corrosion rates and to affect the distribution of certain fission products within the reactor system; it is readily adjustable via the addition of beryllium metal.

A sealed-tube dissolution technique was applied to the determination of the total reducing power of radioactive MSRE fuel. An apparatus (Fig. 3.1) was required that provides a way to evacuate and seal the dissolution tubes and is compact enough to be transferred between hot cells. Fragments of salt (250 mg) are placed in a dissolution tube. The tube is fitted with a "breakseal" and contains a thin-walled ampul that holds ~6 ml of evacuated 6 *M* HCl-H_3BO_3 dissolver solution (Fig. 3.2). The neck of the tube is then passed through the platinum-wound furnace and connected to the manifold via a standard-taper joint. The dissolution tube is evacuated with the cryogenic sorption pump (13X molecular sieve at liquid-nitrogen temperature) and sealed off with the furnace. The ampul is broken by shaking, and the dissolution tube is sealed in a steel bomb and heated to 250°C under a suitable overpressure for 16 hr. When the dissolution is completed, the breakseal end of the tube is connected to the left-hand port of the manifold, and a receiver tube is connected through the furnace and is evacuated. After the dissolver solution is frozen in liquid nitrogen, the breakseal is broken, and the evolved hydrogen is distributed through the system. The receiver tube is also cooled in liquid nitrogen to minimize thermal diffusion effects, and a standard addition of helium (1 cc at 38 mm pressure) is made through the stopcock at the top of the apparatus. After a 15-min equilibration, the

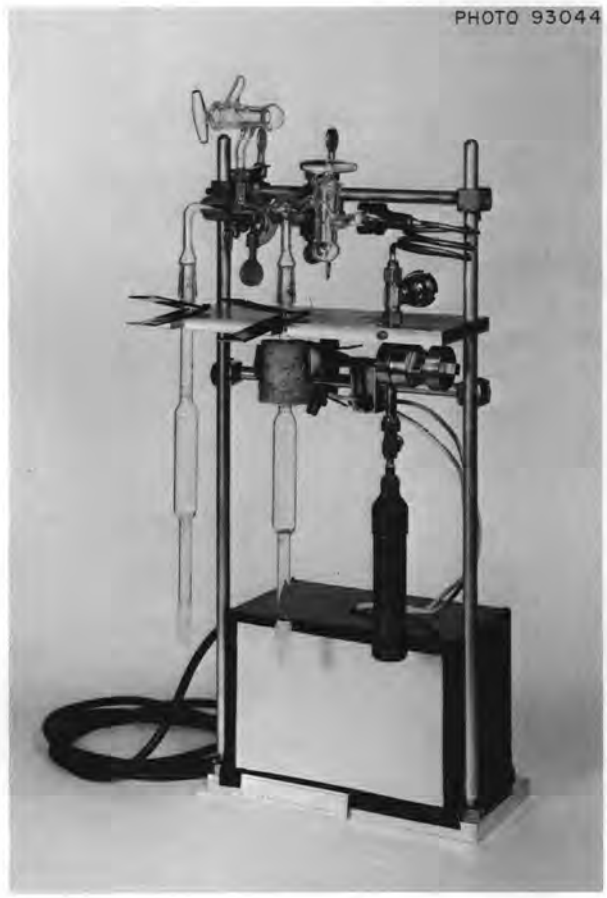

Fig. 3.1. Hot-Cell Apparatus for Use in Determining Total Reducing Power of MSRE Salts.

receiver tube is sealed off and removed from the hot cell for mass spectrographic determination of the H_2/He ratio. The solution is transferred to an empty dissolver tube, which is sealed off and carried through the entire procedure to determine the amount of hydrogen generated by radiolysis.

A modification of the dissolution technique was necessary for hot-cell operations. The conventional technique of adding a weighed amount of Dry Ice to the bomb provides a suitable overpressure for laboratory dissolutions, but an undetermined quantity of the Dry Ice evaporates during the longer period required for remote sealing. Simulated tests indicate that ~50% of the dissolution tubes would be broken during hot-cell dissolutions. A comparison of the vapor-pressure curves of the common stable organic reagents with extrapolated values of the pressures reported for 6 *M* HCl[3] revealed that the pressure of the dissolver solution was most closely paralleled by that of methanol. When 10 ml of methanol was sealed into the bombs, no breakage occurred.

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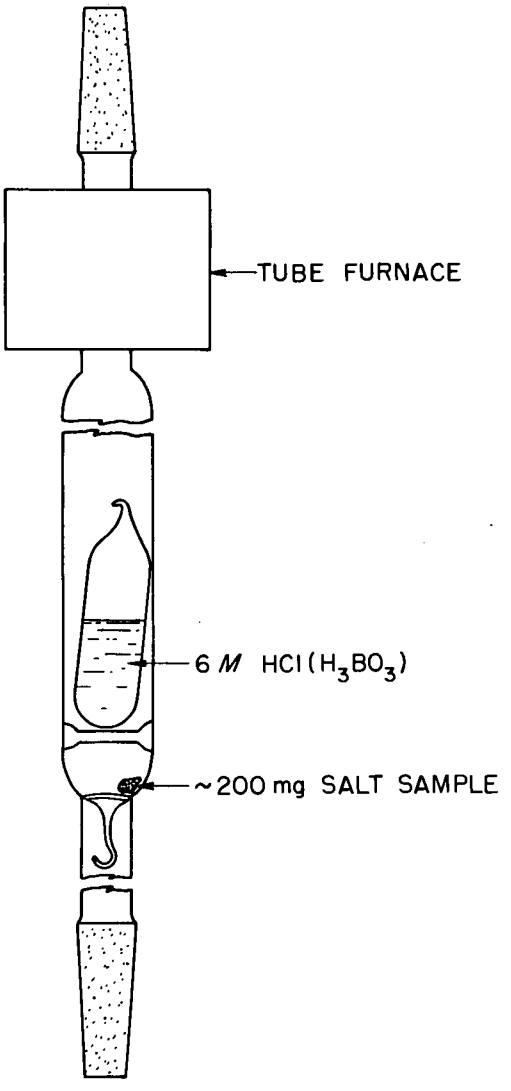

Fig. 3.2. Dissolution Bulb for Use in Determining Total Reducing Power of MSRE Salts.

The method was originally developed to determine free metals in the reprocessed solvent salt. When the apparatus was completed, the addition of uranium to the fuel had already been started, and an attempt was made to apply the method to the determination of U(III). Table 3.1 summarizes the results of the analyses of two fuel samples.

During the period between samplings, sufficient beryllium metal was added to the reactor fuel to increase the yield of hydrogen by 0.55 μmole/g. Also, some $LiUF_5$ fuel concentrate was added. The calculated increase in the hydrogen yields was not obtained. It is evident that the method in its present form is not suitable for the determination of U(III) in these samples. The hydrogen is derived predominantly from metal dispersed in the samples. Because a fragment that represents only ~2% of the sample is taken, any inhomogeneities in the distribution of the metals in the frozen salt would yield nonrepresentative samples. Also, the weighing and transfer of the salt fragments in the hot cell is

[3] Z. I. Simkhovich, "Relationship Between the Temperature and the Vapor Pressure of 20% Hydrochloric Acid," *J. Appl. Chem. USSR* 30, 1312 (1957).

Table 3.1. Total Reducing Power of Fuel Salt

	Sample FP-15-5	Sample FP-15-25
Uranium, ppm	4730	7340
Nickel, ppm	46	51
Iron, ppm	140	140
Chromium, ppm	42	50
Total H_2, μmoles/g	4.07	3.69
Blank corr., μmole/g	0.22	0.22
Nickel corr., μmole/g	0.78	0.87
Iron corr., μmoles/g	2.51	2.51
Chromium corr., μmole/g	0.11	0
U^{3+} equivalent	0.44	0.09
$U^{3+}/\Sigma U$, %	4.3	0.6

time-consuming, and this atmospheric exposure may introduce significant indeterminate oxidation errors.

The technique offers high sensitivity, and a specialized sampling approach will undoubtedly prove of value for reducing-power measurements of other types of MSRP samples. As time permits, studies will be resumed to determine its ultimate sensitivity and precision and its limitations as to sample size and types of salts that can be dissolved.

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

J. M. Dale J. E. Caton[4]

The application of the hydrogen-reduction method to determine U(III) in the original ^{235}U fuel and the development of a remote method to measure HF generated by the hydrogenation of the current ^{233}U fuel were described previously.[5] When the precision of the remote determination of HF was demonstrated to be adequate for the measurement of HF in the effluent from the hydrogenation of ^{233}U fuel, an apparatus was assembled to simulate the hydrogenation of radioactive salt samples. This apparatus consisted of a copper-block furnace fitted with a nickel liner of internal diameter equal to that of a sample ladle, appropriate sparging and exit tubes, and an addition port. The hydrogenator was heated with a precision furnace that maintained a temperature differential <5°C over the region occupied by the sample. Provision was also made to change the depth of the melts in order to determine whether equilibrium was closely approached during the hydrogen transpirations.

Before salt was added to the hydrogenator, pulses of HF in a stream of hydrogen were passed through the system to determine the time constant of the apparatus. A portion of the HF was detained in the apparatus. This delay is apparently caused by adsorption on the surface of the hydrogenator, since the effect becomes less pronounced when successive pulses are introduced. The period of delay is about 1 hr and is the result of chemisorption, since the retention period increases with temperature. The adsorption is stronger on nickel than on copper surfaces. By comparison of the shape of the HF pulses with those of an unadsorbed gas (argon), the quantities of HF adsorbed were estimated to be about 2 to 3 μmoles. Adsorption of such quantities of HF would not introduce a significant error in the results of the analysis of the ^{235}U fuels, but a complex correction would be required for the lower yields expected from the ^{233}U fuel. Although the adsorption of HF complicates batch analyses of the fuel, it would not interfere with in-line methods. Surfaces would become rapidly saturated, so that the steady-state concentration of HF in the effluent would correspond to its rate of production in the fuel.

Study of the method was discontinued temporarily, because the voltammetric method (Sect. 3.1.f) gives promise of more immediate results.

3.1.f Voltammetric Determination of U(IV)/U(III) Ratios in MSRE Fuel

J. M. Dale R. F. Apple

An apparatus was designed to make electrochemical measurements on samples of radioactive MSRE fuel. The objective is to measure the half-wave potential ($E_{1/2}$) of the voltammetric reduction U(IV) → U(III) vs the potential (E_{eq}) of a platinum reference electrode that is poised by the bulk concentrations of U(IV) and U(III) in the melt. The difference ($E_{1/2} - E$) is a measure of the U(IV)/U(III) ratio (Fig. 3.3). This relationship has been verified with nonradioactive melts (Sect. 3.2.a).

The electrochemical cell (Fig. 3.4) was designed so that the measurements can be made directly in the sampling ladle. The ladle is cut at a prescored point below the level of the salt, and the lower portion, which

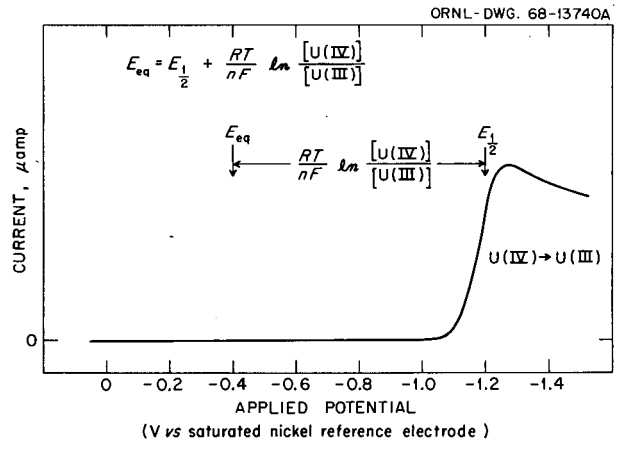

Fig. 3.3. Relationship Between Equilibrium Potential (E_{eq}) and Half-Wave Potential ($E_{1/2}$) for Voltammetric Reduction of U(IV) to U(III).

[4] Analytical Biochemistry Group.

[5] J. M. Dale, A. S. Meyer, R. F. Apple, J. E. Caton, and C. M. Boyd, "Determination of Uranium(III) in Radioactive MSRE Fuel by Hydrogen Reduction," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968,* ORNL-4343, p. 25.

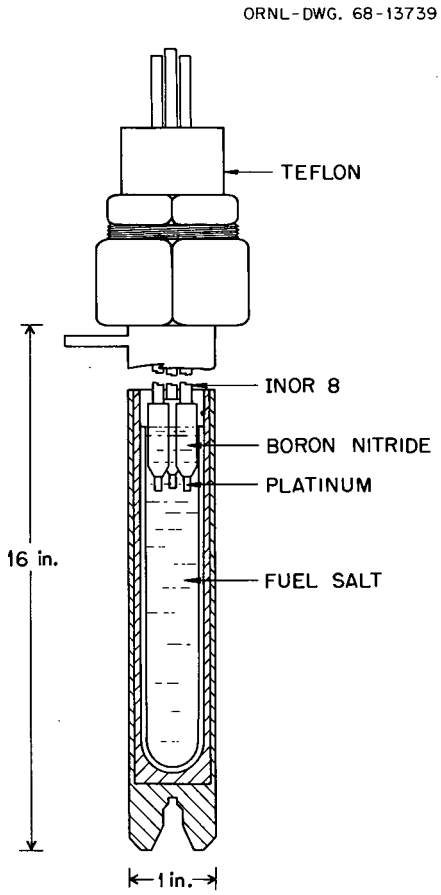

Fig. 3.4. Electrochemical Cell for Determining the U(IV)/U(III) Ratio in MSRE Fuel.

contains the bulk of the salt, is placed in a nickel liner. The liner containing the lower portion of the ladle is then placed in the cell. To minimize atmospheric contamination of the sample, this operation is completed in ~5 min. The top of the cell is fitted with a Teflon bushing, which is penetrated by three $^1/_8$-in.-diam metal electrode-support rods and a sparge tube. The electrodes (~50-mil-thick platinum-rhodium wires) are sheathed with boron nitride insulators, which define the electrode areas and prevent electrical contact between electrodes or with the cell wall.

This cell was used in the laboratory to make measurements on a sample of simulated MSRE fuel. It was shown that the U(III) in the molten salt was stable in the system and that a 5-min exposure of the surface of the solid salt to the atmosphere did not cause the U(III) concentration of the salt to change when the salt was remelted.

The electrochemical cell and other equipment necessary for making the voltammetric measurements were installed at the hot cell. After some leaks in the gas system were repaired, a radioactive molten sample could be maintained in the apparatus in the reduced state. This possibility was demonstrated by reducing a radioactive sample with hydrogen and observing that the $E_{1/2}$ of the U(IV) → U(III) reduction is stable with time.

From equilibrium considerations, it was concluded that knowledge of the U(III) concentration at 500°C is not sufficient to predict the U(III) concentration at reactor temperature (650°C) due to the buffering action at 500°C of the chromium (70 ± 10 ppm) present in the MSRE fuel. Voltammetric measurements on a radioactive fuel sample indicated that the U(III) concentration does increase when the sample is heated to 650°C and decreases when cooled to 500°C. Meaningful U(III) concentration measurements on fuel samples by any analytical technique should, therefore, be made on samples at reactor temperature.

Voltammetric measurements on an MSRE fuel sample, received and analyzed on May 8, indicated that ~0.4% of the uranium in the sample was present as U(III). At that time, enough zirconium metal had been dissolved in the fuel in the reactor to reduce 1% of the U(IV) to U(III). However, through equilibrium calculations it can be shown that ~60% of the zirconium could have been used up in reducing 6 ppm Fe(II) if the starting concentration of U(III) were ~0.08% and the activity of the reduced iron were 1. Another possibility for the nonstoichiometric agreement between the zirconium dissolved and the U(III) formed is discussed below.

An experimental setup in the Fluoride Research Laboratory at the Y-12 Site is being used to follow the change in the U(IV)/U(III) ratio in a simulated MSRE fuel as either the U(IV) is reduced or the U(III) is reoxidized. The system contains 15 kg of LiF-BeF_2-ZrF_4-UF_4 (64.4-30.2-5.2-0.27 mole %) and is maintained at 650°C under argon. Two methods are being used to follow the change in the U(IV)/U(III) ratio; (1) the voltammetric method with three platinum electrodes and (2) a potentiometric method with a reference electrode designed by C. F. Baes[2] and B. F. Hitch.[2] In the first experiment, the melt was reduced in steps by immersing zirconium rods until the voltammetric measurements indicated that 10% of the 1000 meq of U(IV) present had been reduced to U(III). Results for various stages of reduction from both the above methods and results from spectrophotometric measurements by J. P. Young are shown in Table 3.2. The spectrophotometric results 1.5 and 3.5% may be high because of an experimental problem, which will be investigated.

Table 3.2. U(III) Concentration in Simulated MSRE Fuel

Comparison of Results Obtained by Three Methods[a]

Spectrophotometric	Potentiometric	Voltammetric
<0.2	0.06	0.01
1.5	0.9 to 1.0	0.7 to 1.0
3.5	1.7 to 1.8	1.1 to 1.5
12.0	9.7	10.0

[a]All results expressed as percent of total uranium present.

At the 10%-reduction level, the loss in weight of the several zirconium rods amounted to a total of 670 meq. Even after allowance is made for the reduction of ~100 meq of known impurities in the salt, the value 580 meq of zirconium remains, which would correspond to a 57% reduction of U(IV) to U(III). Two possibilities exist – either a large amount of the zirconium metal goes into the salt as coated particles and cannot dissolve or the solution used to clean the salt from the zirconium rods before they are weighed is removing some of the zirconium. It has been reported that the cleaning solution does not attack the surface of unused zirconium rods.

After the reduction with zirconium, the U(IV)/U(III) ratios were followed as the fuel was reoxidized with NiF_2. A total of ~156 meq of NiF_2 was required to reoxidize the U(III) to U(IV) with only 0.02% U(III) remaining. The amount of U(III) oxidized plus the 21 meq each of iron and chromium totals ~146 meq. This stoichiometric agreement obtained from the oxidation of the fuel confirms that not all the zirconium lost in the reduction experiments was available to the fuel for a reduction process.

A piece of equipment is now being fabricated that will permit the reduction of the fuel with zirconium wire. It is planned that all the wire added to the fuel will be consumed and no second weighings will be necessary. Also a boron-nitride-sheathed working electrode will be used for the voltammetric measurements to better define the reduction waves.

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

3.2.a Electromotive Force (EMF), Voltammetric, and Spectrophotometric Studies of the U(IV)/U(III) Couple in Molten $LiF\text{-}BeF_2\text{-}ZrF_4$

H. W. Jenkins[6] D. L. Manning
Gleb Mamantov[7] J. P. Young

The U(IV)/U(III) ratio in the MSRE reflects the existence or lack of reducing conditions that are desirable to minimize corrosion in the reactor.

Potentiometric determination of this ratio requires knowledge of the value of the standard electrode potential of the U(IV)/U(III) couple relative to that of a reference couple. The standard potential of the U(IV)/U(III) couple was calculated with respect to a hypothetical unit-mole-fraction Ni/Ni(II) electrode (arbitrarily set at 0.0 V) from emf measurements on the galvanic cell Pt|U(IV), U(III)||Ni(II)/Ni. The cell consisted of a Ni/Ni(II) reference electrode contained in a boron nitride compartment and a platinum wire immersed in a melt that contained U(III) and U(IV). The cell potential was measured as a function of the U(IV)/U(III) ratio, which was varied by the controlled reduction of U(IV) to U(III) with a zirconium rod immersed in the solution. Measured cell potentials were corrected by means of the Nernst equation to values vs a unit-mole-fraction Ni/Ni(II) electrode. The corrected potentials were plotted vs log [U(IV)]/[U(III)]; the slope of a line through these points agreed well with the theoretical slope predicted from the Nernst equation. The standard potential of the U(IV)/U(III) couple, corresponding to a U(IV)/U(III) ratio of one, was found to be 1.48 ± 0.01 V at 500°C.

A simple voltammetric method was also developed to determine the U(IV)/U(III) ratio when [U(IV)] > [U(III)]. This method should be generally applicable to the determination of the ratio Ox/Red, where Ox and Red are the soluble oxidized and reduced forms of the reversible couple $Ox + ne \rightleftharpoons Red$. The method involves the measurement of the difference between the equilibrium potential of the melt, measured by an inert platinum electrode immersed in the melt, and the voltammetric equivalent of the standard potential of the U(IV)/U(III) couple, $E_{1/2}$.

For linear-sweep voltammetry at a stationary electrode, the polarographic half-wave potential, $E_{1/2}$, corresponds to the potential on a voltammogram at which the current is equal to 85% of the maximum current, exhibited as a peak on the voltammogram. This relationship is based on the assumption that the couple is reversible and that both the oxidized and reduced forms are soluble, which is the case for the U(IV)/U(III) couple.

The equilibrium potential (E_{eq}) of the U(IV)/U(III) couple is given by the equation

$$E_{eq} = E^0 + \frac{2.3RT}{nF} \log \frac{[U(IV)]}{[U(III)]} ,$$

[6]ORAU Fellow from the Graduate School of Chemistry, University of Tennessee, Knoxville; Sept. 1, 1967–Mar. 5, 1969.

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

and, since $E_{1/2}$ is approximately equal to E^0, it follows that

$$E_{eq} - E_{1/2} = \frac{2.3RT}{nF} \log \frac{[(IV)]}{[(III)]} .$$

Simultaneously with the emf and voltammetric measurements on the U(IV)/U(III) couple, samples were withdrawn for direct spectrophotometric determination of both U(IV) and U(III) in the fused salt. The samples were contained in windowless graphite cells. Each sample was allowed to solidify and then was transferred to a heated spectrophotometric facility. The complete operation was performed without exposing the sample to the atmosphere; such exposure would probably alter the oxidation state of the solute. The sample was remelted in a helium atmosphere, and its spectrum was obtained. The absorbance of U(IV) was determined at 630 nm and that of U(III) at 360 or 890 nm, depending on its concentration level.

The spectrophotometric and voltammetric results for the [U(IV)]/[U(III)] determined on a melt that contained ~0.3 mole % uranium follow:

Spectrophotometric	Voltammetric
6.7	7.1
10.0	13.8
29.3	29.3
140	175

The results are in good agreement. The voltammetric method has the advantages that, in order to determine the U(IV)/U(III) ratio, no other reference electrode is required, no electrode potentials need be known, and the electrode area need not be known.

A paper on this work was accepted for publication.[8]

3.2.b Computer-Operated Voltammetric Determination of U(IV)/U(III) Ratios

M. T. Kelley R. W. Stelzner[9] D. L. Manning

In one of the cells used to study U(IV)/U(III) ratios in molten $LiF\text{-}BeF_2\text{-}ZrF_4$ by the voltammetric method (Sect. 3.2.a), it was observed that the U(IV)/U(III) ratio slowly increased with time. From a starting U(IV)/U(III) ratio of ~10/1 (0.25 mole % total U), the U(III) would essentially all be oxidized to U(IV) in about two to three days. The U(III) could be regenerated with zirconium metal.

The cell assembly consists of a graphite cell that contains the melt (~50 ml); the assembly is enclosed in a quartz jacket to maintain a vacuum or controlled atmosphere. The cap of the jacket is held in place with an O-ring seal, and the electrodes are positioned by means of Cajon O-ring fittings. Although the system appears to be tight, it seems feasible that traces of impurities generated within the cell (SiF_4 and possibly O-ring contaminants) could account for the disappearance of the U(III). The data indicate that the rate of disappearance is first-order with respect to U(III), which rate order suggests that the disappearance of the U(III) may be caused by its reaction with an impurity.

To study the problem further and also to gain practical experience in adapting small on-line computers to electroanalytical methods, a PDP-8/I computer was attached to the controlled-potential controlled-current cyclic voltammeter. The computer-interfacing phase of the work is also discussed in Sect. 1.20. Two computer programs were written for the determination of U(IV)/U(III) ratios. One program is in machine language, the other in FOCAL (*FO*rmula *CAL*culator). In the FOCAL program, the computer takes a data point for the current measurement (actually the time derivative of the current measurement) every 3 mV on the voltammogram until a total of 100 points are taken. For each data point, which is taken in 1/60 sec, 128 readings are taken and averaged. This step is designed to average out a small 60-cycle ripple that is superimposed on the signal. The computer next performs a five-point smooth on the 100 points, locates the maximum of the derivative curve, and from this maximum calculates the numerical value of the U(IV)/U(III) ratio after each voltage scan. The machine-language program is similar except that the computer takes a data point every 1.5 mV on the curve for a total of 256 points. Both programs yield results with a relative standard deviation consistently less than 5%.

The procedure was made completely automatic; the computer furnishes all command signals and computes the U(IV)/U(III) ratio from the recorded time derivative of the U(IV) → U(III) reduction wave. The sequence of operations involves bringing the electrodes from open circuit to a controlled-potential configuration. After ~30 sec, a series of five voltammograms are recorded at ~1-min intervals. The U(IV)/U(III) ratio from each voltammogram is computed, and, at the end of the five scans, the average result and the relative standard deviation for the five runs are typed out. The

[8] H. W. Jenkins, G. Mamantov, D. L. Manning, and J. P. Young, "Emf and Voltammetric Measurements on the U(IV)/U(III) Couple in Molten $LiF\text{-}BeF_2\text{-}ZrF_4$," accepted for publication in the *Journal of the Electrochemical Society.*

[9] Analytical Instrumentation Group.

cell is then placed at open circuit and, after 15 min, the procedure is repeated. The results obtained thus far indicate that small on-line computers can be adapted satisfactorily to electroanalytical methods.

3.2.c Electroanalytical Studies of the Ni/Ni(II) Couple in Molten Fluorides

D. L. Manning H. W. Jenkins[6] Gleb Mamantov[7]

Emf studies on the Ni/Ni(II) couple in molten fluorides[10] have indicated that the nickel system appears to be a good reference electrode for use in molten-fluoride salt systems. The Ni/Ni(II) couple exhibits Nernstian reversibility, has only one valence state, and is relatively noble; these are among the characteristics desired for a reference electrode.

Measurements of the reversibility of the Ni/Ni(II) couple more quantitative than demonstration of Nernstian reversibility were made kinetically. From such experiments, it was hoped to evaluate basic kinetic parameters such as exchange current, transfer coefficient, and heterogeneous rate constant for the reaction $Ni^{2+} + 2e \rightarrow Ni^0$. The definition of exchange current (i_0) is

$$i_0 = nFk\, C_{Ox}^{(1-\alpha)} C_R^{\alpha} ,$$

where k is the heterogeneous rate constant, C_{Ox} and C_R are the concentrations of the oxidized and reduced forms of the couple, and α is the transfer coefficient. Two methods were used for these experiments, the relaxation voltage-step method of Vielstich and Delahay[11] and the chronocoulometric method of Christie, Lauer, and Osteryoung.[12] In the voltage-step method, a small voltage step (few millivolts) is applied to the system. Then the faradaic current, which is observed after the charging current has become negligible, is extrapolated back to zero time. The kinetic parameters are then calculated from the measured "zero-time" current, the known voltage step, and the known resistance of the system. Also for the chronocoulometric method, a potential step is applied to the cell. The number of coulombs that passes after application of the potential step is measured as a function of time. The coulombs are associated with both diffusion and nondiffusion processes; their distinction permits the evaluation of kinetic parameters k and α.

Table 3.3 summarizes the kinetic parameters for the Ni/Ni(II) couple in molten $LiF\text{-}BeF_2\text{-}ZrF_4$, which are believed to be reliable. The agreement between the two methods is good, and the kinetic parameters for the nickel system appear to be of the correct order of magnitude. The values for k and i_0 are somewhat smaller than those for the Ni/Ni(II) couple in other media. This discrepancy suggests that the charge transfer for the $Ni^{2+} + 2e \rightarrow Ni^0$ reaction in molten fluorides may correspond more to a quasi-reversible process than to a truly reversible process. The reason for the discrepancy is not known, although the great complexing affinity of fluoride melts, which results in associated fluorocomplex species, may be partly responsible. However, from a practical standpoint, as long as the exchange current is large compared with the net current that a reference electrode is required to pass, the reference electrode should remain poised. For most electroanalytical applications involving controlled-potential, controlled-current, and potentiometric techniques, this relationship is generally the case.

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

H. W. Jenkins[6] D. L. Manning Gleb Mamantov[7]

The program of emf measurements in molten fluorides[13] conducted by H. W. Jenkins[6] as part of his

[10] H. W. Jenkins, G. Mamantov, and D. L. Manning, "Emf Measurements on the Nickel-Nickel(II) Couple in Molten Fluorides," *J. Electroanal. Chem.* 19, 385 (1968).

[11] W. Vielstich and P. Delahay, "Voltage Step Method for Kinetic Study of Fast Electrode Reactions," *J. Am. Chem. Soc.* 79, 1874 (1957).

[12] J. H. Christie, G. Lauer, and R. A. Osteryoung, "Measurement of Charge Passed Following Application of a Potential Step – Application to the Study of Electrode Reactions and Adsorption," *J. Electroanal. Chem.* 7, 60 (1964).

[13] H. W. Jenkins, D. L. Manning, and G. Mamantov, "Electromotive Force (EMF) Measurements in Molten Fluorides," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968,* ORNL-4343, p. 28.

Table 3.3. Kinetic Parameters for the Ni/Ni(II) Couple in Molten $LiF\text{-}BeF_2\text{-}ZrF_4$ at 500°C

	Method	
	Voltage-Step	Chronocoulometric
Standard rate constant (k), cm/sec	$\sim 1 \times 10^{-3}$	$\sim 1.7 \times 10^{-3}$
Transfer coefficient (α)	0.68 to 0.45	~0.6
Molar exchange current (i_0), A/cm^2	0.06 to 0.2	

Table 3.4. Standard Electrode Potentials of Electrode Couples in Molten Fluorides

Electrode Couple	E^0(V)		
	Maximum[a]		Calculated[b]
	$LiF\text{-}BeF_2\text{-}ZrF_4$	LiF-NaF-KF	$LiF\text{-}BeF_2$
Be(II)/Be	−2.120		−2.211
Zr(IV)/Zr	−1.742		−1.772
U(IV)/U(III)	−1.480		−1.517
Cr(II)/Cr	−0.701		−0.789
Cr(III)/Cr	−0.639		
Cr(III)/Cr(II)	−0.514		
Fe(II)/Fe	−0.410	−0.390	−0.413
Fe(III)/Fe	−0.218	−0.327	
Ni(II)/Ni	0.000	0.000	0.000
Fe(III)/Fe(II)	+0.166	−0.200	

[a]The standard state for all the solutes listed except Be(II) is the hypothetical unit mole fraction. The Be(II) standard state is the Be content of the solvent $LiF\text{-}BeF_2$ (66-34 mole %).

[b]Values calculated by Baes[14] and converted to 500°C.

doctoral thesis research was concluded. Table 3.4 summarizes the electrode potentials measured thus far.

The Ni/NiF_2 reference electrode[10] consists of a thin-walled boron nitride (BN) compartment that contains the fluoride melt, NiF_2 at a fixed concentration, and a nickel electrode. For comparison, the standard electrode potentials (E^0's) calculated by Baes[14] for the indicated electrode couples in $LiF\text{-}BeF_2$ (66-34 mole %) at 500°C are included in Table 3.4 if they were available. The trend in the values determined for the couples in $LiF\text{-}BeF_2\text{-}ZrF_4$ agrees well with that in the values calculated by Baes for the couples in $LiF\text{-}BeF_2$; however, in all instances, the values determined in this study are lower than those calculated by Baes. The 91-mV difference between the two values for the Be(II)/Be couple could be due partially or totally to the inclusion of a junction potential in the value determined in this study. The other differences range from 3 mV for the Fe(II)/Fe couple to 88 mV for the Cr(II)/Cr couple. These differences may be due to the greater stability of the higher oxidation state of the couple relative to its lower oxidation state in $LiF\text{-}BeF_2$ than in $LiF\text{-}BeF_2\text{-}ZrF_4$. Such an effect may be caused by the formation of a very stable ZrF_x^{4-x} complex, which would make $LiF\text{-}BeF_2\text{-}ZrF_4$ a more acidic solvent, according to the modified Lux-Flood concept, than $LiF\text{-}BeF_2$ by the reduction of the activity of the basic species, free F^-. The same effect, in a more pronounced example, is observed when the relative stability of Fe(III) is compared with that of Fe(II) in LiF-NaF-KF and in $LiF\text{-}BeF_2\text{-}ZrF_4$. In LiF-NaF-KF where all the fluoride is present as free F^-, the standard potential of the Fe(III)/Fe(II) couple is 0.190 V anodic to the Fe(II)/Fe standard potential. However, in $LiF\text{-}BeF_2\text{-}ZrF_4$ where the free F^- activity is reduced by the formation of BeF_4^{-2} and ZrF_x^{4-x} complexes, the standard potential of the Fe(III)/Fe(II) couple is 0.576 V anodic to the Fe(II)/Fe couple. Thus, relative to Fe(II), Fe(III) is 0.386 V (or 8.8 kcal) more stable in LiF-NaF-KF than in $LiF\text{-}BeF_2\text{-}ZrF_4$.

A paper that describes this work was accepted for publication.[15]

3.2.e Facility for Spectrophotometric Studies of Radioactive Materials

J. P. Young

The development of a spectrophotometric facility for hot-cell use has continued.[16] Although it will be used initially to demonstrate the spectral determination of U(III) in MSRE fuel, it can also be used to study the spectra of highly radioactive liquids, gases, or solids.

The design and fabrication of the optical components for the hot-cell spectrophotometer were completed by Cary Instruments of Varian Associates. Figure 3.5 shows the complete unit mounted on a temporary stand. The source compartment and the monochromator-detector compartment are shown in the front and rear, respectively, at the left in the photograph. By the addition of the necessary optical elements, the light beam of the spectrophotometer is directed through the 3-ft-long wall embedment (the shiny metal bulk shown in the center of the photograph) and into the hot cell. The portion of the facility that is within the hot cell is the darkened area at the right. Both sample and reference beams pass into and out of the hot cell; they are displaced 6 in. vertically.

[14]C. F. Baes, Jr., "The Chemistry and Thermodynamics of Molten-Salt Reactor Fluoride Solutions," pp. 409–43 in vol. 1 of *Thermodynamics*, International Atomic Energy Agency, Vienna, 1966.

[15]H. W. Jenkins, G. Mamantov, and D. L. Manning, "Electrode Potentials of Several Redox Couples in Molten Fluorides," accepted for publication in the *Journal of Electrochemical Society*.

[16]J. P. Young, "Facility for Spectrophotometry of Radioactive Samples," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968*, ORNL-4343, p. 29.

Fig. 3.5. Spectrophotometer Designed for Use in a Hot Cell.

The molten-salt spectral furnace will be located in the upper sample beam.

The completed spectral apparatus was checked out at the vendor's site and has now been delivered for final tests here at ORNL.

The equipment described previously[16] for transferring the MSRE sample from reactor to spectral furnace has been fabricated; also, it has been tested both mechanically in a mock-up to evaluate manipulation operation and thermally to evaluate performance.

At present, the sample-loading furnace[16] is not satisfactory. Design modifications are being considered to improve its performance. The spectral furnace and top assembly have been used routinely for studies in the laboratory since they were fabricated. The former works very well; minor problems in the latter are to be corrected. It is planned that the entire system will be in operation within six months.

3.2.f Spectrophotometric Determination of Uranium(III) in Molten Fluoride Salts

J. P. Young

In a cooperative venture with H. W. Jenkins,[6] D. L. Manning, and G. Mamantov,[7] both voltammetric and spectrophotometric measurements of U(III) in a LiF-BeF_2-ZrF_4 melt were made; the work is also discussed in Sect. 3.2.a. A paper on the results of this study was accepted for publication.[8] From a melt for which the U(III)/U(IV) ratio had been determined voltammetrically, a sample was withdrawn into a windowless graphite cell,[17] was allowed to freeze, and was transferred without exposure to the atmosphere to a vacuum

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

dry box and then into a spectral furnace. The sample was remelted, the spectra of U(III) and U(IV) were observed, and the concentration of U(III) was determined. A number of U(III) determinations were then made for other persons, both members and nonmembers of our Division. In later determinations, a new way to obtain a sample was devised. A sample was withdrawn from a melt through a fritted copper filter into a ¼-in.-diam copper tube. The tube was placed in a sealed container for transportation to a vacuum dry box. This entire sampling operation was performed without exposing the sample to the atmosphere. After the sample assembly was loaded into the dry box, small sections (⅜-in.-long) that contained the solidifed filtered salt were cut out of the copper tube. A section was placed in the upper portion of a windowless graphite cell[18] and was transferred, in the absence of air, to the spectral furnace for remelting. The melt ran out of the tubing and into the cell for spectral study. The technique worked satisfactorily, and reasonable values of U(III) concentration were obtained on a number of samples.

Recently, L. M. Toth[2] has gathered data with the diamond-windowed cell[18] which indicate that the molar absorptivity at 360 nm of U(III) in BeF_2-based salts is greater than the value reported originally;[17] our values for the absorptivity at 890 nm agree well. Work is now under way to explain the discrepancy of the 360-nm values. Possibly, with the instrumentation used, light scattered by the molten sample got through to the detector. The effect would have been to reduce the apparent absorbance of light of a given wavelength; the effect would be most pronounced with short-wavelength light. However, if the sample were placed reproducibly in the optical system – and that was done to get maximum light transmission through the sample – this scattering error should have been relatively constant. Thus, the results should be self-consistent. The agreement between voltammetric and spectrophotometric results bears out this conjecture (Sect. 3.2.a). The reason for the high spectral results given in Table 3.2 of Sect. 3.1.f is not now understood.

3.2.g Spectrophotometric Cells for Molten Fluoride Salts

J. P. Young

A container of fixed pathlength through which light can be transmitted is needed for melts. With windowless cells,[19] the pathlength can be determined but is not fixed. A diamond-windowed cell[18] is an excellent fixed-pathlength cell that is compatible with many fluoride melts, but the small size (5 X 5 X 1 mm) of presently available windows and their possible embrittlement in radioactive environments precludes their use in special situations, such as the hot-cell spectrophotometer. Study was begun of a new type of windowless fixed-pathlength cell that is made of "porous metal," a product developed by Mallory Metallurgical Company, Indianapolis, Ind. Porous metal is a foil that contains a number of small irregular pits formed by a proprietary electrochemical process. Many of the pits are etched completely through the foil so that light is transmitted through the metal. Cells made of porous stainless steel adequately hold aqueous solutions for spectral study. Mallory Metallurgical Company has agreed to fabricate experimental batches of porous Hastelloy N from material furnished by ORNL. If the product is satisfactory, it should be possible to fabricate a fixed-pathlength cell entirely of Hastelloy N, which is compatible with radioactive MSRE fuel.

3.2.h Absorption Spectra of 3d Transition-Metal Ions in Molten Fluoride Solutions

J. P. Young

Spectral study of the 3d transition-metal ions in molten fluoride solution continues. Figure 3.6 summarizes the absorption spectra of the ions that have been studied in molten Li_2BeF_4. Horizontal lines show general areas of absorption; vertical lines denote peak and shoulder positions. The regions where the absorption of these solutions becomes very intense are shown as non–d-d transitions. The solvent Li_2BeF_4 is transparent over the wavelength range 250 to 1800 nm. At least one example of each $3d^n$ configuration, except $3d^5$, has been observed. Other than the spectra of Fe(II), Ni(II), Cr(II), and Cr(III), which were described earlier,[16,17] the figure is comprised of data collected this year. Molar absorptivities are not known accurately but, in all cases, appear to be <20 liters $mole^{-1}$ cm^{-1}. From the positions and intensities of the spectral peaks involved, it is reasonable to assume that the indicated solute species are situated in high symmetry, possibly O_h, in these melts. Exceptions to this generality are Ti(III) and Fe(II), which may be distorted by Jahn-Teller effects.

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[18] L. M. Toth, J. P. Young, and G. P. Smith, "Diamond-Windowed Cell for Spectrophotometry of Molten Fluoride Salts," *Anal. Chem.* 41, 683 (1969).

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

ORNL-DWG. 68-14183A

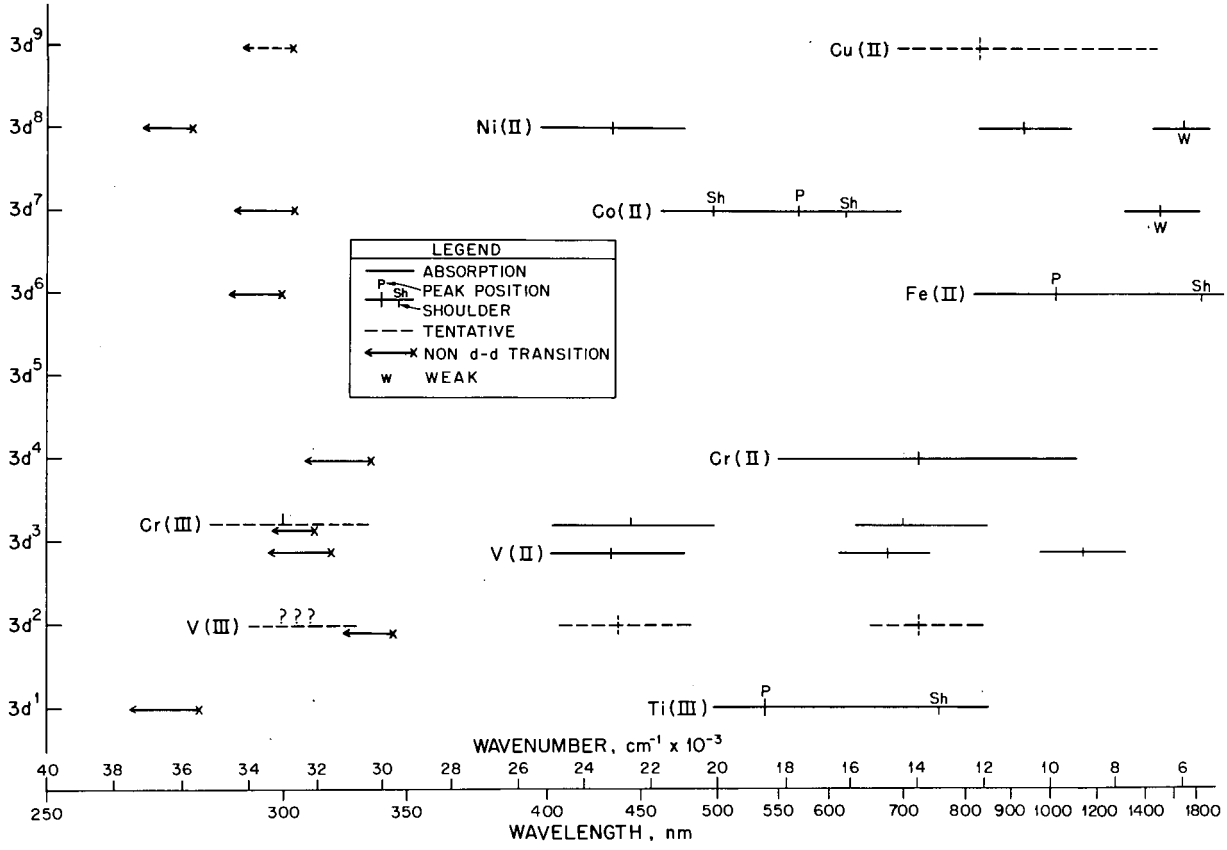

Fig. 3.6. Absorption Peaks of 3d Solute Ions in Molten Li_2BeF_4 at 550°C.

The spectral study of the effect of solvent composition on the coordination of solute species in fluoride melts is also continuing.[20] It has been observed that the spectrum of Co(II) is essentially the same in Li_2BeF_4 melts – even those that are BeF_2-rich to the composition LiF-BeF_2 (60-40 mole %) – as in molten LiF-NaF-KF. If stabilization energies for octahedral coordination are considered, Co(II) appears to be the ion whose coordination would most easily be changed from O_h to T_d as the concentration of free fluoride ion is reduced by coordination with excess BeF_2 to make BeF_4^{2-}. Since the Co(II) spectrum does not change, it is concluded that no change in the type of coordination occurs.

Spectral studies of Co(II) and Ni(II) in molten and glassy BeF_2 are under way. The spectra of Co(II) in liquid or solid BeF_2 correspond well to the spectrum reported by Pappalardo[21] for Co(II) in BeF_2 glass, which he interprets to indicate T_d coordination of Co(II). The Ni(II) species seems to be only slightly soluble in BeF_2, but the spectra obtained appear to arise from Ni(II) that is still in O_h coordination – a puzzling result that needs further study.

3.2.i Spectrophotometric Evidence for the Existence of the Superoxide Ion in Molten Fluoride Salts

F. L. Whiting[22] J. P. Young Gleb Mamantov[7]

The absorption spectrum of the superoxide ion (O_2^-) in a molten salt was observed for the first time; Fig. 3.7 is the spectrum of O_2^- in LiF-NaF-KF. A paper on this

[20] J. P. Young and J. C. White, "Absorption Spectra of Molten Alkali Salts; Solutions of Several Metal Ions in Molten LiF-NaF-KF," *Anal. Chem.* 32, 799 (1960).

[21] R. Pappalardo, "Evidence for Tetrahedrally Co-ordinated Co^{2+} in Beryllium Fluoride Glasses," *Spectrochim. Acta* 19, 2093 (1963).

[22] ORAU Graduate Fellow from the Graduate School of Chemistry, University of Tennessee, Knoxville.

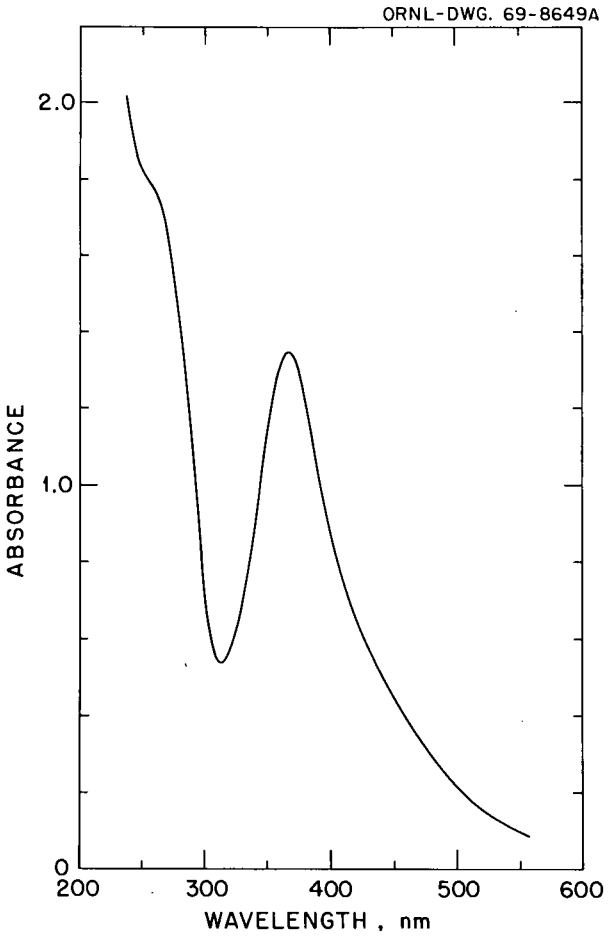

Fig. 3.7. Absorption Spectrum of Superoxide Ion (O_2^-) in Molten LiF-NaF-KF at 500°C.

observation was accepted for publication[23] and also was given orally;[24] the abstract of the oral presentation follows.

"Evidence derived from spectral studies has shown that the superoxide ion, O_2^- exists in molten LiF-NaF-KF (46.5-11.5-42.0 %) at 500°C. This solute species can be added directly to the molten solvent or can be generated by the addition of oxidants to an oxide containing melt.

"The superoxide species is generated by the addition of either MnF_3 or CoF_3 to the molten solvent which contains oxide ion. In the first case a spectrum of Mn(III), in essential O_h symmetry, is observed, and a second absorbing species is also observed which exhibits a peak at 372 nm and a shoulder at 260 nm. In the latter case, the spectrum of Co(II) is observed as well as the spectrum of the second species. The 372 nm peak agrees with the spectrum ascribed to superoxide ion in other media. Addition of either NaO_2 or Na_2O_2 to the solvent also produces the expected spectrum. The spectrum is much more intense with NaO_2, however.

"The solute species is relatively stable in SiO_2 containers or in platinum windowless containers. A molten solution containing the oxidant in a SiO_2 cell can be maintained under a vacuum of *ca* 5μ for a period of two hours with no decrease in the absorbance of the 372 nm peak. The species decomposed slowly in graphite windowless cells perhaps by reaction with the graphite. Spectral evidence indicates that the superoxide species is also generated electrochemically at a platinum anode in an oxide containing LiF-NaF-KF melt."

Since the abstract was written, it was found that O_2^- can be generated in molten LiF-NaF-KF, 2LiF-BeF_2, LiF-BeF_2-ZrF_4 by oxidation of O^{2-} with O_2 gas and in molten KHF_2 at 280°C by oxidation of O^{2-} with CoF_3. Also, it can be generated electrochemically at a pyrolytic or regular graphite electrode in molten LiF-NaF-KF at 500°C.

3.3 NUCLEAR SAFETY

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

C. M. Boyd A. L. Travaglini A. S. Meyer

The system[25] for the analysis of the gases from the in-reactor tests of Gas Cooled Reactor (GCR) type fuel-element specimens was tested extensively and modified. This system is needed to measure the gases that are evolved when a small simulated GCR fuel element in a rapidly flowing stream of helium that contains as much as 30% steam is inserted into the reactor. Both rapid transients and steady-state levels in the concentrations of H_2, CO, CO_2, CH_4, water vapor, and atmospheric contaminants are to be measured. On the basis of competitive bidding, a General Electric Monopole 600 Residual Gas Analyzer (RGA) was selected for this application. Figure 3.8 is a schematic diagram of the gas system to be used, together with a system for preparing calibrating-gas mixtures. The section downstream of the special T valve, which was fabricated to minimize delays caused by dead volumes, is essentially the analytical system to be installed at the

[23] F. L. Whiting, G. Mamantov, and J. P. Young, "Spectral Evidence for the Existence of the Superoxide Ion in Molten LiF-NaF-KF," accepted for publication in the *Journal of the American Chemical Society*.

[24] F. L. Whiting, G. Mamantov, and J. P. Young, "Spectral Evidence for the Existence of the Superoxide Ion in Molten LiF-NaF-KF," presented at the 158th Meeting of the American Chemical Society, New York, Sept. 7–12, 1969.

[25] C. M. Boyd, W. F. Peed, and A. S. Meyer, "Analysis of Purge Gas from In-Reactor Tests of Reactor Fuel Elements," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968*, ORNL-4343, p. 32.

ORR. The remaining components are used for the dynamic preparation of calibrating-gas mixtures.

The capillary in the helium supply line furnished reproducible flows of 50 to 500 cc/min. With steam generated by an Aerograph model 675 gas chromatographic steam-supply unit, water vapor can be added at accurately measured flows as fast as 60 cc/min. Other gas additions can be made through the third capillary at rates from 0.2 to 6-10 cc/min, the upper limit varying with the viscosity of the added gas. It is thus possible to prepare helium standards that contain as much as 30% steam and permanent gases in concentrations from 400 ppm to 10%. On the basis of the reproducibility of flow measurements for the capillaries, the error in this preparation of standard is estimated to be ~2% in the middle concentration range. This accuracy has, so far, exceeded the accuracy of the analytical system.

In the analysis of off-gas samples, a small portion of the effluent gas from the experiment will be drawn into the intermediate-pressure region through the entrance capillary. By selection of pressure-regulating capillaries, intermediate pressures of 2, 7, and 17 mm can be obtained. At the 7-mm upstream pressure, the inlet capillary introduces about ~5 μcc of helium per second, which is about the maximum flow that the ion pump can tolerate for extended periods. With a small-bore (6-mil) entrance capillary, the time constant of the sample-introduction system is ~15 sec. Where possible, nickel is used for these sampling components, because stainless steel capillaries catalyze the disproportionation of CO.

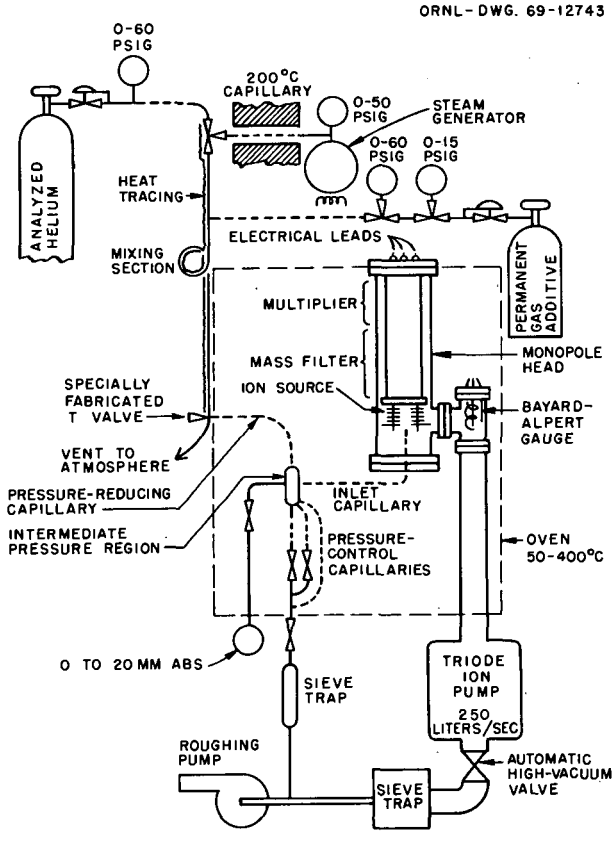

Fig. 3.8. Analytical System for the Determination of the Products of the Reaction of Graphite with Steam-Helium Mixtures.

Also included as a part of the analytical system are the electronic controls for the Monopole head and a read-out system, which includes a Keithly model 418A remote ranging picoammeter that drives an oscilloscope and a two-pen $\frac{1}{8}$-sec recorder.

The operation of the Monopole has been beset by many problems. These problems are associated with error in design or fabrication of the Monopole head that was manufactured early on the first production run, the attempt to apply the instrument for purposes for which it was not designed, and some problems associated with inadequately designed electronics. Some of these problems will be described briefly.

It was necessary to return the head of the instrument to the vendor twice; first to gold-plate the field-defining surfaces and second to replace a magnetic component that was inadvertently included as a structural member of the resolving section.

Although the instrument met or exceeded specifications for sensitivity (100 A/torr) and resolution, it was impossible to fully exploit this sensitivity for quantitative applications. The response of the instrument decreases with ion current. This behavior leads to nonlinear calibration curves when a single gas is added in percentage concentration to the helium and to an unpredictable decrease in its sensitivity for trace constituents. This phenomenon is believed to result from "beam spreading" caused by mutual ion repulsion in the resolution section of the instrument. It can be minimized at the expense of sensitivity by operating the instrument at reduced ionization current. This mode of operation increases the detection limits from 1 to ~20–50 ppm. It is believed that compromise operating conditions can be selected that will accommodate most of the requirements for the off-gas analysis.

One of the most basic problems encountered is that the instrument is designed primarily to measure residual gases in large vacuum systems and thus includes only a simple ion source in which the ionization takes place in an open-mesh cage. A large fraction of the signal,

therefore, resulted from gases desorbed from the envelope walls rather than from the sample introduced through the inlet capillary. At the suggestion of R. B. Crawford,[26] who had encountered a similar problem with another type of RGA, the ionization region was largely isolated from the remainder of the vacuum system by the substitution of a low-gas-conductivity ion chamber for the open-grid structure. We are indebted to J. R. Sites[27] and W. H. Montgomery[2] for suggestions and aid in the fabrication of this chamber. The new ionization chamber consists of an ~2-cc gold box perforated by 0.020- × ⅛-in. electron-entrance slits, a 1-mm ion-exit hole and a sample-inlet port. The inlet capillary is connected to this chamber with a ceramic insulating bushing. The calculated conductivity of the openings in the chamber is ~356 cc/sec, so that with the 5-μcc/sec flow of sample a differential pressure between the chamber and the vacuum system of $\sim 10^{-5}$ torr is established. With a well-baked system, the background of the most persistent peak (mass 28) of the mass spectrum is reduced to a height that corresponds to ~50 ppm in the sample.

Some problems in maintaining precise potentials on the elements in the ion source were solved by substituting low-impedance power supplies (Hewlett-Packard model 6205 B) for those installed by the manufacturer. The observed voltage fluctuations have been attributed to the formation of slightly conductive films on the insulators used to isolate the ion-source elements. With the simple power supplies furnished with the instrument (impedance 50 to 200 kilohms), small leakage currents can produce significant changes in the voltages of these elements. This problem is particularly important, because resistance of the films is not constant but is influenced by constituents introduced with the samples.

Some additional electronic problems believed to be associated with rf circuitry are still being experienced. These will be corrected after the monopole is installed, because the necessary changes in electrical leads introduced during installation may influence the resonant circuits.

The Monopole is now being installed at the ORR, where it will be given additional calibration tests before application to off-gas samples.

[26] R. B. Crawford, Lawrence Radiation Laboratory, Livermore, Calif., personal communication to A. S. Meyer, November 1968.

[27] Mass Spectrometry Service Group.

3.3.b Analysis of Natural-Gas Samples

C. M. Boyd A. L. Travaglini A. S. Meyer

The evaluation of underground nuclear explosions to enhance production from natural-gas wells required special analytical methods to determine the extent of radioactive contamination of the gas and to study techniques for reducing it. Of prime interest is the concentration of the long-lived radioactive gases ^{85}Kr and ^{3}H, both present at the μcurie/ft^3 level, and the distribution of ^{3}H among the following chemical constituents of the samples: H_2, H_2O, CH_4, and higher hydrocarbons. The Oak Ridge National Laboratory is constructing an apparatus to make these analyses.

Representatives of ORNL visited the Lawrence Radiation Laboratory (LRL) at Livermore, where Project Gasbuggy samples are being analyzed. The information obtained has been of great help to us in designing the

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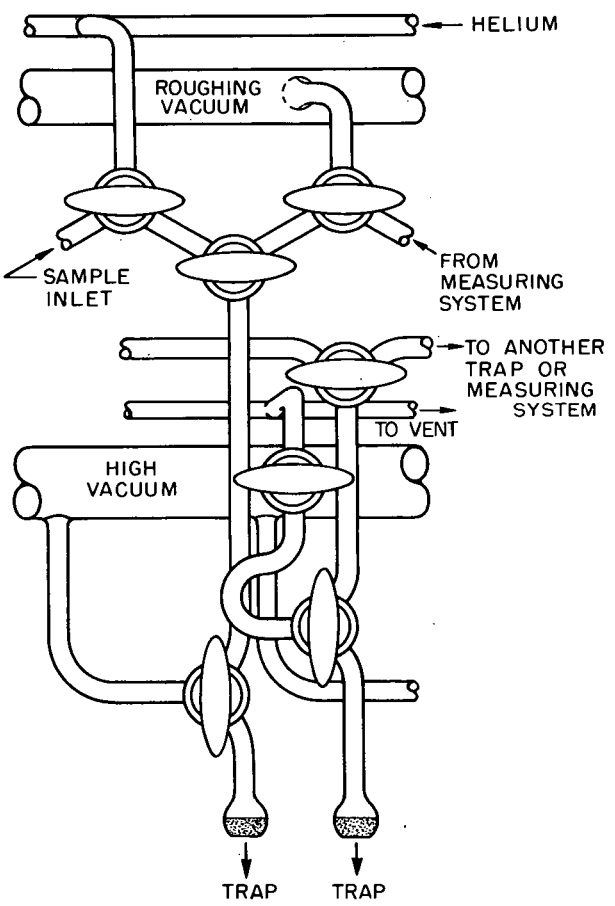

Fig. 3.9. Arrangement of a Stopcock Unit for the Natural-Gas-Analysis Train.

ORNL gas-analysis system. C. F. Smith[28] has offered complete cooperation, including exchange of analyzed samples, to establish agreement between the two laboratories. We will perform the analyses in a manner similar to that used at LRL but with some modifications to make the apparatus more compact and to use liquid-scintillation counting.

In the technique developed at LRL, the chemical composition of the samples is determined mass spectrometrically. Then the radioactive constituents from ~1-liter samples are fractionated for counting by successive elution with helium from large charcoal and molecular-sieve traps at selected temperatures – essentially a large-scale gas chromatographic technique. The separated ^{85}Kr is counted in thin-window cells, and the tritiated species are counted by internal gas-proportional counting.

The LRL gas-separation train cannot be used at ORNL without modification, because there is no available C-zone area large enough to accommodate it. This train includes eight traps, some of which serve both as gas collectors and gas-chromatographic columns. For optimum versatility, it is necessary to be able to connect any trap with any other trap in the system and to elute gases from one trap, pass them through a measuring system (thermal-conductivity cell plus an ionization chamber), and collect the condensable gases on a second trap. Also, paths must be provided for evacuating or venting any of the traps. The use of three-way precision-ground pressure stopcocks (Lab Glass, Inc., Kingsport, Tenn.) effected ~40% reduction in the length of this section. Figure 3.9 shows the arrangement of these stopcocks in one of the units that is repeated in the train. This configuration uses fewer stopcocks than the original design and eliminates dead legs. The possibility of ejection of stopcock plugs by pressure within the system is eliminated.

We are supplementing the LRL counting techniques with liquid-scintillation counting of ^{3}H. The tritiated species will be converted to $^{1}H^{3}HO$ by heated copper oxide. A multiple-pass oxidation system is being incorporated in the system to ensure the quantitative oxidation of CH_4.

Except for the interconnections and some of the traps, fabrication of the glassware is completed. This apparatus is now being installed in Building 2026. Electronic equipment and special gas-counting cells are being furnished by the Instrumentation and Controls Division. The system will be used to analyze samples of natural gas from the Rulison test.

[28] Radiochemistry Division, Lawrence Radiation Laboratory, Livermore, Calif.

4. Analytical Biochemistry

Gerald Goldstein

4.1 MACROMOLECULAR SEPARATIONS PROGRAM

4.1.a Statistically Designed Procedure for Optimizing Conditions for the Determination of a Specific Transfer Ribonucleic Acid (tRNA)

I. B. Rubin T. J. Mitchell[1] Rae Lyn Athanacio[2]

Studies to develop statistically designed procedures for establishing optimum conditions for the determination of purified tRNA's have been continued.[3,4] In the past, optimization studies were made by testing one variable at a time. This process is very tedious and requires ~1000 assays, because each variable must be tested at several levels of all the other variables. However, with statistically designed experiments, the number of assays can be decreased by at least 60%, and various interactions can be studied as well. By the use

[1] Mathematics Division.

[2] ORAU Student Trainee from the University of Nevada, Reno.

[3] I. B. Rubin, "A Fractional Factorial Method for Optimizing Conditions for the Determination of Transfer Ribonucleic Acid (tRNA)," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968*, ORNL-4343, p. 34.

[4] "An Experimental Optimization Program for the Chemical Assay of tRNA," *Math. Div. Ann. Progr. Rept. Dec. 31, 1968*, ORNL-4385, p. 83.

of a suitable initial design, most of the variables can be tested simultaneously in a relatively few trials. As the influence of each variable is established, simpler designs can be constructed to study the more influential variables in more detail and to determine what quantitative combination of these variables provides maximum aminoacylation.

In these optimization studies, 2^{k-p} fractional factorial designs are used.[5] Table 4.1 shows a design for the first, or screening, experiment; it is basically a 2_{IV}^{8-4} factorial design in eight blocks of two. Some or all of these trials may be duplicated, and four trials using points midway between the high and low values may also be included. It is desirable to run a blank assay with each trial. The trials, each of which is an individual assay, are run in random order. Eight variables are assigned randomly to the first eight columns, pH is assigned to the ninth, and four buffers are assigned to columns 10 and 11. In the preparation of this design, three-factor interactions are assumed to be negligible, but columns 9, 10, and 11 are each confounded with four sets of two-factor interactions, which may or may not be significant. Thus, there is a certain amount of ambiguity in the results of this type of experiment. Results can be evaluated by the Yates algorithm,[6] which is a means of summing up the results of n trials in n different ways. Table 4.2 shows the assignment to the variables of the estimates from the solution of the Yates algorithm. These answers are then ranked 1 through $(n - 1)$ by absolute values, excluding the average estimate. These values are plotted as a half-normal curve on probability paper. Table 4.3 presents as an example the results from the glutamic acid-accepting tRNA ($tRNA^{glu}$) screening experiment, and Fig. 4.1 is the half-normal plot. The points that are significantly below the line, that is, those with the largest estimates, represent the factors or interactions that need to be examined in the next design. In the case of qualitative variables, the low point may mean that one variable has such a strong negative influence on the reaction that the variable can be dismissed.

Factors involved in the aminoacylation of tRNA are (1) ^{14}C-labeled amino acid concentration; (2) pH; (3) type of buffer; (4) enzyme concentration; (5) Mg^{2+} concentration; (6) adenosine triphosphate (ATP) concentration; (7) type of monovalent cation, usually NH_4^+ or K^+; (8) concentration of this cation; (9) reducing agent, usually glutathione (GSH) or 2-mercaptoethanol (ME); (10) concentration of this reagent; and (11) time. Temperature has been held constant in these studies, and the effect of divalent metals other than Mg^{2+} will be treated

Table 4.1. Factorial Design for Screening Experiment[a]

Trial	Variable										
	1	2	3	4	5	6	7	8	9	10[b]	11[b]
1	+	–	–	–	+	+	+	–	–	–	–
2	–	+	+	+	–	–	–	+	–	–	–
3	+	+	–	–	–	–	+	+	+	–	–
4	–	–	+	+	+	+	–	–	+	–	–
5	+	–	+	–	–	+	–	+	–	+	–
6	–	+	–	+	+	–	+	–	–	+	–
7	+	+	+	–	+	–	–	–	+	+	–
8	–	–	–	+	–	+	+	+	+	+	–
9	+	–	–	+	+	–	–	+	–	–	+
10	–	+	+	–	–	+	+	–	–	–	+
11	+	+	–	+	–	+	–	–	+	–	+
12	–	–	+	–	+	–	+	+	+	–	+
13	+	–	+	+	–	–	+	–	–	+	+
14	–	+	–	–	+	+	–	+	–	+	+
15	+	+	+	+	+	+	+	+	+	+	+
16	–	–	–	–	–	–	–	–	+	+	+

[a]The signs indicate high (+) or low (–) values of quantitative variables or the presence of one (+) or the other (–) of the qualitative variables.

[b]The signs in column 10 and 11 indicate buffers, which are identified as follows: buffer 1, ––; 2, +–; 3, –+; and 4, ++.

Table 4.2. Assignment of Results from Screening Experiment[a]

Estimate[b]	Variable Assignment
1	Average
2	1
3	2
4	9 + (1,2) + (3,5) + (4,6) + (7,8)
5	3
6	10 + (1,3) + (2,5) + (4,7) + (6,8)
7	(9,10) + (2,3)
8	5 + (1,2,3)
9	4
10	11 + (1,4) + (2,6) + (3,7) + (5,8)
11	(9,11) + (2,4)
12	6 + (1,2,4)
13	(10,11) + (3,4)
14	7 + (1,3,4)
15	8 + (2,3,4)
16	(9,10,11) + (1,2,3,4)

[a]After arrangement of results in the standard order.

[b]Calculated from Yates algorithm.

[5]G. E. P. Box and J. S. Hunter, "The 2^{k-p} Fractional Factorial Designs. Part 1," *Technometrics* 3, 311 (1961).

[6]W. G. Cochran and G. M. Cox, *Experimental Designs*, pp. 158–61, Wiley, New York, 1957.

in a separate study. Optimum conditions have been established for the determination of lysine-accepting tRNA ($tRNA^{lys}$), and several statistical experiments have been completed for the determination of arginine- and glutamic acid-accepting tRNA's ($tRNA^{arg}$ and $tRNA^{glu}$).

$tRNA^{lys}$. – The results of the first four experiments in this study have been reported.[4] A total of six experiments were carried out. The fifth experiment was essentially a central composite rotatable design[7] that tested four variables: concentrations of ^{14}C-lys, ATP, and enzyme and pH with each of two buffers. The sixth experiment was a recheck of these same four factors over very narrow ranges with three buffers. An anomalous situation with regard to sodium cacodylate as a buffer was encountered. The highest response with cacodylate present was at pH 9.8, a value that is well out of its buffering region and one at which hydrolysis of the aminoacylated tRNA should be rapid.

Table 4.4 gives the optimum conditions for the determination of $tRNA^{lys}$. Although the results of the first experiment indicated that time is not a significant factor after 15 min, a subsequent time study showed that aminoacylation continues to increase for up to at least 1 hr. The increase between 30 and 60 min is ~10%; this increase could be an evaporation effect. Consequently, 30 min will be used as the incubation period pending further investigation.

$tRNA^{glu}$. – Five statistically designed experiments have been completed in the study of $tRNA^{glu}$. The first three were factorial designs for eleven, five, and four variables. Incubation was set arbitrarily at 30 min, because this type of experiment is difficult to perform when time is varied. Four buffers were tested initially, as indicated in Table 4.1, rather than only two as in the $tRNA^{lys}$ study. At the completion of the third experiment, the levels of all the factors except the ^{14}C-glu concentration were well established. However, it was considered desirable at that point to make a buffer-pH study by using as many of those buffers as were available with pK_a's in the region of the indicated optimum pH. A simple experiment was made that tested five buffers at four pH values. The results confirmed the pH and buffer conditions indicated by the results of an earlier design. The fifth experiment was a central composite rotatable design with three variables: pH and concentrations of ATP and amino acid. The results indicate that the optimum values for these variables are very near the center point of the design. However, a confirmatory experiment will be performed before the project will be considered finished. Table 4.5 gives the tentative conditions for the determination of $tRNA^{glu}$.

$tRNA^{arg}$. – Three statistically designed experiments have been completed in the study of $tRNA^{arg}$. The designs and results of these experiments were similar to those in the $tRNA^{glu}$ investigation. At the completion

Table 4.3. Results from $tRNA^{glu}$ Screening Experiment

Rank	Estimate	Variable Assignment
	915	Average
1	20	Conc. of K^+ or NH_4^+
2	41	pH
3	74	B_2[a]
4	−90	Enzyme
5	170	B_1[a]
6	179	K^+ or NH_4^+
7	−180	Mg^{2+}
8	−216	pH, B_2[a]
9	353	pH, B_1,[a] B_2[a]
10	372	Conc. of ME[b] or GSH[c]
11	553	pH, B_1[a]
12	−560	ATP[d]
13	−566	ME[b] or GSH[c]
14	764	^{14}C-glu[e]
15	−948	B_1,[a] B_2[a]

[a]B_1 and B_2 refer to columns 10 and 11 of Table 4.1.
[b]2-Mercaptoethanol.
[c]Glutathione.
[d]Adenosine triphosphate.
[e]^{14}C-labeled glutamic acid.

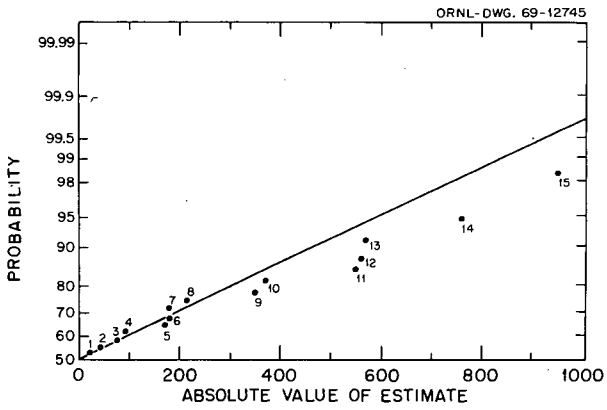

Fig. 4.1. Half-Normal Plot from $tRNA^{glu}$ Screening Experiment.

[7]W. G. Cochran and G. M. Cox, *Experimental Designs*, pp. 344–52, Wiley, New York, 1957.

of the third experiment, the levels of all the variables were well established except for some small doubt as to the interaction of pH with some of the other variables. A fourth experiment was a simple study of five buffers at four pH values. Results of the fourth experiment indicated that maximum aminoacylation can take place with any of three buffers and in each of two pH ranges. The fifth experiment was a central composite rotatable design with one buffer and one pH range; the other variables were concentrations of amino acid and enzyme. The optimum values of these variables were within the response surface. The final experiment will be a design to recheck several of the variables with each of the three buffers in each of the two pH ranges.

Table 4.5 gives the tentative conditions for the determination of $tRNA^{arg}$. With the six experiments described above, the total number of assays for each study will reach ~350; this number includes duplicates and a blank for each trial.

Table 4.4. Optimum Conditions for the Determination of $tRNA^{lys}$

Variable	Value
Final test-solution volume, ml	0.25
pH[a]	8.5
Buffer (HEPES[b]), μmoles	25
^{14}C-Lys, pmoles	375
Mg^{2+}, μmoles	4
ATP, μmole	0.9
K^+, μmoles	1.25
ME, μmoles	2.5
Enzyme, μg of protein	~50

[a]Measured at 23°C.
[b]*N*-2-Hydroxyethylpiperazine-*N'*-2-ethanesulfonic acid.

Table 4.5. Tentative Optimum Conditions for the Determination of $tRNA^{glu}$ and $tRNA^{arg}$

Variable	Value	
	$tRNA^{glu}$	$tRNA^{arg}$
pH[a]	6.5	7.2
Buffer, μmoles	25 (MES[b])	25 (HEPES)
^{14}C-Amino acid, pmoles	600	1000
Mg^{2+}, μmoles	2.5	5
ATP, μmole	1	1
K^+, μmoles	1.25	1.25
ME, μmoles	2.5	2.5
Enzyme, μg of protein	~50	~50
Time, min	30	30

[a]Measured at 23°C.
[b]2-(*N*-Morpholino)ethanesulfonic acid.

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

I. B. Rubin

One of the more recent tRNA's to be produced by the groups of the Chemical Technology Division working under the Macromolecular Separations Program has been formylmethionine-accepting tRNA ($tRNA^{fmet}$). Therefore, it has been necessary to ascertain the optimum conditions for the assay of this material. This $tRNA^{fmet}$ differs from normal methionine-accepting tRNA ($tRNA^{met}$) in that the free amino group of methionine can be enzymatically formylated when esterified with tRNA. The $tRNA^{fmet}$ is assayed by measuring formyl acceptance.

A scan of the literature revealed no definitive conditions that are used by a majority of the researchers to determine $tRNA^{fmet}$. A few preliminary experiments indicated that most of the conditions are the same as those for the determination of $tRNA^{met}$ by methionine acceptance.[8] In the determination of $tRNA^{fmet}$, the tRNA is first acylated with unlabeled methionine by means of met-tRNA synthetase, and the resulting met-$tRNA^{fmet}$ is formylated with ^{14}C-labeled N^5,N^{10}-methenyltetrahydrofolate (^{14}C-MTHF) as the formyl donor by means of fmet-tRNA transformylase. The preparation of the mixed synthetases normally used by the Bioanalytical Service Group did not contain a sufficient concentration of the transformylase to effect quantitative formylation. Therefore, a new enzyme solution was produced by processing *E. coli* B cells through a modification of the procedure described by Marcker[9] except that the protein was eluted in a single step rather than in two. The ^{14}C-MTHF was prepared by L. R. Shugart.[10] Since the concentration of ^{14}C-MTHF varied from batch to batch, a fixed ^{14}C count rate was used in each assay rather than a fixed volume of ^{14}C-MTHF solution. Calculations indicated that a minimum eight- to tenfold molar excess of ^{14}C-MTHF over $tRNA^{fmet}$ is necessary for maximum formylation.

[8]D. W. Hatcher, "Determination of Methionine-Accepting Transfer Ribonucleic Acid ($tRNA^{met}$)," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968,* ORNL-4343, p. 34.

[9]K. Marcker, "The Formation of *N*-Formyl-Methionyl-sRNA," *J. Mol. Biol.* 14, 63 (1965).

[10]Biology Division.

Table 4.6 lists the optimum conditions for the determination of $tRNA^{fmet}$. The blank count rate increases significantly with only several minutes of drying time, and this change in the blank value is not necessarily reflected in the sample count rate. Therefore, the filter-paper disks should be mounted individually and put into the wash solution within 15 to 20 sec after deposition of the sample. Also, the completeness of the "charging" is more dependent on $tRNA^{fmet}$ concentration than had been found for other tRNA's.

4.1.c Effect of Organic Compounds on Aminoacylation of Transfer Ribonucleic Acids (tRNA's)

I. B. Rubin

The samples of tRNA received by the Bioanalytical Service Laboratory have been processed through steps that require the use of a variety of organic reagents. Samples from the early processing stages may contain phenol, ethanol, or isopropanol. Samples from the reversed-phase chromatography columns may contain more than trace quantities of the quaternary amines that are used on these columns. They may also pick up minute quantities of various immiscible liquids used to prepare the columns or to act as antimold agents. Very little work has been done to establish what effects these, or other, organic compounds have on the aminoacylation of tRNA. Sarin and Zamecnik[11] examined the influence of several organic solvents on the aminoacylation of several tRNA's in a crude *E. coli* sRNA and found different effects for different solvents ranging from almost complete inhibition to 90% enhancement. Ritter, Kull, and Jacobson[12] reported results that suggest the occurrence of some interaction between solvent (dimethyl sulfoxide or ethanol) and buffer (Tris or cacodylate) in a heterologous system.

We have begun research to delineate the effects of a variety of organic compounds on all the tRNA's for which optimum conditions of assay have been defined. First, we are studying the effects of eight water-miscible organic liquids on several partly purified tRNA's. The tRNA solutions were provided by H. O. Weeren.[13] These tRNA's had been separated initially on a DEAE-cellulose column and purified further on a reversed-phase chromatography column. The solutions were dialyzed against 0.001 *M* EDTA of pH 7.5 and then against double-distilled water. The solvents were all reagent grade and were diluted with water to the desired concentrations. Our usual aminoacylation procedure[14] was used; the relative standard deviation of the assay as calculated from duplicates is ~3%. The data (Table 4.7) indicate that the various solvents have a wide range of effects. Inhibition at the higher solvent level is particularly evident in the determination of $tRNA^{lys}$. However, in the determination of $tRNA^{leu}$ or $tRNA^{val}$, there is little effect in most cases. Each of the immiscible solvents benzene, toluene, xylene, Freon 214, amyl acetate, and chloroform was equilibrated with a buffered salt solution, and the aqueous phases were tested with all the above tRNA's except $tRNA^{leu}$. None of the solvents had a significant effect on any tRNA.

Future work will include finding the limits of each of the above water-miscible solvents that can be tolerated in the determination of the various tRNA's. Particular attention will be given to determining what, if any, the effects of the various quaternary amines are on these assays.

Table 4.6. Optimum Conditions for the Determination of $tRNA^{fmet}$

Variable	Value
Final test-solution volume, ml	0.25
pH[a]	8.0
Buffer (HEPES), μmoles	25
^{14}C-MTHF, counts/min	~30,000
^{12}C-Methionine, pmoles	750
Mg^{2+}, μmoles	2.5
ATP, μmoles	1
K^+, μmoles	1.25
ME, μmoles	2.5
Enzyme, μg of protein	~50
tRNA, A_{260}/ml:	
Pure	0.7 to 1.4
Crude	2 to 16
Incubation time, min	30

[a]Measured at 23°C.

[11]P. S. Sarin and P. C. Zamecnik, "Modification of Amino Acid Acceptance and Transfer Capacity of S-RNA in the Present of Organic Solvents," *Biochem. Biophys. Res. Commun.* **19**, 198 (1965).

[12]P. O. Ritter, F. J. Kull, and K. B. Jacobson, "Effects of Tris and Dimethylsulfoxide on the Aminoacylation of *Escherichia coli* Valine Transfer RNA by *Neurospora crassa* Phenylalanyl Transfer RNA Synthetase," *Biochim. Biophys. Acta* **179**, 524 (1969).

[13]Chemical Technology Division.

[14]I. B. Rubin and G. Goldstein, "An Ultrasensitive Isotope Dilution Method for the Determination of L-Amino Acids," *Anal. Biochem.*, in press (1969).

Table 4.7. Effect of Organic Compounds on the Aminoacylation of Partly Purified tRNA's

Organic Compound		Aminoacylation (%)				
Identity	Amount in Test Solution (vol %)	$tRNA^{leu}$	$tRNA^{lys}$	$tRNA^{met}$	$tRNA^{phe}$	$tRNA^{val}$
(Control)		100	100	100	100	100
Ethanol	2	103	93	100	106	111
	10	104	17	103	120	95
Isopropanol	2	101	96	93	96	83
	10	110	17	108	104	97
Glycerol[a]	2	99	69	91	85	80
	10	93	28	62	45	98
Ethylene glycol	2	98	78	81	94	97
	10	96	34	61	63	94
Propylene glycol	2	99	92	90	95	103
	10	98	63	76	97	90
Dimethyl sulfoxide	2	106	107	97	99	90
	10	95	124	95	119	98
Ethylene glycol monomethyl ether[b]	2	106	106	99	127	91
	10	92	67	93	136	92
Pyridine	2	91	0	90	19	86
	10	0	0	0	0	0

[a]Quantity added with enzyme negligible.
[b]Also called Methyl Cellosolve.

4.1.d Selection of Staining and Operating Conditions for Gel Electrophoresis of Transfer Ribonucleic Acids (tRNA's)

A. L. Travaglini[15]

Preliminary studies[16] indicated that gel electrophoresis of tRNA's is feasible. A survey was made of numerous dyes and stains, and a solution of Pyronin Y (1 w/v %) and $KCr(SO_4)_2 \cdot 12H_2O$ (2 w/v %) in 15 vol % acetic acid was chosen for fixing and staining the tRNA bands. Electrolytic destaining does not remove the dye that is bound to the tRNA.

Studies of buffers and pH showed that good separations are obtained where Tris buffer at pH 8.9 is incorporated in the gel and the same buffer is in both electrode compartments.

[15]Reactor Projects Group.

[16]A. L. Travaglini, "Gel Electrophoresis of Ribonucleic Acid (RNA)," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968,* ORNL-4343, p. 38.

4.1.e Polyacrylamide Disk Gel Electrophoresis of Ribonucleic Acids (RNA's)

J. E. Caton

We have used disk gel electrophoresis to qualitatively assay both tRNA and rRNA. The experimental apparatus for this work was described by Travaglini.[17] All gels used were some variation of polyacrylamide; however, in general, tRNA assays are accomplished on 10% gels, and rRNA is assayed on gradient (2.5 to 12%) gels. Details of the experimental procedures will be given in a forthcoming report.

The electrophoresis of tRNA samples on 10% gels yields consistent and reproducible results. The photograph (Fig. 4.2) of several tRNA samples on 10% gels stained with Pyronin Y (Sect. 4.1.d) illustrates the typical sharp separation that is obtained. Table 4.8 shows the similarity of different low-molecular-weight

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

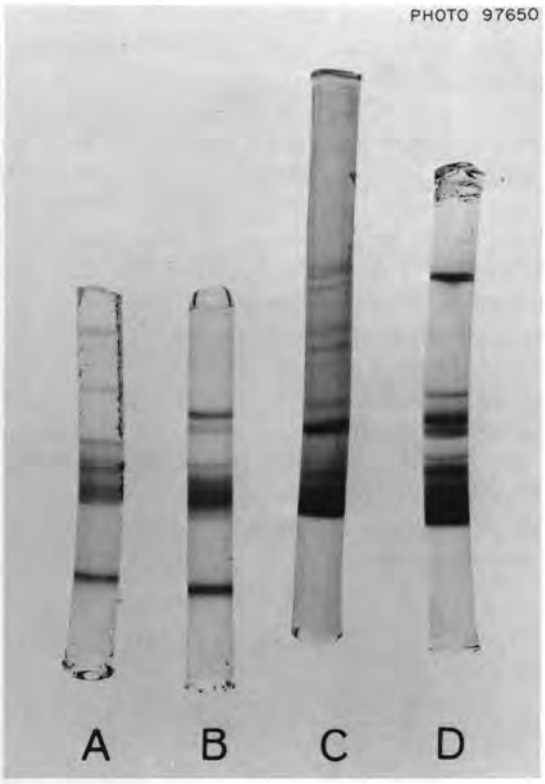

Fig. 4.2. Photograph of Electrophoretic Gel Disks of 10% Polyacrylamide on Which tRNA's from Different Samples Were Separated. Sources of samples are: yeast (A), rat liver (B), *B. subtilis* (C), and strain K-12 *E. coli* (D). Note the absence of a "front running" band in C and D.

RNA samples. Such results have been duplicated readily in many separate electrophoresis assays. The results of electrophoresis assays are recorded by scanning the stained gel on a densitometer; we use the Joyce-Loebl Chromoscan. Figure 4.3 shows typical scans that illustrate well the usefulness of disk gel electrophoresis as a qualitative assay for tRNA; two RNA samples from the same source (*E. coli* K-12) but prepared at different sites are compared. The samples are identical except that one contains an additional species of R_s = 0.53 (Table 4.8). The most intense band is thought to be that of 4-S tRNA. The sharp "front-running" band (R_s = 0.96) was probably an artifact; it has been eliminated in more recent experimental procedures (Fig. 4.2).

It may be possible to quantitatively determine the tRNA components in a sample by careful analysis of the scan of a stained gel. Figure 4.4 shows the variation of absorbance of the most intensely colored band (disk) of the developed gel as a function of the volume of the tRNA sample, *E. coli* K-12, obtained from General Biochemicals, Chagrin Falls, Ohio. The linear relationship indicates that Pyronin Y does stain at least one tRNA species in a reproducible manner. However, little work has been done on the quantitative aspects of disk gel electrophoresis. Thus, future studies should be devoted to such analyses.

Ribosomal RNA's (rRNA's) can also be characterized readily by disk gel electrophoresis. A gradient poly-

Table 4.8. R_s Values[a] of RNA's Isolated from Several Low-Molecular-Weight RNA Samples by Gel Disk Electrophoresis on 10% Polyacrylamide

Crude sRNA[b]	*E. coli*[b] B	*B. subtilis*[b] (6009)	Yeast[b]	Rat Liver[b]	*E. coli*[b] K-12	*E. coli* (ORNL) K-12 "MO"
0.40		c	0.41			
0.44						
0.48						
0.54	0.53	0.55		0.55	0.53	
	0.57		0.58	0.60	0.60	0.60
0.65 (?)	0.66 (?)				0.64, 0.65	0.63, 0.64
				0.69	0.69	0.68
0.72, 0.77	0.74			0.76	0.76	0.76
0.83, 0.87		0.92	0.84, 0.86, 0.92		0.87	0.88
0.98	0.96	0.98	0.98	0.98	0.96	0.96

[a]R_s is the ratio of the distance a given species traveled along the gel to the distance the solvent front traveled.

[b]Purchased from General Biochemicals. The source from which the RNA was extracted is indicated.

[c]*B. subtilis* has a broad band at R_s = 0.17.

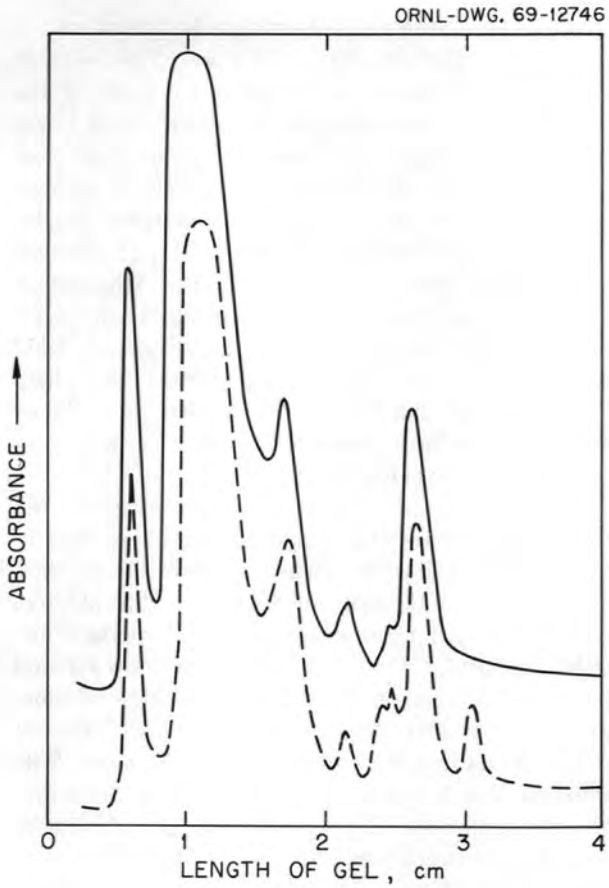

Fig. 4.3. Scans of Electrophoretic Gel Disks of 10% Polyacrylamide on Which tRNA's from *E. coli* K-12 Were Separated. (—, *E. coli* DEAE 77 from ORNL; - - -, *E. coli* from General Biochemicals.)

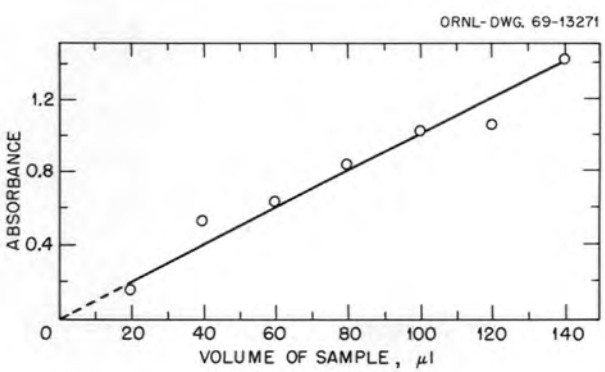

Fig. 4.4. Variation of Absorbance of Most Intensely Colored Band (Disk) with Volume of tRNA Sample. (Gel, 10% polyacrylamide; sample, *E. coli* K-12 from General Biochemicals.)

acrylamide gel is the best support. The gradient gels that have been used have a 2.5% polyacrylamide concentration at the top (sample-accepting end) of the gel and are 10 to 12% acrylamide at the bottom. Details for preparing these gels and for analyzing rRNA samples are recorded elsewhere. The bands are very sharp (Figs. 4.5 and 4.6). The 16-S, 18-S, 23-S, 28-S rRNA's are resolved easily. In addition, many other minor rRNA species can be separated; for example, those from rat-spleen rRNA (Fig. 4.5 and 4.6). The possibility of using gradient gels to quantitatively assay rRNA should be studied. Such a procedure would have many advantages. Gradient gels can be prepared readily in large quantities and stored satisfactorily for at least four months. Also, they are easier to handle and to preserve than 10% gels. They offer a possible means to the simultaneous assay for both tRNA and rRNA that justifies more study.

Fig. 4.5. Photograph of Electrophoretic Gradient Gel Disks of 2.5 → 12% Polyacrylamide on Which rRNA's from Different Samples Were Separated. [Sources of samples are: Q-13 strain *E. coli* (A), rat liver (B), mixture of Q-13 strain *E. coli* and rat liver (C), and rat spleen (D).]

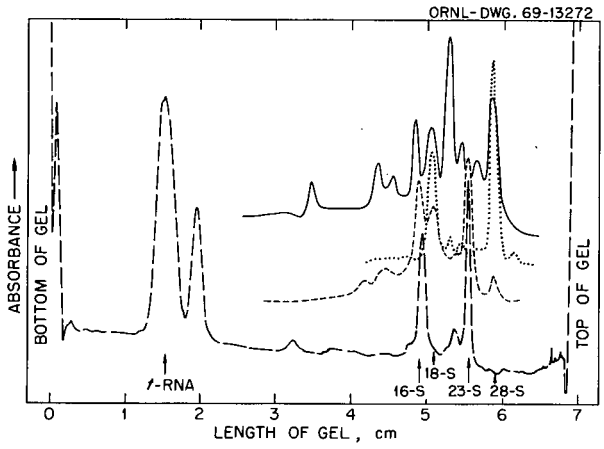

Fig. 4.6. Scans of Ribosomal RNA Separations on 2.5 → 12% Polyacrylamide Gradient Gels. — rRNA from *E. coli* plus tRNA; ---- rRNA from *E. coli* plus rRNA from rat liver; .... rRNA from rat liver; — rRNA from rat spleen.

4.1.f Nucleoside and Nucleotide Analysis with the High-Pressure UV Analyzer

J. E. Caton

A high-pressure UV Analyzer, Prototype Mark II, was built by the Instrumentation and Controls Division for our Division. This analyzer is a duplicate of those instruments in use in the Body Fluids Analyses Program. We are using it to separate, identify, and quantitatively measure nucleosides and nucleotides that result from the hydrolysis of RNA's. With such data one can do much toward characterizing a given RNA species.

For the nucleosides and nucleotides listed in Table 4.9, we have determined the elution characteristics under the elution conditions described by Pitt, Johnson, and Scott.[18] Also, we have chromatographed hydrolysates of several samples of different RNA's. We are now devising methods to determine base ratios, and possibly molecular weights, directly from data recorded by the monitoring of the chromatogram of a RNA hydrolysate. This work must still be regarded as being in the developmental stage, but the data for two very similar hydrolyzed tRNA samples (Table 4.10) are promising.

4.1.g Isolation of 310-nm-Absorbing Nucleosides from Transfer Ribonucleic Acid (tRNA)

J. C. Wolford[19] Gerald Goldstein

The ultraviolet absorption spectrum of crude tRNA from *E. coli* has a large peak at ~260 nm and a much smaller peak (~5% of the 260-nm absorption) at 310 to 340 nm depending on pH. Most of the 310-nm absorption is due to 4-thiouridine, a component of some tRNA's. However, our experience indicated that there must be at least one more contributing nucleoside. In our initial attempts to separate these nucleosides, recovery of 310-nm-absorbing material was low; this result suggests that the 310-nm-absorbing nucleosides are unstable. Consequently, we tested columns of the gel-filtration materials dextran and polystyrene beads. These materials are chemically inert and can be used at pH 7. Although used mainly as molecular sieves to separate large molecules of different sizes, they have also been used to separate small molecules by adsorption and weak ion-exchange effects. The nucleosides obtained by alkaline hydrolysis of tRNA and treatment of the hydrolysate with alkaline phosphatase are separated nicely on a 2.5- by 100-cm column of Sephadex G-10 by elution with water (Fig. 4.7). The four large off-scale peaks were found to result from the major ribonucleosides uridine, guanosine, cytidine, and adenosine, which eluted in that order. By simultaneously monitoring the effluent at 260 and 310 nm, it was possible to recover 90% of the 310-nm-absorbing nucleoside while separating it into three pools, as shown (Fig. 4.7). The material in pool C was then rechromatographed on a 1- by 30-cm column of Biogel P-2, also with water elution; the results are shown in Fig. 4.8. The 310-nm-absorbing component is completely separated from the 260-nm-absorbing material and was identified from its absorption spectrum as 4-thiouridine. The materials in pools A and B are not pure; work on them is continuing.

4.1.h Improved Methods for the Determination of Ribonucleic Acids (RNA's) and Deoxyribonucleic Acids (DNA's)

D. W. Hatcher Gerald Goldstein

An article was published[20] that describes improved methods of determining ribonucleic acids (RNA's) and deoxyribonucleic acids (DNA's); the summary from the article follows:

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[18] W. W. Pitt, W. F. Johnson, and C. D. Scott, *Operation Manual for the UV Analyzer, Prototype Mark II*, ORNL-TM-2593 (August 1969).

[19] Predoctoral Student Guest from the Graduate School of Chemistry, University of Tennessee, Knoxville.

[20] D. W. Hatcher and G. Goldstein, "Improved Methods for the Determination of RNA and DNA in Bacterial Extracts," *Anal. Biochem.* in press (1969).

Table 4.9. Elution Characteristics of Some Nucleotides and Nucleosides on the UV Analyzer

Compound	Elution Volume (ml)	Relative Elution Volume[a]	Peak Half-width[b] (ml)
Adenosine	154	37	7.5
2′-Deoxyadenosine	161	41	2.2
Adenosine-2′-monophosphate	580	134	8.3
Adenosine-3′-monophosphate	545	126	9.5
Adenosine-5′-monophosphate	482	117	7.6
Adenosine-5′-diphosphate	736	169	10.8
Adenosine-5′-triphosphate	947	218	16.9
Cytidine	38	9	3.4
2′-Deoxycytidine	35	8	3.9
Cytidine-2′-monophosphate	433	104	6.4
Cytidine-3′-monophosphate	418	101	6.4
Cytidine-5′-monophosphate	368	88	3.3
Cytidine-5′-diphosphate	593	143	6.0
Cytidine-5′-triphosphate	800	193	7.1
Guanosine	247	60	18.5
2′-Deoxyguanosine	230	55	22.5
6-Mercaptoguanosine	423	102	10.0
Guanosine-2′-monophosphate	615	150	8.6
Guanosine-3′-monophosphate	587	142	8.2
Guanosine-5′-monophosphate	571	137	7.7
Inosine	106	25	8.6
2′-Deoxyinosine	103	25	7.2
Inosine-6′-monophosphate	465	115	6.6
Pseudouridine B	32	8	3.3
Pseudouridine C	40	10	4.4
Pseudouridine-2′-monophosphate	420	101	7.0
Pseudouridine-3′-monophosphate	450	113	7.0
Thymidine	91	23	8.2
Thymidine-5′-diphosphate	720	182	9.4
Uridine	64	15	6.1
2′-Deoxyuridine	66	16	5.2
4-Thiouridine	350	84	5.4
Uridine-2′-monophosphate	510	121	8.9
Uridine-3′-monophosphate	501	119	5.3
Uridine-5′-monophosphate	419	105	5.7
Xanthosine	374	93	5.5
Xanthosine-5′-monophosphate	635	153	5.8
6-Thioxanthine	460	108	10.8

[a]This is the elution volume when a value of 100 is assigned to the difference between the actual elution volumes of guanosine-5′-monophosphate and adenosine, that is: relative elution volume = (100/difference) (measured elution volume).

[b]Peak half-width is the width of the peak at the peak height where the absorbance is equal to one-half the absorbance at the peak maximum.

"Nucleic acids (RNA and DNA) can be separated from extraneous materials by precipitation as their cadmium salts. This separation substantially improves the reliability of subsequent determinations of RNA by the orcinol method and of DNA by the diphenylamine method. In addition, color formation in the orcinol reaction is stabilized by addition of *n*-butanol to the reaction mixture thereby improving the precision of the method."

Table 4.10. Results of Analysis of Two Similar Samples of tRNA with the UV Analyzer

Component	Mole %	
	Sample 1	Sample 2
Cytidine	31.6	29.8
Uridine	14.7	13.8
Adenosine	20.3	22.5
Guanosine	29.4	29.2
4-Thiouridine	1	
42[a]	1.5	1.6
60[a]	0.6	0.3
90[a]	1	0.8
390[a]		0.6
412[a]		0.6
467[a]		0.5

[a]Elution volume (ml) of an unidentified trace component. Results are only approximations, because molar absorptivity was assumed to be 10,000 and molecular weight 300.

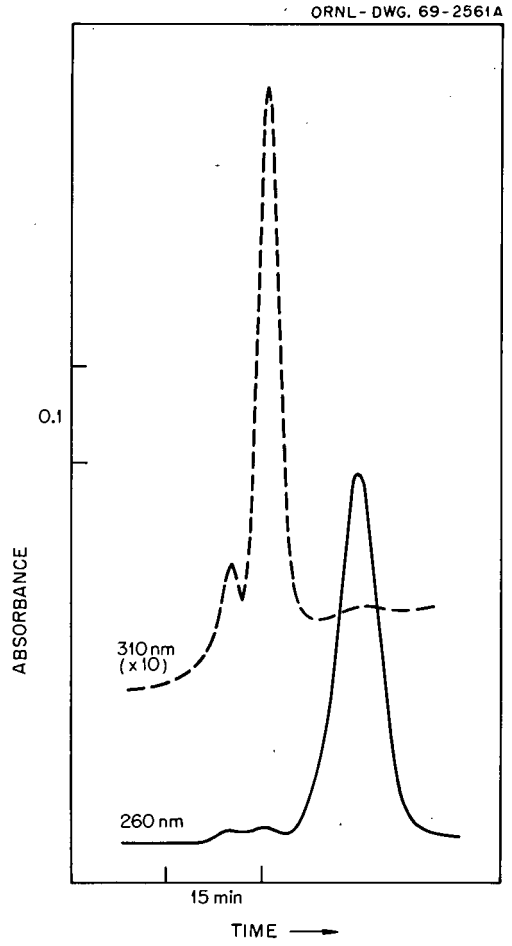


Fig. 4.8. Chromatograms Obtained on Rechromatography of Pool C on Biogel P-2.

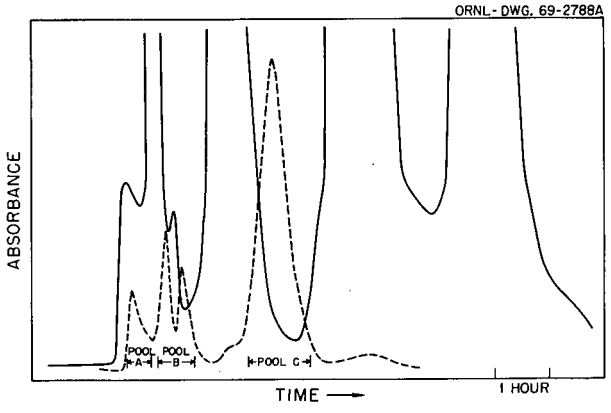


Fig. 4.7. Chromatogram of tRNA Nucleosides on Sephadex G-10, pH 7. (— 260 nm; --- 310 nm.)

4.1.i Gas Chromatography of Nucleosides and Nucleic Acid Bases

W. C. Butts

Because of the interest in identifying nucleic acid components, the gas chromatography of nucleosides and of the purine and pyrimidine bases is being investigated. In all cases, trimethylsilyl (TMS) derivatives have been formed prior to the gas chromatography. Fifteen bases have been chromatographed successfully; the MU values (Sect. 4.2.a) range from 13.30 (uracil) to 22.04 (kinetin). Many of these bases can be separated well (Fig. 4.9).

With the exception of cytidine, substituted cytidines, and unusual substituents (e.g., iodocompounds), the nucleosides also chromatograph well. As expected, the lower-molecular-weight deoxycompounds elute slightly

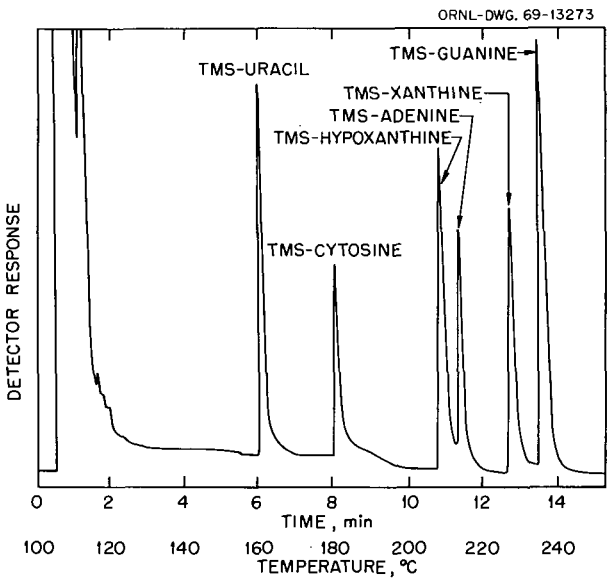


Fig. 4.9. Gas Chromatogram of the Trimethylsilyl Derivatives of Purine and Pyrimidine Bases. [Conditions: 6-ft by $\frac{1}{3}$-in.-OD column of OV-101 (5%) on 80/100-mesh Chromosorb W(HP); nitrogen flow, 90 cc/min; temperature programmed from 100 to 320°C at 10°C/min.]

earlier than the corresponding ribosides. Figure 4.10 shows the separation of the TMS derivatives of six ribonucleosides. Derivatives other than the TMS derivatives are being studied to improve the chromatographability of cytidines.

4.1.j Ultrasensitive Isotope-Dilution Method for the Determination of L-Amino Acids

I. B. Rubin Gerald Goldstein

Work to develop a procedure for the determination of subnanomole quantities of L-amino acids was completed.[21] An oral presentation of this work was given,[22] and a paper was accepted for publication.[14] The summary of the paper follows:

"A method for the determination of picomolar quantities of L-aminoacids is presented. No reaction occurs with D-amino acids. This procedure combines an isotope dilution technique with the standard aminoacylation method for the determination of tRNA. The method is highly specific for most of the common amino acids. As little as 50 pmoles of L-amino acid in the test portion of the sample can be detected, while the optimum range for quantitative determination is about 200 to 1500 pmoles. Accuracy is very good, and a relative standard deviation of ~5% is attained. The method is particularly useful when a few amino acid assays in a large number of samples are required; one analyst can perform at least 250 assays per day; the procedure is adaptable to semi-automation. Analyses of standard amino acid mixtures by this method and by ion-exchange chromatography give comparable results. This procedure has been used for the determination of L-amino acids in skin secretions, peptide hydrolyzates and DL-amino acid mixtures. Several additional applications are suggested."

[21] I. B. Rubin, "Determination of Subnanomole Quantities of L-Amino Acids by Isotope Dilution–Aminoacylation," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968,* ORNL-4343, p. 39.

[22] I. B. Rubin and G. Goldstein, "An Ultrasensitive Isotope Dilution Method for the Determination of L-Amino Acids," presented at the Southeastern Regional Meeting, American Chemical Society, Tallahassee, Fla., Dec. 4–7, 1968.

4.2 MOLECULAR ANATOMY (MAN) PROGRAM

4.2.a Identification of Urinary Constituents Collected from the UV Analyzer

W. C. Butts W. T. Rainey[24]
C. A. Burtis[23] S. Katz[13]

About 150 ultraviolet-absorbing components of urine have been resolved by high-pressure anion-exchange chromatography.[25] The principal methods used to

[23] Present address: Varian Aerograph, 2700 Mitchell Dr., Walnut Creek, Calif. 94598.

[24] Mass and Emission Spectrometry Development Group.

[25] C. D. Scott and S. Katz, *Body Fluids Analyses Program. Progress Report for the Period September 1, 1968 to February 28, 1969,* ORNL-TM-2551 (July 1969).

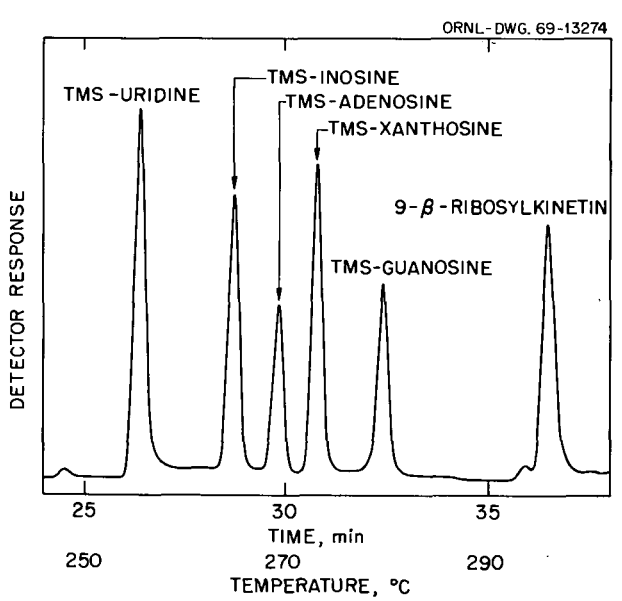


Fig. 4.10. Gas Chromatogram of the Trimethylsilyl Derivatives of Some Ribonucleosides. [Conditions: 6-ft by $\frac{1}{4}$-in.-OD column of SE-30 (5%) on 80/100-mesh Chromosorb W(HP); nitrogen flow, 90 cc/min; temperature programmed from 150 to 320°C at 4°/min.]

identify them are gas chromatography and mass spectrometry; the steps required are: (1) collection of a selected fraction from the UV Analyzer, (2) lyophilization of the fraction to remove the aqueous ammonium acetate–acetic acid buffer, (3) dissolution of the lyophilized residue in methanol and transfer to a 3-ml septum-capped vial (half of this solution is used for mass spectral analysis), (4) evaporation of the methanol under a stream of nitrogen, (5) derivatization of the residue by addition of 50 μl each of pyridine (solvent) and Regisil-TMCS (a commercial preparation of trimethylchlorosilane-catalyzed bistrimethylsilyltrifluoroacetamide, and (6) direct injection of a 2- to 5-μl aliquot of the reaction mixture into the gas chromatograph.

The trimethylsilyl derivatives of the urinary constituents are formed to increase their volatility and to allow their successful gas chromatography. A series of normal hydrocarbons is added to the sample mixture to permit the determination of accurate retention data expressed as methylene-unit (MU) values; a compound that elutes halfway between C_{22} and C_{24} hydrocarbons has an MU value of 23.00.

Figure 4.11 shows the gas chromatograms of a urinary constituent identified as pseudouridine and a sample of chemically synthesized pseudouridine to which C_{22} and C_{24} hydrocarbons have been added. The results are consistent with the facts that metabolic pseudouridine exists only as the β anomer and that the synthetic material is a mixture of the α and β forms. The agreement between the MU values for the urinary and reference samples (23.69 and 23.72) is typical of the precision obtained by determining retention data in this manner.

The mass spectra of 4-hydroxyacetanilide and 3-methoxy-4-hydroxy-acetanilide (Fig. 4.12) were obtained from UV Analyzer fractions collected from the urine of a patient who had been given large doses of 4-ethoxyacetanilide (phenacetin). Although reference spectra were not available for the two metabolites of the phenacetin, the spectra of the urinary samples agree with the proposed structures and are consistent with that of the parent compound for which the reference spectrum was available (top spectrum, Fig. 4.12). A

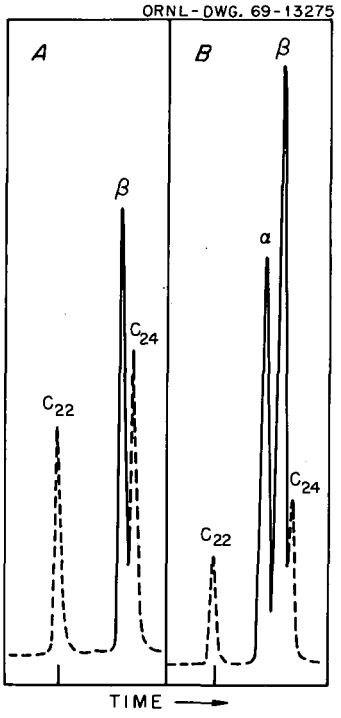


Fig. 4.11. Gas Chromatograms of Urinary (*A*) and Chemically Synthesized (*B*) Pseudouridine as Their TMS Derivatives.

paper on this work was presented orally[26] and has been accepted for publication.[27]

A total of 18 compounds have been identified by gas chromatography and mass spectrometry (Table 4.11), and 41 other compounds have been tentatively identified by these two methods, chemical testing, ultraviolet spectra, or co-chromatography on the UV Analyzer. At this time last year, these numbers were 3 and 8 respectively.[28]

4.2.b Identification of Urinary Carbohydrates Collected from the Carbohydrate Analyzer

W. C. Butts

A gas chromatographic procedure similar to that for identifying ultraviolet-absorbing constituents in urine is used to identify urinary sugars separated by high-

[26]C. A. Burtis, W. C. Butts, W. T. Rainey, Jr., and C. D. Scott, "Separation and Identification of Drug Metabolites in Physiologic Fluids by High-Resolution Anion Exchange Chromatography," presented at the First Annual Symposium on Automated, High-Resolution Analyses in the Clinical Laboratory, Oak Ridge, Tenn., Mar. 13–14, 1969.

[27]C. A. Burtis, W. C. Butts, W. T. Rainey, and C. D. Scott, "Separation of the Metabolites of Phenacetin in Urine by High-Resolution Anion Exchange Chromatography," *Am. J. Clin. Pathol.* in press (1969).

[28]C. A. Burtis and S. Katz, "Identification of Urinary Constituents Isolated by Anion-Exchange Chromatography," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968,* ORNL-4343, p. 43.

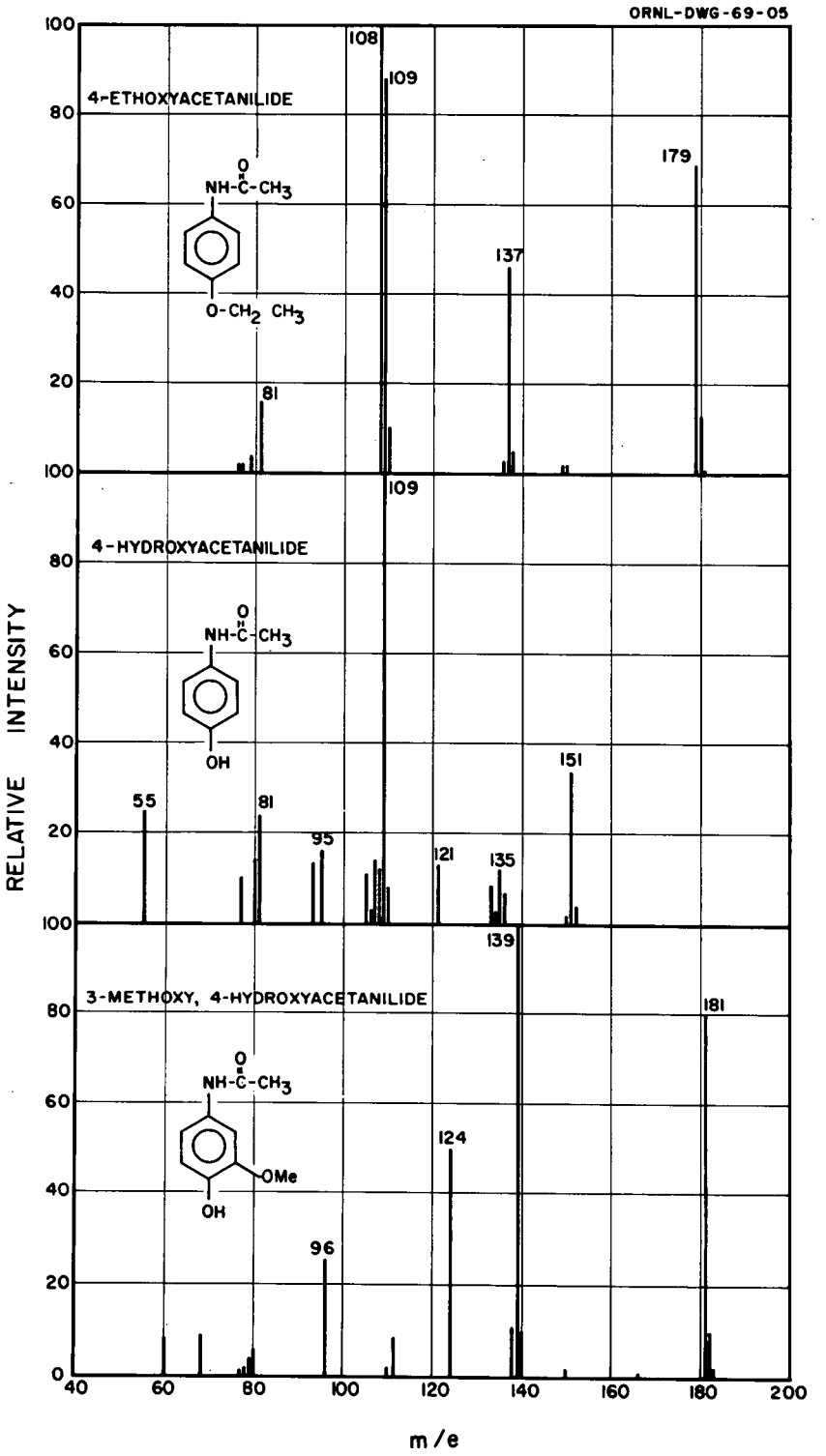


Fig. 4.12. Mass Spectra of 4-Ethoxyacetanilide (Phenacetin) and Its Urinary Metabolites.

pressure anion-exchange chromatography. The sodium borate–boric acid buffer used for the carbohydrates is removed by cation-exchange chromatography followed by vacuum distillation with methanol to remove the boric acid as methyl borate. Trimethylsilyl derivatives are formed with TRI-SIL-Z (a commercial preparation of trimethylsilylimidazole in pyridine).

Figure 4.13 is a carbohydrate-analyzer chromatogram of the carbohydrates in normal human urine. The nine carbohydrates (each from both reference and urinary examples) that have been identified are indicated in Fig. 4.13 and are listed in Table 4.12, along with the GC retention data.

Table 4.11. Urinary Constituents Identified by Gas Chromatography (GC) and/or Mass Spectrometry (MS)

5-Acetylamino-6-amino-3-methyluracil		MS
Allopurinol	GC	MS
Alloxanthine	GC	MS
Benzoic acid	GC	MS
Creatinine	GC	MS
3,4-Dihydroxyphenylpropionic acid		MS
Hippuric acid	GC	MS
Homovanillic acid	GC	MS
4-Hydroxyacetanilide	GC	MS
4-Hydroxyacetanilide glucuronide	GC	MS
2-Hydroxybenzoic acid		MS
4-Hydroxyphenylacetic acid	GC	MS
3-Methoxy-4-hydroxyacetanilide	GC	MS
3-Methoxy-4-hydroxyacetanilide glucuronide	GC	MS
3-Methoxy-4-hydroxymandelic acid	GC	MS
Pseudouridine	GC	
Uracil	GC	MS
Uric acid	GC	MS

The components associated with peaks *A* and *D* as well as with several small peaks in the chromatogram have not yet been identified. In addition, GC data indicate that at least three more compounds are present in the broad peak that corresponds to fructose, arabinose, and fucose. Additional reference data are necessary to establish the identity of the compounds that

Table 4.12. Gas-Chromatographic Retention Data for Reference and Urinary Carbohydrates

Carbohydrate	Source	MU Value		
		Peak 1	Peak 2	Peak 3
Arabinose	Reference	16.47	16.78	17.10
	Urinary	16.45	16.75	17.07
Fructose	Reference	18.60	18.71	
	Urinary	18.57	18.71	
Fucose	Reference	16.71	17.06	17.46
	Urinary	16.75	17.07	17.48
Galactose	Reference	18.68	19.11	19.53
	Urinary		19.08	19.55
Glucose	Reference	18.89	19.35	20.31
	Urinary		19.31	20.25
Lactose	Reference	27.34	28.60	
	Urinary	27.31	28.58	
Sorbose	Reference	18.41	19.09	20.00
	Urinary		19.16	20.00
Sucrose	Reference	27.46		
	Urinary	27.42		
Xylose	Reference	17.38	17.94	
	Urinary	17.44	18.00	

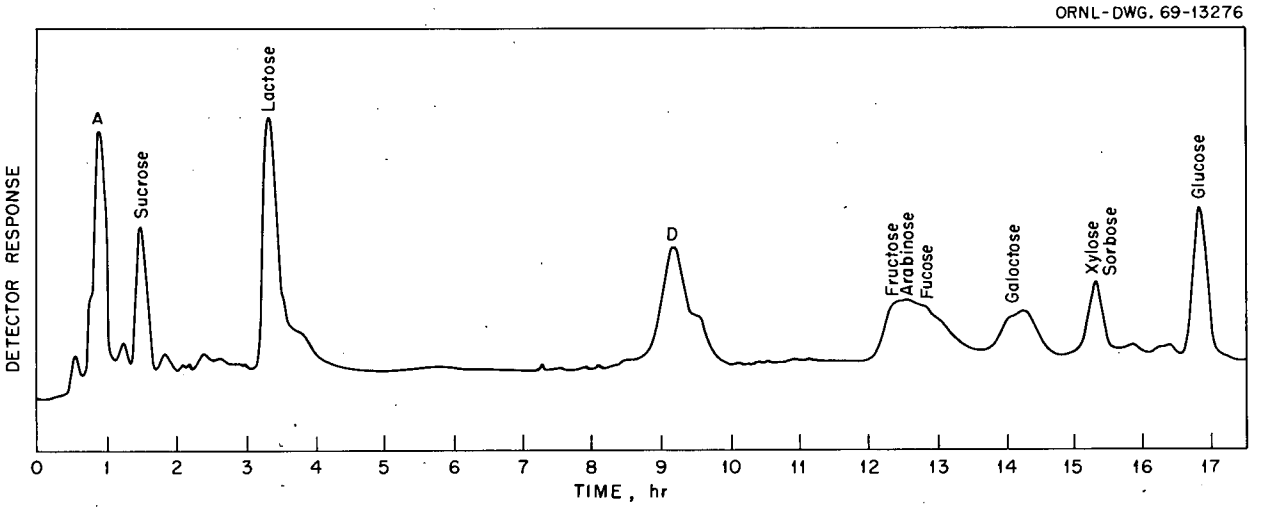


Fig. 4.13. ORNL Carbohydrate-Analyzer Chromatogram of Normal Human Urine.

cause peaks *A* and *D*. The GC retention data for the two peaks indicate that if the compounds are simple carbohydrates, that associated with *A* is a pentose and that with *D* is a hexose. Table 4.13 lists the GC retention data for the reference carbohydrates chromatographed to date. Multiple peaks occur because many sugars exist in more than one anomeric form. Nonreducing sugars (e.g., sucrose and raffinose), which cannot form anomers, yield only one peak.

4.2.c Computerized Data Handling for Identifications Programs

W. C. Butts

Four computer programs have been written to handle the data generated in the identification of compounds resolved by the two body-fluids analyzers. The programs are:

MS2: Input: Raw mass spectral data.

Output: Normalized mass spectral data, calculation of probable metastable transitions, and a plot of the mass spectrum. Figure 4.14 is a computer-plotted spectrum of a urinary constituent identified as uracil.

MULIST: Input: Alphabetized list of compound names and GC MU values.

Output: Same as input and a listing of MU values in increasing numerical order with associated compound name.

MW: Input: Alphabetized list of names of compounds reported to be in urine (currently 775 entries) with associated empirical formulas and indication of active hydrogens and glucuronide formation.

Output: Same as input plus associated molecular weight and a listing by increasing molecular weight with associated input

Table 4.13. Gas-Chromatographic Retention Data for Trimethylsilyl Derivatives of Carbohydrates

Carbohydrate	MU Value			
	Peak 1	Peak 2	Peak 3	Peak 4
Arabinose	16.47	16.78	17.10	
Ascorbic acid	20.00			
Cellobiose	27.90	28.87		
Deoxyribose	15.12			
Erythrose	14.21			
Fructose	18.60	18.71		
Fucose	16.71	17.06	17.46	
Galactosamine	18.84			
Galactose	18.68	19.11	19.53	
Gentiobiose	29.94			
Glucosamine	18.15	19.30		
Glucose	18.89	19.35	20.31	
Glucuronic acid	19.54	20.15	20.61	
Lactose	27.34	28.60		
Lyxose	16.44	16.86		
Maltose	27.45	27.81	28.17	
Mannoheptulose	21.36			
Mannosamine	18.48	19.27		
Mannose	18.57	19.58		
Melezitose	36.55			
Melibiose	29.68			
Methyl glucoside	19.11			
Methyl mannoside	18.17			
Methyl xyloside	16.75			
Raffinose	3560			
Rhamnose	16.60	17.32		
Ribose	16.88	17.04		
Ribulose	16.50	16.70	17.11	17.70
Salicin	25.65			
Sedoheptulose	19.54			
Sorbose	18.41	19.09	20.00	
Sucrose	27.46			
Trehalose	28.44			
Turanose	28.10			
Xylose	17.38	17.94		

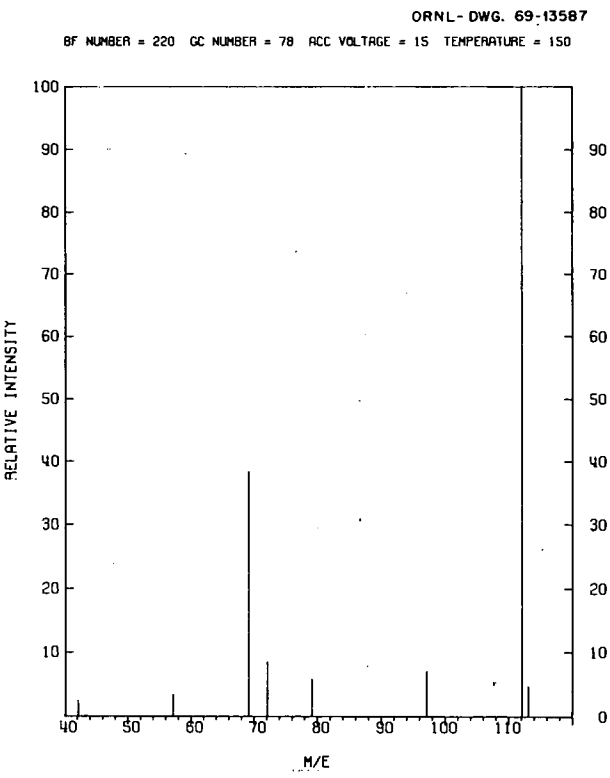


Fig. 4.14. Computer-Plotted Mass Spectrum of a Urinary Constituent Identified as Uracil (mol wt 112).

data. This second listing contains additions for possible glucuronide formation and trimethylsilyl-derivative formation.

DATA: Input: Sample number of fraction collected from analyzer, MU values for as many as four peaks, wavelengths of maximum and minimum ultraviolet absorption, elution time from analyzer, elution time of uric acid in same run, two possible parent peaks from mass spectrum, three most intense masses in mass spectrum, and name of compound if identified.

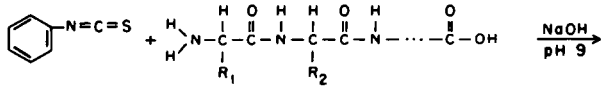
Output: Same as input plus elution time relative to uric acid. Also, a listing in increasing order of any of the input variables can be called.

Several of these programs are, of course, being used for projects other than body-fluids analyses.

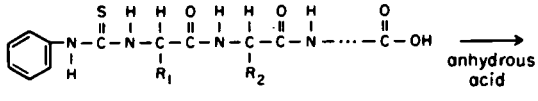
4.2.d Automatic Amino Acid Sequencing

J. E. Attrill

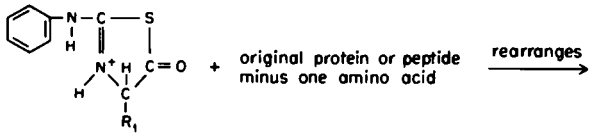
An automatic amino acid sequenator based on the same principal as one built by Edman and Begg[29] has been built for use in the Molecular Anatomy (MAN) Program. This instrument will automatically determine the sequence of the amino acids in a protein or peptide by a chemical reaction sequence with phenylisothiocyanate in which the *N*-terminal amino acid of the protein is removed and identified; the reaction sequence is



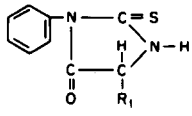
phenylisothiocynate + a protein or peptide



an addition product



phenylthiazolone amino acid (unstable)



a phenylthiohydantoin amino acid

When the formation of the phenylthiohydantoin (PTH) of the *N*-terminal amino acid is complete, the derivative is extracted from the remaining protein and identified, and the cycle is repeated. We have spent some time in testing and reviewing several thin-layer and paper chromatographic methods for determining these PTH derivatives. We obtained excellent results with the method developed by Sjöquist.[30,31]

4.2.d.a Methylthiohydantoin (MTH) Derivatives of Amino Acids. – A number of people[32-34] have reported difficulty in obtaining satisfactory results using the PTH method of sequencing because of the relative insolubility of the compounds formed and their low volatility. In anticipation of this problem, we are making experiments based on the suggestion that methylisothiocyanate be used in place of the phenyl compound thus yielding compounds of smaller size with greater solubility and volatility. The methylthiohydantoin (MTH) derivatives of 16 amino acids have been synthesized, and their separation by gas chromatography is being studied (Sect. 4.2.e). We plan to use this reagent and a gas chromatograph for on-line

[29] P. Edman and G. Begg, "A Protein Sequenator," *Eur. J. Biochem.* 1, 80 (1967).

[30] J. Sjöquist, "Paper Strip Identification of Phenyl Thiohydantoins," *Acta Chem. Scand.* 7, 447 (1953).

[31] J. Sjöquist, "Determination of Amino Acids as Phenyl Thiohydantoin Derivatives. III. Quantitative Determination of 3-Phenyl-2-Thiohydantoin from Paper Chromatograms," *Biochim. Biophys. Acta* 41, 20 (1960).

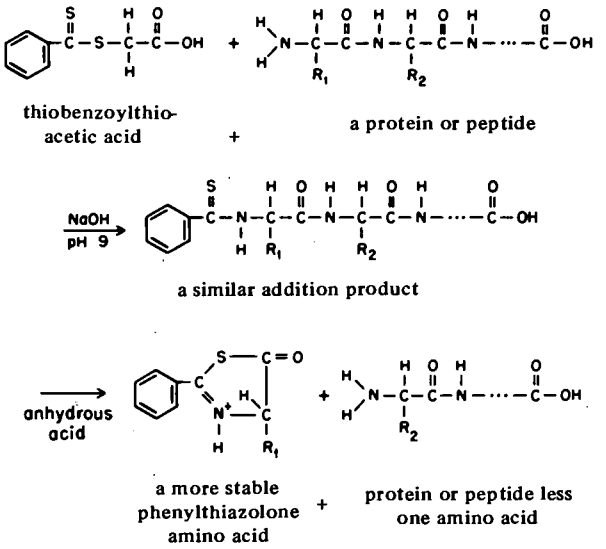
[32] J. T. Potts, A. Berger, J. Cooke, and C. B. Anfinsen, "A Reinvestigation of the Sequence of Residues 11 to 18 in Bovine Pancreatic Ribonuclease," *J. Biol. Chem.* 237, 1851 (1962).

[33] D. G. Smyth, W. H. Stein, and S. Moore, "On the Sequence of Residues 11 to 18 in Bovine Pancreatic Ribonuclease," *J. Biol. Chem.* 237, 1845 (1962).

[34] V. M. Stepanov and V. F. Krivtsov, "2-Methyl-2-Thiohydantoins of Amino Acids 1. Synthesis and Properties of 2-Methyl-2-Thiohydantoins of Some Aliphatic Amino Acids, Phenylalanine, and Tyrosine," *Zh. Obshch. Khim.* 35, 53 (1965).

identification of sequenced amino acids. Some work on the paper and thin-layer chromatography of these compounds has also been started.

4.2.d.b Phenylthiazolone Derivatives of Amino Acids. – Another possible reagent for sequencing amino acids in protein has been suggested by J. W. Holleman,[35] namely, thiobenzoylthioacetic acid. It is known to react with the *N*-terminal amino acid of a protein in a manner similar to the reaction of phenylisothiocyanate;[36,37] the reaction sequence is



The reaction stops with the phenylthiazolone; the product does not rearrange to the thiohydantoin. Use of thiobenzoylthioacetic acid should then shorten the reaction time required in the sequencing and yield a stable, soluble, and relatively volatile compound. We are now preparing thiobenzoylamino acids according to the methods of Holmberg,[36] of Barrett,[37] and of Kurzer[38] and are characterizing the acids by mass spectrometry and gas chromatography. When all mechanical problems with the sequenator are eliminated, we hope to develop a method based on the use of thiobenzoylthioacetic acid as a sequenating reagent.

[35] Molecular Anatomy (MAN) Program.

[36] B. Holmberg, "Thiobenzoylamino Acids," pp. 299–308 in The Svedberg Memorial Volume, ed. by A. W. Tiselius, Almqvist and Wiksells, Uppsala, Sweden, 1944 (ORNL-tr-2060).

[37] G. C. Barrett, "Cleavage of *N*-Thiobenzoyl-dipeptides with Trifluoroacetic Acid: the Basis of a New Stepwise Degradation of Polypeptides," *Chem. Commun.* No. 10, 487 (1967).

[38] F. Kurzer, "Thioacylation with (Thiobenzoylthio)-Acetic Acid," *Chem. Ind. (London)* 1333 (1961).

4.2.e Gas Chromatography of Amino Acid Derivatives

W. C. Butts G. L. Bangs[39]

In conjunction with the development of an amino acid sequenator (Sect. 4.2.d), J. E. Attrill has prepared methylthiohydantoin (MTH) derivatives (Fig. 4.15) of the 19 amino acids listed in Table 4.14. W. T. Rainey[24] has confirmed the structures of these compounds by mass spectrometry. Initially, the sequenator will be used to split off the protein amino acids as MTH derivatives. Gas chromatography is expected to be one method of identifying the isolated amino acids. Table 4.14 lists MU values for 16 of the MTH derivatives on two GC columns, one coated with nonpolar SE-30 and the other with polar OV-17, both silicone gums. Unique retention values are obtained for the MTH derivatives of the 16 amino acids on the pair of columns. Figure 4.16 is a chromatogram of a mixture of these 16 MTH-amino acids on SE-30; threonine-alanine (THR-ALA) and

[39] ORAU Student Trainee from Arkansas Polytechnic College, Russellville.

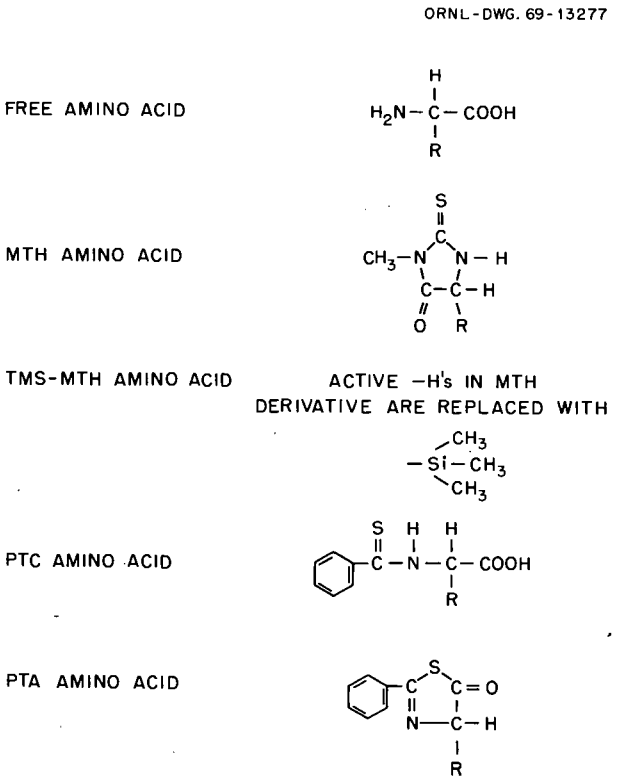


Fig. 4.15. Formulas for Several Types of Amino Acid Derivatives.

Table 4.14. Chromatographic Retention Data for Several Amino Acid Derivatives

Amino Acid	MU Values			
	3% OV-17 Column	5% SE-30 Column		
	MTH[a]	MTH[a]	MTH-TMS[b]	PTC[c]
Glycine	19.08	14.63	17.68	15.46
Valine	18.68	15.12	15.72	
Proline	19.95	15.49	15.76	18.53
Threonine	19.64	15.75	16.61	
Alanine	20.82	15.81		16.94
Isoleucine	19.78	16.14	16.70	17.60
Leucine	19.81	16.26	16.74	
Glutamic acid	23.70	17.80	20.36	20.63
Methionine	23.76	18.88	19.16	
Phenylalanine	24.88	19.61	20.20	21.78
Asparagine	26.35	21.28	20.51	
Glutamine	28.00	22.00	21.72	
Lysine	29.98	23.07	23.48	
Tyrosine	29.47	23.17		24.89
Histidine	30.16	23.78	22.85	
Tryptophan	34.25	26.77	26.70	
Serine	(no peak)	(no peak)	17.30	
Aspartic acid	(no peak)	(no peak)	19.05	
Arginine	(no peak)	(no peak)	24.93	

[a]MTH = methylthiohydantoin derivative.
[b]TMS = trimethylsilyl derivative.
[c]PTC = phenylthiocarboxy derivative.

lysine-tyrosine (LYS-TYR) pairs that are not resolved on this column are well resolved on OV-17. The MTH derivatives of serine, aspartic acid, and arginine are apparently unstable under the chromatographic conditions used; the three give a common peak that occurs early in the chromatogram.

Silylation of the MTH derivatives of these three amino acids gives products with good GC properties. The MTH derivatives of several other amino acids, especially asparagine and glutamine, chromatographed significantly better as trimethylsilyl (TMS) derivatives. Table 4.14 lists MU values for the TMS-MTH derivatives of 17 amino acids; MTH-alanine and MTH-tyrosine were not available for this study.

Phenylthiazol-5-one (PTA) derivatives of the amino acids (Fig. 4.15) are also being considered for use with the sequenator. These derivatives have not yet been prepared by J. E. Attrill, but his early attempts to make them produced instead phenylthiocarboxy (PTC) derivatives of amino acids (Fig. 4.15), which can be gas chromatographed (Table 4.14). Silylation of the PTC derivatives would undoubtedly improve their GC characteristics because of the silylation of the free carboxy group.

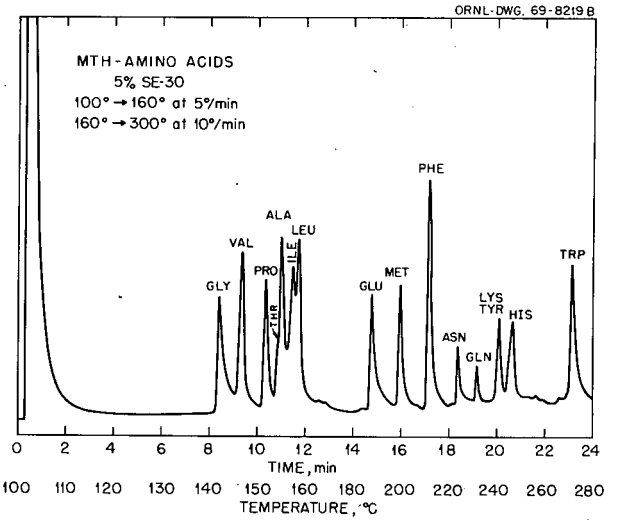


Fig. 4.16. Gas Chromatogram of a Mixture of the Methylthiohydantoin Derivatives of 16 Amino Acids.

Work with the phenylthiohydantoin derivatives of amino acids is also discussed in Sect. 2.2.c.c.

4.2.f GeMSAEC Fast Analyzer: A New Analytical Tool for Clinical Chemistry

D. W. Hatcher

The development by N. G. Anderson of a multiple-cuvette rotor – the GeMSAEC Fast Analyzer – for a new microanalytical system was reported recently.[40] Versions of this instrument are now available from Union Carbide Corporation as the CentrifiChem System and from Electro-Nucleonics as the GeMSAEC.

We have developed procedures for its clinical use and will develop procedures for its nonclinical use as well (Sect. 2.6).

4.2.f.a Design. – The GeMSAEC consists of a flat circular rotor with a number of cuvette-type chambers on the perimeter. The chambers are formed by compressing a 1-cm-thick X 6-in.-OD X 4.5-in.-ID annulus of Teflon between a glass annulus of the same dimension and a 1.25-cm-thick glass disk. The Teflon annulus has a number of indentations on its inner perimeter that lead to the chambers. The three parts are bolted together between two stainless steel flanges. A unique spectrophotometer is made by spinning this rotor between a fixed light source and a fixed photomultiplier tube. The electrical signal from the photo-

[40]N. G. Anderson, "Analytical Techniques for Cell Fractions. XII. A Multiple-Cuvet Rotor for a New Microanalytical System," *Anal. Biochem.* 28, 545 (1969).

multiplier tube can be monitored by an oscilloscope, when synchronized appropriately, or by a computer. Each rotation provides all the basic data required for quantitation, including dark-current reading (zero transmittance or infinite absorbance of the metal flanges between cuvettes), a water blank in cuvette number one as a reference (assigned 100% transmittance or zero absorbance), and signals that represent values between these extremes and that correspond to the transmittance by the other cuvettes.

4.2.f.b Rapid Determination of Total Serum Protein by the Biuret Reaction. – An article was published[41,42] that describes the adaptation of the biuret reaction for determining serum protein with the GeMSAEC Fast Analyzer; the summary from the article follows:

"A method is described for determining total serum protein using a new and very fast microanalytical system in which quantification depends upon the color developed during the first few seconds of the biuret reaction. The method is in excellent agreement with Kjeldahl nitrogen analysis for determining serum protein."

4.2.f.c Rapid Determination of Total Serum Cholesterol. – A paper was presented[43] that described the rapid determination of serum cholesterol with the GeMSAEC Fast Analyzer; the summary from the paper follows.

"Total serum cholesterol can be measured accurately between 25 and 45 seconds after initiating the reaction between cholesterol, ferric ions, and strong mineral acid. Sera samples (5–15 μl) are mixed with reagent solutions (0.4 ml total volume) in the peripheral cuvettes of the rotor in the GeMSAEC Fast Analyzer.

"Several modes of acquiring and handling the data are presented in detail. The simplest requires only an oscilloscope and camera. A PDP-8/I computer on line with the Fast Analyzer reduces the analysis time for 14 sera samples (or samples and standards) to five minutes, three to four minutes of which are involved in pipetting the sera samples."

4.2.f.d An Optical-Density Thermometer for Measuring Rotor Temperature. – The problem of measuring rotor temperature either in or near the sample cell of an analytical ultracentrifuge has been considered repeatedly,[44,45] but no simple solution was found. The same problem was encountered with the GeMSAEC Fast Analyzer rotor.

We devised a convenient means of measuring accurately the temperature of a rotor and published an article[45] that describes it; the summary from the article follows:

"An optical density thermometer is described for determining the temperature of centrifuge rotors. Quantification is based on the change in the concentration of the vapor phase of a volatile substance, such as bromine, with temperature. Centrifugal force holds the liquid phase away from the light path and removes condensation from the optical windows."

4.3 CHARACTERIZATION AND PURITY OF ORGANIC COMPOUNDS

4.3.a Clinical Standards

Gerald Goldstein J. C. White

On behalf of the Clinical Chemistry Task Force Committee, National Committee for Clinical Laboratory Standards, we have analyzed five different inorganic salts, six samples of urea from different manufacturers, and one sample each of uric acid, creatinine, and cholesterol. The Committee is attempting to establish specifications for standard materials for clinical laboratories and will use this information as background to show what is commercially feasible. The analytical techniques used included emission spectrography; gas chromatography; and infrared, NMR, and mass spectrometry.

The five inorganic compounds NaCl, KCl, $CaCO_3$, Li_2CO_3, and MgO, which were supplied by a reputable manufacturer of analytical-grade reagents, were found to be of unusually high purity, probably 99.9+%. The samples of urea differed in purity and were all easily distinguishable. The poorest (~97% pure) contained nickel (1000 ppm) and detectable degradation products of urea; the best appeared to be ~99.3% pure. Of the other organic compounds, the sodium salt of uric acid was detected in uric acid, and creatine in creatinine, but no quantitative estimate of these impurities was made. Compared with NBS-standard cholesterol (certified as 99.4 ± 0.3%), the clinical-standard cholesterol had an impurity amounting to ~0.5%, as assayed by gas chromatography.

[41] D. W. Hatcher and N. G. Anderson, "GeMSAEC: A New Analytical Tool for Clinical Chemistry. Total Serum Protein with the Biuret Reaction," *Am. J. Clin. Pathol.* in press (1969).

[42] D. W. Hatcher and N. G. Anderson, "GeMSAEC: A New Analytical Tool for Clinical Chemistry. Total Serum Protein with the Biuret Reaction," as a detailed abstract of the paper, *J. Am. Med. Assoc.* in press (1969).

[43] D. W. Hatcher, "Total Serum Cholesterol: Adaptation to the GeMSAEC Approach," presented at the 13th Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Sept. 30–Oct. 2, 1969.

[44] L. Gropper and W. Boyd, "Temperature Measurement and Control of Analytical Rotors in the Ultracentrifuge," *Anal. Biochem.* 11, 238 (1965).

[45] D. W. Hatcher and N. G. Anderson, "An Optical Density Thermometer," *Anal. Letters* 2, 373 (1969).

4.3.b Chemical Mutagenesis Program

Gerald Goldstein

Many of the compounds used in the Chemical Mutagenesis Program in the Biology Division are not available commercially but are synthesized by individuals or received as gifts. If the compound seems interesting, the concerned investigator would then like analytical information about the identity of the compound, its purity, and its stability under normal storage conditions. To date, we have examined one compound, *N*-acetoxy-2-acetylaminofluorene (*N*-acetoxy-AAF), rather extensively by infrared, NMR, and mass spectrometry and by thin-layer chromatography and have done preliminary work on a complex acridine derivative known as ICR-170.

Our analyses confirmed that the compound labeled *N*-acetoxy-AAF was indeed *N*-acetoxy-AAF. No impurities were observed by infrared, NMR, or mass spectrometry, which results indicated that the sample was at least 95% pure. However, several spots were detected by thin-layer chromatography (Sect. 2.3.c.c).

4.3.c National Academy of Sciences–National Research Council (NAS-NRC) Specifications and Criteria for Biochemical Compounds

Gerald Goldstein

In an effort to improve the quality of chemicals available for biochemical research by establishing criteria, standards, and specifications with regard to purity, the Committee on Biological Chemistry, National Academy of Sciences–National Research Council (NAS-NRC) established a number of subcommittees to periodically assess the purity of commercially available biochemicals. The reports of these subcommittees are published.[46] On behalf of the Subcommittee on Nucleotides and Related Compounds, the Analytical Chemistry Division has agreed to evaluate 58 compounds in preparation for the third edition of this publication.

As a quantitative measure of impurities, the Subcommittee recommends two procedures – paper chromatography and/or paper electrophoresis in four specified solvents and spectrophotometric analysis. If a sample shows no visible impurities (ultraviolet illumination) on paper chromatograms run under specified conditions *and* its molar absorptivity at a specified wavelength in a specified medium agrees to within ±3% with a value given in the publication, then the sample is said to meet the NAS-NRC specifications. In addition to these two tests, we are also obtaining infrared and NMR spectra on all the compounds and gas chromatograms and mass spectra on those compounds that are sufficiently volatile or have volatile derivatives. Compounds that appear to meet NAS-NRC specifications are also analyzed for water by Karl Fischer titration and for trace metallic contaminants by emission spectroscopy. To date, we have reported our results on eight compounds; work is almost completed on 15 others.

Analytically speaking, probably the most significant outcome of this work is the use of gas chromatography to separate and determine TMS derivatives of purine and pyrimidine bases and of nucleosides. Gas chromatography is the best single method of analysis for many of the compounds. Good separation can be made easily (Sect. 4.1.i), and quantitative methods can undoubtedly be developed. An example of the gas-chromatographic separation of TMS derivatives is given in Sect. 4.3.d.

4.3.d Gas Chromatographic Analysis of NAS-NRC Biochemicals

W. C. Butts

In conjunction with the NAS-NRC program on Specifications and Criteria for Biochemical Compounds, trimethylsilyl (TMS) derivatives of eight bases, eight ribonucleosides, and seven deoxyribonucleosides were gas chromatographed to determine their purity. Three of the deoxy compounds were cytidine derivatives and were not sufficiently stable to be gas chromatographed; apparent decomposition was also encountered with 5-isouridine. With the exception of 2-dimethylamino-6-hydroxypurine, which apparently contains two impurities that total 30%, the remaining compounds contained 0.0 to 5.0% impurities. Figure 4.17 shows the chromatogram of the derivatized sample of uridine, which contained 1.0% uracil and unidentified contaminants *A* (2.4%) and *B* (1.6%).

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[46] Committee on Biological Chemistry, Division of Chemistry and Chemical Technology, National Academy of Sciences, and the National Research Council, *Specifications and Criteria for Biochemical Compounds,* 2d ed., National Academy of Sciences–National Research Council, Washington, D.C., 1967 (Publication 1344).

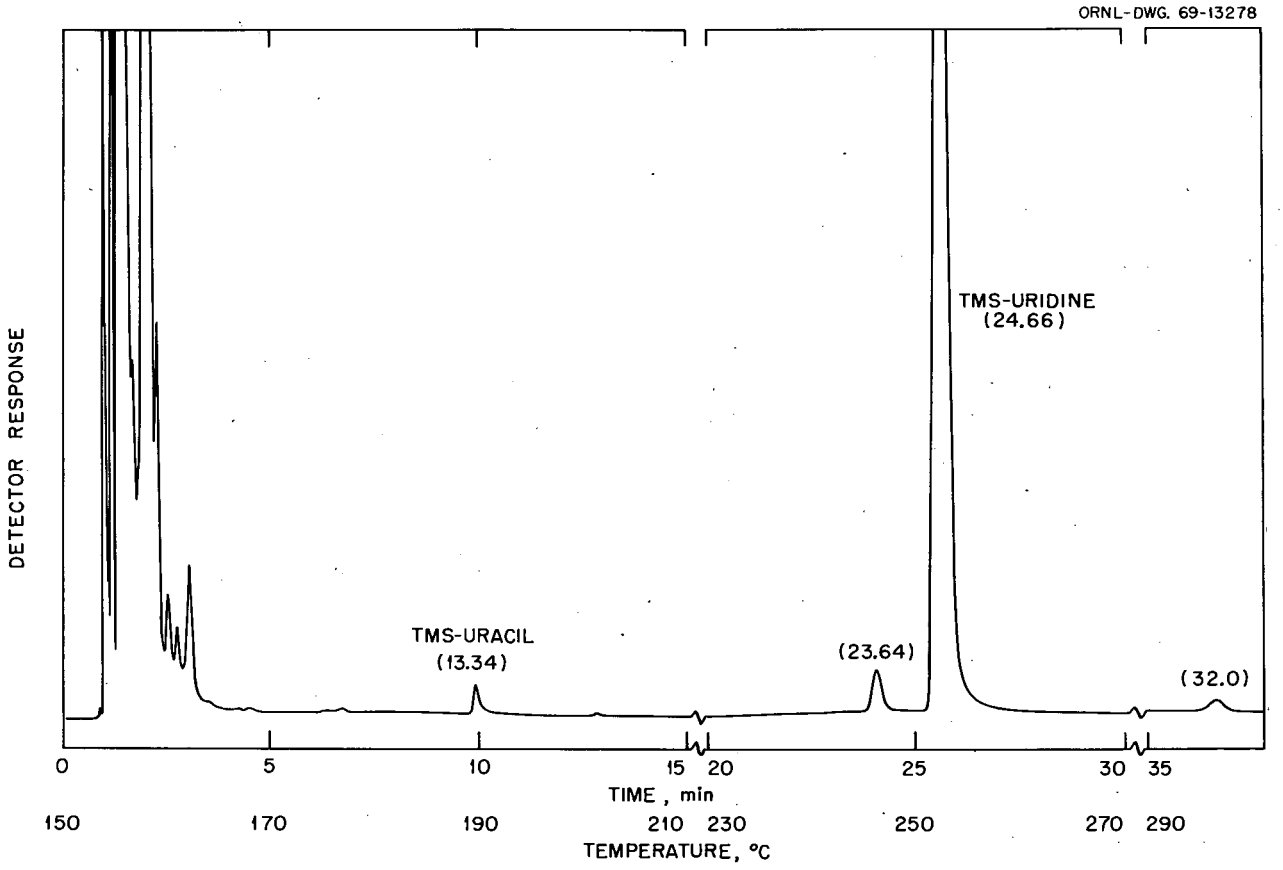

Fig. 4.17. Gas Chromatogram of a Trimethylsilyl (TMS) Derivative of Commercially Prepared Uridine. [Conditions: 6-ft by ¼-in.-OD column of SE-30 (5%) on 80/100-mesh Chromosorb W(HP); nitrogen flow, 90 cc/min; temperature programmed from 150 to 320°C at 4°C/min.]

5. Nuclear and Radiochemistry

W. S. Lyon
H. H. Ross

5.1 ACTIVATION ANALYSIS

5.1.a Analysis of Drug Samples

L. C. Bate F. F. Dyer J. F. Emery

The program to measure trace elements in drugs was completed.[1] The purpose was to explore the use of trace elements as a means to distinguish drugs from different sources (e.g., lot, manufacturer). Samples (138) of barbiturates and amphetamines and their chemical derivatives, collected from 18 manufacturers by the Federal Bureau of Drug Abuse Control, were analyzed. Table 5.1 lists the 13 elements and their concentration ranges found in most of the drugs. Of the elements listed, those least often present were Th, Sb, Br, and Cd; in a few samples, Cu, Au, Sr, Ag, and Fe were not found. No correlation was found between a

[1] L. C. Bate, "Trace Elements in Drugs," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968,* ORNL-4343, p. 66.

Table 5.1. Trace Elements Detected in Drugs

Element	Concentration Range (μg/g)
Zn	1.8×10^{-1} to 3.2
Sr	7.8×10^{-3} to 8×10^{-2}
Ag	6.2×10^{-3} to 3.4×10^{-1}
Hg	2.1×10^{-3} to 5.5×10^{-1}
Sb	3.5×10^{-4} to 5.2×10^{-2}
Sc	1.3×10^{-4} to 1.9×10^{-3}
Br	8.9×10^{-2} to 3.2
Cs	2.3×10^{-4} to 1.4×10^{-2}
Au	3.3×10^{-4} to 6.4×10^{-2}
Cr	2.5×10^{-1} to 2.7
Fe	1.1 to 41
Co	1.3×10^{-3} to 5.0×10^{-2}
Th	9.2×10^{-4} to 1.4×10^{-1}

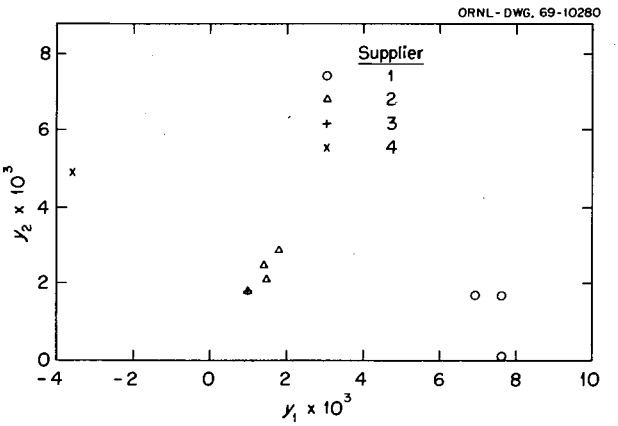

Fig. 5.1. Cluster Diagram for Trace Elements in D-Desoxyephydrine Hydrochloride.

drug supplier and the presence or absence of any element.

Replicate samples of a specific drug from a particular supplier were seldom available. A limited statistical analysis of the results was made only for D-desoxyephydrine hydrochloride, D,L-desoxyephydrine hydrochloride, and sodium pentobarbital. Two statistical tests were made. The first consisted in a one-way analysis of variance (ANOVA) to compare the between-supplier concentration variances with the within-supplier (among lots) concentration variances. F statistics (variance ratios) calculated for each element (taken separately) indicated that Br, Mg, and Sb might be useful in distinguishing suppliers. The F ratios were not significant for other elements. The second test was made by cluster analysis, a new statistical procedure being developed by the ORNL Statistics Group. This technique consists in forming two vectors, y_1 and y_2, as linear functions of n trace-element concentrations, that is,

$$y_1 = \sum_1^n a_i x_i ,$$

$$y_2 = \sum_1^n b_i x_i ,$$

where x_i is the concentrations of the ith element in a sample and a_i and b_i are linear coefficients. Each sample yields values for y_1 and y_2 that can be entered as a point on a two-dimensional plot. The plot is called a cluster diagram. If significant differences in the trace-element concentrations exist among the drugs from various suppliers, the points on the plot will cluster into a separate area for each supplier. Figure 5.1 shows a cluster diagram for D-desoxyephydrine hydrochloride; these results were calculated for ten drug samples from four manufacturers. Although too few samples were available to permit definite conclusions, the sample points appear to cluster and to distinguish suppliers 1 and 4 from each other and from 2 and 3 but not to distinguish 2 and 3. A more comprehensive evaluation of this statistical test is believed to be warranted.

5.1.b Delayed-Neutron Method for Determination of Fissionable Materials

J. F. Emery

The determination of fissionable materials by the delayed-neutron method[2] was extended to ^{233}U and ^{239}Pu. A ^{233}U (99.54%) standard and an NBS standard ^{239}Pu sample were used for comparison. The detection limits for ^{233}U, ^{235}U, and ^{239}U are 1×10^{-9}, 5×10^{-10}, and 7×10^{-10} respectively. The results follow for the determination of ^{239}Pu in thenoyltrifluoroacetone extracts of three MSRE fuel-salt samples:

Sample	^{239}Pu (ppm)
FP-15-68	145
FP-18-22	141
FP-18-43	137

[2]F. F. Dyer, J. F. Emery, and G. W. Leddicotte, *A Comprehensive Study of the Neutron Activation Analysis of Uranium by Delayed-Neutron Counting*, ORNL-3342 (July 27, 1962).

5.1.c Influence of Channeling in Conventional ^{3}He Activation Analysis

Enzo Ricci

A paper on the influence of channeling in conventional ^{3}He activation analysis was published;[3] the abstract follows.

"Channeling effect results when energetic charged particles enter a crystal with small angles relative to its lattice rows or planes; the ions suffer small-angle collisions with lattice atoms that tend to channel their paths inside the crystal. The channeling effect offers advantages and drawbacks to charged-particle activation analysis. To investigate whether this effect should be expected in routine analytical bombardments, a germanium single-crystal ingot was cut into numerous slices, perpendicular to its (1,1,1) axis. Each of these slices was mounted on a 0.2° precision goniometer, and bombarded at different angles with 7.8 ± 0.2 MeV helium-3 ions. To ensure the validity of the results over a wide area in charged-particle activation, the alignment was much more precise than in ordinary bombardments, so that observation of channeling could reasonably be expected. As activation analysis is based on counting of induced radioactivity, this was preferred to measurement of prompt nuclear reaction products. As the channeling critical angle in our investigations was 0.56° ± 0.01°, the bombardment angles used (beam with respect to (1,1,1) axis of sample) were −1.0°, −0.8°, −0.6°, −0.4°, −0.2°, 0°, 0.2°, 0.4°, 0.6°, 0.8°, 1.0°, 4.0°, 4.8°, 5.0°, 5.2°, 5.6° and 6.0°. The products of the germanium bombardment, viz., arsenic-71, arsenic-72, arsenic-74, arsenic-76 and selenium-73, were identified and the individual radioactivities, as well as their sum, were plotted against irradiation angle. Results indicate that the induced radioactivity is independent of angle, i.e., channeling effect is not observed. Because of the conditions chosen for these experiments it can be concluded that the channeling effect cannot influence most conventional charged-particle activation analyses."

5.1.d Proton-Reaction Analysis for ^{12}C, ^{13}C, and ^{15}N

Enzo Ricci

The principle and advantages of proton-reaction analysis (proton microprobe) have been discussed by several authors,[4-6] who have applied it to problems in many areas. In collaboration with the Physics Division, we have evaluated the applicability of this method to biomedical studies. In particular, there is considerable interest in the determination of the ratio $^{13}C/^{12}C$ because of the possibility of using ^{13}C as a tracer in humans. Since ^{13}C is nonradioactive, its advantages over ^{14}C for that purpose are evident.

We bombarded thick freeze-dried samples of human tissue (brain and lung) and *E. coli,* as well as thick graphite and uranium nitride standards, with 0.65- and 1.00-MeV protons at ORNL's 3-MV Van de Graaff generator. Prompt gamma-ray spectra produced during the nuclear reactions of the protons with ^{12}C, ^{13}C, and ^{15}N were measured with a multichannel analyzer and a pair of 5 by 5-in. NaI(Tl) detectors. The crystals face each other on a line perpendicular to the beam at the center of the target, which is 4.4 cm from each crystal. The reactions used and their corresponding resonance energies are $^{12}C(p,\gamma)^{13}N$, 0.450 MeV; $^{13}C(p,\gamma)^{14}N$, 0.554 MeV; $^{15}N(p,\alpha\gamma)^{12}C$, 0.429 and 0.898 MeV.[7]

Analytical sensitivities were measured with the inorganic standards. These values, expressed as counts min^{-1} μa^{-1} per unit percent of nuclide, follow for 0.65- and 1.00-MeV protons, respectively: ^{12}C, 117 and 109; ^{13}C, 990 and 990; ^{15}N, 2.6×10^4 and 3.16×10^5. As expected, the second ^{15}N resonance causes the enhancement of its sensitivity at 1.00 MeV. Figure 5.2 shows a typical spectrum of sample of human lung; this enhancement is clearly displayed. Human brain and *E. coli* gave very similar spectra.

Interesting conclusions can be drawn from our experiments. Sensitivities are good, and the gamma-ray spectra are easy to analyze. Thus, the ratios $^{13}C/^{12}C$ and $^{15}N/^{12}C$ (equivalent to N/C in natural samples) can be determined. A 1% change from normal isotopic concentration can be detected. The ^{15}N sensitivity is particularly outstanding. Further, ^{15}N peaks do not interfere with other peaks of biological interest, even when 1.00-MeV protons are used. However, since the ^{15}N peaks are also well-defined for 0.65-MeV protons, it is not necessary to use protons that have higher energy. Therefore, one advantage is a drastic reduction in the price of the charged-particle accelerator needed for the determinations. Another is a lessened sensitivity to interferences. Sample preparation is simple compared with the relatively elaborate techniques necessary for mass spectrometric determinations of isotope ratios.

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[3] E. Ricci, "Influence of Channeling in Conventional Helium-3 Activation Analysis," *Analyst* **94**, 435 (1969).

[4] E. Möllet and N. Starfelt, "Micro Analysis of Fluorine in Zircaloy by the Use of the $^{19}F(p,\alpha\gamma)^{16}O$ Reaction," *Nucl. Instr. Methods* **50**, 225 (1967).

[5] R. L. Macklin, J. H. Gibbons, E. Ricci, T. Handley, and D. Cuneo, "Proton Reaction Determination of Lithium and Fluorine in Molten Salt Reactor Graphite," *Nucl. App.* **5**, 269 (1968).

[6] T. B. Pierce, P. F. Peck, and D. R. A. Cuff, "The Microanalysis of Surfaces by Scanning with Charged Particle Beams," *Nucl. Instr. Methods* **67**, 1 (1969).

[7] F. Ajzenberg-Selove and T. Lauritsen, "Energy Levels of Light Nuclei. VI," *Nucl. Phys.* **11**, 1 (1959).

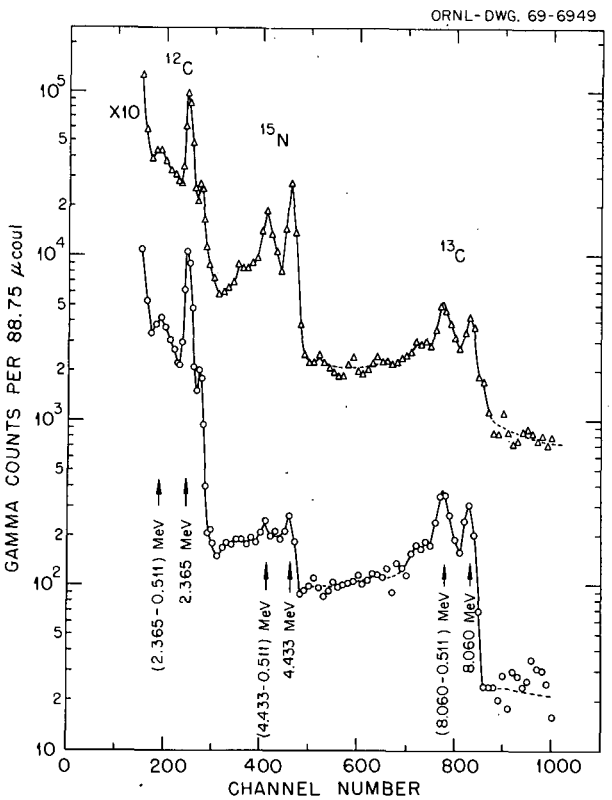

Fig. 5.2. Prompt Gamma-Ray Spectra from a Freeze-Dried Sample of Human Lung Tissue Bombarded with Protons of 0.65-MeV (○) and 1.00-MeV (△) Energies.

Furthermore, the proton microprobe lends itself to sample scanning (mass spectrometry does not), thus making it possible to study spatial distributions with a resolution of 0.05 to 0.10 mm.[6]

5.1.e Determination of Oxygen by 14-MeV Neutron Activation

J. E. Strain

The facility designed to determine oxygen in alkali and refractory metals operated throughout the year except two weeks when a neutron-generator malfunctioned because of a high-voltage breakdown that resulted from a foreign deposit in the deuterium-supply system. Replacement of the polyethylene tubing that conveys the deuterium to the palladium leak-valve corrected the difficulty.

As a result of a sequence-timer modification, oxygen in zirconium metal can now be determined. When irradiated with 14-MeV neutrons, Zr produces copious quantities of ^{90m}Zr, which decays by emission of a 2.32-MeV gamma ray ($t_{1/2}$ = 0.8 s). The resulting high level of radiation saturates the electronics and causes pulses to be recorded in the 4- to 7-MeV portion of the spectrum. By incorporating a delay timer in the control circuits, it is possible to start the counting sequence after the decay of the ^{90m}Zr; a 5-sec delay is sufficient. The lowest oxygen concentration that has been determined by this technique is 15 ppm in a 2-g sample.

The inability to determine oxygen in sodium by 14-MeV neutron activation was investigated fully. There is no nuclear interference in the form of high-energy radiation. The problem is saturation of the electronics by the intense 1.60-MeV gamma radiation from ^{20}F. Because ^{20}F has a half-life of 11.6 s, a delay in the start of the counting sequence is not possible, as is so with Zr. A variety of detectors [NaI(Tl), plastic scintillator, and Cerenkov] and absorbers (Pb, Al, Be, Fe, and plastic) were tested without success in an effort to overcome the saturation problem. In all cases, the reduction of the unwanted radiation also reduced the detectability of oxygen in the sodium sample. The only solution is to dispose of the bulk of the sodium prior to activation. This solution will be undertaken with a rapid distillation process now under development. The determination of oxygen in lithium and in potassium has become routine; the lowest oxygen values measured have been 19 ppm in lithium and ≤5 ppm in potassium.

5.1.f Neutron Generator Target Development Program

J. E. Strain

For several reasons, no further work was done to develop neutron-generator targets. Our 150-kV Texas Nuclear Corporation neutron generator was inspected by the ORNL Hot-Cell and Source Committee and was found to be potentially unsafe. The recommended ventilation, warning lights, bells, and redundant interlocks must be installed (cost ~$6000) before neutron production can be resumed. A target of the new design described earlier[8] has not yet been fabricated. The work load associated with the determination of oxygen in alkali and refractory metals (Sect. 5.1.e) is such that manpower is not available for the program.

[8] J. E. Strain, "Neutron Generator Target-Development Program." *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968*, ORNL-4343, p. 76.

5.1.g Isotopic Neutron Source Application

J. E. Strain

In cooperation with E. T. Creech,[9] an instrument was developed that continuously monitors the degree of oil contamination in a chlorinated organic coolant used in a machining operation. The device consists of a 2-curie ^{241}Am-B neutron source and a $^{10}BF_3$ thermal-neutron detector. The neutron source produces 2×10^5 neutrons/sec; the average energy of the neutrons is 2.5 MeV. The hydrogen in the oil acts as a moderator to slow the neutrons to thermal energy so that they can be detected by the $^{10}BF_3$ counter. The increase in count rate is proportional to the oil content of the coolant. The device is stable, insensitive to the low levels of radioactivity introduced in the coolant during the industrial process, and capable of measuring oil contamination in the range 1 to 20%.

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

Enzo Ricci T. H. Handley

Partial results of the work on activation analysis with a ^{252}Cf source, done jointly with the Transuranium Research Laboratory, were reported earlier.[10] A paper was submitted for publication;[11] its abstract follows.

"We show that a ^{252}Cf spontaneous-fission neutron source placed in the center of a 1-foot cube of moderator can be considered a portable, maintenance-free, quasi reactor by the activation analyst. One such source was calibrated by activation to have a 4π neutron emission intensity of 8.6×10^8 sec^{-1} (i.e., 0.37 mg ^{252}Cf); the thermal, epithermal, and fast neutron fluxes in the moderating cube were 9.7×10^6, 9.4×10^4, and 10^8 cm^{-2} sec^{-1} per mg ^{252}Cf, respectively, and the cadmium ratio 7.6. The average cross section for the reaction ^{27}Al(n,α)^{24}Na was calculated for the ^{252}Cf neutron spectrum to be 1.35 mb; use of analogous values, corresponding to the ^{235}U fission spectrum in ^{252}Cf calculations is shown to result in serious error. A pneumatic activation-analysis system was built; interference-free sensitivities and detection limits, determined for 13 elements of geological and biomedical interest, were found quite satisfactory for activation analysis. The system was also reliable and had good irradiation and counting-position reproducibilities. As a consequence, a mobile-power-free, maintenance-free sensitive and reliable activation analysis unit is suggested for field work and discussed. Increasing demand is also predicted for more and larger ^{252}Cf sources for use both in American biomedical centers and in research institutes of other countries, where maintenance of a nuclear reactor is not economically feasible."

5.1.i High-Flux Activation Analysis Laboratory

F. F. Dyer L. C. Bate

Funds ($102,000) for construction of the High-Flux Activation Analysis Laboratory in the HFIR[12] were approved. The engineering design of the laboratory and pneumatic tube was finished. The laboratory (Fig. 5.3) will consist of a counting room and a "hot" room in which the pneumatic-tube loading station and controls will be placed. The loading station will be located in a

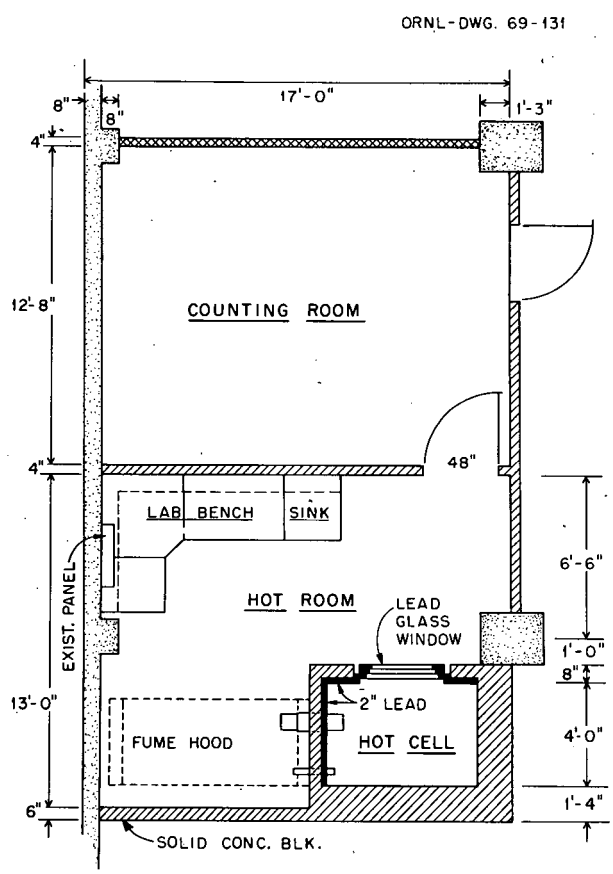

Fig. 5.3. Floor Plan for High-Flux Activation Analysis Laboratory in the HFIR.

[9] Y-12 Plant Laboratory.

[10] E. Ricci and T. H. Handley, "Neutron Activation Analysis with a ^{252}Cf Source," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968*, ORNL-4343, p. 63.

[11] E. Ricci and T. H. Handley, "Activation Analysis with a ^{252}Cf Source," submitted for publication in *Analytical Chemistry*.

[12] F. F. Dyer and L. C. Bate, "Proposed HFIR Activation Analysis Laboratory," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967*, ORNL-4196, p. 72.

steel hot cell (5-in. wall thickness) provided with a viewing window and remote manipulators.

The essential operating features of the pneumatic tube were tested with a mock-up. The pneumatic tube will operate with air blowing into both ends. Four exit holes for the air will be placed around the tube at the midplane of the reactor core. Thus, a rabbit will be shot through the tube and stopped on a countercurrent column of air. After the preset irradiation time, the rabbit will be returned automatically to either the loading station or a temporary holding station in the reactor pool. Samples that are too radioactive for direct return to the hot cell will be stopped at the temporary holding station. When returned to the loading station, a rabbit will again be stopped on a countercurrent column of air. To enhance the sensing of rabbit arrival at the reactor mid-plant, only one of the air-exit holes will be open while the rabbit is traveling. When arrival is sensed, a preset command will cause either three or four holes to open. With three holes open, the rabbit will remain stationary. With four holes open, the rabbit will vibrate and rotate slightly to average out variations of neutron flux over the rabbit.

Construction of the facility began in October 1969; completion is expected in early 1970.

5.1.j Status of the Photon and Fast-Neutron Activation Facility at the ORELA

Enzo Ricci

Work continued on the installation of the pneumatic system at the ORELA for activation analysis.[13,14] All parts of this system have been constructed, and a two-hood rabbit-loading-and-receiving station at Building 6010 is almost completed. On August 25, 1969, ORNL acquired possession of the ORELA. Since then, assemblage of the pneumatic system has proceeded rapidly.

After the installation is complete, experiments will be made to optimize conditions of operation of the air-floating sphere[13] (air pressure, internal balance, etc.) so that reliable and reproducible performance of the facility will be ensured.

A U.S. Patent application was filed on the design and operation of the air-floating sphere.

5.2 EXTRATERRESTRIAL APPLICATIONS AND ANALYSES

5.2.a Meteorite Fall at Pueblito de Allende, Chihuahua, Mexico: Preliminary Information

E. A. King, Jr.[15] Ernest Schonfeld[15]
K. A. Richardson[15] J. S. Eldridge

Specimens from the meteorite fall at 1:05 AM, February 8, 1969, at Pueblito de Allende, Chihuahua, Mexico, were recovered and taken to the Lunar Receiving Laboratory (LRL), Manned Spacecraft Center, Houston, Texas. Specimens were introduced into the low-background gamma-ray spectrometer within 101 hr after their fall. The rapid recovery was accomplished by E. A. King, Jr., who made a personal survey of the site and immediately transferred the specimens to the LRL.

Preliminary chemical analyses were sufficient to establish that the Pueblito de Allende meteorite is a C Group Chrondrite; petrographic data indicate that the specimen analyzed should be classified as type C4 (or C3).

One of the prime objectives in obtaining portions of this fall was to take as quickly as possible to the LRL Radiation Counting Facility specimens that were suitable for total gamma and gamma-gamma-coincidence spectrometry in order to analyze for short-half-life radionuclides. The following radionuclides were identified: ^{22}Na, ^{24}Na($t_{1/2}$ = 15.0 h), ^{26}Al, ^{40}K, ^{54}Mn, ^{60}Co, ^{238}U(^{214}Bi), and ^{232}Th(^{208}Tl). Further detailed analyses of the gamma-ray spectral data are being made: the results will be reported later.

The analysis of meteorites of short terrestrial age, such as the Pueblito de Allende meteorite, was an excellent rehearsal for the LRL Radiation Counting Facility, because it was expected that many of the same radionuclides would be present in the samples to be returned from the moon by the Apollo 11 project; this expectation was found to be true (Sect. 5.2.f).

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[13] E. Ricci, T. H. Handley, and M. G. Willey, "Air-Floating Sphere for Homogeneous Photon and Neutron Irradiation in Remote Facilities of Uneven Flux," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968*, ORNL-4343, p. 62.

[14] E. Ricci and M. G. Willey, "Photon and Fast-Neutron Activation Analysis at the Oak Ridge Electron Linear Accelerator (ORELA)," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968*, ORNL-4343, p. 62.

[15] Lunar Receiving Laboratory, NASA Manned Spacecraft Center, Houston, Texas.

A paper on this work was published.[16] Also, the analysis of the meteorite specimens by neutron activation analysis is discussed in Sect. 5.2.b.

5.2.b Nondestructive Neutron Activation Analysis of the Specimens from Pueblito de Allende Meteorite Fall

J. F. Emery G. D. O'Kelley[17]
J. E. Strain W. S. Lyon

Fourteen elements, including all major constituents, were determined in specimens from the Pueblito de Allende meteorite fall (Sect. 5.2.a); Ge(Li) and NaI(Tl) gamma-ray spectrometry were used to identify the radionuclides induced by reactor neutron irradiation. Two elements were determined by 14-MeV neutron activation. A paper on this subject was published.[18]

5.2.c Radiotracer Evaluation of a Lunar Surface Sampler

H. H. Ross

One objective of the United States lunar-landing program is to obtain aseptic samples of the lunar surface and to return them uncontaminated to earth. However, the sampling problem is complicated, because the astronauts themselves are a continuous source of biological contamination to the lunar surface. Estimates of the contamination level range from 1 to >1000 biota per square centimeter of lunar surface. Thus, any practical sampler must incorporate some method to either sterilize or remove the surface contamination before taking the sample. The most straightforward design approach appeared to be strictly mechanical; some portions of the lunar surface would be scraped aside, and the sample would be taken from the cleared field under aseptic conditions. We investigated the practical decontamination level that can be achieved by simple mechanical-scraping techniques. The results of this work were published.[19]

5.2.d Radiotracer Evaluation of a Lunar Core Sampler

H. H. Ross

A new type of core sampler was used on the recent Apollo 11 mission to the moon. We were asked by NASA to test an exact duplicate of the sampler with simulated lunar soil. The primary objective was to determine the surface contamination transfer characteristic in the core during a typical sampling operation. The tests were made in the following manner.

Simulated lunar soil was placed in a primary container (24-in. length, 5-in. diam). The soil was packed to density of ~106 lb/ft^3. The surface of the soil was seeded uniformly with 30 mg of MnO_2 dust (1.5-μ particle diam) that had been activated in the ORR. After the sampler was taken to a depth of 12 in., it was carefully removed from the soil, and the central sample tube was taken from the main housing and was sealed. The sample tube and its contents were counted by use of a conventional NaI crystal and a collimator assembly of new design. Radioactivity was determined as a function of distance from the core end, and the percent of original surface radioactivity that was distributed throughout the core was calculated.

Three other similar evaluations were attempted, each with soil of density ~100 lb/ft^3. However, in these tests, the core sampler failed to operate.

The results of all these tests were sent to NASA for further evaluation.

5.2.e Low-Level-Radiation Counting Facility for Geochemical and Returned Lunar Samples

J. S. Eldridge Ernest Schonfeld[15]
V. A. McKay[20] G. D. O'Kelley[17]
P. R. Bell[15] R. T. Roseberry[20]
K. J. Northcutt[21] R. E. Wintenberg[20]

Design concepts for a low-level radiation counting facility described previously[22] were fully implemented in both the ORNL and the Lunar Receiving Laboratory (LRL) facilities. New detectors were fabricated for both laboratories; they effected the expected improvements in background reduction. Cans for the detectors were

[16] E. A. King, Jr., E. Schonfeld, K. A. Richardson, and J. S. Eldridge, "Meteorite Fall at Pueblito de Allende, Chihuahua, Mexico: Preliminary Information," *Science* 163, 928 (Feb. 28, 1969).

[17] Chemistry Division.

[18] J. F. Emery, J. E. Strain, G. D. O'Kelley, and W. S. Lyon, "Non-Destructive Neutron Activation Analysis of the Allende Meteorite," *Radiochem. Radioanal. Letters* 1(2), 137 (1969).

[19] H. H. Ross and T. G. Chapman, "Radiotracer Evaluation of a Lunar Surface Sampler," *Radiochem. Radioanal. Letters* 1(2), 111 (1969).

[20] Instrumentation and Controls Division.

[21] Radioisotopes-Radiochemistry Laboratory.

[22] 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.

formed by a spinning technique in the shops of the ORNL Plant and Equipment Division. This technique resulted in a solid 20-mil-thick can that has much more integral strength than that of the prototype detectors.

Installation of the new detectors in the LRL facility was completed in December 1968. Software modifications for the PDP-9 analyzer system were completed at about the same time, and so the LRL Radiation Counting Laboratory was ready for the first simulation, held in February 1969, of the Apollo 11 mission. Completion of the system occurred at a fortunate time, because the analysis of the Pueblito de Allende meteorite samples (Sect. 5.2.a) was thus possible. The meteorite samples served as simulated lunar samples and provided an excellent opportunity to check the procedures of the LRL Radiation Counting Laboratory. The meteorite counting exercises demonstrated a serious need for adequate "library" spectra and faster means to analyze the gamma-ray spectral data.

Figures 5.4 and 5.5 illustrate background measurements made with the new detectors at the LRL. In Fig. 5.4, an overlay of spectra from both the upper and lower detectors illustrates slight differences in background-spectra shapes. This background run was made with the entire logic system operative and yielded an integral background of 78.9 counts/min (0.015 count min^{-1} cm^{-3} of NaI(Tl)) for the upper detector and 78.4 counts/min (0.015 count min^{-1} cm^{-3} of NaI(Tl)) for the lower one. These values are for the standard energy increment 0.1 to 2.0 MeV (the channels 5

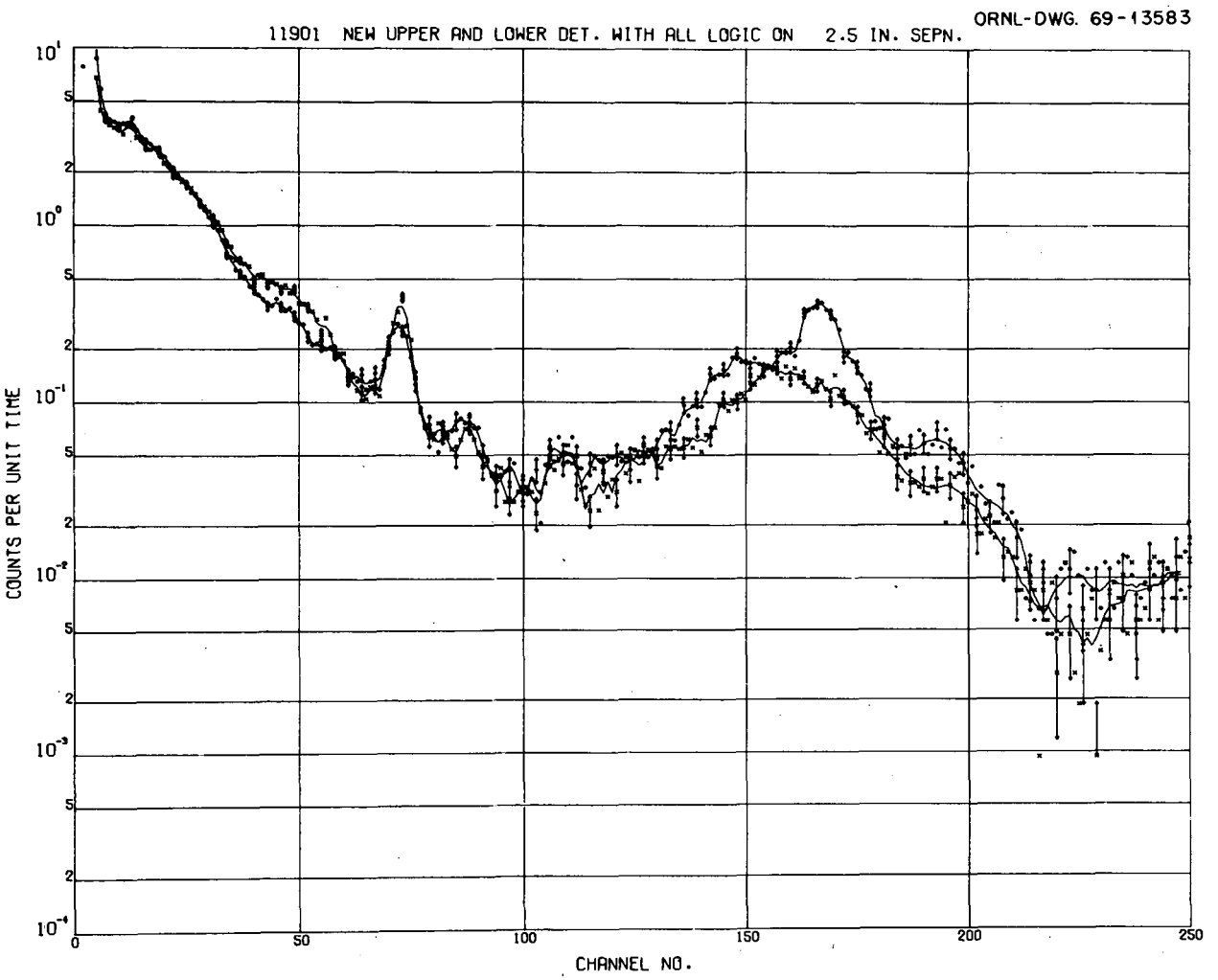

Fig. 5.4. Background Spectra Obtained with the Upper and Lower Principal Detectors of Improved Design in the LRL, Manned Spacecraft Center, Houston.

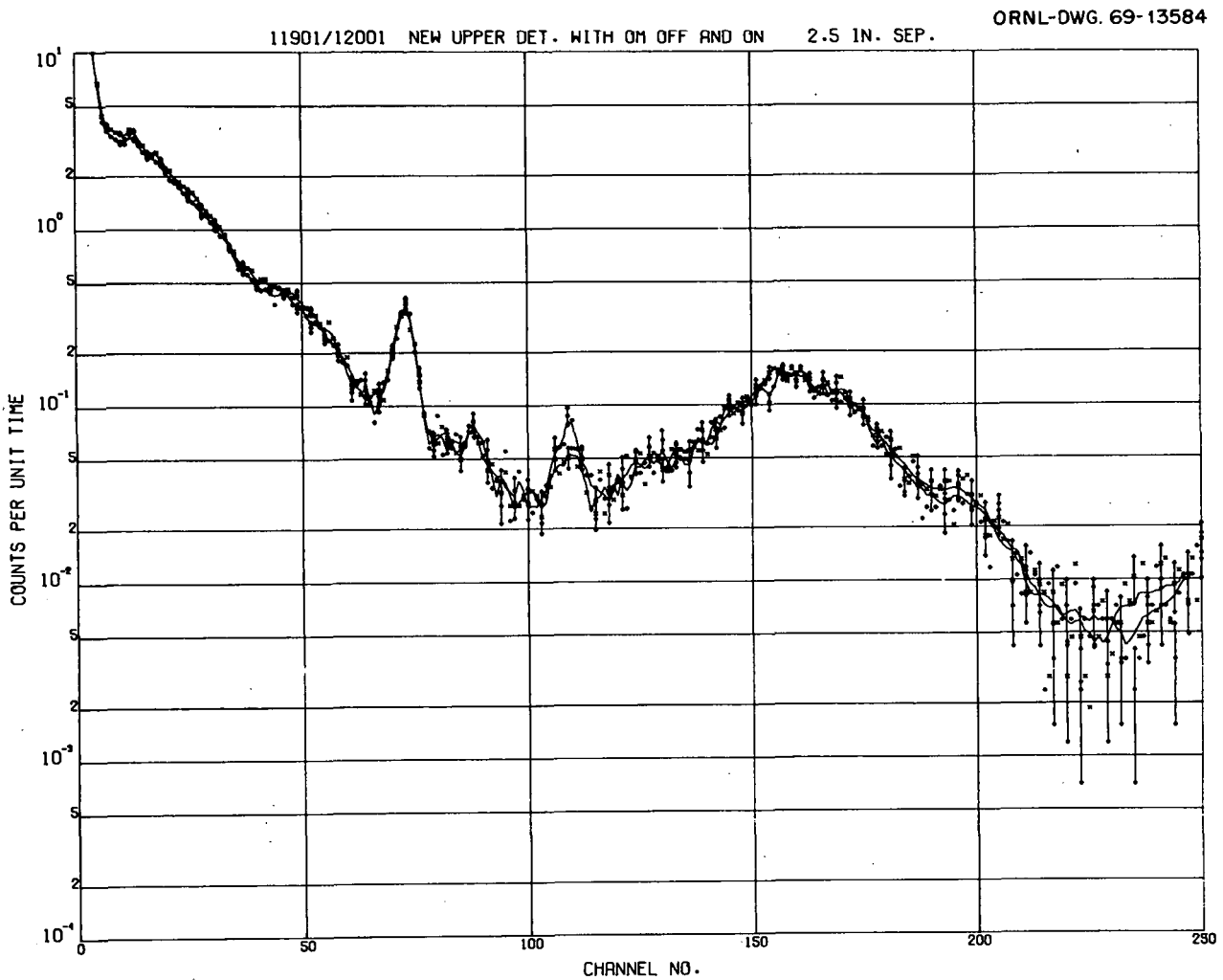

Fig. 5.5. Suppression of the Hydrogen-Capture Peak in the Upper Detector of the LRL.

through 100). In the energy increment 2.4 to 4.8 MeV (channels 120 through 240), the counting rate of the new upper detector is 6.9 counts/min, whereas that of the new lower one is 11.3 counts/min. The difference is caused mainly by differences in heavy-element alpha contamination in the two detectors. Figure 5.5 illustrates the magnitude of the hydrogen-capture peak at 2.2 MeV (channel 110) in the LRL detectors. The upper curve is for the background in the upper detector with the entire logic system operative, whereas the lower curve was obtained with the outer mantle turned off. The net counting rate in the interval 2.0 to 2.4 MeV was 0.39 count/min with the outer mantle off and 0.26 count/min with both mantles on. It is obvious that there is only a slight difference in the two curves. This experiment shows that the design of the counting chamber results in removal of the neutron-producing component of cosmic radiation from the immediate LRL environment; a duplicate experiment at ORNL (~900 ft above sea level) showed a count rate at 2.2 MeV that was almost six times that of the underground measurements at the LRL.

Figure 5.6 illustrates the difference in background response for the LRL lower detector with and without the anticoincidence mantles and shows that the anticoincidence system (lower curve) reduces the background rate over the entire spectral region. Reduction is minimal in the region of channel 170, where heavy-element alpha contaminants are prevalent. This contamination is inherent in the detectors and is not affected by the mantles. For the interval 0.1 to 2.0 MeV, the background rate without anticoincidence is

476 counts/min, compared with 78.4 counts/min with anticoincidence.

Calibration sources were prepared at ORNL and were counted in the LRL facility for library-response functions. Each nuclide expected to be present in a lunar sample must be counted in a variety of geometries. Commercially prepared radionuclides were used whenever practical. Otherwise, the radionuclides were prepared by reactor irradiations or by cyclotron bombardments.

For the preparation of extended sources that approximate large meteoritic or lunar samples, it was found that electrolytically reduced iron powder is sufficiently free of radioactive contamination and has a bulk density of 3.4 to 3.5 g/cm^3. Small assayed portions of each radionuclide were mixed thoroughly with the appropriate amount of iron powder to ensure homogeneity. Then, suitable aliquots of the radionuclide–iron powder mixture were sent to the LRL for the library collection. Standard 25-, 50-, 100-, 200-, 300-, 1260-, and 4100-g samples have been prepared for about one dozen nuclides. Figure 5.7 illustrates the extremes in response obtained with a point-source mount and with a 1260-g sample of the mixture that contained 16-d ^{48}V. These two spectra are from the sum-coincidence mode of analysis in the PDP-9 analyzer system. Note how much of the structural detail is lost through attenuation and scattering by the iron matrix. From such response differences, it is readily apparent that extensive calibration information is necessary for accurate analysis of the expected complex gamma-ray spectra of returned lunar samples.

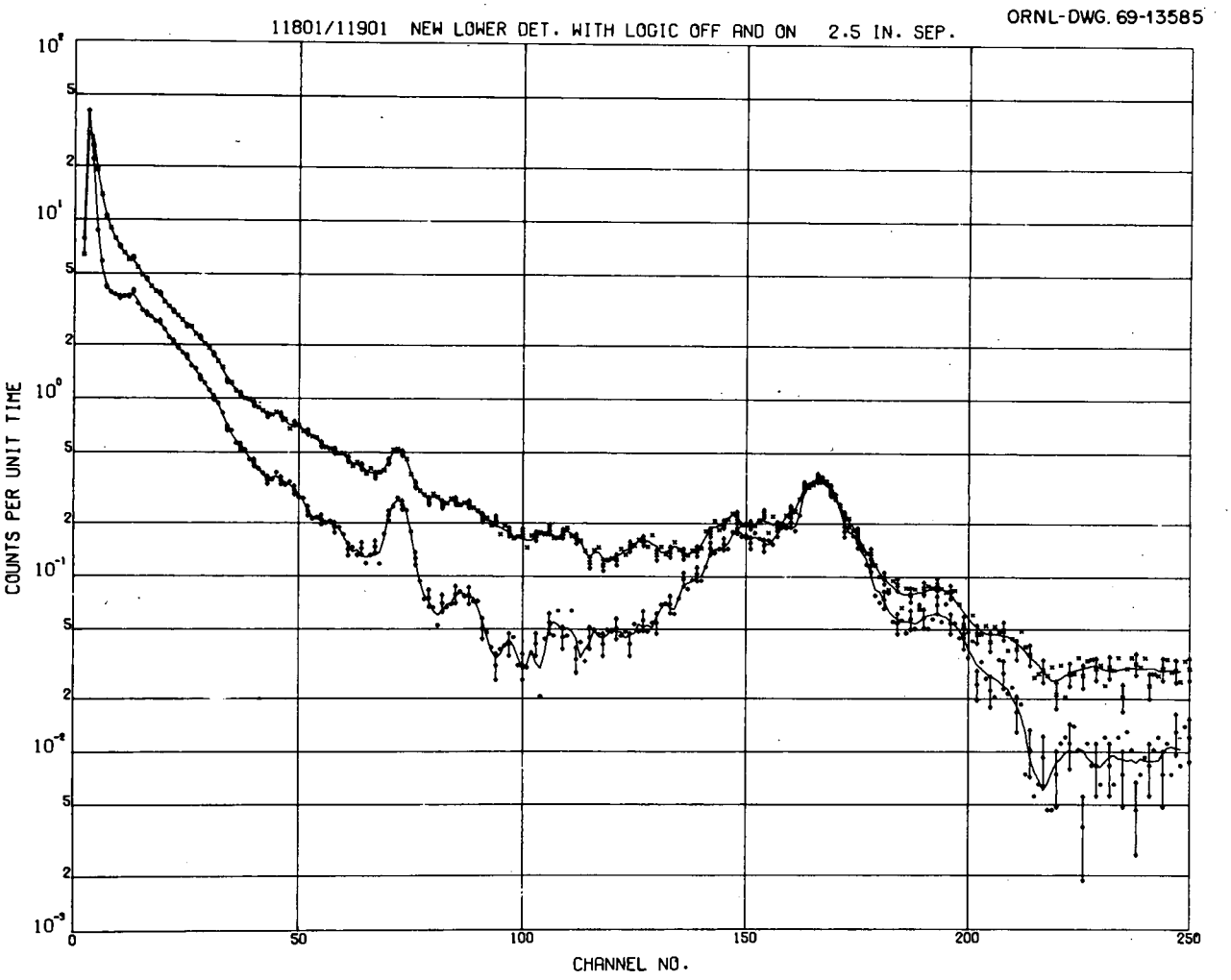

Fig. 5.6. Effect of Anticoincidence Mantles on Background of the Lower Detector in the LRL System.

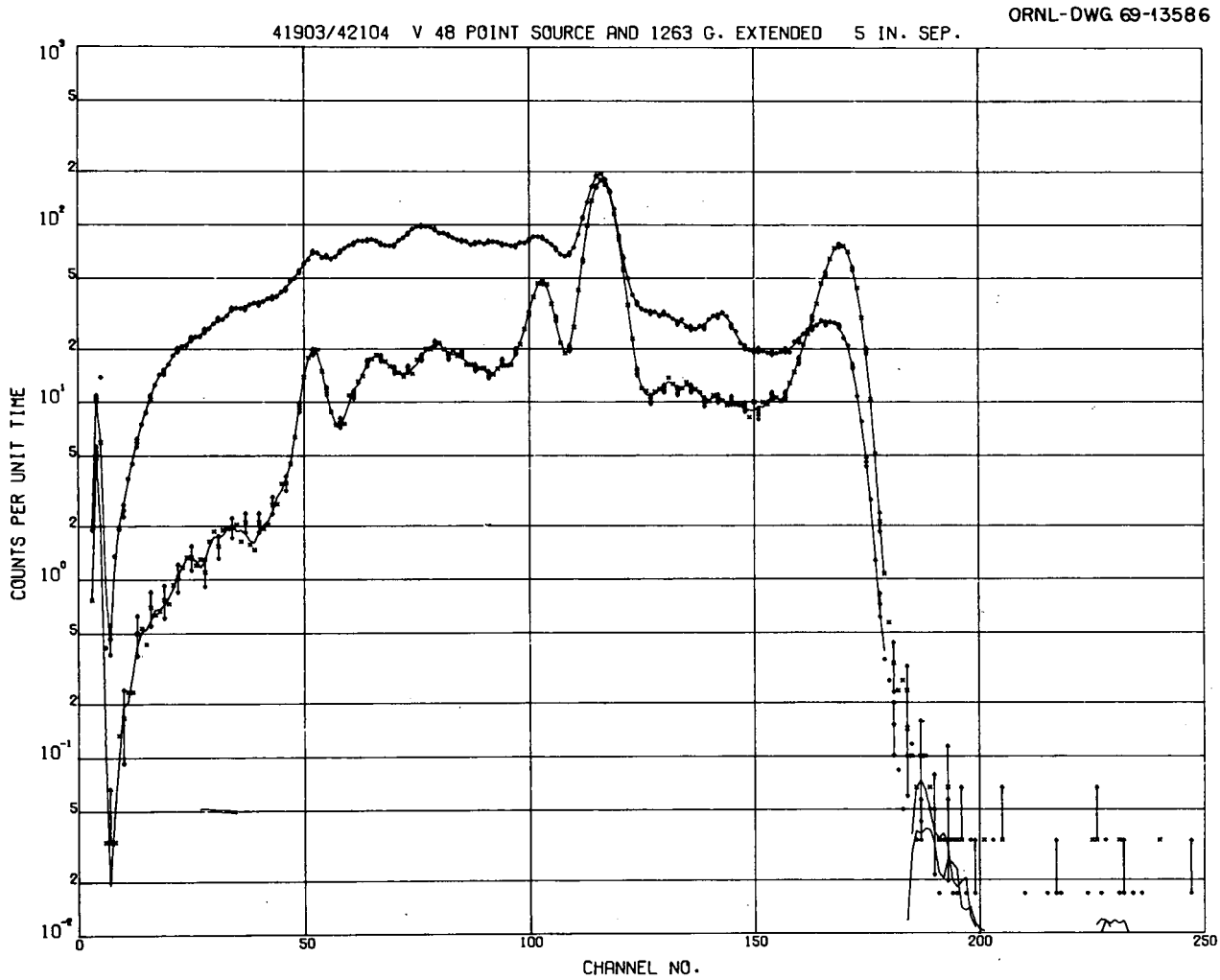

Fig. 5.7. Sum-Coincidence Spectra of ^{48}V Recorded in the LRL System.

5.2.f Preliminary Examination of Lunar Samples from Apollo 11

J. S. Eldridge

Three separate samples (contingency sample, bulk sample, and documented sample) were collected on the lunar surface during the Apollo 11 mission. The Radiation Counting Laboratory (RCL) received eight lunar samples for preliminary study by nondestructive gamma-ray analysis. These included a sample of fines and one rock from the bulk-sample box, one rock from the contingency sample, and five rocks from the documented-sample box. The analyses began on July 29, 1969; Fig. 5.8 is a photograph of one of the samples analyzed.

For the preliminary investigation, the gamma-ray spectrometer was calibrated with the aid of a series of standards prepared by dispersing known amounts of radionuclides in quantities of electrolytically reduced iron powder (Sect. 5.2.e). The amount of each radionuclide in the lunar samples was determined by using the method of least squares to resolve the gamma-ray spectra. Twelve radionuclides were identified, some tentatively; those of shortest half-life were ^{52}Mn (5.7 d) and ^{48}V (16.1 d).

Yields of cosmogenic ^{26}Al were generally high, and the ratio of ^{22}Na to ^{26}Al is considerably less than one in both the lunar rocks and soil. These measurements indicate that the material analyzed in these studies has been exposed to cosmic radiation for at least several million years.

NASA S-69-46273

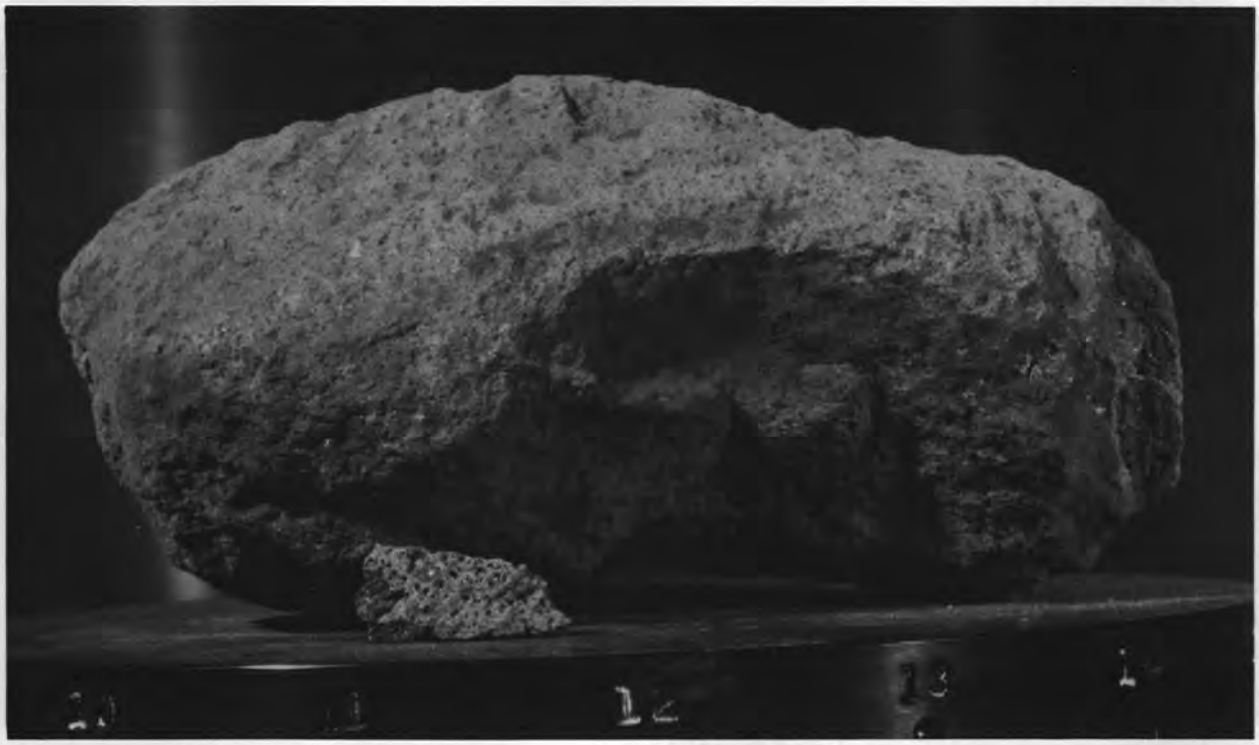

Fig. 5.8. Photograph of Returned Lunar Sample No. 10057.

A paper that describes all the preliminary examinations of Apollo 11 samples was published.[23]

5.3 COOPERATIVE ISOTOPES PROGRAM

5.3.a Technical Assistance

S. A. Reynolds

The joint program[24] of the Isotopes and Analytical Chemistry Divisions in analysis and characterization of isotopes has continued. Important features of the program are discussed below or in other sections of this report. Other work included preparation and reviews of papers and reports and consultation on analytical, radiochemical, and measurement problems, both local and external. The *Isotope User's Guide*[25] was completed. It is expected to be very useful, particularly to students and to others who are unfamiliar with the characteristics and applications of radionuclides and enriched stable nuclides. Also, it will serve as a collection of standard data for use in the Isotopes and Analytical Chemistry Divisions. Many of the recent activities of the Isotopes Division were reported elsewhere.[26]

5.3.b Radioactivity Standards

S. A. Reynolds J. F. Emery

A program is in progress, with support from the National Academy of Sciences (NAS), to survey the status of and need for standard radioactive materials. More than 1000 copies of a questionnaire, with an explanatory letter, were sent to people thought to be

[23] D. H. Anderson, E. E. Anderson, K. Bieman, P. R. Bell, D. D. Bogard, R. Brett, A. L. Burlingame, W. D. Carrier, E. C. T. Chao, N. C. Costes, D. H. Dahlem, G. B. Dalrymple, R. Doell, J. S. Eldridge, and many others, "Preliminary Examination of Lunar Samples for Apollo 11," *Science* **165**, 1211 (1969).

[24] "Cooperative Isotopes Program," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968,* ORNL-4343, p. 76.

[25] F. E. McKinney, S. A. Reynolds, and P. S. Baker, *Isotope User's Guide,* ORNL-IIC-19 (September 1969).

[26] AEC Division of Isotopes Development, Washington, D.C., *Division of Isotopes Development Research and Development Projects: 1968,* TID-24823 (January 1969).

users of radioactivity standards. The proceedings[27] of the American Chemical Society Symposium on Standards in Nuclear Chemistry and Technology is a very convenient aid in this program. Some of the difficulties caused by poor standards are reported for the first time therein. It is hoped that the release of this information will encourage those people contacted for information to report their own puzzling or frustrating experiences.

We are analyzing the returned questionnaires. Also, the NAS Subcommittee on Use of Radioactivity Standards will select a panel of specialists to draw conclusions and make recommendations for appropriate action. The panel report will be prepared at ORNL. A session on standards and a meeting of the NAS Subcommittee were held during the annual Gatlinburg Conference.

Measurements at ORNL have continued on radioactivity standards from NBS, IAEA, and Amersham/Searle Corporation (distributor for British Radiochemical Centre); agreement is entirely satisfactory. For example, eight determinations were made by use of three types of instruments on a ^{106}Ru standard. The mean was 98.6 ± 1.4 μcuries/ml compared with the Amersham/Searle value of 99.0 ± 1.8 μcuries/ml. An ORNL-prepared ^{55}Fe standard was used to determine the disintegration rates of ^{55}Fe standards from IAEA and from the NBS; the results are compared in Table 5.2. The disintegration rates for the NBS and IAEA sources are within the quoted uncertainties for each source.

Table 5.2. Measurements of ^{55}Fe Standards

Standard	Disintegration Rate (dis sec^{-1} g^{-1})	
	Certified Value	Experimental Value
IAEA	$(1.66 \times 10^6) \pm 4.3\%$	1.72×10^6
NBS	$(6.36 \times 10^3) \pm 5.1\%$	6.20×10^3

5.3.c Neutron Cross Sections and Flux Monitoring

S. A. Reynolds W. T. Mullins[21]

The monitoring of thermal and resonance fluxes of neutrons by induced ^{110m}Ag has been recommended. Silver is comparable with gold as a monitor, but its much longer half-life and lower burnout are advantageous. The thermal cross section of ^{109}Ag has been known only very poorly and therefore was determined. It was found to be 4.1 barns, based on the results of the simultaneous irradiation of a Ag-Al alloy and Co-Al standards. This value is in good agreement with the weighted mean of other reported values, 4.0 barns. It is estimated that the resonance integral for production of ^{110m}Ag is 70 barns. In connection with this determination, as well as with the routine assay of ^{110m}Ag products, the decay scheme was reviewed, and a correction was made in the assay calibration.

Reported cross sections of the calcium isotopes have been surprisingly varied, for example, 0.1 to 40 barns for ^{42}Ca. Samples enriched in ^{40}Ca were irradiated a few years ago to produce ^{41}Ca. Mass analyses and ^{45}Ca yields have recently been found to be in accord with the following values:

Isotope	Cross Section (barns)
^{40}Ca	0.41 (ref. 28)
^{41}Ca	4
^{42}Ca	<4, consistent with 0.7 (ref. 28)
^{43}Ca	6.2 (ref. 28)
^{45}Ca	~15

Our activation cross section for ^{44}Ca, 0.86 barn, was assumed. With our earlier value for ^{46}Ca, 0.77 barn, a set of reasonable data has now been completed.

ASTM Committee E-10 and members of the Solid State Division sponsored an interlaboratory test of the method of measurement of reactor fast-neutron flux by fission of ^{237}Np. Quantities (~0.4 mg) of Np were deposited in a set of quartz ampoules by evaporation of portions of solution. The actual amount in each ampoule was measured by counting the gamma radiation of the ^{233}Pa daughter. All ampoules were then irradiated for a short time in the Bulk Shielding Reactor. After decay of short-lived fission products, the 1.6-MeV gamma radiation of ^{140}La was measured nondestructively by gamma spectrometry. Seventeen of the ampoules were then shipped to various participating laboratories throughout the country. Their results were compared with each other (through the known quantities of Np in all ampoules) and with the ORNL measurements. Our statistical analysis showed that the overall precision (S) was 10% and that the only

[27] H. F. Beeghly, J. P. Cali, and W. W. Meinke (eds.), *Nuclear Standards for Chemistry and Technology* (Proceedings of the Symposium on Standards in Nuclear Chemistry and Technology, 156th Meeting of the American Chemical Society, Atlantic City, N.J., Sept. 8–9, 1968), National Bureau of Standards Special Publication 310 (December 1968).

[28] D. T. Goldman, P. Aline, R. Sher, and J. R. Stehn, "Twenty-two Hundred Meter per Second Neutron Absorption Cross Sections," in preparation.

important source of variance was the purported absolute measurement of ^{140}La by each laboratory. The ORNL *S* value was 1.5%, and the ORNL average was 6% below the overall average apparent flux.

5.3.d Radioisotope Characteristics

S. A. Reynolds J. F. Emery

Samples of product ^{133}Xe were used to study its decay scheme and to determine trace impurities. The method involved NaI(Tl) and Ge(Li) gamma spectrometry after various decay times. The experimental findings as of nine days after irradiation are given in Table 5.3. The decay scheme of ^{133}Xe has been known very poorly, largely because of errors in published work. When the data are corrected, the results agree adequately with those of our study. The percent of ^{133m}Xe (0.1%) was surprisingly low compared with that predicted (0.3 to 0.7%) on the assumption that the feeding of the isomer from ^{133}I is 1.4 to 2.9%. If the cross section of ^{133m}Xe is 3×10^4 barns, the observation can be explained. Such a cross section is plausible, since that of ^{133}Xe has been reported to be ~190 barns and, generally, the upper isomer of an active pair appears to have a much higher cross section than the lower. The amount of ^{131m}Xe was in good agreement with that calculated from a recent finding that 1.4% of ^{131}I atoms decay to the isomer.[29]

Our tabulation of the characteristics of ~240 radionuclides prepared for the *Isotope User's Guide* was compared with other recent compilations, including a table,[30] a chart,[31] and a draft report prepared at the Lawrence Radiation Laboratory. Agreement of the selections of properties was very satisfactory.

Table 5.3. Radiochemical Characteristics of Product ^{133}Xe

Radionuclide	Energies of Emitted Gamma Rays (MeV)	Number of Gammas per Disintegration	Impurity in ^{133}Xe (%)
^{133}Xe	0.08	0.36	
	0.16	7×10^{-4}	
	0.30	4.5×10^{-5}	
	0.38	2.4×10^{-5}	
^{133m}Xe	0.23	0.10	0.1
^{131m}Xe	0.16	0.02	0.8
^{131}I	0.36	0.80	0.003
^{85}Kr	0.51	4.2×10^{-3}	3×10^{-4}

5.3.e Half-Life Measurements

J. F. Emery S. A. Reynolds
E. I. Wyatt[21]

New half-life measurements were made on more than 40 radionuclides of interest to the Isotopes and Analytical Chemistry Divisions. Some of the significant values are: ^{28}Al, 3.243 ± 0.005 m; ^{48}V, 15.971 ± 0.004 d; ^{76}As, 26.32 ± 0.07 h; ^{110m}Ag, 252.2 ± 0.3 d; ^{123m}Te, 119.7 ± 0.1 d; ^{131m}Xe, 12.00 ± 0.02 d; ^{133}Xe, 5.29 ± 0.01 d; and ^{194}Ir, 19.40 ± 0.01 h. A number of determinations are in progress that have resulted in the following tentative half-life values: ^{152}Eu, 13.5 ± 0.5 y; ^{154}Eu, 8.5 ± 0.2 y; ^{155}Eu, 4.9 ± 0.1 y; and ^{181}W, 121.6 ± 0.1 d. These values are in agreement with recent unpublished values measured at the Los Alamos Scientific Laboratory and Lawrence Radiation Laboratory.

A new instrument was put into service specifically for precise half-life measurements. It consists of a well-type NaI(Tl) detector, a stable amplifier and single-channel analyzer, and a high-quality scaler.

Radium-226 (1600-y) sources from the National Bureau of Standards are used as reference standards for checking the stability of gamma ionization chambers with which some half-life determinations are made. For measurement of a long-half-life like that of ^{137}Cs, correction for ^{226}Ra decay is necessary. The presence of a contaminate radioactivity such as 5.8-y ^{228}Ra could be a source of error. Measurement of the amount of ^{228}Ra in the ^{226}Ra standards through the 2.6-MeV gamma radiation of its ^{208}Tl daughter indicated $< 0.01\%$ ^{228}Ra, an insignificant amount.

A few papers have reported rather questionable evidence of the dependence of half-lives of beta emitters on their chemical state. The analogous effect on half-lives of electron-capturers is real, although very small. The author of papers on such an effect in ^{64}Cu and ^{131}I inquired whether our observations substantiate his. He was informed that our data indicate no effects, and explanations were suggested for his observations on ^{131}I (volatility) and ^{64}Cu (impurity). He was informed of our new value for the half-life of ^{64}Cu,

[29] G. Graeffe and W. B. Walters, "Decay of ^{129}Cs and ^{131}I to the Levels in ^{129}Xe and ^{131}Xe," *Phys. Rev.* **153**, 1321 (1967).

[30] P. H. Blichert-Toft, *Radioatoms in Nuclear Medicine*, R. Nilsson, Gotenburg, Sweden (1968).

[31] M. Brucer, *Trilinear Chart of the Nuclides*, Mallinckrodt Chemical Works, St. Louis, Mo. (1968).

12.71 ± 0.04 h, based on solid and solution sources. Our 1956 value was higher, as were many others reported in the literature. The presence of radioactive impurities is suspected – in our case probably ^{76}As. Recomputation of our old data to correct for ^{76}As impurity gave the value 12.72 ± 0.04 h, which is in good agreement with the newly determined value.

5.3.f Quality Control

S. A. Reynolds

Sections on analysis and quality control were prepared for a number of radioisotope products; they are to be submitted for inclusion in a new edition of the IAEA *Manual of Radioisotope Production.* A brief summary of analytical quality-control tests on ^{123}I product was submitted to the Food and Drug Administration for consideration. An attempt was made to establish a precedent of not including minor arbitrary details, such as number of channels in an analyzer and its manufacturer. Provision was made to use for assay either an NBS ^{139}Ce standard or an ORNL ^{123m}Te standard. In fact, those concerned have agreed that the former is preferable because of the prestige of NBS standards and the fact that if a user requested an ORNL ^{123m}Te standard, policy forbids furnishing it.

It was observed that the measured ^{137}Cs contents (curies/g) of ^{137}CsCl products sometimes are low, without a corresponding low value for ^{137}Cs abundance as measured by mass spectrometry. Review of the radiometric assay indicated satisfactory performance. Typical overall precision (S) was 2.8%, including the precisions of sampling, dilution, and measurement by 4π ionization chamber. Four measurements of standards in the past two years agreed with the IAEA values to within ±0.2 to ±2.1%. Spectrographic analyses showed no anomalies. Chloride results determined by two independent methods were low on some products and thus indicated the presence of another anion. The possibility of formation of oxide or hydroxide by radiolytic decomposition was contradicted by the observation that the material was neutral. Classical gravimetric sulfate determinations made in a hot cell gave values such that an anion-cation balance was obtained. Sulfate from the alum stage in the process was therefore occasionally appearing in the product by an unknown mechanism. A spectrographic determination of aluminum, as well as consideration of stoichiometry and ^{137}Cs content, indicated that alum was not the source of sulfate. Specifications were set for chloride (18% minimum) and sulfate (3% maximum); these determinations are easily done by existing analytical methods.

Stable-isotope preparations that are loaned out sometimes contain radioactivity when they are returned. A screening test was recommended that consists in measurement of gamma radiations of energies up to 1 MeV by a 3- by 3-in. NaI(Tl) detector. A standard of ^{137}Cs (solid or solution) of about the same "geometry" is used for comparison, in accord with standard ASTM and American Public Health Association (APHA) methods. A test limit was set for gamma radioactivity, including bremsstrahlung, equivalent to 0.001 μcurie of ^{137}Cs. Above that limit, the radioactivity is identified by appropriate analysis, and the extent of health hazard or interference with future use of the material is evaluated.

The author's proposal for a standard statement of the precision of a radiochemical method has been accepted by Subcommittee 4 of ASTM Committee D-19. The approximate wording is:

"The intrinsic [single-operator or multi-laboratory] precision (S_m, standard deviation) of this method is about –%. To obtain the overall precision of a particular analysis, the standard deviation of counting must be combined with the intrinsic precision as follows:

$$S = \sqrt{S_m^2 + \sigma_c^2}\text{''}$$

5.3.g Activation by Radioisotope Gamma Radiation

J. J. Law[32] D. E. Smiley[33]
S. A. Reynolds

Radioactive isomers of certain stable nuclides can be formed by irradiation with gamma photons from radioisotope sources.[34] Silver solutions were irradiated in ^{60}Co fields of intensity as great as 15 megarads/hr by use of sources in the Graphite Reactor canal. The isomers ^{107m}Ag and ^{109m}Ag, with half-lives of 44 and 40 s and gamma radiations of 0.094-MeV (5%) and 0.088-MeV (4%) energies, were measured by gamma-ray spectrometry. Yields were proportional to Ag content and gamma flux and were such that 1 dis/min per gram of Ag was equivalent to 3.4×10^4 rads/hr.[35]

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[32] Department of Natural Sciences, Longwood College, Farmville, Va.

[33] Isotopes Division.

[34] J. Law and F. A. Iddings, "A Study of Photoactivation with a ^{60}Co Source," *J. Radioanal. Chem.* 3, 53 (1969).

[35] J. J. Law, "Measurement of High Dose Rates Delivered by Cobalt-60 Sources with the Aqueous Silver Nitrate System," *Health Phys.* 17, 338 (1969).

5.3.h Special Problems

S. A. Reynolds

Information was requested about measurement of the gamma-radiation field strength at various distances from ^{241}Am sources of microcurie levels. The usual survey instruments were neither sufficiently sensitive nor calibrated for measurement of the 0.060-MeV gamma radiation. Fortunately, standards prepared by IAEA were available. The specific gamma-ray emission of ^{241}Am was calculated to be 0.012 r/hr at 1 m from a 1-curie point source. The corresponding value for a 10.5-μcurie standard at 5 cm is 0.050 mr/hr. It was recommended that a Ge(Li) detector of small volume or a Geiger-Mueller probe be used for the measurements after calibration with the standard; the usual large NaI(Tl) detectors are too sensitive.

A commercial cleaner was found to effectively decontaminate manipulators and other hot-cell devices. It was desired to evaluate its safety with respect to contact with common reagents, since it is advertised to contain "mild alkalis, synthetic detergents, wetting agents, and solvents." General information was obtained by titration with acid, evaporation and ignition, activation analysis, and gas-liquid chromatography. Conclusions from these tests were that the material is not of itself flammable, explosive, or otherwise hazardous. Drop-scale tests were done by mixing portions of the product with concd HCl, concd H_2SO_4, and 2 *M* and concd HNO_3. No significant reaction occurred, except that with H_2SO_4 a gel formed. Elevated-temperature tests were made by a simple adaptation of earlier methods.[36,37] A 0.5-ml volume of the cleaner was mixed with 0.5 ml of acid in a 10- by 75-mm culture tube. The tube was placed in a water bath, and a thermometer was inserted in the mixture. The reference liquid (water) was placed in an identical tube in the bath near the first tube and with an identical thermometer inserted. The thermometers were supported on a common stand so that both could be agitated simultaneously. The difference in temperature was observed as a function of reference temperature. In the mixture of cleaner with concd HNO_3, an obvious temperature excursion occurred just above 80°C. It was concluded that the concentrated cleaner should not be mixed with concentrated acid. In any case, such mixing would probably destroy its effectiveness.

[36] W. H. Baldwin, *Differential Thermal Analysis. Qualitative Study of Nitrate–Butyl Phosphate Systems*, Jan. 25, 1960 (unpublished).

[37] L. Silver, "Screening for Chemical Process Hazards," *Chem. Eng. Progr.* 63(8), 43 (1967).

5.3.i Special Analytical Assistance for Isotopes Development Center Activities

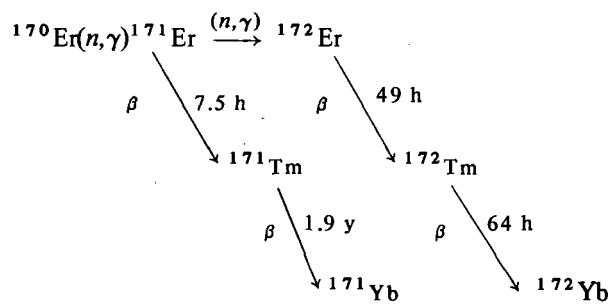
J. S. Eldridge W. S. Lyon
K. J. Northcutt[21]

As part of a continuing program,[38] radiochemical assays were performed for various members of the Isotopes Development Center. Some samples were measured for radionuclide content, whereas others were checked for product purity. Some samples were carefully assayed in order that they might serve as secondary standards for on-line analyses in other laboratories. Samples were assayed that contained the following nuclides: ^{37}Ar, ^{43}K, ^{68}Ge-^{68}Ga, ^{85}Kr, ^{85m}Sr, ^{123}I, ^{144}Ce, ^{199}Au, and ^{228}Th.

5.3.j Separation and Identification of New Isotope Products

T. H. Handley W. S. Lyon
J. K. Poggenburg, Jr.[33]

In a cooperative effort with the Isotopes Division, a sample of enriched ^{170}Er that had been irradiated in the HFIR to produce ^{172}Er by the reactions



was analyzed for all radioactive members of these decay chains. The Er and Tm fractions were separated by ion exchange, and the nuclides were identified and quantitatively measured by NaI(Tl) and Ge(Li) gamma-ray spectrometry. On the basis of these results, a second series of irradiations was made, but the Er and Tm

[38] J. S. Eldridge and W. S. Lyon, "Special Analytical Assistance for Isotopes Development Center Activities," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968*, ORNL-4343, p. 80.

fractions were not separated. The $^{171}Er/^{172}Er$ ratio was measured for different irradiation times and, from these data, the cross sections and optimum irradiation times for production of the two radioisotopes will be determined.

5.3.k Radioactive Source of ^{241}Am for Excitation of Characteristic X Rays

T. H. Handley R. S. Pressly[33]

An ^{241}Am source was constructed according to a design suggested by R. D. Giauque.[39] The source is made in such a way that the 60-keV gamma radiation from ^{241}Am can be used either to excite a sample directly or to excite characteristic x rays of a secondary target (Se, Sn, or Gd), which are then used to excite characteristic x rays of the chosen sample. Thus, improved sensitivity and selectivity are attained. Although considerable development work has been done on the uses of radioactive sources for x-ray fluorescence, no applications have been made at ORNL.

5.3.1 Radioisotopic Tagging of Copper Cable with ^{110m}Ag

L. C. Bate F. F. Dyer

Tagging with ^{110m}Ag has been demonstrated as a feasible way to identify stolen copper electrical cable. The radiotag can be applied in solution with a hypodermic syringe by a semiskilled person with a minimum of training. The ^{110m}Ag is permanently attached to the interior surfaces of the cable. After cable so tagged was exposed to washing and to simulated weather conditions for 1½ yr, followed by burning and rewashing, the total ^{110m}Ag that remained on the cable was ~92% of the original. The presence of the radiotagged cable in a pile of scrap copper can be detected by monitoring with a survey type radiation meter.

5.4 LIQUID SCINTILLATION AND CERENKOV COUNTING

5.4.a Applications of Cerenkov Radiation

H. H. Ross

We have completed an extensive study of the variables that determine the Cerenkov counting response of a variety of radionuclides. The results indicate that Cerenkov techniques can be applied to many radiochemical problems with significant advantage. From our results, the optimum counting conditions and the approximate counting efficiency can be determined for most radioisotopes. Also, we have suggested a new design for a Cerenkov counter. The results of this work were published.[40]

5.4.b Measurement of ^{14}C by Use of Cerenkov Radiation

H. H. Ross

Aithough the maximum beta energy of ^{14}C in water is well below the Cerenkov threshold, under certain conditions this nuclide can be assayed successfully in Cerenkov counting systems. This unusual response can be observed by using solvents that exhibit the effect of anomalous refractive dispersion. Among the advantages of the technique are a complete absence of response to tritium and the ability to quantitatively recover the sample without contamination.

The general theoretical principles of these measurements and typical experimental results were presented at a recent meeting[41] and will appear in the conference proceedings.

5.4.c Absolute Assay of ^{129}I

H. H. Ross

We have developed a new liquid-scintillation technique for the absolute assay of ^{129}I. The iodide-containing sample is reacted with H_2O_2 in acid solution to generate elemental iodine. The iodine is extracted into toluene and is then reacted with 2-methyl-2-butene in the presence of short-wavelength ultraviolet radiation. The resulting iodobutane is colorless in toluene and exhibits no significant quenching effect in standard liquid-scintillator "cocktails." An aliquot of the product is added to a BBOT-toluene liquid scintillator; the observed count is corrected for counting efficiency by use of a ^{14}C internal standard. The results obtained are in very good agreement with values calculated from mass and half-life data and also

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[39] Analytical Chemistry Division, Nuclear Chemistry Department, Lawrence Radiation Laboratory, Berkeley, Calif.

[40] H. H. Ross, "Measurement of β-Emitting Nuclides Using Cerenkov Radiation," *Anal. Chem.* 41, 1260 (1969).

[41] H. H. Ross, "The Measurement of Carbon-14 Using Cerenkov Radiation," presented at the Symposium on the Current Status of Liquid Scintillation Counting, Cambridge, Mass., Mar. 31–Apr. 3, 1969.

Table 5.4. Tritium Counting Efficiencies Obtained with Various Liquid Surfactants

Composition of test solutions: toluene, 2 vol parts
surfactant, 1 vol part
PPO, 5.5 g/liter
dimethyl POPOP, 0.1 g/liter
$^3H_2O + H_2O$ (quencher), indicated vol %

Surfactant		Tritium Counting Efficiency in Indicated Percent Water (%)			
Source	Code	0.33%	6.45%	12.02%	16.90%
Continential Oil Company	Alfonic 1012-6	23.90	22.52	*a*	*a*
	1412-4	24.03	*a*	*a*	*a*
General Aniline & Film Corporation	Igepal CA620	22.61	16.68	15.53	*a*
	CO630	22.84	17.47	15.45	*a*
	CA720	24.28	18.60	16.85	16.85
Monsanto Chemical Company	Sterox DF	25.05	20.31		
	DJ	23.28	21.75	17.24	16.16
	NJ	18.94	14.86	*a*	*a*
Jefferson Chemical Company, Inc.	Surfonic N95	24.99	19.27	17.79	*a*
Proctor & Gamble	Kyro EOB	25.89	18.87	*a*	*a*
Rohm & Haas	Triton X-100	23.86	18.12	16.20	14.63

[a] $<$10.00%.

with x-ray photon measurements. An article on this work was accepted for publication.[42]

5.4.d Polyethoxylated Nonionic Liquid Surfactants in Toluene for Liquid-Scintillation Counting of Tritium Samples in Aqueous Media

S. B. Lupica

Twelve polyethoxylated nonionic liquid surfactants were examined in a toluene scintillator system for liquid-scintillation counting of tritium samples in aqueous media. Of the surfactants tested, only Sterox DJ and Igepal CA720 give counting efficiencies higher than that with Triton X-100 (Table 5.4). Texofor 128 (Glovers Chemicals Ltd.) was among the surfactants tested; because it gives color quenching, it is not included in Table 5.4. At water concentrations less than ~6.5 vol %, any of the ten surfactants listed can be used instead of Triton X-100. At higher water concentrations, Igepal CA720 and Sterox DJ are superior; they give ~13% higher counting efficiencies than does Triton X-100.

The infrared and NMR spectra of these surfactants indicate that those compounds which have relatively simple spectra produce less quenching and have a high counting efficiency for tritium.

5.5 STUDIES OF NUCLEAR PROPERTIES

5.5.a Nuclear Spectroscopy of Erbium Radionuclides $158 \leqslant A \leqslant 161$

T. H. Handley Ben Harmatz[43]

A systematic study of the properties of low-lying nuclear levels populated by electron-capture decay of erbium to holmium is in progress. Erbium radionuclides in the mass interval $158 \leqslant A \leqslant 161$ are produced in the ORIC by (α,xn) reactions on enriched dysprosium isotopes and are separated chemically from the target by ion-exchange techniques. Conversion-electron spectrographs of ~0.1% resolution and ~0.01% transmission are used to unravel the complex spectra that follow the Er → Ho decay. Isotopic and element assignments of observed electron lines are based on relative activation

[42] H. H. Ross, "Absolute Assay of ^{129}I," accepted for publication in *Radiochemical and Radioanalytical Letters.*

[43] Electronuclear Division.

yields obtained with different isotopes and bombarding energies and on the decay rate of electron line intensities. Transition multipolarities are determined either from K/L or K/L-subshell intensity ratios or from K conversion coefficients.

Our earlier studies of neutron-deficient holmium nuclei did not disclose rotational excitation of the proposed single-particle and vibrational states. In the present work, rotational configuration may be built from the new transition data. Experimental rotational constants tend to characterize the intrinsic states excited in holmium in the framework of the Nilsson model.

5.5.b Search for ^{172}Hf Alpha Decay

T. H. Handley R. L. Hahn[44]
K. S. Toth[43] C. E. Bemis, Jr.[44]
M. F. Roche[45]

Naturally occurring ^{174}Hf has been observed to decay with a half-life of $(2.0 \pm 0.4) \times 10^{15}$ y by emitting alpha particles that have an energy of 2.5 ± 0.3 MeV. The alpha-decay energy of the neighboring doubly even radionuclide ^{172}Hf (with an electron-capture half-life of 5 y) has been estimated to be ~2.7 to 3.0 MeV. If the higher energy value were correct, then the alpha-decay half-life would be sufficiently short that the alpha-decay branch of ^{172}Hf could be detected by standard radiochemical techniques. Thus, a search for possible alpha decay in ^{172}Hf is under way.

An article on this work was accepted for publication.[46]

5.5.c Fast Neutron Cross Sections

W. S. Lyon J. F. Emery
J. D. Jenkins[47]

In a cooperative program between the Reactor and Analytical Chemistry Divisions, a number of foils for flux monitoring were prepared and shipped to Gulf General Atomic, San Diego, Calif. There, the foils were irradiated with fast neutrons whose neutron-energy spectrum was known. The foils that produced radionuclides of half-lives less than 16 h were counted at Gulf General Atomic, but the remainder of the foils were sent to ORNL for measurement of absolute radioactivity by calibrated NaI(Tl) and Ge(Li) gamma-ray spectroscopy for all nuclides except ^{32}P, which was determined by liquid-scintillation counting. The nuclides measured were: ^{24}Na, ^{32}P, ^{46}Sc, ^{47}Sc, ^{48}Sc, ^{55}Co, ^{58}Co, ^{60}Co, ^{59}Fe, ^{114m}In, ^{126}I, ^{140}Ba, ^{198}Au, ^{233}Pa, and ^{239}Np. From these measurements, it will be possible to check cross sections by using the known neutron flux. A second series of irradiations is planned for November 1969.

5.6 APPLICATIONS TO REACTOR PROGRAMS

5.6.a Uranium Isotope Analysis

J. F. Emery

The ^{232}U content of calutron-separated ^{233}U products was determined by gamma-ray spectrometry.[48] For the determination of ^{232}U, the radiation measured is the 2.614-MeV gamma ray from ^{208}Tl, the final radioactive daughter in the thorium series. The decay of ^{232}U is by alpha emission to ^{228}Th ($t_{1/2}$ = 1.9 y), and the ^{208}Tl grows in with the ^{228}Th. Early after separation, the ^{208}Tl radioactivity should grow rapidly; however, the ^{208}Tl activities of these samples were growing only ~2%/week, which rate corresponded to a separation date of 1.2 years. Contamination of the separated product with 0.5% of the feed material accounts for the small observed growth of the ^{208}Tl. Correcting the observed ^{208}Tl radioactivity for this contamination produced a normal growth curve. For two ^{233}U products, the ^{232}U contents, calculated from the growth of the ^{208}Tl plus the amount of ^{232}U present from contamination, were 8.6 and 13 ppb.

5.6.b ^{134}Cs Source Scanning

F. F. Dyer L. C. Bate

A graphite tube (80-mil wall thickness) that contained evaporated ^{134}Cs was scanned to determine the distribution of the ^{134}Cs on the tube. The tube was

[44] Chemistry Division.

[45] Formerly of the Electronuclear Division; present address: Argonne National Laboratory, Argonne, Ill.

[46] K. S. Toth, R. L. Hahn, C. E. Bemis, Jr., T. H. Handley, and M. F. Roche, "Search for ^{172}Hf Alpha Decay," accepted for publication in the *Journal of Inorganic and Nuclear Chemistry*.

[47] Reactor Division.

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

scanned from different directions to ascertain whether the ^{134}Cs was evenly distributed. Later, a section of the outer surface of the tube and successive layers were removed to determine the depth of penetration of the ^{134}Cs through the graphite. The data show that the ^{134}Cs penetrated through the wall of the graphite tube to the outer surface.

5.6.c ^{244}Cm Source Scanning

L. C. Bate

The distribution of the ^{244}Cm throughout two ^{244}Cm sources in sealed metal tubes was measured with the collimated scanner. A gamma-ray spectrum was taken, and the gamma radioactivity was identified as ^{239}Np and a fission continuum of ^{244}Cm. The source was scanned by use of the gross radioactivity, each gamma-ray peak, and the fission continuum. The data indicated that the radioisotopes of the ^{243}Cm and ^{244}Cm decay chains were fairly evenly distributed in the source. The ^{239}Np is the daughter of ^{243}Am, an impurity in the ^{244}Cm.

5.6.d Fission-Product Gas Analysis

L. C. Bate

Several samples of short-cooled (~3 hr) fission-product gases were submitted for analysis. The samples were mixtures of the krypton and xenon radioisotopes ^{85m}Kr, ^{87}Kr, ^{88}Kr, ^{133}Xe, and ^{135}Xe. The gamma-ray spectra from an NaI(Tl) detector showed that the gamma-ray photopeaks interfere with each other in the region 0.10 to 0.40 MeV. To measure the radioactivity, the samples were counted on the Ge(Li) diode, where adequate resolution was achieved for the measurements. Also, an additional gamma ray (the 0.166-MeV gamma of ^{88}Kr) that was not detectable by NaI(Tl) was resolved and measured by use of a Ge(Li) diode, and ^{131}I was detected but not measured. The use of the Ge(Li) diode for this type of sample that has many gamma-ray photopeaks is demonstrated to have a tremendous advantage over NaI(Tl) detectors (Fig. 5.9).

5.6.e Fission-Product Release from Reactor-Fuel Microspheres

L. C. Bate

The study of fission-product release from reactor-fuel microspheres[49] is a cooperative program with the Reactor Chemistry Division. The first phase of this program on the release of ^{131}I and ^{137}Cs from cracked fuel microspheres was completed.

The next phase, which concerns the release of Sr and Ba, has been started. The initial work was done on microspheres that had been irradiated for 6 hr and allowed to decay for 18 hr before they were heated. These microspheres are not cracked before heating. The spheres are heated in the induction-heater apparatus for definite time periods until essentially all the Ba and Sr are released. The first runs were made at 1700°C with pyrolytic-graphite-coated microspheres. The radionuclides detected were ^{91}Sr, ^{91m}Y, ^{105}Ru, ^{112}Pd-^{112}Ag, and ^{140}Ba-^{140}La. The samples are assayed by either NaI(Tl) or Ge(Li) gamma-ray spectrometry. Immediately after a heating period, the furnace parts are counted for ^{91}Sr and then are allowed to decay until ^{140}Ba and ^{140}La are in equilibrium. The 1.02-MeV gamma peak of ^{91}Sr is used for the ^{91}Sr assay; ^{140}Ba is assayed by use of the 1.60-MeV gamma

[49] L. C. Bate and F. F. Dyer, "Fission-Product Release from Reactor-Fuel Microspheres," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968*, ORNL-4343, p. 75.

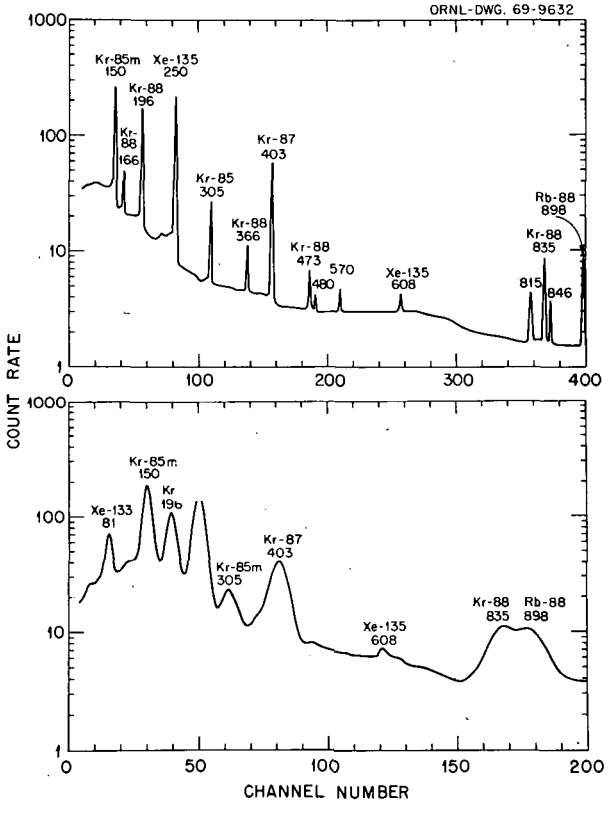

Fig. 5.9. Fission-Gas Spectra Determined with Ge(Li) (Top) and NaI(Tl) (Bottom) After a 3-hr Decay.

peak of ^{140}La. With SiC-coated microspheres, no release of radioactivity was observed until the temperature reached 2000°C, at which time ^{99}Mo was observed.

The next series of microspheres for Sr and Ba were irradiated until the uranium burnup was 15 to 20% and then were allowed to decay ~2 mo before the release rates were determined. The radionuclides measured were ^{89}Sr-^{89}Y, ^{137}Ba, and ^{140}Ba-^{140}La. The ^{137}Cs (0.662 MeV) and ^{140}La (1.60 MeV) were measured on the furnace parts. The measurement of the ^{89}Sr-^{89}Y radioactivity required a chemical separation after the removal of the radioactivity from the furnace parts, and the graphite crucible was dissolved. Several methods of cleaning the furnace parts were tried; $HClO_4$-HNO_3-H_2O was the best cleaning agent and resulted in a solution usable for the subsequent chemistry. The best and fastest way to dissolve the graphite crucible was to use a mixture of $HClO_4$-HNO_3-$CH_2ClCOOH$-$K_2Cr_2O_7$. The Sr yield was obtained by adding ^{85}Sr tracer before dissolution or cleaning of the furnace parts. In some cases, the ^{85}Sr in the separated samples was not measurable by an NaI(Tl) detector due to the high level of the beta bremsstrahlung but could be measured by a Ge(Li) detector. This difference is illustrated in Fig. 5.10.

Ten TRISO-2 UO_2 microspheres were analyzed before and after each annealing at temperatures from 1300° to 2200°C, and the furnace components were analyzed for fission products. Less than 0.1% of the Ce and Zr and ~0.5% of the Ru were released. At 2200°C, ~50% of the Cs was released, which value indicates failure of the SiC layers in the coating. This failure occurred in eight of the ten microspheres.

5.6.f In-Reactor Penetration of Fission Products into MSRE Graphite

L. C. Bate

Additional MSRE graphite samples[50] have been analyzed for fission products. The data are to be correlated with the uranium content of the graphite. The fission products detected and measured were ^{95}Nb, ^{95}Zr, ^{103}Ru, ^{106}Ru, ^{137}Cs, ^{141}Ce, and ^{144}Ce.

[50] L. C. Bate and F. F. Dyer, "In-Reactor Penetration of Fission Products into MSRE and Pyrolytic Graphite," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968,* ORNL-4343, p. 74.

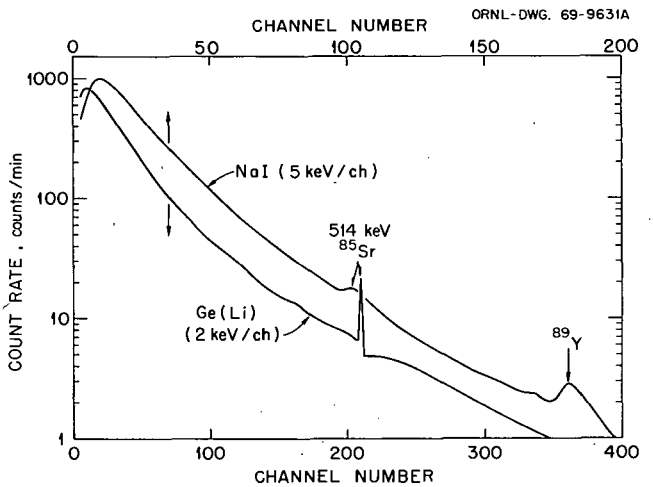

Fig. 5.10. Measurement of ^{85}Sr in $^{89,90}Sr$ by NaI(Tl) and by Ge(Li) Detectors.

5.6.g Measurements of Ge(Li) Gamma-Ray Spectrometry of Fission Products in MSRE Components

F. F. Dyer Aut Houtzell[51]
Robert Blumberg[51]

Because previous measurements of fission products in the fuel-circulation and off-gas systems[52,53] of the MSRE were promising but limited in scope and accuracy, another more comprehensive experiment was undertaken. The objectives are to determine both short- and long-lived fission products, to increase the accuracy of the measurements, and to obtain a more detailed knowledge of the fission-product distribution throughout the MSRE.

The equipment was improved in several ways. Figure 5.11 shows schematically the collimated detector and associated equipment used to manipulate and align the detector. Before the gamma rays from a reactor component enter the detector, they are highly collimated by the 1-in.-diam hole in the collimator and the narrow hole in the collimator insert. A laser is positioned above the collimator so that its beam can be

[51] Reactor Division.

[52] R. Blumberg, F. F. Dyer, T. H. Mauney, and D. Scott, *Measurements of Fission-Product Deposition in the MSRE with Ge(Li) Gamma Ray Spectroscopy,* Nov. 19, 1968 (unpublished).

[53] F. F. Dyer, R. Blumberg, and T. H. Mauney, "Measurement by Ge(Li) Gamma Spectrometry of Fission Products Deposited in Components of the MSRE," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968,* ORNL-4343, p. 74.

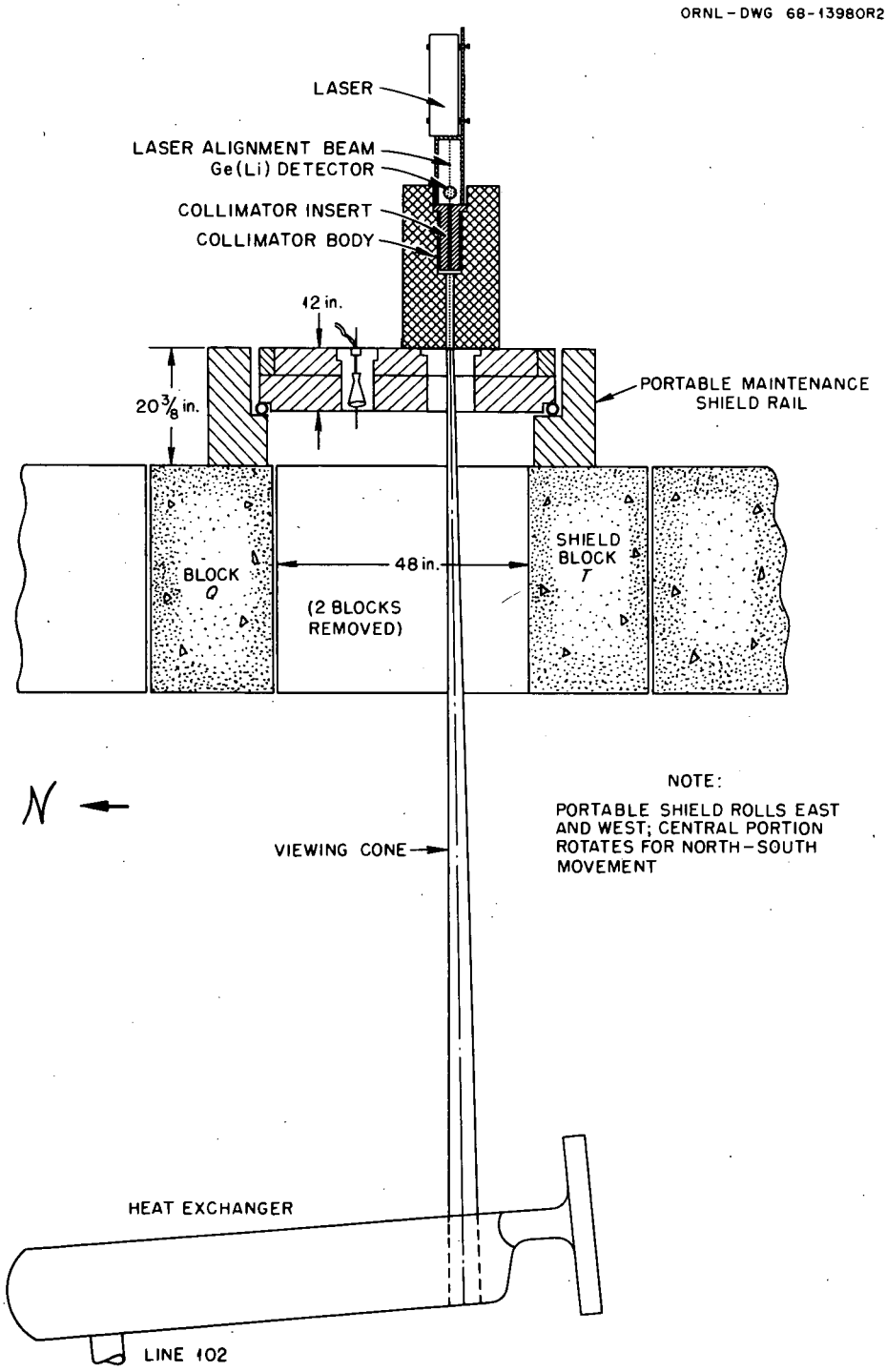

Fig. 5.11. Detector-Collimator Assembly for Remote Ge(Li) Gamma Spectrometry.

directed through the hole and onto a reactor component. This procedure permits accurate visual alignment of the detector above a desired spot on a reactor component. A new premium-quality Ge(Li) diode in a right-angle cryostat is mounted so that it can be moved horizontally in and out of the gamma-ray beam. The detector is positioned reproducibly over the hole by means of the laser. Gamma spectra are obtained with a 4096-channel analyzer and are recorded on magnetic tape. The channel position and areas of full-energy peaks are measured by a computer, which uses the code, GAMSPEC3.[54] This new spectrometry system permits spectral measurements over the energy range 0 to 2 or 0 to 4 MeV with significant separation of lines that differ by only 2 to 3 keV. Thus, the number of detectable peaks (i.e., fission products) and the accuracy of peak-area measurements were greatly increased. Other improvements in the experiment include provisions for more accurate calibration of the detector and four access holes drilled through reactor shielding to permit measurements during and immediately after reactor operation.

About 200 gamma-ray spectra have been recorded. Most of these were obtained above the heat exchanger and the off-gas system during reactor shutdown in June 1969. The fission products detected were those reported previously.[52,53] Recently, spectra have been obtained over an off-gas line during low- and full-power operation and during a reactor shutdown of a few days duration. Preliminary analysis of these spectra indicates that all the gamma-emitting gaseous fission products (and their daughters) with half-lives equal to or greater than that of ^{140}Xe ($t_{1/2}$ = 16 s) were detected. Because each of these spectra has more than 100 full-energy peaks, their analysis would be extremely laborious without GAMSPEC3. Quantitative results for the fission-product measurements will be calculated as soon as absolute calibrations of the detector have been completed.

Discussions of this work were presented orally.[55,56]

[54] A. F. Joseph, *Program GAMSPEC3*, Nov. 15, 1968 (unpublished).

[55] A. Houtzell, R. Blumberg, and F. F. Dyer, "Deposition of Fission Products in a Molten Salt Reactor System," to be presented at the Winter Meeting of the American Nuclear Society, San Francisco, Calif., Nov. 30–Dec. 4, 1969.

[56] R. Blumberg, F. F. Dyer, and A. Houtzell, "Remote Gamma Spectrometry for Fission Product Deposition Studies," to be presented at the Winter Meeting of the American Nuclear Society, San Francisco, Calif., Nov. 30–Dec. 4, 1969.

5.7 EFFECTS OF RADIATION ON METHODS AND MATERIALS

5.7.a Gamma Radiolysis of Iodide Solutions

Hisashi Kubota

Iodide is frequently encountered as a minor anionic constituent, so the radiolysis of iodide was studied on iodide by itself and in the presence of a number of common anions. Neutral, acid, and basic media were used; a special study was made on the effect of nitrate.

A 0.1 *M* KI solution buffered at pH 6.9 (K_2HPO_4-KH_2PO_4) forms free iodine, which is indicated by the growth of the I_3^- spectrophotometric absorption peaks at 283 and 350 nm. The 283-nm peak has the greater sensitivity; but the 350-nm peak suffers less interference, particularly from nitrate. The *G* value for I_3^- formation is ~0.4. This radiolysis is affected very slightly by the presence of 0.1 *M* Cl^-, SO_4^{2-}, or ClO_4^-; 0.1, 1, and 3 *M* NO_3^- increase the *G* value to 2.6, 4.0, and 4.8 respectively.

The rate of formation of I_3^- in the presence of nitrate increases with increase in pH. This formation competes with the reaction between the I_3^- and base to form I^- and IO^-. At pH's up to 10, the formation of I_3^- is faster than the latter reaction, and a net I_3^- absorption can be observed. The second reaction takes over at pH's >10. Thus, free iodine is formed in irradiated basic nitrate medium and then reacts with the base. With respect to both NO_3^- concentration and pH, the rate of I_3^- formation parallels very closely the formation of NO_2^- produced by the irradiation of NO_3^-.[57] Iodide in basic NO_2^- will form I_3^- only when irradiated.

Acid iodide solutions exposed to air are oxidized spontaneously; therefore, the radiolytic studies were made with air-free acid solutions. Nitric acid oxidized I^- immediately both in the presence and absence of free oxygen. The I_3^- formation in deaerated HCl parallels the Cl_2 formation, which depends on the HCl molarity.[58] In a similar manner, the oxidation of I^- in H_2SO_4 parallels the radiolytic behavior of

[57] M. L. Hyder, "The Radiolysis of Aqueous Nitrate Solutions," *J. Phys. Chem.* **69**, 1858 (1965).

[58] H. Kubota, "Chlorine Production by Gamma Radiolysis of Acid Chloride Solutions and Its Effect on Analytical Procedures," *Anal. Chem.* **40**, 271 (1968).

H_2SO_4,[59,60] and a 10^6-rad dose to a solution in 6 *M* H_2SO_4 completely oxidizes all the I^- (0.1 *M*) to free iodine.

5.7.b Gamma-Radiation Stability of Standard pH Buffers

Hisashi Kubota

Some commercially available standard pH buffers were tested for gamma radiolytic stability preliminary to the study on the radiolytic stability of the calomel and glass electrodes (Sect. 5.7.c). The results are shown in Table 5.5. The pH-4 buffer is a solution of potassium acid phthalate; its decomposition gives K^+, which makes the medium more basic. The pH-7 and -10 buffers become colored after a small dose of gamma radiation, but the color eventually disappears. The very small change in the pH's of the pH-7 (phosphate) and -10 (borate) buffers indicates that these buffers are stable. A 0.2 *M* KH_2PO_4 buffer (pH 4.5) was prepared, and its stability was tested. It showed no change after having received a 10^7-rad dose.

5.7.c Stability of the Calomel and Some Indicator Electrodes to Alpha and Gamma Radiation

Hisashi Kubota

The behavior of calomel and certain indicator electrodes in a gamma radiation field was followed. The electrodes tested were the saturated calomel, glass, fluoride-sensitive, and nitrate-sensitive electrodes. The calomel electrode was placed in a partly shielded position within a ^{60}Co-source cavity and was connected to a half-cell that contained a hydrogen electrode placed outside the source. Both half-cells were filled with pH-4.5 phosphate buffer. The behavior of the calomel electrode was followed at gamma-ray dose rates of 40, 90, 250, 1000, and 1.5×10^4 rads/min. No change in response occurred at any of the dose rates. Prolonged exposure at the highest dose resulted in a drift of ~0.2 pH, which was found to be a temperature effect that disappeared when the electrode was cooled to ambient temperature. The calomel electrode is stable in a gamma radiation field up to a dose rate as great as 1.5×10^4 rads/min and a cumulative dose of 2×10^7 rads.

Table 5.5. Gamma-Radiation Stability of Standard pH Buffers

Gamma Radiation Dose (rads)	pH of Buffer		
	pH 4	pH 7	pH 10
0	4.000	7.093	10.021
5×10^5	4.253	7.083	10.030
2×10^6	4.861	7.131	10.035
2×10^7	5.802	7.095	10.041

Similarly, the glass electrode remains stable in radiation fields as intense as 1.5×10^4 rads/min. There was a permanent pH increase of 0.1 to 0.3 after a 10^7-rad dose. It was found that unless the leads to high-impedance electrodes within a gamma-radiation field are shielded adequately and uniformly, they can be a source of trouble. The usual type of junction boxes cannot be used within a source cavity.

The fluoride-sensitive electrode (Orion solid-state) also is stable when first encompassed by a gamma field. However, with a prolonged dose, the calibration curve shifts toward more negative potentials vs S.C.E. (Fig. 5.12). Thus, this electrode can be used in a gamma field provided it is recalibrated periodically with standard solutions.

The nitrate-sensitive electrode (Orion membrane) gives stable readings when initially exposed to a gamma field of dose rate as great as 1.5×10^4 rads/min. It likewise shows a parallel translation of the calibration curve with prolonged dose, after which the curve becomes nonlinear (Fig. 5.13). This nonlinearity is removed when the internal nitrate solution of the electrode is replaced.

A study was begun of the effect of alpha radiation on these electrodes. The calomel and glass electrodes have shown no ill effects after having been immersed in a solution of ^{244}Cm and thus subjected to a cumulative dose in excess of ~10^7 rads.

5.7.d Determination of Hydrogen Peroxide

Hisashi Kubota

A method was developed to determine H_2O_2 in the small concentrations found in radiolytic studies. The method consists in the alkaline oxidation of chromic ion by H_2O_2 and the spectrophotometric measurement at 541 nm of the resulting chromate ion as the diphenyl

[59] J. W. Boyle, "The Decomposition of Aqueous Sulfuric Acid Solutions by Cobalt Gamma Rays. I. Radical and Molecular Product Yields from Ce(IV) Solutions in 0.4 to 18 *M* Acid," *Rad. Res.* 17, 427 (1962).

[60] J. W. Boyle, "The Decomposition of Aqueous Sulfuric Acid Solutions by Cobalt Gamma Rays. II. Yields of Solvent Decomposition and Reducing Radicals from Fe(II) Solutions in 0.4 to 18 *M* Acid," *Rad. Res.* 17, 450 (1962).

semicarbazide. Nitrite ion interferes. A 25-μmoles/liter solution of H_2O_2 will give an absorbance of one unit in a 1-cm-pathlength cell. With the buffered KI procedure commonly used, this absorbance is not attained until the H_2O_2 concentration is 38.8 μmoles/liter.[61] The new procedure is the better one for determining H_2O_2 in radiolytic perchloric acid medium.

[61] D. Katakis and A. O. Allen, "The Radiolysis of Aqueous Perchloric Acid Solutions," *J. Phys. Chem.* 68, 3107 (1964).

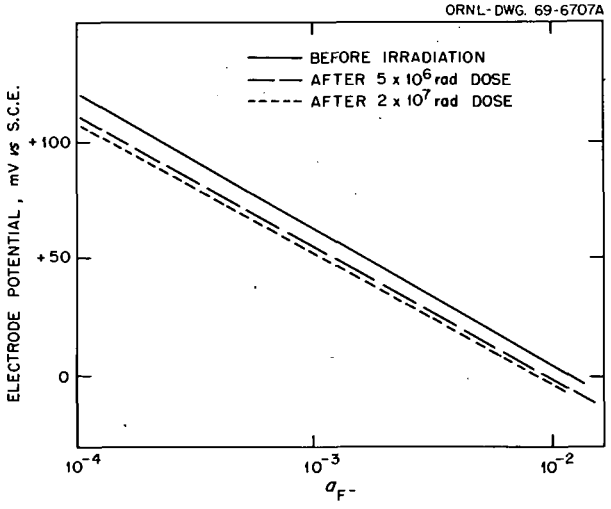

Fig. 5.12. Effect of Gamma Radiation on the Fluoride-Sensitive Electrode (Orion Solid-State).

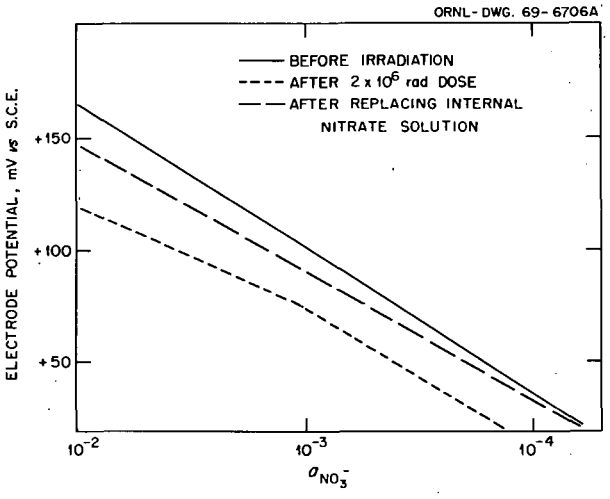

Fig. 5.13. Effect of Gamma Radiation on the Nitrate-Sensitive Electrode (Orion Membrane).

5.7.e Homogeneous Precipitations with Radiolytically Generated Reagents

Hisashi Kubota

The homogeneous precipitation of Ag^+ as silver chloride with hydrochloric acid radiolytically generated from chloral hydrate was reported previously.[62] This precipitation was approached from another angle by releasing Ag^+ from its ammonium complex with hydrochloric acid radiolytically generated from ammonium trichloroacetate. The resulting precipitate is crystalline, in sharp contrast to the consistently amorphous product obtained before. The possibility of effecting precipitations either by the release of anion or cation offers an interesting approach to the study of precipitation phenomena.

5.7.f Spray and Pool Absorption Technology Program Support

H. E. Zittel S. B. Lupica
J. R. Lund[63]

5.7.f.a Radiation, Thermal, and Chemical Stability of Spray Solutions. – Studies on the effects of radiation on radiation-safeguard spray solutions[64,65] were continued. Some of the results were presented orally;[66] a summary follows.

Water sprays possibly useful in suppressing pressure buildup following accidental loss of reactor coolant are currently being tested for concomitant fission-product removal. It is anticipated that these sprays will be composed of the borated reactor-coolant water to which chemicals are added that will react with the fission products (largely ^{131}I). The two proposed sprays now of greatest interest are the basic borate (0.15 *M* NaOH–3 ppb B) and the basic thiosulfate (1 wt % $Na_2S_2O_3$–0.15 *M* NaOH–3 ppb B).

[62] H. Kubota, "Application of Radiolytically Generated Reagents in Analytical Chemistry," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967,* ORNL-4196, p. 14.

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

[64] 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.

[65] H. E. Zittel and J. R. Lund, "Effects of Radiation on Analytical Methods. Radiolysis of Radiation-Safeguard Spray Solutions," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968,* ORNL-4343, p. 80.

[66] H. E. Zittel and T. H. Row, "Radiation and Chemical Stability of Spray Additives," presented at the 169th Annual Meeting of the American Nuclear Society, Seattle, Wash., June 15–19, 1969.

It is estimated that these sprays will be exposed to a maximum gamma-radiation dose of ~3 X 10^8 rads accumulated over a period of several weeks. The temperature of the reactor environment will vary from room temperature to ~150°C. Therefore, it is imperative that any given spray solution be stable at these temperature extremes. We have learned that the basic borate solution is completely stable at 150°C and that the basic thiosulfate solution is acceptably stable (10% decomposition after 72 hr at 140°C). The radiolytic stabilities of the two test solutions, as established by exposure to a ^{60}Co gamma source, follow the same pattern – the basic borate solution is essentially unaffected by a dose of 10^8 rads, whereas the effects on the basic thiosulfate are additive, i.e., radiation at 140°C gives approximately the same degradation as the sum of the radiation and thermal processes carried out separately.

Of great concern is the generation of radiolytic H_2 from the spray solutions. Contrary to results expected for pure water, the radiolytic gases do not reach equilibrium at a low pressure in these solutions; the data of Table 5.6 support this statement. The $G(H_2)$ values indicate that the systems move toward an equilibrium state. However, in neither case is equilibrium attained at a low pressure. The data also show clearly that the radiolytic reaction paths for the two solutions differ. The basic borate solution produces both radiolytic O_2 and H_2, whereas the basic thiosulfate solution used up O_2 and produces H_2. In each case, radiolytic H_2 is formed in amounts sufficient to be a problem.

The compatibility of the proposed spray solutions with metals of the type normally found in reactors was studied under static conditions. Under radiation, copper-bearing alloys catalyze the radiolytic decomposition of the basic thiosulfate solution, whereas none of the metals studied seem to affect the radiolytic behavior of the basic borate solution. The metals themselves show relatively little change, as might be expected under static conditions. However, aluminum alloys corrode in both test solutions; copper alloys do also, but to a lesser extent.

Because some of the ^{131}I fission product may be present as $CH_3{}^{131}I$, various additives to enhance the CH_3I "gettering" properties of the spray solutions are under test. The radiation stability of such additives was studied. In general, these additives are polyamines and have sufficient radiation stability for use under conditions expected to obtain in an accident.

5.7.f.b Dynamic Radiation Facility. – All tests carried out previously in the Spray and Pool Absorption

Table 5.6. Data that Show Radiolytic Gas Generation from Proposed Radiation-Safeguard Spray Solutions

Gas/Liquid Ratio in Test Container	ΔO_2 (cc/ml)	ΔH_2 (cc/ml)	$P(H_2)$ (atm)	$P(H_2 + O_2)$ (atm)	$G(H_2)$
Solution: 0.15 *M* NaOH–3 ppb B; gamma radiation dose = 1.2 X 10^8 rads					
25	+0.05	+1.06	+0.04	+0.25	+0.43
5	+0.48	+1.40	+0.28	+0.57	+0.57
1	+0.46	+1.14	+1.14	+1.79	+0.46
0.5	+0.36	+0.82	+1.66	+2.53	+0.33
0.1	+0.15	+0.39	+3.92	+5.58	+0.16
Solution: 1 wt/% $Na_2S_2O_3$–3 ppb B–0.15 *M* NaOH; gamma radiation dose = 1.1 X10^8 rads					
25	–3.26	+0.97	+0.04	+0.01	+0.40
5	*a*	+1.21	+0.24	+0.24	+0.50
1	*a*	+1.17	+1.16	+1.16	+0.48
0.5	*a*	+1.12	+2.24	+2.24	+0.46
0.1	*a*	+0.73	+7.40	+7.40	+0.30

[a]All O_2 has disappeared.

Technology Program have been done under static conditions, that is, capsule-type irradiation. Because the applicability of the results of such static tests to the real case is questionable, a dynamic test facility is being established. A miniature stainless steel system was fabricated which simulated the thermal and radiation conditions that might obtain during a loss-of-coolant accident. Figure 5.14 is a block diagram of the system, which consists of a radiation capsule of variable capacity (10 to 100 ml), a reservoir also of variable capacity (100 to 5000 ml), and a variable-speed flow pump, all connected together with ¼-in. stainless steel tubing. There are provisions for following radiolytic buildup of gas pressure and for sampling at desired intervals. With this system, the effects of such variables as flow rate, temperature, and gas pressure on the radiolytic behavior of the proposed sprays will be studied.

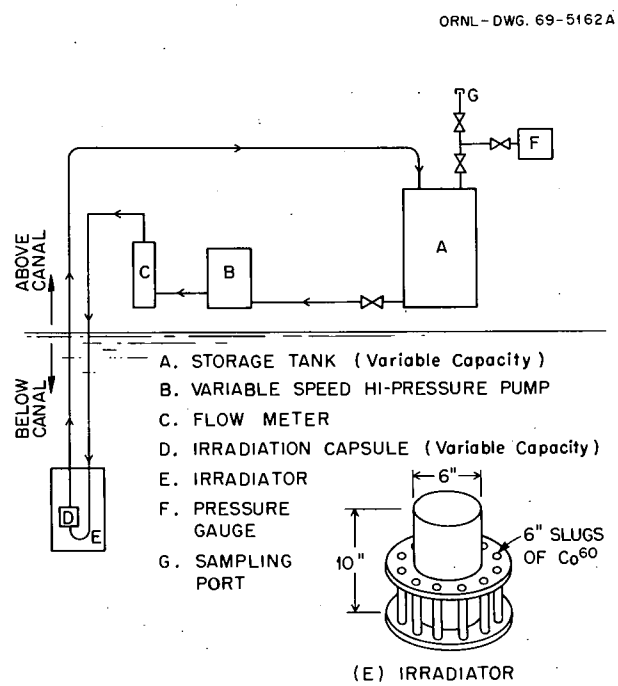

Fig. 5.14. Schematic Diagram of Dynamic Radiolysis Loop.

5.7.g Alpha-Radiation Damage in Inorganic Phosphors

H. H. Ross

The program to study alpha-radiation damage in organic phosphors is being continued.[67] The oversized glove-box assembly was fabricated and installed; the box is located in the High-Radiation-Level Analytical Laboratory. The optical measuring equipment has been placed in the box, interfaced with an external electronic package, and tested for proper response. The preparation of the phosphor-isotope (^{241}Am) mixtures has been started, and some preliminary intensity measurements have been completed. However, no significant results have as yet been collected.

[67]H. H. Ross, "Alpha-Radiation Damage in Inorganic Phosphors," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968,* ORNL-4343, p. 68.

5.7.h Dihydroxyanthraquinone Complexes of Zirconium

H. E. Zittel

An article on the nature of the zirconium complexes of alizarin red S, alizarin, and hystazarin was accepted for publication.[68]

[68]T. M. Florence, Y. J. Farrar, and H. E. Zittel, "Dihydroxyanthraquinone Complexes of Zirconium," accepted for publication in the *Australian Journal of Chemistry.*

6. Methods Development and Evaluation

L. T. Corbin
P. F. Thomason
D. A. Costanzo

6.1 DETERMINATION OF TRANSURANIUM ELEMENTS

6.1.a Absorption Spectrophotometric Determination of Americium

D. A. Costanzo

Absorption spectrophotometry was used for the rapid and precise determination of americium in the presence of an ~100-fold weight excess of curium. Radiochemical and coulometric-titration methods for the determination of ^{243}Am in the presence of the large amounts of ^{244}Cm encountered in TRU process samples have not been entirely satisfactory.

In 1 *N* $HClO_4$, Am(III), which is the stable oxidation state of americium in aqueous solution, exhibits a single, sharp, analytically useful absorption peak at 5034. At this wavelength, Cm(III) does not absorb and the absorbance of Am(III) obeys Beer's law.

Standard spectral data were obtained for Am(III) in the various aqueous media encountered in analytical sample preparation and TRU processing, that is, perchloric, hydrochloric, sulfuric, and nitric acids and potassium carbonate and carbonate-nitrate media. Figure 6.1 shows the gross effects of these reagents on the shape of the Am(III) absorption peak; Fig. 6.2 shows the relationship between the molar absorbance of Am(III), measured at the absorption maximum, and the concentrations of the various reagents.

The Cary model 14 spectrophotometer and glove-box system[1] was used to obtain spectral data. With a 0- to 0.1-absorbance-unit slide wire and micro absorption cell, it was possible to measure 1 to 2 μg of ^{243}Am in the presence of a 100-fold excess of ^{244}Cm.

6.1.b Absorption Spectrophotometric Determination of Plutonium

D. A. Costanzo

Absorption spectrometry was used routinely to determine the valence-state distribution of plutonium for the conditions under which it occurs in solution during processing, purification, and sol-gel formation and to follow the course of the reaction of plutonium during sol preparation. A Cary model 14 spectrophotometer and glove-box system[1] are used to obtain spectral data. Analysis of the data for a multicomponent system, which may contain ionic Pu(III), Pu(IV), Pu(V), and Pu(VI) in equilibrium with each other and in the presence of polymeric Pu(IV), is accomplished by means of the Com-Share computer.

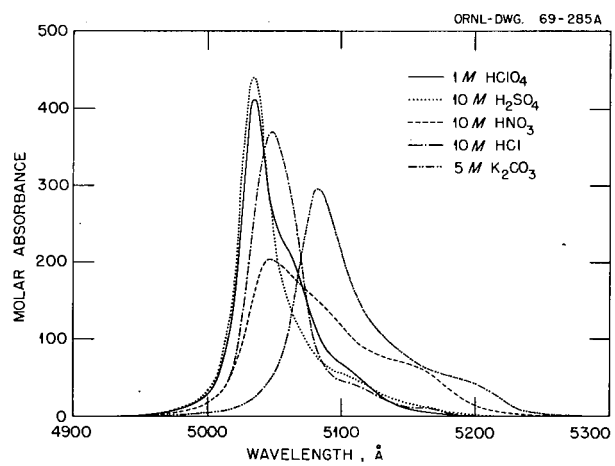

Fig. 6.1. Absorption Peak of Am(III) in Various Aqueous Media.

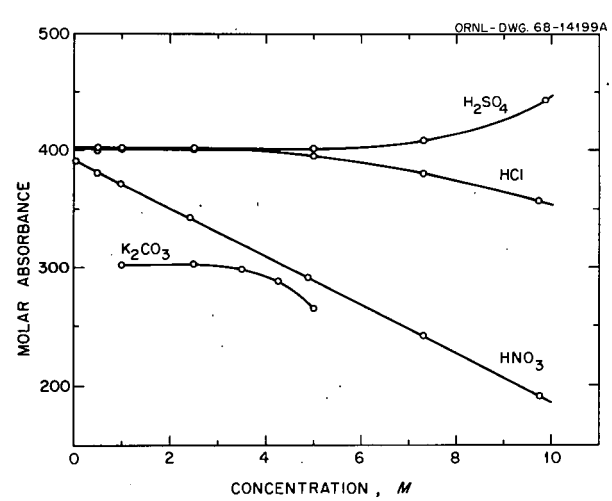

Fig. 6.2. Molar Absorbance of Am(III) in Various Aqueous Media Measured at the Absorption Maximum.

[1] D. A. Costanzo and L. T. Corbin, "A Glove Box System for a Spectrophotometer," *Nucl. Appl.* 5, 333 (1968).

6.1.c Self-Oxidation of ^{238}Pu(IV) in Nitric Acid

D. A. Costanzo

In a study done in cooperation with R. G. Haire,[2] it was observed spectrophotometrically that when a concentrated solution of ^{238}Pu(IV) in strong nitric acid is diluted with more-dilute nitric acid, ^{238}Pu(IV) is oxidized to ^{238}Pu(VI). This self-oxidation occurs under conditions of acidity and temperature for which ^{238}Pu(IV) heretofore was thought to be stable. As Figs. 6.3 and 6.4 show, the rate of self-oxidation of ^{238}Pu(IV) to ^{238}Pu(VI) is a function of acidity and plutonium concentration. This type of oxidation behavior has not been observed for ^{239}Pu(IV); it is thought to be due to the reaction of the ^{238}Pu(IV) with the radiolysis products produced by the intense alpha radiation from ^{238}Pu.

6.1.d New Solvent-Extraction Method for the Rapid Separation of Cerium from Berkelium and Other Elements

F. L. Moore

Research and development studies were completed on a new liquid-liquid extraction method for the rapid separation of cerium from berkelium and many other elements. The method affords the first practical liquid-liquid extraction separation of Ce(IV) from Bk(IV). The work is described in a current article,[3] the abstract from which follows:

"A new liquid-liquid extraction method for the rapid separation of cerium from berkelium and many other elements is described. The method is based on the preferential extraction of cerium(IV) from dilute nitric acid solution containing bromate ion with the quaternary ammonium nitrate, Aliquat 336-S-NO_3, dissolved in xylene. A postulate for the marked difference in extraction behavior of berkelium(IV) and cerium(IV) is advanced. Several useful analytical and process applications are discussed."

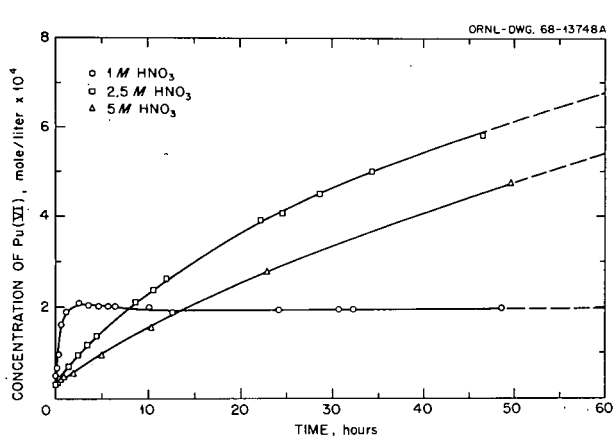

Fig. 6.3. Plots that Show Self-Oxidation of ^{238}Pu(IV) to ^{238}Pu(VI) in Aqueous Nitric Acid Solutions of Various Concentrations. (Total Pu concentration = 2.5×10^{-2} *M*.)

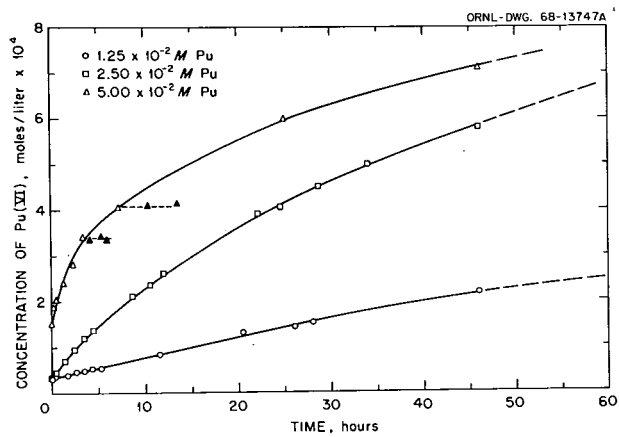

Fig. 6.4. Plots that Show Self-Oxidation of ^{238}Pu(IV) to ^{238}Pu(VI) in Solutions of Various Total ^{238}Pu Concentrations. (Medium, 2.5 *M* HNO_3.)

6.2 ANALYSIS OF REACTOR FUELS

6.2.a Precise Determination of Uranium in MSRE Fuel

Gerald Goldberg

To increase the precision of the determination of uranium in MSRE fuel, a method was developed that involves the preliminary separation of the uranium from the fuel within a hot cell.[4] By high-temperature fluorination of the fuel, the uranium is converted to gaseous UF_6, which is collected in a trap of sodium fluoride. Although the beta-gamma radioactivity of the sample is high, the radioactivity within the trap is <20 millirem. Therefore, the trap can be removed from the hot cell, the contents dissolved, and the uranium – now free from interfering ions – determined with a high-sensitivity controlled-potential coulometric titrator. The alternative is to dissolve the fuel sample in the hot

[2] Chemical Technology Division.

[3] F. L. Moore, "New Liquid-Liquid Extraction Method for the Rapid Separation of Cerium(IV) from Berkelium(IV) and Other Elements," *Anal. Chem.* 41, 1658 (1969).

[4] G. Goldberg, "Precise Determination of Uranium in MSRE Fuel," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968*, ORNL-4343, p. 89.

cell and to titrate the uranium remotely in the presence of the other ions.

Preliminary results were encouraging, although the recovery of the uranium was less than the anticipated 99.8%. A second NaF trap in series with the collection trap was void of uranium after a fluorination. Finally, it was determined that the illusive uranium was deposited on the cooler upper section of the reaction vessel along with other constituents of the MSRE fuel. Since this deposition was believed to be due, in part, to sublimation, the fluorination temperature was reduced from 600° to 525 to 550°C.

A series of samples was taken from a container of simulated MSRE fuel. Since there was no radioactivity present, the samples were analyzed for uranium on the bench top rather than remotely in the hot cell, as well as by high-temperature fluorination. The results by both methods agreed to within ±0.2%, as did these same results with the theoretical value for the uranium added as UF_4 to prepare the simulated fuel. No salt was deposited on the upper section of the reaction vessel at the lower fluorination temperature (525°C). This method will be used to determine uranium in the MSRE fuel when necessary.

The fluorination apparatus has been used for determinations other than that of uranium. It permits the rapid separation of the uranium from the radioactive fission products contained in the fuel, as well as from the fuel. Thus, a more precise isotopic analysis of the uranium is possible. The UF_6 is desorbed from the NaF trap at 400°C and is collected in a cold trap. A number of samples for isotopic analysis have been handled in this manner.

The separation of uranium from the fuel also simplifies the determination of plutonium, which is present in milligram amounts in the salt sample. The residual salt, after fluorination, is removed from the reaction vessel and is dissolved in nitric acid. The solution is passed through an anion-resin column in the nitrate form, and the plutonium is retained by the resin. Eventually, the plutonium is stripped from the resin and is determined coulometrically. Uranium would also remain on the resin and would interfere with the subsequent titration. An alternative is a liquid-extraction procedure to separate the plutonium from the uranium and the other components of the salt. This type of separation is difficult to carry out within a hot cell.

6.2.b Inverse Polarographic Determination of Bismuth

P. F. Thomason

It was mentioned previously[5] that inverse polarography was to be studied for the determination of bismuth at the ppb concentration level. It has been found that a purified metal-free 1.5 *M* solution of HCl permits the determination of bismuth in the presence of copper up to a 1:5 ratio by weight. The Bi(III) → Bi reduction shows an anodic peak at –0.08 V vs S.C.E., and Cu(II) → Cu reduction appears at –0.18 V vs S.C.E. Calibration curves are fairly linear for the Bi concentration range 1.3 to 10 ppb. Special samples from the Chemical Technology Division that contained Li, Be, U, and Th as fluorides were analyzed successfully for Bi by this method. Synthetic MSRE samples will be analyzed soon.

6.2.c Controlled-Potential Coulometric Titration of Uranium and Plutonium

P. F. Thomason

Three new all-solid-state controlled-potential coulometric titrators were obtained from the M-T Electronics Company, San Leandro, Calif. They were checked and calibrated according to the instructions and were found to be very stable and accurate. They have been placed in service for analyzing samples that contain uranium and plutonium.

Three types of titration cells for the controlled-potential coulometric titration of uranium were evaluated; the results were presented orally.[6]

6.2.d Proposed Reactor Fuels

6.2.d.a Uranium Nitride and Uranium Carbonitride (J. L. Botts). – In cooperation with the Metals and

[5] P. F. Thomason, "Thin-Film Mercury Electrode Voltammetry of Bismuth," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968*, ORNL-4343, p. 88.

[6] P. F. Thomason and W. R. Laing, "Precision Studies for the Controlled-Potential Coulometric Titration of Uranium and Plutonium – Comparison of Various Electrolysis Cells," 13th Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Sept. 30–Oct. 2, 1969.

Ceramics Division, work was begun in February on the analytical requirements of the Liquid Metals Fast Breeder Reactor (LMFBR) Program. The main effort has been toward updating our methods to better characterize the uranium nitride and uranium carbonitride fuels and, to some extent, the oxide fuels. Two interlaboratory checks were initiated to compare our analytical capability with that of other sites doing this type of work. Also, one intralaboratory check was made to compare glove-box with bench-top operations for a single method. New equipment has been ordered for use in this work. A more detailed discussion of the work follows.

At the beginning of the uranium nitride and uranium carbonitride phase of the LMFBR Program, very little analytical work had been done on these materials. Because of the reactivity of these fuels with atmospheric oxygen and water, the data available were unreliable. To establish some confidence in the analytical methods, the Metals and Ceramics Division initiated an interlaboratory check. Specimens of uranium nitride were fabricated into pellets (~3 g) and sintered at 1650°C in argon. Four groups of these specimens were chosen at random, packaged in an inert atmosphere, and sent to laboratories of the National Aeronautics and Space Administration (NASA), Cleveland, Ohio, Battelle Memorial Institute, Columbus, Ohio, LeDoux & Company, Teaneck, N.J., and to our General Analyses Laboratory. At each laboratory, the pellets were opened and prepared for analysis in an inert atmosphere. For the determinations, aliquots for each analysis were then transferred outside the special atmosphere. These specimens were analyzed for uranium by the gravimetric-combustion method and for nitrogen by the micro Kjeldahl method. Battelle used an open-beaker dissolution technique for the nitrogen determination, whereas, the other laboratories used the sealed-tube dissolution method.[7] Oxygen was determined by inert fusion except that Battelle used vacuum fusion. At each site, carbon was determined by the Leco oxygen-combustion method. The results from all four sites were in very good agreement except the nitrogen and oxygen results from Battelle, which were both low. This agreement indicates that the methods are satisfactory for the analysis of uranium nitride.

Another group of specimens were prepared in exactly the same way and distributed to the same laboratories; not all the results are completed. The data returned, including that from our General Analyses Laboratory, again show very good agreement among the laboratories. The levels of concentration are: uranium, 94.50%; nitrogen, 5.30%; oxygen, 0.20%; and carbon, 200 ppm.

Much of the time has been spent in obtaining equipment needed to update or improve the analytical methods to be used in the LMFBR Program. The equipment includes that for methods to be used in glove boxes and hot cells. Since the proposed fuels are very reactive and since the systems will contain plutonium, inert-atmosphere glove boxes are required. Drawings and specifications for eleven of the boxes, including purification units for each, were prepared, and the assemblies were ordered. We will have inert-atmosphere boxes located in the laboratories in Buildings 2026, 3508, and 4500S. The new equipment that has been obtained or is now on order includes: seven solid-state coulometers to replace all existing coulometers, Leco oxygen analyzers and induction heaters to be installed both in glove boxes and in the hot cell, two Aminco porosimeters to be used in glove boxes and in the hot cell, Leco carbon equipment, thermogravimetric-analysis equipment, and surface-area equipment that includes an integrator for the continuous-flow surface-area method.

We are now installing the oxygen equipment in the hot cell in Building 2026. This installation involves the modification of the Leco analyzer and the 7½-kW induction heater. Other equipment, such as that for gas-release, carbon, and density-porosity measurements, will be placed in the cell as it is needed to analyze the irradiated fuels.

6.2.d.b Uranium Carbide–Plutonium Carbide (L. G. Farrar). – Recent interest in carbide nuclear fuels initiated studies to prepare uranium carbide–plutonium carbide by sol-gel techniques. Metal-carbon sols were made into microspheres and were calcined at elevated temperatures to produce the carbides. We have studied the compositions of the products and the effects of various calcination conditions. Thermogravimetric, chemical, and x-ray analyses were made on both the starting materials and the carbide products. At first, plutonium carbide was prepared separately from uranium carbide; analyses of the calcined products for plutonium, oxygen, and carbon gave excellent material balances. In later work on the uranium carbide–plutonium carbide products, fast oxidation of the mixtures caused difficulties in obtaining accurate oxygen analyses and product compositions. The acquisition of inert-atmosphere apparatus for sample preparation and analysis should eliminate the problem of oxidation before analysis.

[7] D. C. Canada, "Sealed-Tube Dissolutions," *Anal. Chem. Div. Ann. Progr. Rept. Oct. 31, 1967,* ORNL-4196, p. 41.

6.2.d.c Urania-Plutonia (P. F. Thomason, L. G. Farrar). – An Ainsworth No. 1015 electronic balance was installed in a double glove box to permit thermogravimetric analysis of alpha-emitting materials. The balance is being used to determine moisture and oxygen-metal ratios in UO_2-PuO_2 samples. Microspheres of various sizes and densities were characterized as to moisture content, weight changes at elevated temperatures, and reduction-oxidation rates at various pressures and temperatures.

7. Mass Spectroscopy

A. E. Cameron

7.1 SPARK-SOURCE MASS SPECTROMETRY

J. A. Carter J. R. Sites

The Associated Electrical Industries, Ltd. (AEI) MS-702 spark-source mass spectrometer (Fig. 7.1) was installed in Room 133 of the new addition to Building 2026. The instrument is to be used for the analysis of alpha-emitting radioactive and transuranium elements. Functionally, with the exception of a demountable source-housing, the MS-702 instrument is very similar to the MS-7,[1] which was installed in 1964 in Building 9734 for the analysis of nonradioactive samples. The demountability of the source-housing facilitates the decontamination of the source after radioactive samples are analyzed. A glove box is fitted onto the source end of the instrument; this arrangement permits the handling of alpha-emitting samples whose radioactivity is as intense as $\sim 10^9$ dis/min.

The MS-702 has high resolution. With a 3-mil slit, the resolution is 4000 at an m/e ratio of 208. With a 10-in.-long photoplate as detector, the instrument spans the m/e range 10 to 313; with a 12-in.-long photoplate, the range is extended to an m/e of $\sim$400. The high resolution permits the use of graphite as a substrate for transuranium samples without interferences from hydrocarbons or singly charged carbon-polymer ions that may be formed by the rf-spark ion source. In general for the transuranium elements, a solution that contains 1 to 10 μg of sample in a reasonable volume is spiked with a standard solution of normal erbium, and the resulting solution is evaporated on the tips of two small spectrographically pure graphite, pure gold, or pure niobium electrodes. The microphotometric data from the photoplate is reduced by means of an IBM-1130 computer program. This method of trace analysis permits the determination of as little as 0.01 wt % impurity in a 2-μg sample.

The MS-702 instrument has been used to determine impurities in samples of the following isotopes of the transuranium elements: ^{241}Am, ^{243}Am, ^{246}Cm, ^{248}Cm, ^{249}Bk, ^{249}Cf, and a ^{232}Th-^{241}Am (24:1)

Fig. 7.1. Associated Electrical Industries, Ltd. (AEI) MS-702 Spark-Source Mass Spectrometer Installed at ORNL.

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

alloy. In the characterization of the results of an irradiation of enriched ^{136}Ce to produce ^{137}La, the MS-702 instrument was used to analyze a few micrograms of the highly radioactive solution left after most of the total cerium was chemically removed. In the same manner, two 1-μg samples of molybdenum that had plated out on MSRP graphite surveillance specimens were analyzed. The estimated relative abundances of the natural and fission-product molybdenum isotopes showed that niobium deposited more strongly than molybdenum.[2]

The MS-7 spark-source mass spectrometer continued to be used mainly to determine trace elements in special alloys and in pure metal oxides. Samples of isotopically enriched metallic elements were also analyzed, for example, ^{28}Si, ^{90}Zr, ^{92}Zr, ^{94}Zr, ^{157}Gd, and ^{167}Er (only a few milligrams of each was consumed). Samples of the following were analyzed: Fe, V, Nb, W, W-Re-Mo, Au, Th, and U. For an EBR-II Reactor Study, several samples of residue from the distillation of very pure metallic sodium were analyzed.

7.2 ORGANIC MASS SPECTROMETRY

W. T. Rainey W. H. Christie

As in the past, the organic samples for mass spectrometry were predominantly from biologically oriented programs. The Body Fluids Analyses Program has furnished a large number of samples, both knowns for comparison purposes and unknowns for qualitative identification. Some components contained in fractions from the liquid chromatographic columns were identified, especially when gas-chromatographic data suggested possible compound types. It was observed that acidification with HCl and subsequent evaporation of methanol solutions of unknowns often resulted in the formation of methyl esters. Recognition of this possibility was an asset in understanding the mass spectra of some samples.

Mass spectra were useful in determining the efficiency of the preparation of the derivatives used for amino acid sequence determination (Sect. 4.2.d) in the Molecular Anatomy (MAN) Program. Proof of identity and estimate of purity were possible with many phenyl- and methylthiohydantoins and phenylthioazolones.

Assistance was also given in the National Academy of Sciences–National Research Council (NAS-NRC) program on Specifications and Criteria for Biochemical Compounds (Sect. 4.3.c). Nucleosides, nucleotides, and free nucleic acid were studied with varied success. The spectra obtained from bases and nucleosides were as expected, but those from nucleotides were of little value. Impurities were detected in several of the materials.

Several compounds of interest in the Chemical Mutagenesis Program (Sect. 4.3.b) were studied for possible impurity content. Work has continued for the Y-12 Development Group in attempts to understand the mechanism of formation of various products found after the polymerization of indene. Further work has also been done to identify products formed by gamma irradiation of hexane-naphthalene mixtures and by radiation-induced oxidation of various organic molecules.

The design of the direct-inlet probe was improved to permit use of a 0.125-in.-diam sample tube. The probe may be cooled rapidly to about $-150°C$ or heated to $350°C$. Several helium separators have been studied for interfacing a gas chromatograph with a mass spectrometer. The sintered-glass Biemann-Watson type proved to be difficult to adjust and resulted in long memory. Single- and double-stage polymethylsilicone-membrane (General Electric Company's Permselective Membrane) separators showed much promise and warrant further study. A sintered stainless steel tubular separator modeled after those in use by Krueger and McCloskey[3] has been satisfactory and will be used on the low-resolution mass spectrometer.

Work is progressing to develop a combination electron-bombardment (EB)–field-emission (FI) ion source. Field-emission ionization of organic molecules usually results predominantly in the formation of molecular ions and is of great value in the determination of molecular weights. Very promising results have been obtained by using commercial razor blades and etched tungsten sheet as ion emitters. With an FI source, a mixture of acetone, benzene, toluene, *o*-xylene, and mesitylene gave only molecular peaks. An FI emitter was installed in the EB source of the low-resolution mass spectrometer. Both EB and FI spectra were recorded, but conversion of one source type to the other and instrument refocusing were slow.

[2] E. L. Compere, S. S. Kirslis, and J. A. Carter, "Composition of Molybdenum on Run 14 Surveillance Specimens," *Molten-Salt Reactor Program Monthly Report for June 1969*, MSR-69-57 (July 1, 1969).

[3] A. M. Lawson, F. A. J. M. Leemans, and J. A. McCloskey, "Characterization of the Carbonyl Position in Keto-Steroids by O^{18} Exchange Reactions," presented at the 15th Annual Conference on Mass Spectrometry and Allied Topics, Denver, Colo., May 14–19, 1967.

All calculations on the organic mass spectra are now being made by the IBM-1130 computer. The lack of interfacing equipment, which is on order, requires that measurements of peak heights and mass numbers be made manually and be hand-punched for computer input. Programs in use allow normalization of the peak intensities and calculation of possible transitions that are indicated by peaks associated with metastable ions. Also, the metastable ions must be identified, their intensities measured, and the data fed in by punched cards. Output is in the form of printed mass numbers and associated normalized peak intensities. Additional output in the form of punched cards is available for plotting bar-graph spectra. A plotter has just been received that is to be attached to the computer. Until now, all plotting has been done by the ORNL computing service.

Other computer programs are in use for magnetic-disk storage and retrieval of organic mass spectra. The data stored are spectrum numbers, molecular weights, masses of metastable ions, and the six most intense peaks in the spectrum together with their associated intensities. A program is available to search the stored library for a known spectrum that matches an unknown spectrum. Comparison is made by means of selected options, either molecular weight or fragment ions.

Lack of interfacing equipment has also prevented full use of the double-focusing spectrometer, which has been used primarily as a test instrument for developing ion sources. Its output is fed to an oscillographic recorder.

7.3 SURFACE-IONIZATION STUDIES

D. H. Smith

The investigation into the mechanism of surface ionization was continued. The surface-ionization comparison technique[4] was used to determine the first ionization potentials of Th, Pu, and Np.[5] The values obtained were: Th, 7.5 ± 0.3 eV; Pu, 5.71 ± 0.06 eV; and Np, 6.16 ± 0.06 eV.

An attempt is now being made to determine the energy of desorption of neutral U from a hot tungsten surface. If it is successful, values for the desorption energies of Th, Pu, and Np will be determined.

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

[5] D. H. Smith and G. R. Hertel, "First Ionization Potentials of Th, Np, and Pu by Surface Ionization," *J. Chem. Phys.* in press (1969).

8. X-Ray and Optical Spectroscopy

Cyrus Feldman

8.1 X-RAY SPECTROSCOPY

H. W. Dunn

The computer codes for calculating results in x-ray fluorescence and electron-probe work were improved by the insertion of routines to calculate and plot least-squares curves in the form of direct or inverse power series up to the ninth power. In a test of the determination of sulfur/phosphorus (S/P) ratios by electron probe,[1] a mixture of mercaptoguanosine and guanosine was used as a reference sample. It was found that if a solution of the mixture is evaporated by freeze-drying on copper foil and the data are taken with a defocused beam in the scanning mode, the homogeneity of the deposit is satisfactory. The results shown in Table 8.1 were obtained with such deposits. In each case, the areal density of P was 20 $\mu g/mm^2$; 100 readings were taken of each mixture.

[1] H. W. Dunn, "X-Ray Spectroscopy," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968*, ORNL-4343, p. 53.

8.2 X-RAY ANALYSIS OF RADIOACTIVE SAMPLES

R. L. Sherman H. W. Dunn

Preliminary sketches were drawn and apparatus lists were compiled for the construction of a laboratory for

Table 8.1. Electron-Probe Measurements of Sulfur/Phosphorus (S/P) Ratios in Residues of Mercaptoguanosine-Guanosine Solutions

Sulfur ($\mu g/mm^2$)	S/P Ratio	S/P Mean X-Ray Intensity Ratio	Relative Probable Error of Mean (%)	
			10 Readings	100 Readings
0.020	0.001	0.00049	±46	±14
0.200	0.010	0.00390	±2	±6.5
2.000	0.100	0.0360	±5.4	±1.7

the analysis by x-ray diffraction, fluorescence, and/or absorption of samples that emit high levels of alpha radiation and moderate levels of beta and gamma radiation. The facility would be located in Building 2026.

8.3 BEAM-SPLITTER FOR USE IN CALIBRATING SPECTROGRAPHIC EMULSIONS

Cyrus Feldman

Reproducibility tests were made on a beam-splitter whose partially transmitting mirror was a 51%-transmittance[2,3] platinum-coated quartz plate. The beam-splitter was located at the slit of a 2.25-m Ebert (Jarrell-Ash Company) spectrograph. Illumination was accomplished by the intermediate-image technique. A real image of an iron arc was thrown onto a diaphragm located 25 cm from the slit; a 1-mm-high by 6-mm-wide slot in the diaphragm, centered on the optic axis, passed only the center portion of the arc image. The lens at the entrance of the beam-splitter projected two images of this slot onto the collimating mirror of the spectrograph; the totally reflecting mirror of the beam-splitter was adjusted to make these images coincide. The height of the spectra was controlled by a diaphragm on the optic axis immediately in front of the beam-splitter. Each exposure thus produced two spectra that differed in intensity by a factor of 3; the spectra were ~1.6 mm high and were 5 mm apart. This study was confined to the 3100- to 3200-Å region. Since the beam-splitter was located at the stigmatic position of the spectrograph, the splitter was slowly oscillated sidewise about its mean position during exposures in order to minimize the possible effects of any local inhomogeneities that might exist in the platinum coating.

Reproducibility of results was tested by making six exposures on SAI 35-mm roll film each day for five days, removing all apparatus from the optical bench each day, and replacing it the next. A fresh portion of developer was used each day; the developer was kept at 20° ± 0.2°C. The densitometric measurements from a given day's exposures (~88 pairs/day) were combined into a single (linear) Seidel-function preliminary curve[4] whose slope and intercept were calculated on the IBM 360 by a least-squares procedure. The mean Y intercept was $\Delta = -0.610 \pm 0.015$ Seidel units.

Since the intensity ratio of the beams that produced this preliminary curve is fixed, this variability means that in the 50 to 80% transmittance region, the slope of the corresponding emulsion-calibration curve is uncertain to within ±0.015/0.610 = ±2.5%. The degree of uncertainty that will attach to an intensity ratio measured on this curve segment will depend on the ratio itself. For an intensity ratio of 1, the uncertainty would, of course, be zero; for a ratio of 3, the uncertainty would be $\pm\{\log^{-1}[(1.025)(0.47712)]\}/3 = \pm 2.7\%$.

8.4 CHEMICAL PRECONCENTRATION OF TRACE ELEMENTS IN BONE ASH: REMOVAL OF PHOSPHATE FROM BONE-ASH SOLUTIONS

Cyrus Feldman F. S. Jones

8.4.a Electrodialysis Procedure

The presence of phosphate in bone-ash solutions interferes with the chemical preconcentration of trace elements in such solutions by the Mitchell-Scott pro-

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

[3] C. Feldman, "Beam Splitter for Use in Calibrating Spectrographic Emulsions," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968,* ORNL-4343, p. 53.

[4] C. Feldman, "Recommended Practices for Photographic Photometry in Spectrochemical Analysis (E 116-67)," *Methods for Emission Spectrochemical Analysis,* 5th ed., American Society for Testing and Materials, Philadelphia, Pa., 1968.

cedure.[5] Since tests[6] have shown that a two-compartment electrodialysis cell rapidly removes the phosphate from a weakly acid solution of bone ash, further tests were made to see how well trace metals are retained in the cathode compartment. If they are retained, they can then be concentrated by the above-mentioned procedure. For each test, a small amount (usually 2.5 μg) of the trace metal in question was treated with an appropriate radioactive tracer and was added to a solution of bone ash. This solution was electrodialyzed to remove phosphate, and the percentage recovery of the trace metal was measured as the sum of the percentage of tracer that remained in the cathode cell and and the percentage that was strippable from the rhodium cathode. In Table 8.2 the results are listed and are correlated with the tendency of each metal to form anionic phosphate complexes.[7] Although it had been realized that such complexes existed in some cases, it had been hoped that their comparatively low mobility, together with the displacement of complexing equilibria resulting from migration of the phosphate, would act to keep metals in the cathode chamber. Retention was satisfactory for Fe (75.7%) and Bi (83.1%), excellent for Co, Eu, Cu, Mn, and Zn (>93%), but unsatisfactory for the remaining 12 elements. Thus, the electrodialysis process can be used to remove phosphate only if the elements (of those tested) to be collected subsequently are Fe, Bi, Co, Cu, Mn, Zn, and the rare-earth elements. It was decided to search for a more generally applicable procedure.

8.4.b Hydrous Zirconium Oxide Procedure

Ion-exchange columns formed from hydrous zirconium oxide proved to be capable of removing as much as 94% of the P from a 3-g sample of bone ash, but the column also retained unacceptably large amounts of trace metals; the recoveries of Sn, Sb, and Cr tracers were only 28, 29, and 69% respectively.

8.4.c Prior Removal of Calcium as the Oxalate

If the Ca (and Sr) are separated from the phosphate in bone ash as the oxalates, the Mitchell-Scott procedure can easily be used to recover, with good collection efficiency, whatever trace elements accompany them (Table 8.3). The phosphate-rich supernatant solution that remains after removal of the Ca and Sr can be treated to remove excess ammonium oxalate. This solution, which then contains phosphoric acid, alkalies, Mg, and trace metals, can be treated to recover the latter by the Mitchell-Scott procedure, and the two batches of trace metals can be combined for spectrographic analysis.

Table 8.2. Recovery of Trace Metals by Electrodialysis of Bone-Ash Solutions; Correlation with Phosphate Complex Formation

Type of Metal-Phosphate Complex[7]	Metal	Recovery (%)
Cationic and anionic	Fe	75.7
Anionic	Nb	(Loss)
	Mo	7.2
	Cr	14.5
	In	18.2
	Ag	22
Soluble and/or insoluble	Ta	7.5
	Sn	7.9
	Zr	18.7
	Be	45.7
	Bi	83.1
No evidence of complex formation	Co	83.0
	Cu	94.3
	Mn	97.5
	Zn	98.0
	Eu	99.0
No information available	Sb	17.6
	Ru	20.8
	Ga	32.0

Table 8.3. Recoveries of Trace Metals from Bone-Ash Solution by Separate Mitchell-Scott Treatment of Calcium Oxalate Precipitate and Phosphate-Rich Supernatant Liquid (H_3PO_4 not removed)

Metal	Recovery (%)		
	From Calcium Oxalate Fraction	From PO_4^{3-}-Rich Supernatant Liquid	From Combined Mitchell-Scott Precipitates
Be	90.8	0.8	7.2
Cd	95.3	80.9	86.3
Co	>98	89.9	92.3
Cr	81.7	2.3	32.4
Fe	>98	94.7	93.7
Mn	80.0	28.3	64.8

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

[6] C. Feldman and F. S. Jones, "Chemical Preconcentration of Trace Elements in Bone Ash," *Anal. Chem. Div. Ann. Progr. Rept. Sept. 30, 1968*, ORNL-4343, p. 54.

[7] L. G. Sillén, *Stability Constants of Metal-Ion Complexes; Section I: Inorganic Ligands*, Special Publication No. 17, The Chemical Society, London, 1964.

If trace elements are recovered from the phosphate-rich fraction without first eliminating the phosphoric acid from the solution, recovery of some trace elements is hindered (Table 8.3). If elimination of the phosphoric acid from this fraction can be accomplished without loss of trace elements, as seems likely from the literature,[8] their recovery from both phases should be satisfactory.

8.5 DETERMINATION OF MOISTURE AND FAT CONTENTS OF BONE

F. S. Jones

The moisture content of bone samples was determined by weighing the samples before and after freeze-drying. Portions were then removed from different parts of the dry samples with the aid of a specially designed burr grinding tool. The fat was extracted from each portion by the Folch procedure,[9] dried at 50°C under nitrogen and then over P_2O_5, and weighed. The interior of a beef rib bone was found to contain 4.7% fat and the exterior 0.67% fat. Measurements will be made on human-autopsy bone samples.

8.6 DETERMINATION OF TRACES OF BISMUTH AND RARE-EARTH ELEMENTS IN MSRE FUEL

Cyrus Feldman Anna M. Yoakum

To spectrographically determine Bi in MSRE fuel (a mixture of LiF, BeF_2, ThF_4, and UF_4) the sample is fused with NH_4HSO_4 to eliminate fluoride, and the excess NH_4HSO_4 is fumed off. The residue is dissolved in dilute acid, the pH of the solution is adjusted to 1.5, and the Bi is extracted with dithizone in CCl_4. The organic phase is evaporated onto graphite powder and is analyzed spectrographically. In a 10-g fuel sample, as little Bi as 5 ppb can be determined.

To determine rare-earth elements, the residue from the evaporation of NH_4HSO_4 is dissolved in 1 *M* HNO_3. The solution is made 8 *M* in HNO_3 and is passed through an anion-exchange column to remove Th. The eluate is treated with HF, NH_4F, and La carrier. The resulting precipitate, which carries traces of rare-earth elements, is analyzed spectrographically. In a 10-g sample of fuel salt, as little Gd as 2 ppb can be determined.

8.7 DISSOLUTION OF HASTELLOY N AND GeO_2

Cyrus Feldman

A solution of Hastelloy N in 1% H_2SO_4 was desired for spectrographic analysis. Ordinarily, this alloy must be dissolved with aqua regia, in which case the precipitation of molybdic acid sometimes results. Trials showed, however, that if a 2-mm-diam rod of the alloy serves as one electrode and a graphite rod as the other, this alloy can be dissolved in 1% H_2SO_4 by ac electrolysis at the rate of 16 mg/min.

Ordinarily, it is difficult to dissolve some preparations of GeO_2 because of the presence of a relatively insoluble glassy phase. It was found that if the GeO_2 is first boiled vigorously for 5 min in a 1:1 glycerol-water solution, the resulting sol can be diluted with water and then the diluted sol can be boiled to give a clear solution.

8.8 PRELIMINARY STUDIES OF CARRIER-DISTILLATION SPECTROGRAPHIC PROCEDURE FOR USE IN UO_2-PuO_2 ANALYSIS FACILITY

Anna M. Yoakum

With UO_2-base standards that contained 5% of an In_2O_3-SrF_2 (2:1 wt) mixture as carrier, a sensitivity of 0.1 ppm for the spectrographic determination of B and Cd was achieved in the first order. Maximum emission intensity was obtained in the 0- to 30-sec period after ignition for Be, As, Al, B, Si, Hg, Sb, Ge, Tl, Pb, Sn, Ga, Bi, Ti, V, Zr, Ti, and Cr and in the 30- to 70-sec period for Cd, Fe, Mn, Mo, Cu, Ag, Co, Ni, Th, Ba, and Ca.

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[8] F. H. Cripps, *A Solvent Extraction Procedure for the Quantitative Separation of Phosphoric Acid from Solutions Containing Metal Salts*, CRL/AE-49, Mar. 1950.

[9] J. Folch, M. Lees, and G. H. Sloane-Stanley, "A Simple Method for the Isolation and Purification of Total Lipides from Animal Tissues," *J. Biol. Chem.* 226, 497 (1957).

9. Optical and Electron Microscopy

T. E. Willmarth
L. D. Hulett[1] T. G. Harmon

9.1 RESEARCH ASSISTANCE IN ELECTRON MICROSCOPY AND ELECTRON DIFFRACTION

The use of electron microscopy to characterize graphically a wide variety of materials associated with nuclear technology and other interests at ORNL has continued. Where necessary for identification or structure studies, either the highly sensitive technique of selected-area-transmission electron diffraction or reflection electron diffraction was used. Light optics was used principally in the preparation of materials for electron optical investigations.

9.1.a Lanthanide Hydroxide Sols

The hydroxide sols of certain lanthanides (Ce, Yb, and Tm) are being characterized according to their microsphere-forming properties. A program is in progress to study systematically lanthanide- and actinide-element chemistry by electron microscopy and by electron and x-ray diffraction. This work includes studies of solid compounds produced in solution under a great variety of conditions. Complex formation will be studied in particular.

9.1.b Crystalline Urania Sols Program (CUSP)

A new process has been developed[2] to prepare concentrated crystalline urania (UO_2) sols by solvent extraction. In this development, electron microscopy and electron diffraction were used to determine what changes occur in the physical properties of the product with such process variables as off-gas temperature, crystallizing temperature, duration of initial nitrate extraction, uranium-feed concentration, methanol addition, and duration of aging. Figure 9.1 is an electron micrograph of a representative product sol; it shows the typical clusters (micells) of UO_2 crystals. These clusters are of very uniform diameter (250 to 300 Å) and are composed of crystals whose average diameter is 40 Å. There is also evidence that a binder – possibly organic – holds the crystals in place. Further study will be required to determine the exact mechanism of agglomerate formation.

9.1.c Other Research Assistance

Other materials examined by electron microscopy and electron diffraction included gold colloids, high-nitrate thoria-urania sols, carbonized rayon, ion-exchange resins, tobacco-mosaic virus, stainless-steel aerosols, carbon fibers, graphite, magnesium oxide, thorium oxide, boron, a silver iodide deposit on silver plate, vaporized thulium, boron nitride, beryllium oxide, titanium hydroxide and hafnium hydroxide sols, water-scale deposits, aluminum metal films, diamond cut-off wheels, carbide cutting tools, boron carbide, and simulated Molten Salt Reactor products.

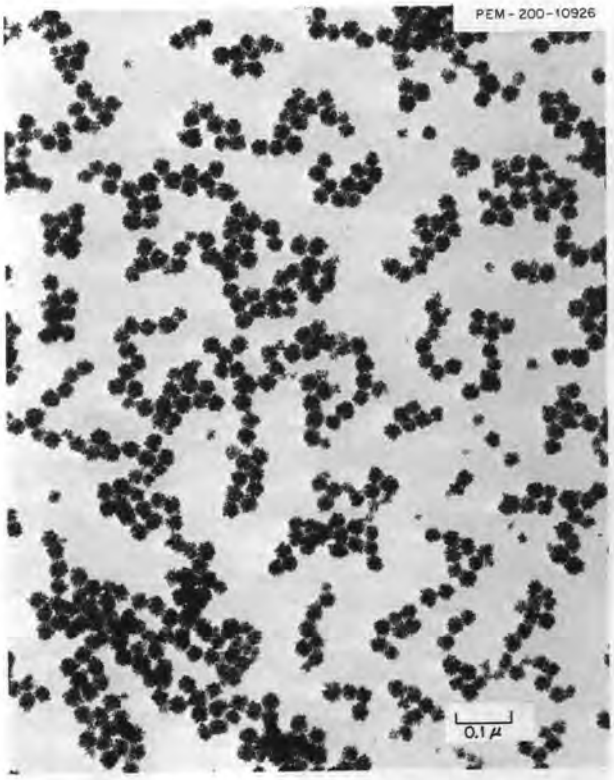

Fig. 9.1. Typical Crystalline Clusters of UO_2 Seen in CUSP Preparations. (Magnification: 183,000X.)

[1] On loan from the Solid State Division.

[2] J. P. McBride, K. H. McCorkle, and W. L. Pattison, "The Preparation of Concentrated Urania Sols by Solvent Extraction," presented at the 15th Annual Meeting, American Nuclear Society, Seattle, Wash., June 15–19, 1969; abstract in *Trans. Am. Nucl. Soc.* **12**, 206 (1969).

9.2 ELECTRON MICROSCOPY OF RADIOACTIVE MATERIALS

Compounds of californium, berkelium, and einsteinium were studied to learn more about their separation chemistry and how their physical appearances and crystallinities are related to the separation method. For the samples observed thus far by electron microscopy, it has not been possible to identify individual components or even to distinguish an actinide-related material from an artifact. With no ASTM x-ray data available on the compounds of these elements, electron diffraction of material in such samples, although possible, is rather meaningless. Improved methods of separation, aided in part by the present studies, will result in faster specimen preparation, more rapid observations, and – it is hoped – in real basic information.

Plutonia (PuO_2), UO_2, and PuO_2-UO_2 sols were studied by electron optical methods to relate process variables with such changes in the final product as crystallite size, degree of crystallinity, presence of amorphous material, and degree and nature of agglomeration. Figure 9.2 is a photomicrograph of a typical PuO_2-UO_2 sol; the size of the PuO_2 crystal is ~20 Å, and that of UO_2 crystals ~80 Å.

Fig. 9.2. Electron Micrograph of a PuO_2-UO_2 Sol. (Magnification: 181,200X.)

The Mound Laboratory was assisted in the electron microscopy of $^{238}PuO_2$ sols prepared at their site. Similar work, including particle-size-distribution measurement, was done on UO_2 prepared at the Nuclear Fuel Services Plant, Erwin, Tennessee.

9.3 INSTRUMENTATION

9.3.a Evaluation of Electron Spectroscopy for Chemical Analysis (ESCA)

New or improved electron optical methods that have a definite use in analytical chemistry are always of interest. One such method is so-called electron spectroscopy for chemical analysis (ESCA); it is described below and evaluated in terms of certain problems of interest at ORNL.

Photoelectron spectroscopy allows very accurate measurement of the binding energies of electrons in atoms. This subject was reviewed recently.[3] Fundamental research in this field is being conducted conjointly with the Physics Division. A spectrometer capable of resolving electron energies to one part in 10^3 has been constructed and is being used to evaluate ESCA for analytical chemistry purposes.

The ESCA principle is very simple. The specimen is bombarded with photons of known energy. From a measurement of the kinetic energy of the electrons ejected from the surface of the sample, the binding energies are deduced. Valence-electron binding energies can be measured by irradiating with ultraviolet photons; if x rays are used, electrons are ejected from K and L shells. Siegbahn[3] has shown that the ESCA technique permits much higher resolution in measuring binding energies than do x-ray methods. Qualitative elemental analysis can be done by measuring inner-shell binding energies; ESCA can be applied to all elements except hydrogen. There are slight shifts (<10 eV) in binding energies, the magnitude of the shift depending on the valence of the element. The high-resolution capability of ESCA thus allows estimates of valence – probably the most outstanding feature of the technique. The method is nondestructive if the compound does not decompose under irradiation. The required specimen quantities are <1 μg.

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[3] K. Siegbahn, C. Nordling, A. Fahlman *et al.*, *Electron Spectroscopy for Chemical Analysis*, Technical Report AFML-TR-68-189, October 1968, Air Force Materials Laboratory, Air Force Systems Command, Wright-Patterson Air Force Base, Dayton, Ohio.

Three nucleotides from tRNA have been studied; they are 4-thiouridine (dimerized form), thioguanosine, and 7-methylguanosine. The objectives of the study were to distinguish 4-thiouridine from the thioguanosine and to test the capability of ESCA for revealing different valence states of elements. The specimen chosen was 7-methylguanosine, which exists in a zwitter ion form. It has a positively charged nitrogen atom and a negatively charged oxygen atom. The effect of these charges on electron binding energies was studied. The results showed that 4-thiouridine can easily be distinguished from thioguanosine by comparing the ratio of the intensity of electrons emitted from nitrogen to that from sulfur atoms for one of the two compounds with the ratio for the other compound. In the spectrum of 7-methylguanosine there is a distinct broadening of the energy distribution of electrons emitted from nitrogen atoms; probably, the broadening is caused by the charges on the nitrogen atoms. The effect of charge on the energy distribution of electrons from oxygen is not pronounced.

Other compounds of interest in analytical problems at ORNL will also be studied.

9.3.b Scanning Electron Microscopy

Although the demand for the service of scanning electron microscopy is greater now than it was a year ago when we recommended the purchase of a scanning electron microscopy, budget limitations still prevent its purchase. In connection with our request, available commercial instruments have been studied extensively in relation to the type of problems encountered at ORNL. One microscope, more flexible than the rest, offers vacuum conditions of an order that also allow the use of the instrument for microanalytical studies of surface chemical composition by the recently developed Auger-electron-analysis technique.[4] This method is especially useful for the low-atomic-number elements from beryllium to calcium, which are not detected readily with the x-ray microprobe.

9.3.c Transmission Electron Microscopy

The Philips EM 300 electron microscope has been installed and is being used to study the actinides and related materials. The beam-neutralizer device for the electron-diffraction instrument has been modified to accept standard Philips EM 100 filaments.

9.4 PREPARATION OF METALLOGRAPHIC SPECIMENS FOR TRANSMISSION ELECTRON MICROSCOPY

To obtain information about the internal structure of solids by conventional electron microscopy, the solid must be sufficiently thin ($\leqslant$1000 Å) to allow transmission of the electron beam without undue scatter. Biological specimens are often thinned by sectioning with a microtome. However, sectioning is not satisfactory for most types of metallographic samples, because mechanical deformation may introduce artifacts that obscure the information desired. For example, in studying the effect of irradiation on solids, one looks for point-defect clusters and other imperfections produced by the radiation. If specimens for such studies are thinned by microtoming or grinding, the dislocations introduced by the thinning technique might be mistaken for effects due to irradiation.

The most effective way to thin specimens without deforming them mechanically is electropolishing or chemical polishing. These techniques require special photoelectric apparatus for controlling the degree of polishing so that the desired thinness is obtained before the specimen is consumed. Such apparatus has been acquired and used to thin copper, zirconium-titanium alloys, and graphite. We have developed a technique for thinning graphite by electrolysis at high current density in a concentrated aqueous solution of KOH. The method shows promise of being quicker and easier than others. Since graphite is of great importance in reactor development, considerable effort is being expended to perfect this technique.

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[4] L. A. Harris, "Auger Electron Emission Analysis," *Anal. Chem.* 40(14), 24A, 1968.

10. Inorganic Preparations

D. E. LaValle
R. B. Quincy

The Solid State Division continued to provide the major demand for inorganic preparative work with the high-purity materials program and the requests for ferrite materials for single-crystal growth. The former program, for the Research and Development of Pure Materials Group, has now been completed with the accumulation of 15 lb of high-purity MgO. In the continuing ferrite program for the Neutron Spectrometry Group, the following substituted magnetites were prepared:

$^{7}Li_{0.5}Mn_{0.1}Fe_{2.4}O_4$	$Mn_{1.0}Ga_{0.2}Fe_{1.8}O_4$
$^{7}Li_{0.5}Mn_{0.2}Fe_{2.3}O_4$	$Mn_{1.0}Ga_{0.3}Fe_{1.7}O_4$
$^{7}Li_{0.5}Mn_{0.3}Fe_{2.2}O_4$	$Mn_{1.0}Ga_{0.4}Fe_{1.6}O_4$
$^{7}Li_{0.5}Mn_{0.4}Fe_{2.1}O_4$	$Mn_{0.65}Zn_{0.35}Fe_{2.0}O_4$
$^{7}Li_{0.5}Mn_{0.5}Fe_{2.0}O_4$	$^{7}Li_{0.4}Fe_{2.6}O_4$
$^{7}Li_{0.5}Ti_{0.2}Fe_{2.3}O_4$	$^{7}Li_{0.5}Fe_{2.5}O_4$
$Ni_{0.5}Fe_{2.5}O_4$	$^{7}Li_{0.6}Fe_{2.4}O_4$
	$^{7}Li_{0.8}Fe_{2.2}O_4$

For another section of the same group, some large crystals of NH_4Cl and ND_4Cl were grown. In addition, attempts were made to incorporate 10 mole % of KCl as a solid solution in crystals of NH_4Cl. All attempts resulted in crystals that contained 90 mole % KCl. For still another phase of the Group's research, the reduction of 60 g of $^{163}Dy_2O_3$ and 70 g of $^{170}Er_2O_3$, both of ~96% enrichment, to the metals was undertaken by a procedure similar to that used last year to prepare ^{160}Gd.

Other work for the Solid State Division included the preparation of several 100-g batches each of high-purity MnF_2, $KMnF_3$, and MgF_2 for the Lattice Defects in Halides Group. For the Neutron Diffraction Group, the disulfides of Ni, Co, and Fe were prepared in 50-g batches and the solid solution $Fe_{0.5}Co_{0.5}S_2$. Attempts to make the corresponding iron-nickel compound resulted in only partial solution and mixed phases. Also prepared were the intermetallic compound MnSb (100 g), anhydrous NiF_2 (35 g), and V_2O_3 (50 g) by reduction of mixed higher oxides with H_2 at 1200°C. The usual service of annealing or otherwise heat-treating numerous alloys was also continued.

For the Superconductivity Group, several preparations of rhenium nitride and technetium nitride were made by heating the respective ammonium perrhenates and pertechnetates in ammonia gas at several different temperatures between 300° and 700°C. Also, pure CuS was made by the reaction of fine Cu powder and S in CS_2 as a solvent in a sealed tube at 105°C.

The other significant amount of work was done for the Low Temperature, Nuclear, and Solid State Physics Group of the Physics Division. One phase consisted in the preparation of small quantities (500 mg) of alloys of enriched materials such as ^{64}NiV (86 at. % ^{64}Ni), $^{61}NiAu$ (1 at. % ^{61}Ni), and $^{73}GeFe$ (0.75 at. % ^{73}Ge). Other alloys prepared for the group were $GdAl_2$, $GdFe_2$, $Gd_{0.05}Y_{0.95}Al_2$, CaAu (1 at. % Au), InAu (4 at. % In). The compound $NaNiO_2$ was also prepared.[1] In another phase of work, the solid solutions of the mixed disulfides $Fe_{0.7}Co_{0.3}S_2$, $Fe_{0.5}Co_{0.5}S_2$, and $Fe_{0.25}Co_{0.75}S_2$ were prepared.[2]

The preparation of fused salts was at a minimum. For the Chemistry of Nonaqueous Inorganic Systems at High Temperatures Group of the Chemistry Division, YCl_3, $GdCl_3$, and $ScCl_3$ were prepared and various mixtures of ThF_4 with LiF and with Li_2BeF_4 were dehydrated. For the Ion Spectroscopy Group of the Metals and Ceramics Division, a quantity of anhydrous fused LiI was prepared, but many attempts to prepare pure fused CsCl failed because the original CsCl was contaminated with organic material that could not be removed. The problem was finally solved by finding a new source of supply of CsCl that contained no organic matter.

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[1] L. D. Dyer, B. S. Borie, Jr., and G. P. Smith, "Alkali Metal–Nickel Oxides of the Type $MNiO_2$," *J. Am. Chem. Soc.* **75**, 1499 (1954).

[2] R. J. Bouchard, "The Preparation of Pyrite Solid Solutions of the Type $Fe_xCo_{1-x}S_2$, $Co_xNi_{1-x}S_2$, and $Cu_xNi_{1-x}S_2$," *Mat. Res. Bull.* **3**, 563 (1968).

Part B. Service Analyses

The table below gives a summary of the service analyses made by the laboratories of the Analytical Chemistry Division.

Summary of Analytical Service Work

Group Making Analyses	Average Number in Group		Number of Results Reported																							
			For ORNL Divisions															For Others								
	Scientists	Technicians	Analytical Chemistry	Biology	Chemical Technology	Chemistry	Electronuclear	Health	Health Physics	Isotopes	Metals and Ceramics	Neutron Physics	Operations	Physics	Reactor	Reactor Chemistry	Solid State	Argonne National Laboratory	Brookhaven National Laboratory	General Electric Co.	Los Alamos Scientific Laboratory	Miscellaneous	Nuclear Fuel Services, Inc.	Westinghouse Atomic Power Development Division	Y-12	Total
Mass Spectrometry Service Laboratory	2	3	60		415	110			98	8,917	934			22	535	650	30					6504			478	18,753
Methods Development and Evaluation Laboratory	5	1																	6							6
Process Analyses																										
General Analyses Laboratory	11	19	307	2243	94,989	443			453	308	7,290	109	720	17	2,802	4,967	816					761			772	116,997
General Hot-Analyses Laboratory	11	8			6,561					664	1,283		7,340		2,494	2,607				8				15		20,972
High-Level Alpha Radiation Laboratory	8	10			20,555		1		4	587	471	4	10		43	717			3		98				2	22,495
Radioisotopes-Radiochemistry Laboratory	7	12	12		6,604	21			1884	6,107	576	39	9,914	617	2,274	13,710	421	174		176		62				42,591
Spark-Source Mass Spectrometry Laboratory	1	1			265	204			20	684	2,671	65		40	594	299	852					650			882	7,226
Spectrochemistry (Y-12)	2	1	493		964	110		18,162	140	15,285	4,249	445	20	30	1,669	3,130	2661					336			969	48,663
Transuranium Mass Spectrometry	2	1	908		1,250	333				871	283	12	75		342	246					455	28				4,803
X-Ray and Spectrochemistry	8	4	159	1271	7,235	918			1823	2,197	14,343	524	510	1108	1,055	4,152	679					613	71			36,658
Total	57	60	1939	3514	138,838	2139	1	18,162	4422	35,620	32,100	1198	18,589	1834	11,808	30,478	5459	174	9	184	553	8954	71	15	3103	319,264

11. Spectrometry

A. E. Cameron

11.1 MASS SPECTROMETRY

11.1.a Transuranium Mass Spectrometry

R. E. Eby

The Transuranium Mass Spectrometry Laboratory reported over 4800 results, a 12% increase over last year. The Chemical Technology Division requested slightly over a quarter of the work, and the Isotopes Division was the next largest customer, requesting ~20% of the work. About 10% was done for other plants, primarily for C. F. Metz of the Los Alamos Laboratory.

Isotopic determinations were made on samples of the following elements: Ca, Sr, Zr, Mo, Cs, La, Ce, Sm, Eu, Gd, Pb, Po, U, Np, Pu, Am, Cm, Bk, Cf, and Es. Samples of the following isotopes, whose isotopic purity was >99.99%, were analyzed: ^{233}U, ^{236}U, ^{238}U, ^{237}Np, ^{239}Pu, ^{240}Pu, ^{241}Am, ^{249}Cf, and ^{253}Es. On other samples, quantitative determinations of uranium, plutonium, americium, and samarium were made by isotope-dilution techniques.

In collaboration with J. Halperin[1] and R. E. Druschel,[1] the thermal-neutron-capture cross sections and resonance integrals of ^{245}Cm and ^{246}Cm were determined.[2] An unusual series of samples from the Isotopes Division consisted of calutron-separated curium isotopes. Samples of Es and Cf from irradiated Cf rabbits were analyzed for R. D. Baybarz.[3] A number of uranium and plutonium samples from the MSRE were analyzed. The quantitative uranium value was obtained on some of these by use of a ^{236}U-spike method.

The Transuranium Mass Spectrometry Laboratory is also responsible for the preparation of sample filaments for the Referee Analysis Laboratory and the Mass Spectrometry Service Laboratory. More than 6000 filaments were prepared, an increase of 20% over the previous year. The filament bake-out rack was changed to double the number of filaments that can be degassed at one time.

[1]Chemistry Division.

[2]J. Halperin, R. E. Druschel, and R. E. Eby, "Thermal Neutron Cross Sections and Resonance Integrals of ^{245}Cm and ^{246}Cm," *Chem. Div. Ann. Progr. Rept. May 20, 1969,* ORNL-4437, p. 20.

[3]Chemical Technology Division.

11.1.b Mass Spectrometry Services

J. R. Sites E. J. Spitzer

The number of analyses of separated stable isotopes reported to the Isotopes Division increased 10%. The certification of cylinders and trailers of argon and helium and the analysis of fission-product xenon and krypton continued as in previous years. A set of nitrogen samples from an ecology experiment was analyzed for ^{15}N content after hypobromite oxidation of ammonium to N_2 in Rittenberg tubes directly on the inlet system of the mass spectrometer.

11.2 EMISSION SPECTROMETRY (Y-12)

J. A. Carter S. A. MacIntyre

The Spectrochemistry Laboratory analyzed 2700 samples, an increase of 8%, and reported ~49,000 results. The Reactor Chemistry Division was the largest supplier of samples (29%, a twofold increase). Most of the samples were Bi, Pb, or Sn metal from the experimental MSRE salt clean-up program. The Paschen direct-reading spectrometer was used to analyze them for Th, Li, Be, and other elements of interest.

The Isotopes Division submitted 16% more samples than last year. Trace impurities were determined in samples of the following 47 stable isotopes: Ag, B, Ba, Br, Ca, Cd, Ce, Cl, 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.

The Y-12 Plant submitted three times as many samples as last year; many were alloys for composition as well as specification analyses.

11.3 INFRARED SPECTROMETRY

M. M. Murray

Constant involvement of the Infrared Laboratory with biological compounds has instigated further study of sample-preparation techniques. Last year's report described a small lyophilization apparatus for preparing KBr-sample mixtures to be pressed into disks for infrared spectrometry. This technique has shortcomings, among which are water absorption and KBr-sample interaction. Many biological compounds pressed into KBr pellets decompose when dried by heat-vacuum

procedures. Recently, a mulling technique has been used almost exclusively for solid biological compounds. Samples are mulled with mineral oil between KBr plates for the low-frequency range of the spectrum (300 to 1350 cm^{-1}) and with a perfluorinated hydrocarbon for the high-frequency range (1350 to 4000 cm^{-1}). In general, the resulting spectra are better defined and cleaner than those obtained by the "alkali-halide-disk" method. Further, if the samples themselves are dry, there is no problem of absorbed water. Occasionally, the Christiansen effect or excessive scatter in the high-frequency region occurs. The Christiansen effect is inherent in the relationship between the indexes of refraction of sample and mulling agent. The scatter might be eliminated by grinding the sample extensively before mulling it. In a program supported jointly by the National Academy of Sciences, National Research Council, and ORNL (Sect. 4.3.c), a number of purine and pyrimidine derivatives (Table 11.1) were examined, primarily by the mulling technique.

Other materials studied have included the biologically related substances derived from *Artemia* (brine shrimp) and *Artemia* cysts, carcinogens, a defoliant, and unidentified components of urine; the commercial products brazing flux, gasket materials, paper, surfactants, particulate Teflon for TLC, and lubricating oils; such inorganic samples as lithium ferrite ($Li_2Fe_{2.2}O_4$), Si^0, Ge^0, $Mg(OH)_2$, $Mg(OH)HOCO_2$, a basic magnesium nitrate, and $NaBrO_4$; and also control solutions from experimental processes and fractions from the synthesis and oxidation of some organic compounds.

For reference, the spectra of the purine and pyrimidine derivatives and of the other known compounds were recorded and listed in a card file.

Table 11.1. Compounds Examined by Infrared Spectrometry

Adenine	Guanosine
Adenosine	Hypoxanthine
Adenosine-5′-diphosphate	Inosine
Adenosine-2′-monophosphate	5-Iodouridine
Adenosine-2′,3′-monophosphate	Kinetin
5-Bromo-2′-deoxycytidine	6-Methylaminopurine
Cytosine	5-Methylcytosine
2′-Deoxyadenosine	5-Methyl-2′-deoxycytidine
2′-Deoxycytidine	Orotic acid
2′-Deoxyguanosine	9-β-D-Ribosylkinetin
2′-Deoxyinosine	Thymidine
2-Dimethylamino-6-hydroxypurine	Uridine
6-Dimethylaminopurine	Xanthine
Guanine	Xanthosine

11.4 NUCLEAR MAGNETIC RESONANCE SPECTROMETRY

J. R. Lund

The demand for the services of the N.M.R. Laboratory has continued to increase. A wide variety of organic samples were leached with CCl_4 from the charcoal filters of the U.S.S. Savannah. The spectra of fractions of these leachings were recorded and were compared with the spectra of the original oils and hydraulic fluids from which they were filtered.

The Y-12 Organic Preparations Group has studied the effects of various curing agents on resins and the effectiveness of separating the monomers, dimers, and polymers of resins by molecular distillation. Although the NMR spectra of many polymers are not well resolved, the NMR spectra of many para-disubstituted benzenes fall into a special category in which the lines form a distinctive pattern and can be assigned in a predictable manner. Since many of the resins examined by NMR spectrometry contained units of the type "*p*-...C–Ø–O...", which fall into this special category, the NMR spectra of these samples provided significant information about them. The NMR spectra of resins that contained such terminal groups as $-SiCH{=}CH_2$, $-SiH$, or the various amide groups were also very useful.

The National Academy of Sciences' Subcommittee on Nucleotides and Related Compounds has set tentative specifications for compounds to be designated as NRC grades (Sect. 4.3.c). Of the 23 pyrimidine and purine bases, nucleosides, and nucleotides received for spectral analysis, NMR spectra were recorded of adenine, adenosine, 2′-deoxyadenosine, 6-dimethylaminopurine, 6-methylaminopurine, 5-methylcytosine, 2′-deoxycytidine, 5-bromo-2′-deoxycytidine, 2-dimethylamino-6-hydroxypurine, guanosine, 2′-deoxyguanosine, hypoxanthine, inosine, 2′-deoxyinosine, xanthosine, orotic acid, kinetin, and 9-β-D-ribosylkinetin. Attempts to use a common solvent for these compounds have been unsuccessful. Polysol, a secret formulation sold by the Stohler Isotope Company, Rutherford, N.J., and touted as a better solvent than deuterodimethyl sulfoxide, $(CD_3)_2SO$, was no better than $(CD_3)_2SO$ as a solvent for the bases and not as good as deuterotrifluoroacetic acid, CF_3COOD, or deuteropyridine, C_5D_5N. For the nucleosides and nucleotides, $(CD_3)_2SO$ was found to be the best solvent of those tried if adequate precautions are taken to keep this very hygroscopic solvent from absorbing water during its use.

The NMR spectra, along with the mass and infrared spectra, were used to determine the structure of the mutagens 2-aminofluorene, 2-acetamidofluorene, 2-diacetylaminofluorene, and *N*-acetoxy-2-acetylaminofluorene.

The NMR spectra of numerous materials were recorded for the purpose of identification and/or comparison; they included tris(2,2,3,3,4,4,4-heptafluorobutyl) phosphate, tris(1,1,5-trihydrooctafluoropentyl) phosphate, silver 1-methylpyrimidine-4-sulfenate, thymine monomer and dimer, homovanillic acid, dioctylphthalate, *N,N'*-dimethylbiacridan, cacodylic acid, and the *exo* and *endo* isomers of 5,6-diaminonorbornene.

12. Process Analyses

L. T. Corbin

12.1 HIGH-LEVEL ALPHA RADIATION LABORATORY

J. H. Cooper

The High-Level Alpha Radiation Laboratory reported >22,000 results, a slight decrease from last year; more than 90% of these were for the Chemical Technology Division.

In the determination of plutonium in samples that contain curium and radiozirconium in high levels, good decontamination of plutonium from both these elements is desirable. The method that has been used routinely consists in extracting the plutonium into thenoyltrifluoroacetone (TTA) and counting an aliquot of the organic phase to measure the plutonium radioactivity. This method does not adequately separate plutonium from radiozirconium, which interferes in the alpha counting and is a health hazard because of its intense gamma radioactivity. This interference can be eliminated by backextracting the plutonium into 8 to 10 *M* HNO_3. An alternate method to determine plutonium has been investigated. Plutonium is extracted from a solution of aluminum nitrate into hexone, and an aliquot of the hexone solution is counted for plutonium radioactivity. However, this method does not give the desired decontamination from curium. We are now using the TTA extraction method with backextraction into nitric acid.

A sealed-bomb dissolution of refractory plutonia-zirconia (PuO_2-ZrO_2) was made with a Teflon-lined bomb. A Teflon liner in a steel case was used to contain PuO_2-ZrO_2 in an HCl-HF mixture. The oxide was dissolved overnight at 135°C with 0.5 *M* HF in concentrated HCl.

Laboratory 4 in Building 3508 is being remodeled extensively. All the glove boxes were disposed of, and new individual boxes are being installed.

12.2 GENERAL ANALYSES LABORATORY

W. R. Laing

The total number of results reported by the General Analyses Laboratory increased from ~111,600 last year to 117,000. The work load in analytical biochemistry increased 14% over last year. Some 80,000 determinations of tRNA were made, compared with 70,000 last year; the average cost for this analysis was reduced from $1.62 to $1.33.

Studies of aminoacyl synthetase stability were completed. It was found that enzyme stored frozen in liquid nitrogen remains fully active for at least 6 mo. Enzyme stored in a commercial freezer begins to lose activity in 4 to 6 weeks.

An improved method to determine RNA and DNA[1] was used to analyze processed *E. coli* cells (Sect. 4.1.h).

A Beckman Amino Acid Analyzer was used to determine amino acids in a variety of biological materials. This analysis is a new addition to the available service analyses.

Two methods to determine bismuth in fluoride salts were developed – an iodide spectrophotometric method[2] for concentrations >10 ppm, and a dithizone

[1] D. W. Hatcher and G. Goldstein, "Improved Methods for the Determination of RNA and DNA in Bacterial Extracts," *Anal. Biochem.* in press (1969).

[2] E. B. Sandell, *Chemical Analysis. Vol. III. Colorimetric Determination of Traces of Metals,* pp. 332–37, 3d ed., Interscience, New York, 1959.

5

extraction–spectrographic method for concentrations as low as 0.1 ppm. The latter was developed in cooperation with J. A. Carter[3] and was used to separate bismuth from thorium. A dithizone extraction–spectrophotometric method[4] was evaluated; it is too slow.

An apparatus was constructed to release ^{4}He formed in pure metals and alloys by the (n,α) reaction of fast neutrons. The volume of the system is made small, because the largest amount of gas calculated to be released from a 10-mg sample is 1 μl. During the melting of the sample, a known amount of a ^{3}He-Ne gas mixture is added to the furnace section. The gas is then transferred to a breakseal holder for determination of the ^{4}He/^{3}He ratio by mass spectrometry.

Zirconium was separated from large amounts of thorium by TTA extraction. After a wet-ashing, the zirconium was determined by the arsenazo III method.[5]

Sample holders described by Smith, Hansel, and Waterbury[6] were used to hold aliquots of uranium mononitride (UN) powder for oxygen determination. For this extremely reactive powder, any exposure to air – or even exposure of the closed holder that contains the sample – increases the oxygen content. In the final technique, closed holders that contained the sample aliquots were sealed in individual Mason jars under argon. Analyses were completed the same day the samples were taken. Consistent results of 90 to 100 ppm oxygen were obtained on the better UN preparations.

The iodine titration method of Fritz and Schenk[7] for the determination of As(V) was adapted to micro levels. All the reagents were added and titrated to the end point before the test aliquot was added. The amounts of As(V) titrated ranged from 0.05 to 3.0 mg.

The Aminco porosimeter was used to force mercury at increasing pressures into pyrolytic carbon coatings on microspheres. X-ray photographs of the mercury-impregnated specimens gave a picture of the pores within the coatings.

Eight chemists and six technicians completed refresher courses taught by J. A. Dean.[8] The idea of refresher courses was received well, and plans to continue these are being made.

Over 3000 lb of mercury was cleaned for coulometric, polarographic, and General Stores' use.

12.3 General Hot-Analyses Laboratory

C. E. Lamb

Of the some 21,000 determinations made by the General Hot-Analyses Laboratory, >99% were for only six ORNL divisions: Operations, Chemical Technology, Reactor, Reactor Chemistry, Metals and Ceramics, and Isotopes.

Samples from the Chemical Technology Division were submitted by the Pilot Plant Section from their fuel-development studies and by the Hot-Cell Section from their development and testing of proposed head-end operations for HTGR and LMFBR fuels. Metals and Ceramics Division's samples also resulted from HTGR and LMFBR fuel-preparation studies. Samples of the MSRE fuel were received from the Reactor Division to secure operating data. Special test samples originated with the Reactor Chemistry Division's study of fission-product behavior. The Operations Division sampled for the control of discharged radioactive waste solutions. The samples from the Isotopes Division were mostly ^{137}CsCl products and fabricated sources.

Three laboratories were added to the High-Radiation-Level Analytical Laboratory area. The Spectrochemistry Group installed an MS-702 mass spectrograph in one of the laboratories for measuring samples that emit low-intensity alpha and beta-gamma radiation. The alpha glove-box facility previously installed in the cell-access area was relocated to one of the new laboratories.

Interest continued in the determination of gas release, porosity, oxygen, surface area, and carbon on UO_2-PuO_2 samples from the Metals and Ceramics Division. The gas-release system was modified extensively to attain and hold a 1700 to 1800°C temperature with a low blank. Four samples can be loaded into the apparatus, the system evacuated, and the gas released

[3] Y-12 Spectrochemistry Group.

[4] E. B. Sandell, *Chemical Analysis. Vol. III. Colorimetric Determination of Traces of Metals*, pp. 347–49, 3d ed., Interscience, New York, 1959.

[5] S. B. Savvin, "Analytical Use of Arsenazo III, Determination of Thorium, Zirconium, Uranium, and Rare Earths Elements," *Talanta* 8, 673 (1961).

[6] M. E. Smith, J. M. Hansel, and G. R. Waterbury, *An Inert Atmosphere Enclosure for the Preparation of Samples Prior to the Determination of Oxygen*, LA-3344 (Aug. 27, 1965).

[7] J. S. Fritz and G. H. Schenk, Jr., *Quantitative Analytical Chemistry*, pp. 546–48, 2d ed., Allyn and Bacon, Boston, 1969.

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

individually from the samples without need to open the system.

The Leco Nitrox Analyzer was modified slightly to allow the attachment of a special glass sample-holder. A sample can now be admitted into the loading device without opening the device to the atmosphere. The oxygen content of $^{237}NpO_2$ samples, as well as the usual UO_2-PuO_2 samples, was measured.

A digital integrator was incorporated into the surface-area apparatus to reduce the time normally required for calculating the results and to increase the reliability of the measurements. Results were erratic; therefore, further testing is necessary.

12.4 RADIOISOTOPES-RADIOCHEMISTRY LABORATORY

E. I. Wyatt

The various types of analyses performed in the Radioisotopes-Radiochemistry Laboratory have remained about the same for the past three years. The total number of analyses reported this year was only ~85% of that of 1968; personnel were reduced accordingly. This decrease is due primarily to a reduction of funds available to the Reactor Chemistry and Chemical Technology Divisions.

Most of our radiometric analyses were 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 dissolutions and subsequent radiochemical separations on various samples from the core, pump bowl, and off-gas lines of the MSRE.

Most of the work in neutron activation was done to determine trace quantities of rare-earth elements in LiF-BeF_2 salts for the Chemical Technology Division. A large number of samples were activated to measure traces of iodine in gas-liquid systems for the Reactor Chemistry Division. Iodine-129 was determined by neutron activation on a large number of samples released by melting "long-irradiated" and "long-cooled" nuclear fuel. These samples required that they be leached or dissolved in the presence of iodine carrier to correct for chemical yield after subsequent separation from other fission products. Since iodine is evolved during HNO_3 dissolution of the fuel, $HgNO_3$ was also added to hold the iodine. We were able to prove that the interchange of ^{129}I with the ^{127}I carrier in the presence of Hg is complete. Special preparation and handling techniques permitted the determination of <100 ng of ^{129}I per sample.

The presence of ^{125}Sb and the absence of ^{99}Tc in both the MSRE salt and the gas phase were proved. This work was done on samples that had decayed for several months to minimize interference from many of the short-lived fission products. A carrier-free separation of cesium from MSRE salt was done in our Building 3019 hot cells. The purpose was to obtain an isotopic analysis by mass spectrometry. About 1 g of the salt was first leached with water. The cesium was then carried down on a precipitate of $(NH_4)_2SnCl_6$ in HCl-C_2H_5OH. This precipitate proved to be free of other radioactive species, and ~10 ng of it was deposited on a filament for isotopic analysis.

A gamma spectrometric analysis of the NaK coolant from an LMFBR experiment was carried out in the HRLEL facility. This NaK had been exposed recently to the fuel through a rupture in the fuel element. A cursory examination showed that ^{134}Cs, ^{136}Cs, and ^{137}Cs were the only gamma emitters present. It was requested that these isotopes, as well as any other radionuclides detected, be determined quantitatively. These determinations required that a known quantity of NaK be dissolved for further radiochemical separations. G. Goldberg[9] supervised the dissolution at the HRLEL; an ice bath and a mixture of isopropyl alcohol and a few drops of water were used. We then removed the radioactive cesium by adsorption on a column of potassium hexacyanocobalt(II) ferrate(II) (BioRad KCF-1). Iodine-131 was the only fission product found in the effluent; ^{110}Ag was detected as a contaminant, presumably from neutron activation of ^{109}Ag in the system.

Several experiments were made to assist in the solution of TRU processing problems with the evolution and trapping of ^{131}I. It was demonstrated that the iodine cannot be sparged from HCl solutions of the molarities encountered in their process, because H_2O_2 forms under the condition of high levels of radiation. Attempts to extract iodine from 6 *M* HCl were successful with diisobutyl carbinol in heptane, Adogen-364 in diethylbenzene, and iodine in diethylbenzene as extractants.

The feasibility of determining the ^{233}U and ^{235}U contents of the graphite moderator from the MSRE was demonstrated by use of a known amount of ^{236}U in the separation of uranium and subsequent mass spectrometric analyses.

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[9]Methods Development and Evaluation Laboratory.

A paper was published on gamma-ray spectral resolution in which fission-product mixtures receive particular emphasis.[10]

12.5 RADIATION CONTROL AND SAFETY

C. L. Burros[11] U. Koskela

Several contaminated glove boxes were transferred from Building 3019 to Building 2026 for temporary storage with no detectable spread of contamination. To improve visibility through the storage-cell window in HRLAF (Building 3019), the oil in the window was changed. No contamination was detected during the operation.

Permission was granted to several investigators to handle unusual amounts of radioactive samples and/or tracers. Approval was also given to some investigators to handle radioactive material within newly designed facilities.

The annual safety audit was conducted in February by C. L. Burros[11] and D. C. Gary.[12] Improper electrical cords and/or grounding were the main faults. In April, the Division cooperated in a plant-wide "Major Hazards Survey" required by the Union Carbide Corporation's New York Office.

During the year, there were 27 medical-treatment cases; one case was serious. The majority of the cases were injuries from broken glass or burns.

12.6 QUALITY CONTROL

G. R. Wilson

For several of the control programs, the fixed 2*S* values were changed during the year. It was necessary to increase the 2*S* value for the MSRE iron, nickel, and chromium controls from 10 to 15% because of the very low concentrations of these elements in the MSRE samples. Several of the control programs have shown a marked improvement in the found 2*S* values over the last few quarters; if the trend continues, the fixed 2*S* values should be reduced. The found 2*S* value for the fluorometric uranium program in the General Hot-Analyses Laboratory in Building 2026 has decreased considerably, probably because more analyses are made by fewer people. The change in the polarographic cell by the General Analyses Laboratory has caused a reduction in the 2*S* value of the polarographic Uranium Control Program.

The quality level by laboratory is shown in Table 12.1. Table 12.2 gives the number of control results and their specific characteristics.

Table 12.1. Distribution by Laboratories of Control Tests for July 1968–June 1969

Laboratory	Number of Control Results		Quality Level (%)[a]	
	Total	Outside Fixed Limits	1968	1969
General Hot-Analyses	1070	170	90.2	84.1
General Analyses	1666	72	93.52	95.7
High-Level Alpha Radiation	150	26	62.07	82.7
Total	2886	268	92.00	90.7

[a]Control results within prescribed 2*S* limits.

Table 12.2. Distribution by Methods of Control Results for July 1968–June 1969

Type of Method	Constituent	Number of Control Programs		Number of Control Results	
Amperometric	Chromium	1		107	
	Zirconium	1	2	75	182
Colorimetric (Spectrophotometric)	Aluminum	1		22	
	Chromium	1		67	
	Iron	2		208	
	Molybdenum	1		89	
	Nickel	2		160	
	Thorium	4		307	
	Uranium	4	15	116	969
Coulometric titration	Plutonium	2		81	
	Uranium	3	5	987	1068
Flame photometric	Lithium	1	1	11	11
Fluorometric	Uranium	2	2	331	331
Gravimetric	Carbon	2	2	165	165
Photoneutron	Beryllium	1	1	38	38
Polarographic	Uranium	1	1	76	76
Volumetric	Aluminum	1	1	46	46
Total			30		2886

[10]M. E. Pruitt, S. H. Prestwood, and H. A. Parker, "Some Practical Applications of Gamma-Ray Spectra Resolution to Radionuclide Analysis," *Nucl. Appl.* 5, 253 (1968).

[11]Deceased March 18, 1969.

[12]Health Physics Division.

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 1968. However, they will be in 1969 to indicate changes made in the content of the *Manual* during 1967, 1968, and 1969.

13.2 MAINTENANCE OF THE *ORNL MASTER ANALYTICAL MANUAL*

No supplement to the reprinted form of the *ORNL Master Analytical Manual* was published in 1968. The tenth supplement will be published in 1969 and will contain the new methods and revisions issued in 1967, 1968, and 1969.

Two new methods were added to the *Manual* (See "Presentation of Research Results.") Thirty-one methods were discontinued; thirty of these were used in the analysis of radioisotopes that are now no longer produced at ORNL.

Part D. Activities Related to Education

14. Activities Related to Education

Certain activities come into existence when the interest, purposes, and problems relative to education overlap those of the Oak Ridge National Laboratory 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 168 formal presentations of the research of the Division made during the past year, about 12% 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[a]
University of Illinois

Gleb Mamantov
University of Tennessee

G. H. Morrison[a,b]
Cornell University

M. L. Moss[a,b]
Previous address: Institute for Muscle Disease, Inc.
Present address: ORNL Analytical Chemistry Division

A. O. C. Nier
University of Minnesota

L. B. Rogers[a]
Purdue University

[a]Advisory Committee Member
[b]Term expires December 31, 1969

14.2 ORAU RESEARCH PARTICIPANTS

Three ORAU Research Participants were with us in the summer of 1969.

A. Y. Herrel, Acting Head of the Chemistry Department, Knoxville College, Knoxville, Tenn., learned to use the Philips electron microprobe for the qualitative and quantitative analysis of 1-μ^2 areas on various specimens. He worked under the direction of *H. W. Dunn,* X-Ray and Spectrochemical Analysis Group.

L. J. Sacks, Professor of Chemistry, Hampton Institute, Hampton, Va., worked with *H. H. Ross,* Nuclear and Radiochemistry Group, on the structure and reactivity of Cr(III) complexes. This work was begun at Hampton Institute and was extended at ORNL through the use of radioactive tracers.

C. R. Spell, Chairman, Department of Chemistry, Emory and Henry College, Emory, Va., worked in the Analytical Instrumentation Group with *M. T. Kelley,* Director of the Division, to explore instrumentation and methodology for digital flame photometry. With assistance from *T. R. Mueller,* he constructed a flame photometer and used with it a Digital Equipment Corporation PDP-8/I computer. *R. W. Stelzner* assisted with the computer interfacing. Sufficient knowledge was gained to constitute a foundation for the further development of digital flame photometry (Sect. 1.21).

14.3 GRADUATE THESIS RESEARCH PROGRAMS

14.3.a ORAU Graduate Fellowship Programs

J. E. McCracken, an ORAU predoctoral fellow from the Department of Chemistry, University of Missouri, Columbia, is working in the Analytical Methodology Group with *W. D. Shults.* He has developed a programmed-current coulometric titrator and is studying its application (Sect. 2.4.d). His major professor is *J. C. Guyon.*

F. L. Whiting, an ORAU predoctoral fellow from the Department of Chemistry with *Professor Gleb Mamantov* at the University of Tennessee, Knoxville, is continuing his 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).

14.3.b ORNL Loanees

F. R. Clayton, Jr., a graduate student in the Department of Chemistry with *Professor Gleb Mamantov* at the University of Tennessee, Knoxville, is conducting research toward the Ph.D. degree. In the Reactor Projects Group, with *J. C. White* and *A. S. Meyer,* 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 extending the electromotive series in fluoride melts and establishing a series in $NaBF_4$ melts.

R. L. Coleman, a graduate student in chemistry at the University of Tennessee, is conducting predoctoral research in the Analytical Methodology Group. He is working with *W. D. Shults* and *Helen P. Raaen* in thin-layer chromatography to investigate the effects and usefulness of temperature as a variable (Sect. 2.3.b). His major professor is *J. A. Dean.*

R. W. Morrow, a graduate student in chemistry at the University of Tennessee, is conducting predoctoral research in the Analytical Methodology Group. He is working with *M. R. Guerin* and *W. D. Shults* to study various applications of the silicon-selective detector that he has developed (Sect. 2.2.a). His major professor is *J. A. Dean.*

J. C. Wolford, a graduate student in the Department of Chemistry with *Professor J. A. Dean* at the University of Tennessee, 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 (Sect. 4.1.g).

14.4 SUMMER STUDENT PROGRAMS

In the summer of 1969, five students participated in summer student programs.

14.4.a ORAU Student Trainee Program

Rae Lynn Athanacio, now a senior at the University of Nevada, Reno, worked with *I. B. Rubin,* Analytical Biochemistry Group, on the development of statistically designed experiments to optimize conditions for the assay of transfer ribonucleic acids (Sect. 4.1.a).

G. L. Bangs, now a senior at Arkansas Polytechnic College, Russellville, worked with *W. C. Butts,* Analytical Biochemistry Group, on the gas-chromatographic separation of nucleic acid components and compounds found in human urine (Sect. 4.2.a).

Beverly A. Brown, now a senior at Evangel College, Springfield, Mo., worked in the Analytical Methodology Group with *J. R. Stokely* on electroanalytical problems involving both inorganic and organic materials (Sect. 2.4.b).

14.4.b ORNL Summer Technical Student Program

J. L. Bowling, a graduate of Concord College, Athens, W.Va., now a first-year graduate student at the University of Tennessee, Knoxville, worked in the Analytical Methodology Group. With *W. D. Shults* and *M. R. Guerin*, he studied the technique of hydrogenation–gas chromatography as a means for rapid elemental analysis (Sect. 2.2.b).

J. D. Lodmell, a graduate of Concord College, Athens, W.Va., now a second-year 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. With *A. D. Horton* and *A. S. Meyer* he studied the volatile complexes of rare-earth-element chlorides and lithium chloride.

14.5 PROJECT "AVAILABLE; BETTER CAREERS" (ABC)

J. E. Caton of the Analytical Biochemistry Group and **M. R. Guerin** of the Analytical Methodology Group participated in the project "Available; Better Careers" (ABC) designed to acquaint local youngsters with career opportunities available to high school graduates. Brief introductory lectures and simple demonstrations served to inform the students about the activities of a chemical technologist. The students carried out simple experiments and visited informally in the laboratories.

14.6 ALIEN GUESTS IN RESIDENCY

Two scientists from other countries were guests in the Analytical Chemistry Division.

Guest	Sponsor	Division Group
Young Kuk Kim Atomic Energy Research Institute Chyungryang-ri Seoul, Korea	International Atomic Energy Agency	Nuclear and Radiochemistry Group
C. S. P. Iyer Bhabha Atomic Research Centre Trombay, Bombay, India	International Atomic Energy Agency	Methods Development and Evaluation Laboratory

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 returned to the Laboratory in February 1969 following his 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. He did electroanalytical research with T. M. Florence under the direction of Dr. Lloyd Smythe (Sects. 1.10 and 1.16).

A. E. Cameron

Member: International Commission on Atomic Weights (Titular Member)
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)

Committee E-10, Radioisotopes and Radiation Effects
American Society for Testing and Materials (ASTM)

ASTM Committee E-20, Fuel Fabrication

J. S. Eldridge

J. S. Eldridge has participated in the Apollo 11 mission as a member of a Principal Investigator team under the direction of G. D. O'Kelley of the ORNL Chemistry Division. This team operated the Low-Level Radiation Counting Facility in the Lunar Receiving Laboratory, Manned Spacecraft Center, Houston, Texas, during the entire quarantine period of the lunar samples. Information obtained in this investigation was used by the Lunar Sample Analysis Planning Team in deciding the most logical distribution of samples for some 100 scientific experiments to be made on them in laboratories throughout the world. The same team will operate the Facility for the Apollo 12 mission, which is scheduled for November 14, 1969.

Secretary: Subcommittee on the Use of Radioactivity Standards

Committee on Nuclear Sciences

National Academy of Sciences–National Research Council (NAS-NRC)

C. Feldman

Scientific Editor:
Optics and Spectroscopy [English Translation of the Russian journal *Optika i Spektroskopiya*, published by Optical Society of America.]

Member: Subcommittee on Photographic Photometry

Subcommittee on Atomic Absorption Analysis

Committee E-2, Emission Spectrochemical Analysis
American Society for Testing and Materials

Publications Committee
Society for Applied Spectroscopy

D. J. Fisher

Recipient: American Chemical Society Award in Chemical Instrumentation

Member: Editorial Board
Chemical Instrumentation

Session Planner:
Trends in Electroanalytical Instrumentation
1969 Eastern Analytical Symposium
New York, N.Y.

G. Goldberg

Publicity Chairman:
Analytical Group
East Tennessee Section
American Chemical Society

Member: Subcommittee I, Research
Subcommittee VIII, Corrosion of Nuclear Materials
Task Group B, Liquid Sodium Systems Alkali Metal Corrosion
Task Group C, Liquid Potassium Systems
Committee G-1, Corrosion of Metals

Analytical Methods Committee
Alkali Metals Task Force
Nonferrous Metals Division
Committee E-3, Chemical Analysis of Metals

American Society for Testing and Materials

G. Goldstein

Member: Subcommittee on Nucleotides and Related Compounds
Committee on Specifications and Criteria for Biochemical Compounds
National Academy of Sciences–National Research Council (NAS-NRC)

Program Chairman:
Analytical Group
East Tennessee Section
American Chemical Society

C. A. Horton

C. A. Horton returned to the Laboratory in November 1968, after two years at the Seibersdorf Laboratory, International Atomic Energy Agency, Vienna, Austria, where he was head of the analytical chemistry work.

Representative from the American Institute of Chemists (AIC) to:
Inauguration of President John Fincher
Carson-Newman College
Jefferson City, Tenn.

Treasurer and Committee Member:
Tennessee Chapter
AIC

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

Committee on Analytical Chemistry (1966–1969)
Division of Chemistry and Chemical Technology
National Academy of Sciences–National Research Council (NAS-NRC)

Advisory Board
The Analyst

Advisory Committee
Microchemical Journal

Board of Editorial Advisors
Analytica Chimica Acta

Chairman: Symposium, ACS Award in Chemical Instrumentation
Analytical Chemistry Division
157th National Meeting
American Chemical Society
Minneapolis, Minn.

W. R. Laing

Secretary: Subcommittee V
Committee C-26, Fuel, Control, and Moderator Materials for Nuclear Reactor Applications
American Society for Testing and Materials (ASTM)

C. E. Lamb

Member: Executive Committee (1967–1970)
Remote Systems Technology Division
American Nuclear Society (ANS)

National Membership Committee (1968–1969)
(Also of ANS)

W. S. Lyon

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 Standard Institute

Chairman: Referee Committee F, Industrial Wastes
Subcommittee on Radiological Methods for Biological and Environmental Samples
American Public Health Association (APHA)

Associate Editor:
Radiochemical and Radioanalytical Letters

S. A. Reynolds

Member: ASTM Committee D-19 on Water
Subcommittee 4, Methods of Radiochemical Analysis
ASTM Committee D-19

ASTM Committee E-10 on Radioisotopes and Radiation Effects

Subcommittee III, Tracer Applications
ASTM Committee E-10

Subcommittee V, Neutron Dosimetry
(Also of 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)
Subcommittee on Radiological Methods for Biological and Environmental Samples
American Public Health Association (APHA)

W. J. Ross

W. J. Ross is on leave to the Instituto Venezolano de Investigaciones Cientificas (IVIC), Caracas, Venezuela.

Member: Committee D-12, Analysis of Water
American Society for Testing and Materials (ASTM)

W. D. Shults

Chairman-Elect:
Analytical Chemistry Group
East Tennessee Section
American Chemical Society (ACS)

Member: Long-Range Planning Committee
(Also of East Tennessee Section, ACS)

Environmental Pollution Committee
(Also of East Tennessee Section, ACS)

Committee E-19, Gas Chromatography
American Society for Testing Materials (ASTM)

Committee D-19, Water
(Also of ASTM)

J. E. Strain

Secretary-Treasurer:
Tennessee Chapter
American Institute of Chemists (AIC)

Member: Committee E-3, Chemical Analysis of Metals
American Society for Testing and Materials

P. F. Thomason

Recipient: "Citation for Achievement" Award
William Jewell College
Liberty, Mo.

J. C. White

Chairman-Elect:
Division of Analytical Chemistry
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
Committee on Analytical Chemistry
NAS-NRC

Associate Member:
Commission V.7, Analytical Radiochemistry and Nuclear Materials
International Union of Pure and Applied Chemistry (IUPAC)

Anna M. Yoakum

Treasurer: Southeast Section
Society for Applied Spectroscopy

Consultant: Stewart Laboratories, Inc.
Knoxville, Tenn.

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

CONTRIBUTIONS TO BOOKS

AUTHOR(S)	TITLE	PUBLISHER
1 Bird, E. D.,[1] J. F. Emery, S. B. Lupica, W. S. Lyon	"Neutron Activation Analysis of Brain for Copper and Manganese," pp. 138–141 in *Modern Trends in Activation Analysis* (Proceedings of the 1968 International Conference held Oct. 7–11, 1968, in Gaithersburg, Maryland), ed. by J. R. DeVoe, Special Publication 312, Vol. I (1969)	National Bureau of Standards, Gaithersburg, Md., 1969
2 Feldman, C.	"Recommended Practices for Photographic Photometry in Spectrochemical Analysis (E 116-67)" in *Methods for Emission Spectrochemical Analysis,* 5th ed.	American Society for Testing and Materials, Philadelphia, Pa., 1968
3 Lyon, W. S.	"Basic Concepts of Activation Analysis," pp. 439–454 in *Radioisotopes in Medicine in Vitro Studies* (Proceedings of Symposium on Radioisotopes in Medicine in Vitro Studies held Nov. 13–16, 1967, ORAU, Oak Ridge, Tenn.), R. L. Hayes, F. A. Goswitz, and Beverly E. Murphy, ed., CONF-671111	U.S. Atomic Energy Commission, Division of Technical Information Extension, Oak Ridge, Tenn., June 1968
4	"Detectors and Instrumentation, Synopsis of Discussions," pp. 1032–1034 in *Modern Trends in Activation Analysis* (Proceedings of the 1968 International Conference held Oct. 7–11, 1969, in Gaithersburg, Maryland), ed. by J. R. DeVoe, Special Publication 312, Vol. II (1969)	National Bureau of Standards, Gaithersburg, Md., 1969
5 Mamantov, G.[2]	"Electrode Reactions in Molten Fluorides," pp. 529–561 in *Molten Salts: Characterization and Analysis,* ed. by G. Mamantov	Marcel Dekker, New York, 1969
6 Manning, D. L.	"Voltammetric Studies of Chromium(II) in Molten $LiF-BeF_2-ZrF_4$ at 500°C," pp. 563–573 in *Molten Salts: Characterization and Analysis,* ed. by G. Mamantov	Marcel Dekker, New York, 1969

[1] Bowman Gray Medical School, Winston Salem, N.C.

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

7 Ricci, E., R. L. Hahn*	"Present Status of the ^{3}He-Activation Analysis Program at ORNL," pp. 15–29 in *Proceedings of the 2nd Conference on Practical Aspects of Activation Analyses with Charged Particles, Liege, Belgium, September 21–22, 1967,* ed. by H. G. Ebert (EUR 3896 d-f-e)	European Communities, Brussels, March 1968
8 Strain, J. E.	"Standard Gamma-Ray Spectra of the Elements Following 14-MeV Neutron Irradiations," pp. 48–55 in *The Use of Small Accelerators for Teaching and Research* (Proceedings of Conference on the Use of Small Accelerators for Teaching and Research, held April 8–10, 1968, ORAU, Oak Ridge, Tenn.), J. L. Duggan, ed., CONF-680411	U.S. Atomic Energy Commission, Division of Technical Information Extension, Oak Ridge, Tenn., 1968
9 Yoakum, Anna M.	"Recent Advances in Analytical Emission Spectroscopy," pp. 57–66 in *Developments in Applied Spectroscopy,* Vol. 6 (Proceedings of the Eighteenth Annual Mid-America Spectroscopy Symposium held in Chicago, May 15–18, 1967)	Plenum, New York, 1968
10 Young, J. P.	"Spectrophotometric Studies of Solute Species in Molten Fluoride Media," pp. 189–208 in *Molten Salts: Characterization and Analysis,* ed. by G. Mamantov	Marcel Dekker, New York, 1969

THESES

AUTHOR	TITLE	PUBLISHER
11 Armento, W. J.[3]	*The Chemistry of Several Hydroxochloroplatinates(IV) and Their Use in the Isolation of Carrier Free Pt^{197}* (Ph.D. thesis)	Georgia Institute of Technology, Atlanta, June 1969
12 Jenkins, H. W.[4]	*Electrochemical Measurements in Molten Fluorides* (Ph.D. thesis)	University of Tennessee, Knoxville, March 1969

ARTICLES

AUTHOR(S)	TITLE	PUBLISHER
13 Anderson, D. H., E. E. Anderson, K. Bieman, P. R. Bell, D. D. Bogard, R. Brett, A. L. Burlingame, W. D. Carrier, E. C. T. Chao, N. C. Costes, D. H. Dahlem, G. B. Dalrymple, R. Doell, J. S. Eldridge, and many others	"Preliminary Examination of Lunar Samples from Apollo 11"	*Science* 165(3899), 1211 (1969)
14 Belew, W. L., D. J. Fisher, H. C. Jones,[5] M. T. Kelley	"Apparatus for Precision Control of Drop Time of Dropping Mercury Electrode in Polarography"	*Anal. Chem.* 41, 779 (1969)

[3] ORAU Graduate Fellow, June 11, 1963–Apr. 15, 1966; present address: Babcock and Wilcox, Alliance, Ohio.

[4] ORAU Graduate Fellow, Sept. 1, 1967–Mar. 5, 1969.

[5] Present address: Instrumentation Section, Isotopic Analysis Department, Laboratory Division, Oak Ridge Gaseous Diffusion Plant.

15 Bemis, C. E., Jr.,* J. Halperin,* R. Eby	"The Alpha Decay Half Lives of ^{242}Pu and ^{244}Pu"	*J. Inorg. Nucl. Chem.* **31**, 599 (1969)
16 Cameron, A. E., D. H. Smith, R. L. Walker	"Mass Spectrometry of Nanogram-Size Samples of Lead"	*Anal. Chem.* **41**, 525 (1969)
17 Costanzo, D. A., L. T. Corbin	"A Glovebox System for a Spectrophotometer"	*Nucl. Appl.* **5**, 333 (1968)
18 Crist, Linda J.,[6] Helen P. Raaen	"Fluorescence and Phosphorescence Response of Steroids to Sulfuric Acid in Thin-Layer Chromatography at 77°K"	*J. Chromatog.* **39**, 515 (1969)
19 Emery, J. F., J. E. Strain, G. D. O'Kelley,* W. S. Lyon	"Non-destructive Neutron Activation Analysis of the Allende Meteorite"	*Radiochem. Radioanal. Letters* **1**, 137 (1969)
20 Fisher, D. J., W. L. Belew, 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"	*Chem. Instrum.* **1**, 181 (1968)
21	"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"	*Chem. Instrum.* **1**, 225 (1968)
22 Florence, T. M.,[7] W. L. Belew	"Interpretation of the Polarographic Behaviour of Metal–Solochrome Violet RS Complexes"	*J. Electroanal. Chem.* **21**, 157 (1969)
23 Guyon, J. C.,[8] W. D. Shults	"Rapid Phosphate Determination by Fluorimetry"	*J. Am. Water Works Assoc.* **61**, 403 (1969)
24 Halperin, J.,* C. E. Bemis,* R. E. Druschel,* J. R. Stokely	"The Thermal Neutron Capture Cross Section and Resonance Integral of ^{252}Cf"	*Nucl. Sci. Eng.* **37**, 228 (1969)
25 Handley, T. H., J. H. Cooper	"Quantitative Electrodeposition of Actinides from Dimethyl Sulfoxide"	*Anal. Chem.* **41**, 381 (1969)
26 Hannatz, B.,* T. H. Handley	"Nuclear Spectroscopy in the Neutron-Deficiency $173 \leqslant A \leqslant 185$ Region"	*Nucl. Phys.* **A121**, 481 (1968)
27 Hatcher, D. W., N. G. Anderson*	"An Optical Density Thermometer"	*Anal. Letters* **2**, 373 (1969)
28 Jenkins, H. W.,[4] G. Mamantov,[2] D. L. Manning	"Emf Measurements on the Nickel-Nickel(II) Couple in Molten Fluorides"	*J. Electroanal. Chem.* **19**, 386 (1968)
29 Jones, H. C.,[5] W. L. Belew, R. W. Stelzner, T. R. Mueller, D. J. Fisher	"A Solid-State Controlled-Potential DC Polarograph with Cyclic Scanning and Calibrated First- and Second-Derivative Scales"	*Anal. Chem.* **41**, 772 (1969)
30 Katz, S.,* C. A. Burtis[9]	"The Relationship Between Chemical Structure and Elution in an Anion Exchange System"	*J. Chromatog.* **40**, 20 (1969)

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[6] ORAU Student Trainee from the University of Arkansas, Jonesboro; summer 1968.

[7] Chemistry Division, Australian Atomic Energy Commission Research Establishment, Lucas Heights, New South Wales, Australia.

[8] Professor of Chemistry and Chairman of the Department of Chemistry, University of Missouri, Columbia.

[9] Present address: Varian Aerograph, 2700 Mitchell Dr., Walnut Creek, Calif. 94598.

31 King, E. A., Jr.,[10] E. Schonfeld,[10] K. A. Richardson,[10] J. S. Eldridge	"Meteorite Fall at Pueblito de Allende, Chihuahua, Mexico: Preliminary Information"	*Science* **163**, 928 (1969)
32 Kohn, H. W.,* T. E. Willmarth	"Metallic Colloids in Molten Salts"	*Science* **163**, 924 (1969)
33 Lepri, Marcia A.,[11] W. S. Lyon	"Decay of ^{103}Pd–^{103m}Rh"	*Intern. J. Appl. Radiation Isotopes* **20**, 297 (1969)
34 Lyon, W. S.	Book Review of: *Nuclear Activation Techniques in the Life Sciences* (Proceedings of a Symposium, May 8–12, 1967), International Atomic Energy Agency, Vienna, 1967	*Health Phys.* **16**, 405 (1969)
35 Macklin, R. L.,* J. H. Gibbons,* E. Ricci, T. Handley, D. Cuneo*	"Proton Reaction Determination of Lithium and Fluorine in Molten Salt Reactor Graphite"	*Nucl. Appl.* **5**, 269 (1968)
36 Moore, F. L.	"New Extraction Chromatographic Method for Rapid Separation of Americium from Other Transuranium Elements"	*Anal. Chem.* **40**, 2130 (1968)
37	"New Liquid-Liquid Extraction Method for the Rapid Separation of Cerium(IV) from Berkelium(IV) and Other Elements"	*Anal. Chem.* **41**, 1658 (1969)
38 Morrow, R. W.,[12] J. A. Dean,[13] W. D. Shults, M. R. Guerin	"A Silicon-Specific Detector Based on Interfacing a Gas Chromatograph and a Flame Emission or Atomic-Absorption Spectrometer"	*J. Chromatogr. Sci.* **7**, 572 (1969)
39 Pruitt, M. E., Sue H. Prestwood, H. A. Parker	"Some Practical Applications of Gamma-Ray Spectral Resolution to Radionuclide Analysis"	*Nucl. Appl.* **5**, 253 (1968)
40 Raaen, Helen P.	"Improved Thin-Layer Chromatographic Separation of Amanita Toxins on Silica Gel G Chromatoplates"	*J. Chromatog.* **38**, 403 (1968)
41	"Vertical-Orifice Dropping-Mercury Electrode of Polytetrafluoroethylene for Rapid Polarography in Glass-Corroding Media"	*Chem. Instrum.* **1**, 287 (1969)
42	"Polarography of Sulfur Dioxide (Sulfite) in Aqueous Hydrofluoric Acid"	*Anal. Chim. Acta* **44**, 205 (1969)
43 Ricci, E.	"Influence of Channeling in Conventional Helium-3 Activation Analysis"	*Analyst* **94**, 435 (1969)
44 Ricci, E., T. H. Handley	"Neutron Activation Analysis with ^{252}Cf Sources"	*Trans. Am. Nucl. Soc.* **11**, 470 (1968)
45 Ross, H. H.	"Measurement of β-Emitting Nuclides Using Cerenkov Radiation"	*Anal. Chem.* **41**, 1260 (1969)
46 Ross, H. H., T. G. Chapman*	"Radiotracer Evaluation of a Lunar Surface Sampler"	*Radiochem. Radioanal. Letters* **1**, 111 (1969)
47 Stokely, J. R., Jr., W. D. Shults	"Controlled-Potential Coulometric Determination of Americium"	*Anal. Chim. Acta* **45**, 417 (1969)

[10] Lunar Receiving Laboratory, NASA Manned Spacecraft Center, Houston, Texas.

[11] ORAU Student Trainee from Mt. Holyoke College, South Hadley, Mass.; summer 1968.

[12] Predoctoral Student Guest from the Graduate School of Chemistry, University of Tennessee, Knoxville.

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

48 Stokely, J. R., W. D. Shults	"Determination of Plutonium by Controlled-Current Coulometry"	*Anal. Chim. Acta* **45**, 528 (1969)
49 Toth, L. M.,* J. P. Young, G. P. Smith*	"Diamond-Windowed Cell for Spectrophotometry of Molten Fluoride Salts"	*Anal. Chem.* **41**, 683 (1969)
50 Turner, L. P.[14]	"Characterization of Nucleotides and Nucleosides by Pyrolysis–Gas Chromatography"	*Anal. Biochem.* **28**, 288 (1969)
51 White, J. C.	Book Review of: *Survey of Analytical Chemistry*, S. Siggia, McGraw-Hill, New York, 1968	*Anal. Chem.* **41**(3), 63A (1969)
52 Young, J. P.	"Absorption Spectra of Several 3d Transition Metal Ions in Molten Fluoride Solution"	*Inorg. Chem.* **8**, 825 (1969)
53 Zittel, H. E., T. H. Row*	"Radiation and Thermal Stability of Sprays"	*Trans. Am. Nucl. Soc.* **11**, 375 (1968)

REPORTS

AUTHOR(S)	TITLE	REPORT NO. AND DATE
54 Bate, L. C., F. F. Dyer	*Investigations of Radioisotope Tagging of Copper Wire*	June 3, 1969 (unpublished)
55 Belew, W. L.	*Report of Assignment at the Lucas Heights Research Establishment – January 9, 1968 to February 3, 1969*	Mar. 20, 1969 (unpublished)
56 Blumberg, R.,* F. F. Dyer, T. H. Mauney,* D. Scott*	*Measurements of Fission-Product Deposition in the MSRE with Ge(Li) Gamma Ray Spectroscopy*	Nov. 19, 1968 (unpublished)
57 Cameron, A. E.	*Report of Foreign Travel June 30 through July 8, 1969*	July 25, 1969 (unpublished)
58 Corbin, L. T.	*Report of Foreign Travel to Canada – Sept. 8–12, 1969*	Oct. 8, 1969 (unpublished)
59 Goldstein, G.	*Report of Foreign Travel to Great Britain, Denmark, Austria and Switzerland – July 19 to August 17, 1969*	Sept. 11, 1969 (unpublished)
60 Haire, R. G.,* T. E. Willmarth	*Trends and Differences in the Crystallization Behavior of Lanthanide Hydroxide Preparations*	ORNL-TM-2387 (Nov. 1968)
61 Katz, S.,* A. Confer,[15] C. D. Scott,* C. A. Burtis,[9] R. L. Jolley,* N. Lee,* Susan A. McKee,[16] B. A. Maryanoff,[15] W. W. Pitt,* K. S. Warren*	*An Annotated Bibliography of Low-Molecular-Weight Constituents of Human Urine*	ORNL-TM-2394 (Oct. 11, 1968)
62 Kelley, M. T.	*Statistical Quality Control Report, Analytical Chemistry Division, July through September 1968*	Oct. 17, 1968 (unpublished)
63	*Statistical Quality Control Report, Analytical Chemistry Division, October through December 1968*	Jan. 17, 1969 (unpublished)

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

[15] ORNL Cooperative Student in the Chemical Technology Division from the Drexel Institute of Technology, Philadelphia, Pa.; summer 1968.

[16] ORNL Cooperative Student in the Chemical Technology Division, from the University of South Florida, Tampa, Fla.; summer 1968.

64		*Statistical Quality Control Report, Analytical Chemistry Division, January through March 1969*	Apr. 22, 1969 (unpublished)
65		*Statistical Quality Control Report, Analytical Chemistry Division, April through June 1969*	July 29, 1969 (unpublished)
66		*Statistical Quality Control Report, Analytical Chemistry Division, July through October 1969*	Oct. 15, 1969 (unpublished)
67		*Report of Foreign Travel to Birmingham, Harwell, Aldermaston, and London, England – July 21–25, 1969*	Oct. 3, 1969 (unpublished)
68	McKinney, F. E.,* S. A. Reynolds, P. S. Baker*	*Isotope User's Guide*	ORNL-IIC-19 (Sept. 1969)
69	Raaen, Helen P., ed.	*Analytical Chemistry Division Annual Progress Report for Period Ending September 30, 1968*	ORNL-4343 (Dec. 1968)
70	Raaen, Helen P., R. L. Coleman[12]	*Thin-Layer Chromatography of Five Organic Compounds of Interest in Carcinogenesis*	ORNL-TM-2647 (July 18, 1969)
71	Raaen, Helen P., Ann S. Klein,* eds.	*Cumulative Indexes to the Analytical Chemistry Division Annual Progress Reports 1964–1968*	ORNL-3904, rev. 3 (Feb. 1969)
72	Raaen, V. F.,* Helen P. Raaen	*Report of Foreign Travel to England, Norway, and Sweden, July 19–Aug. 16, 1969*	Aug. 27, 1969 (unpublished)
73	Reynolds, S. A.	"Radioisotope Standards, Characterization, and Quality Control" in *Division of Isotopes Development Research and Development Programs: 1968*, pp. 63–64	TID-24823 (Jan. 1969)
74	Row, T. H.,* L. F. Parsly,* H. E. Zittel	*Design Considerations of Reactor Containment Spray Systems – Part I*	ORNL-TM-2412 (Apr. 1969)
75	Sood, R. K.,* W. J. Ross	*Development of Deaerators for Seawater Evaporator Plants. Summary of Work through June 1969*	ORNL-TM-2474 (Sept. 1969)
76	Sood, R. K.,* W. J. Ross, J. W. Hill*	*Performance Tests of Deaerator of 1-Mgd. Clair Engle Desalting Plant*	ORNL-TM-2491 (Sept. 1969)
77	Strain, J. E.	*Foreign Travel Report May 2, 1969 through May 16, 1969*	June 9, 1969 (unpublished)
78	White, J. C.	*Analytical Chemistry Division Research and Development Monthly Summary – October 1968*	Oct. 31, 1968 (unpublished)
79		*Analytical Chemistry Division Research and Development Monthly Summary – November 1968*	Dec. 2, 1968 (unpublished)
80		*Analytical Chemistry Division Research and Development Monthly Summary – December 1968*	Dec. 31, 1968 (unpublished)
81		*Analytical Chemistry Division Research and Development Monthly Summary – January 1969*	Jan. 29, 1969 (unpublished)
82		*Analytical Chemistry Division Research and Development Monthly Summary – February 1969*	Mar. 3, 1969 (unpublished)
83		*Analytical Chemistry Division Research and Development Monthly Summary – March 1969*	Apr. 3, 1969 (unpublished)
84		*Analytical Chemistry Division Research and Development Monthly Summary – April 1969*	May 2, 1969 (unpublished)
85		*Analytical Chemistry Division Research and Development Monthly Summary – May 1969*	June 3, 1969 (unpublished)
86		*Analytical Chemistry Division Research and Development Monthly Summary – June 1969*	July 1, 1969 (unpublished)

87	*Analytical Chemistry Division Research and Development Monthly Summary – July 1969*	July 28, 1969 (unpublished)
88	*Analytical Chemistry Division Research and Development Monthly Summary – August 1969*	Aug. 29, 1969 (unpublished)
89	*Analytical Chemistry Division Research and Development Monthly Summary – September 1969*	Sept. 29, 1969 (unpublished)
90	*Analytical Chemistry Division Research and Development Monthly Summary – October 1969*	Oct. 28, 1969 (unpublished)

METHODS ISSUED TO THE ORNL MASTER ANALYTICAL MANUAL

FORMAL ISSUES

AUTHOR(S)	TITLE	NUMBER(S)	DATE
91 Belew, W. L., H. C. Jones[5]	"Polarographic Drop-Time Controller, ORNL Model Q-2942	1 003044 9 003044	3-1-68
92 Jones, H. C.,[5] W. L. Belew	"Polarographic-Voltammeter, ORNL Model Q-2792, Controlled-Potential, DC"	1 003043 9 003043	3-1-68

Oral Presentations

CONFERENCES ON ANALYTICAL CHEMISTRY IN NUCLEAR TECHNOLOGY

Twelfth Conference

The Twelfth Conference on Analytical Chemistry in Nuclear Technology, sponsored by the Analytical Chemistry Division of ORNL, was held in Gatlinburg, Tennessee, on October 8–10, 1968. Dr. Glenn T. Seaborg, Chairman of USAEC, was the introductory speaker for the series of five sessions on the topic "Analytical Chemistry of the Actinide Elements." Concurrently with this series, a series of six sessions convened on the "Analysis of Environmental Pollutants." Of the total 68 papers presented during the Conference, 33 were given in the sessions on Environmental Pollutants, a topic new to the Gatlinburg Conferences, but one which generated a great deal of interest among attendees.

Members of the Conference Committee were: M. T. Kelley, Division Director; L. J. Brady, Chairman; H. L. Holsopple, Treasurer; H. R. Beatty, Exhibits Coordinator; W. H. Christie; J. H. Cooper; L. T. Corbin; D. A. Costanzo; G. Goldstein; T. R. Mueller; W. T. Rainey; and W. J. Ross.

The total attendance was 320 including 12 representatives from five foreign countries.

Thirteenth Conference

The main topics for the Thirteenth Conference were: "Analytical Chemistry for Fast Breeder Reactor Programs," "Activation and Radiochemical Analysis," and "Automated Methods of Analysis." The Conference was held in Gatlinburg, Tennessee, on September 30–October 2, 1969. Sixty-six papers were presented during the 11 sessions of the Conference. A total of 328 attendees were registered; in addition to chemists from throughout the United States, representatives from France, Germany, Austria, Scotland, Canada, Holland, and Japan attended.

Members of the Conference Committee were: M. T. Kelley, Division Director; L. J. Brady, Chairman; H. R. Beatty, Exhibits Coordinator and Treasurer; C. Feldman; G. Goldberg; D. W. Hatcher; W. R. Laing; W. S. Lyon; W. L. Maddox; and J. C. White.

AT MEETINGS OF PROFESSIONAL SOCIETIES, CONFERENCES, AND THE LIKE

	AUTHOR(S)	TITLE	PRESENTED AT
93	Bate, L. C.	"The Use of a Ge(Li) Diode in Fission Product Analysis"	13th Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Sept. 30–Oct. 2, 1969
94	Bird, E. D.,[1,17] J. F. Emery, S. B. Lupica, W. S. Lyon	"Neutron Activation Analysis of Brain for Copper and Manganese"	International Conference on Modern Trends in Activation Analysis, Gaithersburg, Md., Oct. 7–11, 1968
95	Botts, J. L.	"Analytical Aspects of the Liquid-Metal Fast Breeder Reactor Program – The Methods Being Used and the Problems Involved"	13th Conference on Analytical Chemistry in Nuclear Technology, Sept. 30–Oct. 2, 1969
96	Burtis, C. A.,[9,17] W. C. Butts, S. Katz,* C. D. Scott*	"Chemical Identification of Urinary Constituents Isolated by Anion Exchange Chromatography"	Southeastern Meeting, American Association of Clinical Chemists, Birmingham, Ala., Oct. 11–12, 1968
97	Burtis, C. A.,[9] W. C. Butts, W. T. Rainey, Jr.,[17] C. D. Scott*	"Separation and Identification of Drug Metabolites in Physiologic Fluids by High-Resolution Anion Exchange Chromatography"	First Annual Symposium on Automated, High-Resolution Analyses in the Clinical Laboratory, Oak Ridge, Tenn., March 13–14, 1969
98	Butler, C. T.,*,[17] B. J. Sturm,* R. B. Quincy	"Growth of Pure Single Crystals of Magnesium Oxide by Arc Fusion"	Conference on Crystal Growth, Gaithersburg, Md., Aug. 11–13, 1969
99	Cooper, J. H.,[17] T. H. Handley	"A Study of Parameters Affecting Analytical Alpha Spectrometry"	12th Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Oct. 8–10, 1968
100	Danford, M. D.,*[17] J. H. Burns,* O. L. Keller, Jr.,* J. R. Stokely, W. G. Baldwin*	"Preparation and Volatilities of Some Rare-Earth and Americium Chelates"	Southeastern Regional Meeting, American Chemical Society, Tallahassee, Fla., Dec. 4–7, 1968
101	Dean, J. A.,[13,17] R. W. Morrow,[12] W. D. Shults	"A Silicon Specific Detector Based on Interfacing a Gas Chromatograph and a Flame Emission or Atomic Absorption Spectrometer"	20th Mid-America Symposium, Society of Applied Spectroscopy, Chicago, Ill., May 12–15, 1969
102	Eby, R. E.	"Mass Spectrometric Analysis of Actinide Elements"	12th Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Oct. 8–10, 1968
103			Special Separations Group, Electromagnetic Separations Department, Isotopes Division, Oct. 28, 1968
104	Eldridge, J. S.,[17] V. A. McKay,* G. D. O'Kelley,* R. E. Wintenberg,* P. R. Bell[10]	"Low-Level Gamma Ray Spectrometry for Geochemical and Lunar Samples"	12th Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Oct. 8–10, 1968
105	Emery, J. F.	"Low Energy Peak Efficiencies for Ge(Li) Detectors"	13th Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Sept. 30–Oct. 2, 1969
106	Feldman, C.	"A Beam-Splitter for Use in Calibrating Spectrographic Emulsions"	7th Fall Meeting, Southeastern Section, Society for Applied Spectroscopy, Birmingham, Ala., Oct. 5, 1968

[17]Speaker.

107	"Electrodialysis as an Aid to Spectrochemistry" (Invited Paper)	Pacific Conference on Chemistry and Spectroscopy, San Francisco, Calif., Nov. 7, 1968
108 Fisher, D. J.	"The Circumvention of Chemical Measurement Difficulties through the Design of Appropriate Instrumentation Systems: A Design Philosophy, with Illustrations" (Award Address: American Chemical Society Award in Chemical Instrumentation)	157th National Meeting, American Chemical Society, Minneapolis, Minn., Apr. 13–18, 1969
109 Goldstein, G.	"New Methods in Analytical Biochemistry"	Memorial Research Center, University of Tennessee, Knoxville, Mar. 26, 1969
110 Goldstein, G.,[17] C. A. Burtis[9]	"Newer Column Chromatographic Techniques for the Separation of Organic Compounds"	IUPAC International Symposium on Analytical Chemistry, The University of Birmingham, Birmingham, England, July 21–25, 1969
111 Haire, R. G.,*[17] T. E. Willmarth	"Trends and Differences in the Crystallization Behavior of Lanthanide Hydroxide Preparations"	7th Rare Earth Research Conference, San Diego, Calif., Oct. 28–30, 1968
112 Handley, T. H.,[17] R. L. Macklin,* J. H. Gibbons*	"Proton Reaction Analysis for Lithium and Fluorine in Graphite"	Southeastern Regional Meeting, American Chemical Society, Tallahassee, Fla., Dec. 4–7, 1968
113 Hatcher, D. W.	"Total Serum Cholesterol: Adaptation to the GeMSAEC Approach"	13th Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Sept. 30–Oct. 2, 1969
114 Horton, C. A.	"Uses of Fluorine and Its Compounds in Atomic Energy Programs"	Seminar Series, IAEA, Vienna, Austria, Oct. 15, 1968
115	"IAEA and Austrian Information"	Technical Information Division Luncheon, May 20, 1969
116 Jenkins, H. W.,[4] G. Mamantov,[2,17] D. L. Manning	"A New Voltammetric Method for the Determination of Both Oxidation States of a Reversible Redox Couple"	Meeting of the Electrochemical Society, New York, May 4–9, 1969
117 Jenkins, H. W.,[4] G. Mamantov,[2,17] D. L. Manning, J. P. Young	"Emf and Voltammetric Measurements on the Uranium(IV)/Uranium(III) Couple in Molten $LiF\text{-}BeF_2\text{-}ZrF_4$"	Meeting of the Electrochemical Society, New York, May 4–9, 1969
118 Kelley, M. T.	"Activation Analysis Compared to Other Methods of Analytical Chemistry"	Neutron Activation Course, Special Training Division, ORAU, Oak Ridge, Tenn., July 7–Aug. 29, 1969
119 Kelley, M. T.,[17] D. J. Fisher, W. L. Maddox, W. L. Belew	"High Precision DC Polarography" (Invited Paper)	IUPAC International Symposium on Analytical Chemistry, The University of Birmingham, Birmingham, England, July 21–25, 1969
120 Kelley, M. T., R. W. Stelzner,[17] D. L. Manning	"Computer-Operated, Voltammetric U(IV)/U(III) Ratio Determination in Molten Fluoride Salt Media"	13th Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Sept. 30–Oct. 2, 1969
121 Kubota, H.	"The Radiation Stability of Certain Electrodes"	158th Meeting, American Chemical Society, New York, Sept. 7–12, 1969
122 Leitnaker, J. M.,*[17] R. A. Potter,* K. E. Spear,* W. R. Laing	"Lattice Parameter of U(C,N) as a Function of Composition"	71st Meeting, American Ceramics Society, Washington, D.C., May 5–9, 1969
123 Lyon, W. S.	"Summary of Session Detectors and Instrumentation"	International Conference on Modern Trends in Activation Analysis, Gaithersburg, Md., Oct. 7–11, 1968
124	"Principles of Activation Analysis"	Medical Radioisotopes Course, ORAU, Oak Ridge, Tenn., Nov. 7, 1968

125		"Activation Analysis Using High Resolution Detectors"	22nd Annual Summer Symposium on Analytical Chemistry, Modern Instrumental Techniques for Elemental Analysis, Athens, Ga., June 11–13, 1969
126		"Activation Analysis of Biological Samples"	Multitrack Summer Institute for College Science Teachers, ORAU, Oak Ridge, Tenn., Aug. 6, 1969
127	Mamantov, G.,[2,17] D. L. Manning	"Applications of Controlled-Potential Voltammetry to Molten Salt Research"	157th National Meeting, American Chemical Society, Minneapolis, Minn., Apr. 13–18, 1969
128	Meyer, H. C.,[17] J. M. Peele	"Experiences in Operating the Transuranium Analytical Facility at ORNL"	12th Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Oct. 8–10, 1968
129	Mueller, T. R.,[17] H. C. Jones,[5] D. J. Fisher	"An Integrated Coulometric Titration System"	13th Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Sept. 30–Oct. 2, 1969
130	O'Kelley, G. D.,*[17] J. S. Eldridge, P. R. Bell,[10] K. A. Richardson,[10] E. Schonfeld[10]	"Gamma-Ray Spectrometry of Returned Lunar Samples"	157th National Meeting, American Chemical Society, Minneapolis, Minn., Apr. 13–16, 1969
131	Raaen, Helen P.	"Teflon Layers for Thin-Layer Chromatography"	IUPAC International Symposium on Analytical Chemistry, The University of Birmingham, Birmingham, England, July 21–25, 1969
132	Reynolds, S. A.	"Radioactive Standards – Status and Needs"	ASTM Committee D-19 Meeting, Ft. Lauderdale, Fla., Jan. 23, 1969
133			ASTM Winter Meeting, Denver, Colo., Feb. 3, 1969
134		"Survey of Status of and Needs for Radioactivity Standards"	13th Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Sept. 30–Oct. 2, 1969
135	Ricci, E.	"Charged-Particle Activation"	Multitrack Summer Institute for College Science Teachers, ORAU, Oak Ridge, Tenn., Aug. 8, 1969
136		"Influence of Channeling in Customary ^{3}He Activation Analysis"	International Conference on Modern Trends in Activation Analysis, Gaithersburg, Md., Oct. 7–11, 1968
137		"^{252}Cf as a Neutron Source for Activation Analysis"	Seminar on Production and Applications of the Transplutonium Elements, ORNL, Oak Ridge, Tenn., Jan. 28, 1969
138	Ricci, E.,[17] T. H. Handley	"Neutron Activation Analysis with ^{252}Cf Sources"	Winter Meeting, American Nuclear Society, Washington, D.C., Nov. 11–15, 1968
139	Ross, H. H.	"Theory and Application of Cerenkov Radiation"'	Colloquium in Nuclear Engineering, North Carolina State University, Raleigh, N.C., Nov. 21, 1968
140		"The Measurement of Carbon-14 Using Cerenkov Radiation"	Symposium on the Current Status of Liquid Scintillation Counting, Cambridge, Mass., Mar. 31, 1969
141		"Cerenkov Radiation"	Graduate and Faculty Seminar, University of Missouri, Columbia, Apr. 30, 1969

142		"Instrumentation Logic for Liquid Scintillation Counters"	Special Course on Liquid Scintillation Technology, ORAU, Oak Ridge, Tenn., June 5, 1969
143		"Cerenkov Counting"	Special Course on Liquid Scintillation Technology, ORAU, Oak Ridge, Tenn., June 17, 1969
144		"New Isotope Applications to Chemical Analysis"	U.S. Naval Reserve Seminar, Oak Ridge, Tenn., July 9, 1969
145		"New Methods in Nuclear Instrumentation"	Multitrack Summer Institute for College Science Teachers, ORAU, Oak Ridge, Tenn., Aug. 8, 1969
146	Rubin, I. B.,[17] G. Goldstein	"An Ultrasensitive Isotope Dilution Method for the Determination of L-Amino Acids"	Southeastern Regional Meeting, American Chemical Society, Tallahassee, Fla., Dec. 4–7, 1968
147	Shults, W. D.,[17] J. R. Stokely, J. C. Guyon[8]	"Some Recent Applications of Potentiostatic Coulometry in Transuranium Chemistry"	157th National Meeting, American Chemical Society, Minneapolis, Minn., Apr. 13–18, 1969
148	Silva, R. J.,* R. L. Hahn,* M. L. Mallory,* C. E. Bemis,* P. F. Dittner,* O. L. Keller,* J. R. Stokely, K. S. Toth*	"Nuclear Chemistry with the Oak Ridge Isochronous Cyclotron"	International Conference on the Use of Cyclotrons in Chemistry, Metallurgy, and Biology, Oxford, England, Sept. 22–23, 1969
149	Stelzner, R. W.	"The Design of Measuring Circuits for a DC Polarograph" (Invited Paper)	157th National Meeting, American Chemical Society, Minneapolis, Minn., Apr. 13–18, 1969
150	Strain, J. E.	"Industrial Application of 14-MeV Neutron Generators" (Invited Paper)	IAEA Symposium on Applications of Accelerators, New York, Apr. 8–9, 1969
151		"Research with 14-MeV Neutron Generators"	IAEA Panel Meeting on the Utilization of Neutron Generators in Physics and Chemistry, Budapest, Hungary, May 5–9, 1969
152	Thomason, P. F.	"Instruments for Remote Analysis"	157th National Meeting, American Chemical Society, Minneapolis, Minn., Apr. 13–18, 1969
153		"The Role of the Analytical Chemist in the Nuclear Energy Program" (Award Address "Citation for Achievement" Award William Jewell College Liberty, Mo.)	Student Affiliate Group, American Chemical Society, William Jewell College, Liberty, Mo., Nov. 13, 1968
154	Thomason, P. F.,[17] W. R. Laing	"Precision Studies for the Controlled-Potential Coulometric Titration of Uranium and Plutonium – Comparison of Various Electrolysis Cells"	13th Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Sept. 30–Oct. 2, 1969
155	Wampler, J. M.,[17,18] D. H. Smith, A. E. Cameron	"Isotopic Comparison of Lead in Tektites with Lead in Earth Materials"	3d International Tektite Symposium, Corning, N.Y., Apr. 16–18, 1969
156	White, J. C.	"Standard Reference Materials for Environmental Pollution Analysis as Viewed by the Laboratory Supervisor – A Status Report"	12th Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Oct. 8–10, 1968

[18] Associate Professor of Geophysical Sciences, Georgia Institute of Technology, Atlanta.

157	"The New Image of Analytical Chemistry in Science"	Chemistry Colloquium, University of Florida, Gainesville, Fla., May 16, 1969
158 Whiting, F. L.,[19] G. Mamantov,[2] J. P. Young[17]	"Spectral Evidence for the Existence of the Superoxide Ion in Molten LiF-NaF-KF"	158th Meeting, American Chemical Society, New York, Sept. 7–12, 1969
159 Yoakum, Anna M.	"A Review of Recent Achievements in Analytical Emission Spectroscopy"	20th Annual Mid-America Symposium on Spectroscopy, Chicago, Ill., May 12–15, 1969
160 Yoakum, Anna M.,[17] C. Feldman	"The Spectrochemical Determination of Traces of Bismuth and Rare Earths in Molten Salt Reactor Fuel"	13th Conference on Analytical Chemistry in Nuclear Technology, Gatlinburg, Tenn., Sept. 30–Oct. 2, 1969
161	"Application of Ion-Selective Electrodialysis to the Spectrochemical Determination of Silicon in Curium Oxide ($^{244}Cm_2O_3$)"	Pacific Conference on Chemistry and Spectroscopy, Joint Meeting of Society for Applied Spectroscopy and American Chemical Society, Anaheim, Calif., Oct. 6–10, 1969
162 Zittel, H. E.,[17] T. H. Row*	"Radiation and Chemical Stability of Spray Additives"	15th Meeting, American Nuclear Society, Seattle, Wash., June 15–19, 1969

Patents

AUTHOR(S)	TITLE	PATENT NO.	DATE ISSUED
163 Bate, L. C., F. F. Dyer, L. H. Thacker*	"Radiation Detector and Recorder"	3,431,414	Mar. 4, 1969
164 Moore, F. L.	"Method for Removing Lanthanides and Trivalent Actinides from Aqueous Nitrate Solution"	1,130,044 (British)	Oct. 9, 1968
165	"Method of Separating Berkelium from Cerium"	3,402,027	Sept. 17, 1968
166	"Extraction of Berkelium Values in the Tetravalent State Using 2-Thenoyltrifluoroacetone"	3,409,414	Nov. 5, 1968
167	"Method of Extracting Soluble Metal Complexes Using Amine Solvents"	3,409,415	Nov. 5, 1968
168 Ross, H. H.	"Radioactive Isotope-Activated Light Source for Color Photometer"	3,404,270	Oct. 1, 1968

[19] ORAU Predoctoral Fellow from the Graduate School of Chemistry, University of Tennessee, Knoxville.

Translations

Translator	ORIGINAL REFERENCE			TRANSLATION		
	Author (Transliterated)	Source	Language	Language	Translated Title	ORNL-TR-Number
J. A. Carter	H. Hintenberger	*NATWAY.* 56(5), 262–267 (1969)	German	English	"Mass Spectrographic Analysis of Minerals and Rocks"	2220

Articles Reviewed or Refereed for Periodicals

Reviewer or Referee	Number of Articles Reviewed or Refereed for Indicated Periodical																	
	Anal. Biochem	*Anal. Chem.*	*Anal. Letters*	*Anal. Chim. Acta*	*Chem. Instrum.*	*Environ. Sci. Technol.*	*Intern. J. Appl. Rad. Tech.*	*Isotopes Radiation Technol.*	*J. Chromatogr. Sci.*	*J. Electroanal. Chem.*	*J. Gas Chromatog.*	*J. Phys. Chem.*	*Nucl. Appl.*	*Nucl. Sci. Eng.*	*Radiochem. Radioanal. Letters*	*Special Libraries*	*Talanta*	Total
Belew, W. L.						1												1
Butts, W. C.			1															1
Cameron, A. E.		1																1
Dale, J. M.		1																1
Emery, J. F.		3																3
Feldman, C.		3																3
Fisher, D. J.		1			2													3
Goldstein, G.	1	3			2													6
Horton, C. A.		2																2
Horton, A. D.									1		2							3
Kelley, M. T.						1												1
Lyon, W. S.		7	1										3		4		1	16
Manning, D. L.		1								1								2
Moore, F. L.		3		2										1			1	7
Mueller, T. R.		3			1													4
Raaen, Helen P.																1		1
Reynolds, S. A.		1					2	1					1					5
Ricci, E.		2											1					3
Ross, H. H.		2					2											4
Shults, W. D.		2		1														3
Stelzner, R. W.		1			2													3
Stokely, J. R.		1																1
White, J. C.		1																1
Young, J. P.												1						1
Total	1	38	2	3	7	2	4	1	1	1	2	1	5	1	4	1	2	76

Indexes

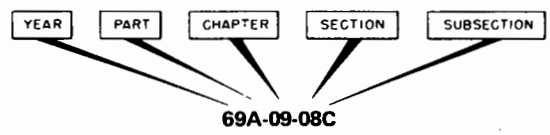
Helen P. Raaen Ann S. Klein[1]

Indexes are a part of the Analytical Chemistry Division annual report for the sixth 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 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.

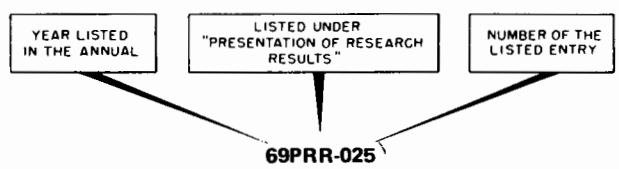
The Author Index is an alphabetical listing of authors, together with number entries that specify the material to which each author contributed. This index includes the authors of material in the body of the report, the authors of the entries listed in the Presentation of Research Results, and the translators 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 69A-09-08C, where 69 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 69PRR-025, where 69 designates the year in which the entry was listed in the annual report, PRR identifies the authored work as being listed in the Presentation of Research Results part of the annual report, and 25 is the number of the entry listed in that part of the report; for example,



In the use of the Index to locate an entry in the Presentation of Research Results part of the report, only the last group of numbers is needed, because the entries are numbered consecutively.

If the entry is in the Translations part of the report, the entry will be identified by the letters TR in the same position as the letters PRR.

Machine limitations necessitate that some entries be in an unusual form. Because special type cannot be used, entries that otherwise would require it are written out; for example, chemical symbols, chemical formulas, Greek letters, *o*-, *m*-, *p*-, etc. Superscript and subscript numbers cannot be printed; hence, valence is designated by a 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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SUBTOTAL	114*	60	17
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STUDENT GUESTS (UT)	4		4
ORAU FELLOWS	2		2
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SUBTOTAL	8		8
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