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# ANALYTICAL CHEMISTRY DIVISION ANNUAL PROGRESS REPORT

For Period Ending December 31, 1984

W. D. Shults, Director

W. S. Lyon, Editor

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MARTIN MARIETTA ENERGY SYSTEMS, INC.
for the
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# EXECUTIVE SUMMARY

W. D. Shults

The Analytical Chemistry Division of Oak Ridge National Laboratory (ORNL) is a diversified analytical chemistry organization which performs research, development, and service work in chemical measurements for a clientele that exists both in and outside of the Laboratory.

**Basic Research and Development.** The division maintains a program to conceptualize, investigate, develop, assess, improve, and implement advanced technology for chemical and physicochemical measurements. Emphasis is on problems and needs identified with ORNL and Department of Energy (DOE) programs, but attention is also given to needs in the analytical sciences themselves. This program is composed of medium- to long-term projects and is supported primarily by the DOE. Fundamental R&D constitutes ca. 25% of our effort.

Several new projects were initiated this year in our basic research program. The spectra of actinide compounds are being studied as a function of pressure. To our knowledge, this is the first study of pressure-induced phase changes in the actinide halides. A supersonic expansion apparatus was set up and used for several interesting spectral experiments, one of which was a comparison of our self-scanning CW dye laser technique (i.e., phase conjugate reflection) with a conventional mechanically scanned laser. We found a 20-fold greater resolution with the self-scanning technique. This approach provides very precise (0.12 $cm^{-1}$) and resolute (0.2 $cm^{-1}$ linewidth) performance as well. Research into our self-scanning laser system has led into work with a degenerative four wave mixing technique which offers even more resolution. This approach may provide an optical means for measuring isotopic ratios. A laser-based system has been designed and is being set up to interrogate, via flourescence or optogalvanic means, chemical vapor deposition processes. Preliminary experiments into the measurement of surface area via positron annihilation have been conducted. Exploration of hollow cathodes as mass spectroscopic sources for intractable elements like Hg, Te, and Os has begun. A thorough investigation of the MS/MS behavior of several nitro-aromatic compounds was initiated this year. Many spectra have been taken using both electron impact and collision-activated dissociation (including positive and negative modes of operation). These spectra will provide the basis for application of the MS/MS technique to important technical problems. We have completed arrangements to acquire a new Fourier Transform Mass Spectrometer and will enter this new R&D area in 1985.

Significant progress also has been made in several on-going projects this year. Research into the secondary ion mass spectrometry (SIMS) of organic compounds has established that the fragmentation patterns of molecular ions from SIMS are similar to those deriving from conventional 70 eV electron impact ionization. These (and other) results suggest that we may be able to develop the theory of organic secondary ion emission as part of this project. Work on laser-ionization in SIMS—our RIMS/SIMS project—has included a study of uranium and samarium. As part of the parallel research project in resonance ionization mass spectrometry (RIMS), we have completed a study of the seven isotopes of neodymium. Ratios can be determined in 40 ng samples with ±1% accuracy, but we cannot go to the desired 10 ng level with existing equipment. An evaluation of the CW mode in RIMS was carried out also. Heavy-ion-induced X-ray satellite emission (HIXSE) studies have expanded into an examination of M X-rays from Pb and Au, using 1.0 MeV/u [illegible] ions: the M spectra vary with the composition of the target which parallels previous work with K and L X-rays. Incorporation of a micro-channel plate detector into our positron spectrometer provided a $10^2$–$10^3$ gain in the rate of data collection and enabled us to complete studies of W, Mo and Cu. A brighter source of positrons (using $^{68}$Ge) was designed, constructed and tested. We acquired a time-of-flight mass spectrometer to augment on-going studies into thermal ionization MS; it will allow us to study in real time the evolution of various species during thermal ionization experiments. Work on the thin layer spectroelectrochemistry of sulfur and $NbCl_5$ in molten salt media is virtually complete.

**Programmatic Research and Development.** The division carries out a wide variety of chemical work that typically involves analytical research and/or development plus the utilization of analytical results or special analytical capabilities to expedite programmatic interests. The effort in this category comes from ORNL and DOE programs and from "Work-for-Others" agreements. Emphasis here is on "applied" chemistry. Programmatic R&D constitutes *ca.* 25% of our work.

Progress in this category this year included two ORNL Exploratory Studies ("Seed Money") projects. Under one of these we developed a means for determining the active cells in affixed biomass samples, based upon ultrasonication and then cell counting with optical and electrical coincidence. The second "seed money" project involved development of a helium ionization detector for gas chromatography, utilizing a pulsed mode of operation. This latter work is being developed as a candidate for technology transfer as is some of the remote instrumentation that we have developed for radiochemical operations. Another instrumentation development, a dosimeter for measuring tobacco smoke exposure in humans, is a candidate for an IR-100 award. In an on-going study of methods for decreasing the toxicological characteristics of fuel refinery products, we confirmed that catalytic hydrogenation is an effective means for destroying mutagens and tumorigens in refinery products and found that the cleanup can be enhanced considerably by a pre-wash with acid. In another study related to environmental issues, the electrochemistry of (trace) munitions components in water has been studied by both voltammetry and pulse polarography. This technique has much potential for screening and monitoring, and for detection of components in liquid chromatography experiments. Considerable effort was devoted to analytical aspects of indoor air pollutants this year, to support work in the Health and Safety Research Division (HASRD) at ORNL. We developed new trapping, thermal desorption and MS monitoring methodologies specifically for the HASRD program. We also carried out an extensive chemical characterization of marijuana tobacco smoke, the first under well defined conditions. A study of ion implanted $^{30}$Si in natural Si was performed using SIMS.

**Technical Service.** The division performs a wide variety of technical service work. Much of it is short-term in nature and comes from other divisions and programs within ORNL. A significant amount originates outside of ORNL, however, and it usually draws upon special talents or capabilities of the division. Technical service work includes chemical and physico-chemical measurements, special projects and/or studies that involve chemistry and chemical measurements, consultation, collaboration, quality assurance related to chemical measurements, and various types of chemical support for the Laboratory in times of urgency. Technical service constitutes *ca.* 50% of our activities.

Several activities in the technical services category warrant mention here. A new internal standard, $K_3Fe(CN)_6$, was implemented for diffuse reflectance Fourier transform infrared spectroscopy. It is applicable to samples that are solid or liquid or both. Upon recommendation of DOE's New Brunswick Laboratory, the division was requested to become a referee laboratory for high-precision analysis of uranium ore concentrate, and we did so. On-going work on remote instrumentation has resulted in development of capabilities to do amperometric titrations remotely, which is important for measuring plutonium. We have initiated work with a fiber optic, modular remote spectrophotometer as part of this project. Development of an X-ray fluorescence system for determining uranium and plutonium in highly radioactive materials has been delayed, pending construction of a suitable monochromator. We have determined U and Th in many semi-conductors and related materials during this report period, for several industrial concerns. During this period also, the division has responded to and participated in many unusual and sometimes very demanding activities that were both stimulating and challenging. Such activities have ranged from participation in a major acid rain program plan with Environmental Sciences Division to characterizing effluent streams prior to the design of a new ORNL waste treatment facility to locating the source of radioactive releases to studying the iodine levels in the thyroids of deer killed on the roads within the reservation. The diversity of analytical work in a multi-program institution like ORNL is tremendous.

The Analytical Chemistry Division is organized into five major sections, each of which may carry out any type of work falling in the three categories mentioned above. Chapters 1 through 5 of this report highlight progress within the five sections during the period January 1, 1984, to December 31, 1984. A short summary introduces each chapter to indicate work scope. Information about quality assurance and safety programs is presented in Chap. 6, along with a tabulation of analyses rendered. Publications, oral presentations, professional activities, educational programs, and seminars are cited in Chaps. 7 and 8. Approximately 55 articles, 24 proceedings publications and 40 reports have been published, and 95 oral presentations were given during this reporting period. Some 274,194 determinations were performed on approximately 152,330 samples in 1984. The overall quality factor was 95%.

# 1. ANALYTICAL METHODOLOGY

W. S. Lyon

Analytical Methodology is composed of two groups: Analytical Instrumentation and Analytical Physics. Major funding for both groups comes from DOE Office of Basic Energy Sciences; major objectives are to develop new instruments and techniques for analytical measurements. In addition the groups perform some development such as remote instrumentation for hot cells or special materials characterization.

Research in Analytical Instrumentation is primarily concerned with lasers and their application. An example of the latter is our cooperative effort with the Mass Spectrometry Section in resonance ionization mass spectrometry (RIMS). Work in Analytical Physics has concentrated on positron spectroscopy, positron source improvement, surface area determination through measurement of positronium formation, and heavy ion induced satellite X-ray emission. The latter is a cooperative program with a number of other ORNL divisions.

## ANALYTICAL INSTRUMENTATION

H. H. Ross

This year, as in the past, our work in analytical instrumentation has been centered in three major areas: analytical spectroscopy, advanced instrumentation, and special services. In each of these areas, we go through a cycle of project initiation, continuation, and completion. Thus, at any given time, several projects are active in each work phase. Occasionally, a particular research effort will be put on "temporary hold" in order to explore an exciting, new research finding that demands immediate investigation. This is frequently the way that we define new program directions and develop fresh research ideas. We are particularly proud of the innovative approaches that we have taken in our work and the success we have had in bringing this creativity to fruition. Much of what follows will support this contention.

### Spectroscopy Research

A new research effort has been initiated that is directed toward laser spectroscopic studies of the fundamental chemical and physical processes that occur in a chemical vapor deposition (CVD) plasma. Although CVD is used extensively for manufacturing microelectronics, electro-optics and wear-resistant coatings, little is understood about the fundamental processes of the deposition. The goal of this project is to understand these processes so that CVD experimental parameters may be manipulated to achieve optimum results. Our initial effort has been directed toward the design and construction of low-pressure CVD cells with built-in features necessary for the detection of laser-induced optical signals. We are presently evaluating two detection schemes: laser-induced fluorescence and optogalvanic detection. Different schemes may be used for different CVD cell designs, plasma types, and chemical reactions to obtain the optimum results. A cell employing a low-pressure radio frequency (RF) plasma has been constructed using a small bench-top RF generator. Different configurations of a RF induction coil were designed and tested to obtain the most efficient and stable plasma. Evaluation of RF optogalvanic detection hardware is underway. A dc plasma cell, to be used for dc optogalvanic detection, is also under construction.

A low-cost microcomputer-based data acquisition and control system, employing an IBM personal computer and other necessary peripheral hardware, has been planned and is being purchased. This system is intended for reliable data acquisition and control of the pulsed dye laser system.

Our continuing effort in resonance ionization mass spectrometry (RIMS) is carried out in cooperation with the Mass Spectrometry Section. We are presently investigating the actinides and lanthanides. We excite and ionize the vaporized atoms of these elements with laser light of a single energy band in the region of 580 to 605 nm. In this wavelength region most, if not all, RIMS transitions involve absorption of 3 photons through two intermediate states to generate the ion. Our laser band width is 3 $cm^{-1}$, and it is interesting that we have found many 3-photon transitions for the f-transition elements. In such processes, the second transition often cannot be assigned because the upper level in the transition has not been identified. RIMS spectral studies can be used to identify these levels. In other work we have evaluated the use of CW lasers for the RIMS uranium determination and found that relatively large samples (>1ug) were needed. It was hoped that CW lasers would improve the sensitivity of RIMS because of the obvious improvement of duty cycle over that of our pulsed laser. We also noted that the static mode structure of the laser caused some severe bias errors in isotope ratio measurements; however, this could be corrected by using a vibrating quartz refraction plate to scramble the modes. We have applied RIMS to a determination of neodymium. The technique is, at present, poorer in sensitivity and accuracy than existing thermal ionization methods, but does not require the difficult chemical separation of other lanthanide isobars. Further description of our results appear in the Mass and Emission Spectrometry Section of this report.

We have begun an investigation of the use of degenerate four-wave mixing (dfwm) in atomic vapors as a method of elemental analysis. In these experiments, three optical beams of identical frequency are crossed in the medium to be analyzed. Two of the beams are counter-propagating with the third at any angle. For parallel polarizations, two of the beams form an interference pattern and when light is resonant with an optical transition of the medium a concentration grating of ground and excited states is produced. For the case where two crossing beams are orthogonally polarized, a grating is formed in the resonant medium with degenerate levels but now it is a grating of coherently aligned atoms.

The third beam can Bragg reflect from these gratings to form a fourth beam. The intensity of this beam will depend on the concentration of the optically resonant species. An attractive feature of this measurement is that it is Doppler free. Thus, in low-pressure gases (1 torr), a hundred-fold resolution enhancement is possible. Such resolution would allow the optical measurement of isotope ratios at much higher abundance ratios than is now possible.

Experiments were carried out using our Nd-Yag pumped dye laser system and a heat pipe oven containing sodium atoms. We observed dfwm signals with various combinations of pump and probe polarizations. Each combination gave specific and different responses. This investigation will continue with a study of two-photon type transitions, and we also plan to set up a high-resolution CW dye laser for Doppler free studies of one-photon transitions.

We previously reported that radiation from an untuned CW dye laser could be spectrally narrowed and passively scanned by feedback from a $BaTiO_3$ passive phase conjugate reflector. This laser has been used as a spectroscopic source and its performance compared with a mechanically tuned CW laser in two experiments. The sodium D lines were measured for the first demonstration. The transitions were monitored through the optogalvanic effect in a hollow cathode discharge lamp.

Also, fluorescence excitation spectra of molecular iodine in a supersonic expansion have been measured to further demonstrate the potential of this new laser system. It is well known that the selectivity of molecular fluorescence measurements can be considerably enhanced by cooling the sample in an expansion from a supersonic nozzle into a vacuum. These high resolution fluorescence excitation spectra place major demands on the linewidth and tuning range of the excitation source. The self-scanning CW dye laser that we reported previously has exactly these attributes. Thus, spectra with more than 5000 resolution elements can be easily measured. These experiments also show for the first time that the scanning characteristics of this laser are sufficiently continuous and monotonic over the entire scanning range for high resolution spectroscopic measurements.

Excitation spectra, using the self-scanning CW dye laser and a conventional mechanically tuned dye laser, were compared for both the supersonic beam and room temperature samples of iodine. A 20-fold greater resolution of the self-scanning dye laser over the mechanically tuned laser is observed in the excitation spectra. The precision of the self-scanning dye laser is demonstrated further by the accuracy with which the transition frequencies can be determined. Frequencies of 13 prominent band origins were measured relative to the 11-0 and 11-2 origins. The standard deviation for the 11 frequencies not used in the calibration was 0.12 cm$^{-1}$. The maximum deviation was 0.2 cm$^{-1}$, about the same as our laser linewidth.

Spectral studies at the Transuranium Research Laboratory have been expanded this year. We have initiated a program to investigate the spectra of actinide compounds as a function of pressure (up to 13 GPa). A diamond anvil cell that would be compatible with our microscope-spectrophotometer was fabricated and tested at the European Institute for Transuranium Elements at Karlsruhe. First results with the cell have been significant and have demonstrated that hexagonal $AmI_3$ is converted to orthorhombic $AmI_3$ at a pressure of 2 GPa and monoclinic $CfBr_3$ is converted to orthorhombic $CfBr_3$ at a pressure of 3 GPa. Both conversions are irreversible; the orthorhombic phase remains on release of the pressure. In the latter case, this is the second synthetic route to orthorhombic $CfBr_3$. This structure cannot be made directly. Previously we had shown that it could be prepared by the $\beta^-$ transformation of orthorhombic $BkBr_3$. These are the first studies of pressure induced phase transformations of actinide halides by any technique. Pressure induced phase transformations of actinide metals and lanthanide halides have been studied by X-ray diffraction techniques, however.

Work continues on spectral studies of Cf(III) doped into lanthanide halides. We have demonstrated that the spectra of Cf(III) doped into lanthanide bromides conforms to the structure of the host and not that of normal $CfBr_3$. This adds proof to the hypothesis we previously set forth that the structure (coordination) of progeny ions growing into solid state materials by radioactive decay is controlled by the host material. In cooperation with workers at the

University of Karlsruhe, who have prepared the samples, we have undertaken spectral studies of mixtures of $EuCl_2$ and $EuCl_3$, including the end members of this series in order to understand the basis for the blue color that is observed in venier compounds of the type $Eu_4Cl_9$ and $Eu_5Cl_{11}$. The spectra of these structures exhibit a broad absorption band centered at 620 nm and a sharp cut-off at 420 nm attributable to Eu(II). We presently believe that the blue absorption is an electron transfer band from Eu(II) to Eu(III) that is possible because of the venier structure.

*(J. M. Ramsey, R. W. Shaw, B. G. Tong, W. B. Whitten, J. P. Young)*

### Advanced Instrumentation

Early this year, we requested and received SEED money support to test a new concept for the measurement of active biomass in attached-growth samples. We had determined that measurement of active biomass in attached multi-species samples might be accomplished by either directly counting active individuals in the cell population, or indirectly by extracting a biochemical marker from the entire sample whose concentration is indicative of only the active cell count. The direct approach represents a digital count of specifically stained cells in a sample, whereas the latter case represents an averaged result dependent upon the types of cellular species present and their given growth stages. If technically feasible, the direct count of active biomass would represent the more accurate analysis procedure. To accomplish this goal, a free suspension of cells must be obtained from the affixed-growth samples. The ability to detach cells from their solid matrix is critical to the development of a direct measurement technique. Several detachment procedures were investigated under this proposal. Cell detachment efficiencies were determined by selectively staining the attached cell sample, performing the detachment procedure, collecting the freed cells and, finally determining the relative concentration of the stain in the freed and remaining attached-cell populations. Submersion of a sample in an ultrasonic water bath for 10 minutes produced the greatest number of freed cells. These studies showed that cellular detachment would not be an obstacle that would prevent the direct measurement of active cell biomass in affixed growth samples.

The efforts of the investigation were then directed towards developing automated instrumentation to provide this analysis. We developed a new instrumentation concept that is an adaptation of the Coulter principle, in which cells are suspended in an electrolyte and counted and sized on the basis of electrical current interruption as cells pass through a restrictive orifice. Fine optical fibers are positioned on either side of the Coulter cell aperture to detect light absorbance or emission from preferentially stained active cells. A coincidence in both the electrical and optical signal triggers data accumulation of the Coulter signal. The resultant data will provide cell size discrimination and count of <u>only</u> the stained active cell population in a given sample. This instrumentation can automatically and rapidly count statistically large cell populations repeatedly in a given sample. The optical signal discriminates against unstained, opaque detritus and can be used in a number of optical alignments to permit turbidometric, nephelometric, absorbance and fluorescent measurement techniques. It was the goal of this initial investigation to provide a proof of principle demonstration of the instrument and then to seek funding for its further development. This goal has been met, and we are looking into funding for further work.

The development of remote analytical instrumentation continues to make excellent progress. The remote titrator was installed in the Transuranium Analytical Laboratory (TRU) in February 1984. Prior to the installation, an operating manual for the system was prepared and training sessions were held with the analytical technicians. Since February the system has performed several thousand titrations in support of TRU operations. In general, the system has proved to be quite reliable; the only significant mechanical or electrical problem was ultimately traced to a failure in an in-cell bulk head electrical connector. Routine use of the instrument uncovered an occasional fatal software error associated with the volume predictor algorithm. This algorithm used a quadratic function as the predictor equation, and due to noise on the signal an attempted division by zero would occasionally occur. A revised predictor algorithm based upon a second order Taylor series was developed to eliminate this problem and an unexpected benefit was a much smoother exit from the endpoint region of the titration curve.

Work on a linear titration system is nearing completion. This system differs from the potentiometric titrator only in the control program and the in-cell transducer electronics. This system will be used for the determination of plutonium in dissolver solutions. Algorithms to automatically sense and calculate the endpoint were developed.

Testing with theoretically calculated titration curves has been completed. The precision of the projected analysis improves as the ratio of titrant to analyte concentration increases; at a ratio of ten, and in the absence of any noise, the precision is 0.1%. Addition of any noise rapidly decreases the precision. A negative bias of 0.1% is projected for all titration conditions. The complete system is undergoing laboratory evaluation with iron serving as a surrogate for the plutonium.

Construction of the remote hardware for two additional titration systems was coordinated; one of these units will serve as the backup system for the unit installed at TRU, and the other will be installed in the High Radiation Level Analytical Laboratory. Calibration and final check-out of a remote pipettor purchased by the Canadian Atomic Energy Commission was completed.

The hot-cell spectrophotometry project has been modified significantly because a commercial system, which uses fiber optic cables to permit remote location of the sample measurement cell, became available during the year. A unit has been procured and will be evaluated for possible use in a hot-cell.

A cooperative program with the Energy Division has been undertaken to develop an in-line analytical sensor to measure the concentration of absorber and refrigerant in experimental commercial heat pumps. The prototype instrument makes use of the relationship between differential refractive index and the loss of transmitted light from an optical fiber. A single compartment, operating heat pump has been constructed and fitted with fiber optic sensors to develop the mathematical relationships and necessary software. When development is complete, it should be possible to measure dynamically the concentration of $H_2O$-LiB, fluorocompounds, or $H_2O$-$NH_3$ in an operating heat pump. This capability will make it possible to design optimum systems to reduce cost and space requirements.

A study of the spectral behavior of the uranium-carbitol-nitric acid complex has been completed and a report written (ORNL/CF-84/283).

The neutron poison monitor has been modified to determine gadolinium in nitric acid in the range of 0.5 g/L to 16 g/L for installation in the integrated equipment test (IET) of the Consolidated Fuel Reprocessing Project (CFRP). The device was also evaluated for stability, precision and electronic interference rejection. A complete test report is now in preparation.

We continue our cooperative program with the Department of Chemistry, the University of Tennessee, for development and application of thin layer spectroelectrochemical techniques to molten salts. The application of an optical multipass technique to enhance the optical signal in these experiments was successfully completed. The optical system consists of two mirrors, placed external to the thin layer electrochemical cell but inside the furnace, which redirect the optical beam several times through the cell. The most significant advantage of the use of external optics is the ease with which one can vary the optical gain of the experiment. In the study of redox systems with multiple steps, especially those involving species with greatly different absorptivities, this technique allows the study of more than one electrochemical step without a change in solute concentration. With molten salt systems the availability of this capability will save a significant amount of time required for melt preparation.

Studies of the first oxidation wave of sulfur in $AlCl_3$-NaCl (63:37 mole%) and of the second reduction wave of $NbCl_5$ in $AlCl_3$-NaCl (65:35 mole %) were completed to demonstrate the capabilities of the technique with molten salt systems. Because of the improved optical signal with the multipass system, the mechanism for the oxidation of sulfur was reinterpreted in terms of $S_8^+$, $S_4^+$, and $S_{12}^{2+}$ only. The product of the Nb reduction was proposed to be $Nb_3^{8+}$; the visible spectrum of this metal cluster was observed. These results were presented at the 188th National ACS Meeting, August, 1984, and received national press coverage in *Chemical & Engineering News* September 10, 1984). This work has been submitted for publication in *Analytical Chemistry*.

A Zymark laboratory robot system is being evaluated for use in the service operations of the division. Several sub-components including the robot arm, control console, dispensing station, and balance have been installed and tested for proper operation. As part of the installation process, we have made a study of the robot programming language and have developed diagnostic programs to test the various system components for proper operation.

A periodic "crash" of the system was traced to a defective processor chip in the console CPU. A new unit was installed along with a completely new operating system (in ROM). This new firmware activates several new features in the robot and extends programming flexibility.

Our first experimental effort was to determine the accuracy and precision of the robot response to program commands. We found that the positional accuracy of the arm is well within the programming resolution. Balance accuracy and precision are essentially those of the balance itself while pipetting and liquid delivery operations are more complicated functions that will probably require calibration under operating conditions.

The General and Environmental Analysis Section requested that we investigate the applicability of the robot to several sample preparation procedures that are in routine use. For the initial demonstration, we selected a liquid scintillation sample preparation procedure. For this work, several pieces of hardware were designed and fabricated to meet special demands of the procedure. The test set-up was run and and a few minor problems were corrected in the program. The robot is now available to prepare these samples on a routine basis.

We have started a second project to develop a procedure to prepare fluorometric uranium samples.

*(D. A. Bostick, L. N. Klatt, H. H. Ross, J. E. Strain, C. S. Alley)*

## Special Projects

Members of the group are frequently asked to lend their expertise to special tasks in other groups or sections. The previously noted robotics work is an instrumentation example. However, we also provide assistance on the solution of "chemical problems". For example in the production of high-purity acids by distillation, the greatest limitation to product purity—aside from contamination by vessel walls—is the carryover of the aerosol resulting from the breaking of bubbles in the distillation pot. It would be highly desirable to eliminate this carry-over while maintaining a high throughput. An aerosol trap has been devised to be placed in the distillation column directly over the distillation pot. The trap operates by constricting the stream of vapor and aerosol and causing the stream to impinge on a liquid surface; the aerosol particles are captured by the liquid surface and the vapor continues through the still. In tests, 500 ml of a solution containing 1 mg Li/ml was placed in the pot, and the still (without the aerosol trap) operated at its maximum rate—360 ml/hr. The distillate showed 2.6 ppm Li; i.e., the distillate was 2.6 x 100/1000 = 0.26% aerosol. With the aerosol trap in place, and the still operated @ 90 cc/hr, the distillate contained $<$0.02 ppm Li; i.e., its aerosol content was $<$0.002%.

For environmental samples the arsenic and selenium values obtained by hydride evolution--atomic absorption began to show poor sensitivity and reproducibility on solutions which had been fumed with $HClO_4$ and $K_2Cr_2O_7$. Investigation showed that the poor reproducibility was caused by internal gas leaks in the Perkin-Elmer 503-MHS 20 instrument, causing the purging gas pressure to oppose the pressure transferring reagent into the reaction flask. The two pressures were adjusted to eliminate this effect, improving both the reproducibility and the accuracy of the arsenic analysis. This adjustment also improved the reproducibility of the selenium determinations, but not their accuracy; the selenium results ranged from 0 to $\sim$80% of the proper values. Selenium is present as $Se^{+6}$ in the processed sample solutions. The $Se^{+6}$ must be reduced to $Se^{+4}$ before hydride evolution is possible. The reduction procedure used previously was found to be ineffective on samples received recently. A new procedure was devised which was quite effective and required almost no attention by the operator. The sample (1-10 ml in a 100 ml volumetric flask) was treated with 35 ml of concentrated HCl. This solution was allowed to stand overnight and then diluted to the mark. Reduction of the selenium to $Se^{+4}$ was complete; values obtained with EPA standards were accurate and precise to within 3% relative.

A request to determine aluminum in $Ni_3Al$ alloy by a precipitation separation led to the discovery that there was only one procedure in the literature that claimed to be able to do this—the homogeneous precipitation of basic aluminum succinate. Although a clean and quantitative aluminum precipitate was obtained when only aluminum and nickel were present, some of the metallic impurities actually present in the alloy were also precipitated. To obtain a correct aluminum figure, the oxide was ignited at 1200°C, weighed, fused with $LiBO_2$ and the melt dissolved. Impurities in this solution were determined by optical emission spectroscopy (ICP) and the appropriate corrections made to the weight of the precipitate.

*(C. Feldman)*

## ANALYTICAL PHYSICS

L. D. Hulett, Jr.

### Positron Spectroscopy

The measurement of surface areas of powdered solids is probably the most practical appplication of positron spectroscopy that has been made recently. Venkateswaran* et al. have shown that when positrons are injected in solid powders the degree of positronium formation is proportional to their surface areas. They prepared a universal curve on which a common plot of points could be made for several different materials, e.g., zeolites, carbon, silica gel. We have applied this method to the measurement of surface areas of fume silicas ("cabosils"). We found that a plot of the degree of positronium formation vs surface area generated a curve parellel to the universal curve of Venkateswaran et al., but the points did not fall on it. Our measurements are preliminary and may have systematic errors; at this point we do not consider the notion of a universal curve to be disqualified. Whether or not a universal curve exists, the positron annihilation method of determining surface areas has some distinct attractions. We will use the method to study other powdered solids.

All who work with low energy positrons are handicapped by the low intensities of monoenergetic positron beams. For purposes of scattering from solid surfaces we have managed to partially overcome this problem by improving spectrometer efficiency. We have returned to the use of the spherical sector spectrometer, with the installation of a microchannel plate detector in the focal plane. Instead of measuring the spectrum by increments of one channel at a time, we now record the entire spectrum over energy spans of 50 - 75 eV. Data collection rates have been improved by factors of 100 - 1000. The positron source and scattering specimen are placed inside a large bell jar coupled to the spherical sector spectrometer at the entrance slit. This eliminates the need to transport the scattered positrons through lenses, for which transmission efficiency is very poor. The large volume of the bell jar allows the source and specimen to be rotated for scattering at different angles, up to about 30°.

Positron and electron energy loss measurements have been made on surfaces of annealed tungsten, molybdenum and copper with the new system. The tungsten energy loss spectra for both positrons and electrons appear the same as our first published results for this material. Similar spectra are also obtained for molybdenum which would not be surprising because it has 4p electrons in the same energy range as the 4f and 5p electrons of tungsten. However, copper which has no electrons in this energy range also shows similar energy loss spectra. This casts doubt on the postulate that the positron energy loss is due to core electron excitation. It is planned to look at a silicon surface which according to our earlier published results gives entirely different energy loss spectra. A freshly deposited gold surface will also be investigated. This more inert surface may shed some light on the possibility that some surface interaction with the atmosphere has occurred on the tungsten, molybdenum and copper surfaces.

Attempts are being made to fabricate a more intense source for the generation of slow positrons. About 80 mCi of $^{68}Ge$ has been electrodeposited inside a platinum annulus, 4 mm inside diameter, 2 mm high. Electrodeposition

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*K. Venkateswaran, Y. C. Jean, K. L. Cheng, *J. Phys. Chem.* <u>88</u>, 2465 (1984) was done using a mixed solution of copper, germanium and cyanide. Deposition time was approximately 40 hrs. A Cu-Ge alloy was formed. Over 90 percent of the $^{68}$Ge was recovered from the solution. We found, however, that the deposit was not completely coherent. Microscopic particles, having activities as much as 100 mr/hr at contact became dislodged. This problem prevented our testing of the source for its efficiency in the generation of slow positrons. We have transported the source back into the hot cell and overplated it with 6 μm of pure copper. We have hopes that this will contain the loose activity so that the source will not be hazardous for use.

*(J. M. Dale, L. D. Hulett, T. M. Rosseel)*

## Heavy-Ion Induced X-ray Satellite Emission

As part of our effort to confirm the sensitivity of heavy-ion induced X-ray satellite emission (HIXSE) to intermediate Z elements and to expand this method to new regions of the periodic table, we have examined the effect of the chemical environment on the Zr L and the Au and Pb M X-ray satellite lines.

High resolution L X-ray satellite spectra produced by 0.7 MeV/u and 1.0 MeV/u Cl ion bombardment of a series of Zr alloys and compounds were obtained at the EN-tandem accelerator (ENTA). As expected, the normalized emission spectra display variations in the intensity profiles of the $L\alpha^nM^m$ and $L\beta^nM^m$ satellites for different targets. Dirac-Fock calculations revealed that the observed fluorescence corresponds mainly to the $L\alpha\ (2p_{3/2} \rightarrow 3d_{3/2,5/2})$ and $L\beta_1\ (2p_{1/2} \rightarrow 3d_{3/2})$ hypersatellite lines ($L^2M^m$ transitions). Examination of the 1.0 MeV/u difference spectra generated using the pure element as a subtrahend corroborate the trends observed in the molybdenum data. For example, the $L_\alpha^2$ region of difference spectra of Zr compounds becomes more negative as the bulk valence electron density (Dv) increases. The Zr-Ti alloy spectra, like the Mo-Ti spectra, exhibit a different trend for the $L_\beta^2$ region. The smaller effect observed for the Zr-Ti alloys was predicted by the smaller relative change in Dv. These results provide additional evidence that HIXSE can distinguish between different types of chemical bonding. It also confirms the sensitivity of the L X-ray satellites to the chemical environment of intermediate Z elements.

Examination of the difference spectra at a projectile energy of 0.7 MeV/u produced unexpected results. Although all of the alloys and some of the compounds exhibited little change (with energy) in their difference spectra, several of the compounds displayed sharp differences. This suggests that certain refilling routes may have been altered due to the change in the degree of the M-shell ionization. Future studies of Nb alloys and compounds at 1.0 MeV/u and 0.7 MeV/u Cl ion excitation and Mo alloys and compounds at 0.7 MeV/u may provide insight into this effect.

The Pb and Au M X-ray satellite spectra were also examined at 1.0 MeV/u. The spectra, like those of Zr and Mo, exhibit variations in the satellite intensity profile due to differences in the chemical environment of the target ion. Like the L spectra, the first two bands in the M spectra are composed of overlapping hypersatellites of the M and M transitions. Attempts to extract chemical information from these spectra are being pursued.

*(T. M. Rosseel, J. M. Dale, L. D. Hulett, J. P. Young, H. F. Krause,[1] P. L. Pepmiller,[1] S. Raman,[1] C. R. Vane[1])*

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[1] Physics Division

# 2. MASS AND EMISSION SPECTROMETRY

J. A. Carter

The Mass and Emission Spectrometry Section is engaged in research and development in four broad areas: inorganic mass spectrometry, organic mass spectrometry, inorganic secondary ion mass spectrometry, and organic secondary ion mass spectrometry. Each area provides effective support for various laboratory programs, both by developing needed new analytical procedures and by providing support analyses. The long-range goal of the section is to develop cost-effective techniques to allow extension of mass spectrometric analysis to new areas; this requires research aimed at understanding the fundamental physical processes involved and using the information thus obtained in development of new analytical procedures and instrumentation. To cite examples in organic secondary ion mass spectrometry, two important breakthroughs occurred: 1) Secondary emission of protonated molecular ions ($MH^+$) can be enhanced by the host matrix; and 2) enhanced emission of molecular ions ($M^+$) and fragment ions by Lewis acid matrices. This information, previously lacking, is invaluable in establishing mechanisms of organic secondary ion emission. Another development with considerable importance to DOE facilities is the detection and identification of organic explosives with few false alarms by MS/MS methodologies. Also, research efforts are under way to improve detection limits by secondary ion mass spectrometry coupled with the resonance ionization technique, possibly to a level where one atom for every 10 atoms available for analysis can be detected. Improvements in the resin bead methodology were realized that allow more DOE laboratories to employ the technique.

About 30% of our research and development support is provided by the Division of Chemical Sciences of the Office of Basic Energy Sciences, Department of Energy. This effort, which is very broad in scope, provides the technical base from which current and future mass spectrometric needs of DOE are addressed. Special projects of diverse origin from the International Safeguards Project Office (ISPO) and from the Office of International Security Affairs (OISA) provide substantial support; the rest of our funding comes through several laboratory programs and various Work-for-Others contracts.

## SECONDARY ION MASS SPECTROMETRY

W. H. Christie

In a further expansion of the study of resonance ionization mass spectrometry, we have included sputtering as a technique for getting neutral atoms into the gas phase for subsequent laser ionization. Ion beam sputtering is an attractive process for coupling with pulsed laser ionization because the process of atom formation can be highly controlled. Knowledge of the energy and angular distribution of the sputtered atoms allows one to calculate the geometrical overlap between a sputter "plume" and a laser beam of given cross-section. In addition, when pulsed sputtering is utilized, the temporal overlap between the atomization and ionization can approach 100%.

A surplused ARL ion microprobe was acquired and made operational. An optical port and mirror assembly was added to the sputtering chamber to facilitate introduction of the laser beam into the sputtering region. This instrument, interfaced with a tunable pulsed dye laser, has many advantages for this work. These advantages include a micro-focused primary ion beam of selectable mass, complete sample manipulation and viewing capability and a double-focusing mass spectrometer for separation and detection of the secondary or laser-generated ions.

Significant redesign of the ion extraction optics was required to allow extraction of laser generated ions which are formed at some distance above the sample surface. This redesign work was facilitated by use of the SIMION computer program. Equipotential surfaces and ion trajectories were modeled with this program to produce a lens system that was highly efficient in steering laser generated ions into the secondary ion mass spectrometer.

In our study, targets of uranium and samarium were used as sources of neutral atoms. Primary ions were generated by using a mixed gas of 2 atom % Ar, 34 atom % $N_2$ and 64 atom % $O_2$ in the microprobe ion source. This allowed the sputter targets to be bombarded with operator-selected mass analyzed ion beams of $Ar^+$, $N_2^+$, or $O_2^+$. As expected, it was found that high current density (micro-focused) $Ar^+$ sputtering beams are more effective than reactive beams such as $O_2^+$ and $N_2^+$ in producing a large sputtered neutral population from reactive targets such as metallic uranium and samarium.

We found that the ion bundle produced in a single laser pulse (nominally 1 μs) long) was 3 ± 1 μs wide at the detector following a delay of 23 μs (for samarium) or 30 s (for uranium) due to time-of-flight through the secondary mass spectrometer. The number of resonance ions in each bundle could be counted using a fast oscilloscope to monitor the output of the ion multiplier preamplifier. The largest signals obtained for $^{238}U$ had over 100 ions per pulse while those for $^{152}Sm$ were found to have 40 ions per pulse. In the absence of information about sputter yields for uranium and samarium under $Ar^+$ bombardment, it is not possible to obtain a reliable measure of the number of resonance ions collected per sputtered atom. However, under the conditions which gave >100 $U^+$ ions per pulse, with a primary $Ar^+$ current of 21 nA, the number of $U^+$ ions detected per incident $Ar^+$ was 1 in 1300. This reveals that a factor of $10^2$ improvement is necessary before reaching the goal of 1 ion per 10 sputtered atoms. We feel that improvements in the laser system and ion extraction optics should increase the overall efficiency.

During the past year, we have continued our collaborative work with several researchers in the Solid State Division. Two of these joint studies will be described. In the first study, secondary ion mass spectrometry (SIMS) has been used to determine the concomitant B and F redistribution in annealed single crystal silicon. $BF_2$ implants were used because they are more economical in that the $BF_2$ ion is directly available from the ion source and does not require expensive mass separation in the accelerator. Using molecular ions such as $BF_2^+$ for ion implantation, more damage energy is deposited by the $F^+$ ions over the same range as the $B^+$ ions and, therefore, allows complete amorphization of the near surface region to be achieved. The molecular ions split, in the near surface region, into atomic species having the same velocity as the molecule. This results in low energy ions that create shallow implants in a thin amorphous near surface region. For example, a 50 keV $BF_2^+$ ion breaks into an 11.2 keV $B^+$ and two 19.4 keV $F^+$ ions. Thus a 50 keV $B^+$ ion has approximately five times the projected range compared to a 50 keV $BF_2^+$ ion. The object of the study was to produce thin amorphous layers having good electrical properties and minimum dopant profile broadening during annealing in order to form shallow junctions.

SIMS depth profiling was performed before and after rapid thermal annealing. The as-implanted specimens showed a Gaussian profile with B peaked at around 40 nm. After thermal annealing the B profile spread toward the surface and into deeper regions of the crystal. As the time or temperature of annealing increased, the amount of spreading increased. The average profile broadening was less than 50 nm even after a 6 s, 1100°C thermal annealing pulse. The amount of boron integrated over depth remained constant (±10%) after various annealing treatments.

The as-implanted fluorine distribution was also Gaussian, peaked at 40 nm. After rapid thermal annealing the fluorine peak was observed at a depth of about 60 nm, which corresponds to a known band of dislocation loops. SIMS and transmission electron microscope (TEM) results show that fluorine is gettered by dislocations.

In laser annealed specimens, boron was shown to move deeper into the crystal, while in sharp contrast the fluorine profile does not spread but moves toward the surface.

In the second study SIMS is being used to measure the broadening of ion implanted $^{30}Si$ in natural Si due to thermal diffusion. Existing measurements of the self-diffusivity for intrinsic silicon show Arrhenius behavior from 1400°C down to 1100°C with a prefactor of 10,000 sq cm/s. Workers in the Solid State Division feel that this large prefactor is inconsistent with a vacancy diffusion mechanism. They feel that it suggests the involvement of an interstitial or more complex defect in the diffusion process at these temperatures. A vacancy mechanism would have a much smaller prefactor and a smaller activation energy and would, therefore, be expected to dominate the diffusion process at some lower temperature. To study this effect, Solid State Division personnel have extended the diffusion measurements to substantially lower temperature using silicon specimens prepared by ion implantation with $^{30}Si$.

High dose implants were epitaxially recrystallized at low temperatures. SIMS was invoked as the analytical technique to measure these redistribution profiles because no other sensitive analytical technique can measure ion implanted $^{30}Si$ in silicon. By using SIMS as the analytical technique, much longer annealing times are possible as compared to radio tracer measurements with $^{31}Si$, which were limited by the short half-life of $^{31}Si$. The data are currently being evaluated.

*(D. L. Donohue, D. E. Goeringer, H. S. McKown, R. E. Eby)*

## RESEARCH IN INORGANIC MASS SPECTROMETRY

R. L. Walker and D. H. Smith

Resonance ionization mass spectrometry (RIMS) has been further developed and investigated. A narrow bandwidth laser was used to probe the fine structure underlying the uranium RIMS transition at 591.54 nm. This study extended our understanding of the sources of bias in isotope ratio measurements using RIMS. A continuous wave, argon pumped dye laser was evaluated for RIMS application with the help of J. M. Ramsey. It was found that, with suitable modification, it held promise with regard to precision, but that its sensitivity was too low for practical use.

Isotopic analysis of neodymium was undertaken with RIMS. We found that we could obtain all six isotopic ratios at precision of about ±1% from 40 ng samples. Unfortunately, our efforts to extend the technique to 10 ng samples were not successful. We must be able to analyze samples of this size to apply the technique to reactor burn-up studies. We are currently laser-limited: we need a higher repetition rate and more power to reach the desired analytical level.

A new isotope ratio mass spectrometer, a VG 354E, is on order and scheduled for delivery by the end of 1984. A state-of-the-art instrument, it features five independently locatable collectors; it also has a pulse-counting detection system for analysis of small samples. It will be equipped with an electron impact ionization source for analysis of elements of high volatility and/or high ionization potential.

Because our present techniques for many of these intractable elements (e.g., Hg, Te, Os) are time-consuming and tedious, an investigation of the applicability of a hollow cathode ion source has been initiated. The most attractive features of such a source are vastly reduced sample preparation (only a solution of the element in question is required) and potential sensitivity. We have on hand no mass spectrometer capable of providing both the necessary high pumping speed (the discharge is supported by a flow of argon) and the data reduction software to perform isotope ratio calculations. Preliminary experiments were carried out on the ion optical bench; further studies were done using the MS-25. This latter instrument seems ideal for the intended purpose. We will be developing software for data acquisition in the near future; data reduction will be accomplished using the extensive isotope ratio computer programs already developed.

A time-of-flight mass spectrometer has been acquired to allow investigation of the temporal behavior of various sample systems. In particular, we hope to study the variation in the evolution of various species (especially oxides) from thermal ionization filaments as functions of time, filament pretreatment conditions, etc. We plan to acquire a PC as a data system for this instrument. We also plan to use the instrument to further our understanding of the physical phenomena involved in the RIMS process; we will be able to probe the populations of various states and how they vary with time.

Modifications have been made to the original resin-bead method which extend its usefulness to breeder spent fuels with U/Pu ratios of 2.5 to 4.0. The original method was designed primarily for spent fuels that had U/Pu ratios of about 100. This approach is made especially attractive because the amount of plutonium on the bead is increased and is high enough to allow the plutonium isotopic analysis to be made at a lower filament temperature thus achieving near complete separation of $^{238}Pu$-$^{238}U$ isobars during plutonium data taking. A ratio of 10:1 $^{239}Pu/^{238}U$ during uranium data taking now seems acceptable for this type sample, at least an order of magnitude more plutonium than allowed by the old method.

*(D. L. Donohue, M. P. May, E. H. McBay, C. A. Pritchard, E. G. Miller, H. C. Smith, M. M. Smaker, L. Quinn)*

## ORGANIC MASS SPECTROMETRY

G. L. Glish

Work in organic mass spectrometry has continued to emphasize research in the area of mass spectrometry/mass spectrometry (MS/MS). A major effort has begun in developing an MS/MS system for the detection of explosive vapors. Initial feasibility studies using the ZAB mass spectrometer have been done. Prior to acquiring MS/MS spectra on the ZAB instrument, it was necessary to modify it for organic MS/MS research. This involved installing a combination chemical ionization/electron ionization source, and a collision region in the second field-free region of the instrument. The collision region was modeled after the one developed and installed on the ORNL triple sector instrument several years ago. Over one hundred MS/MS spectra have been acquired of various explosive compounds under electron ionization, positive chemical ionization and negative chemical ionization. Design of a dedicated MS/MS system to detect these compounds is now under way with the recently developed tandem quadrupole/time-of-flight instrument being used as the basis for the instrument.

In addition to development of the tandem quadrupole/time-of-flight (QT) instrument as an explosive detector, work has been done using it to investigate the effect of internal energy on collision activated dissociation (CAD) MS/MS spectra at low collision energies. Thermal desorption has been used to create ions with different amounts of internal energy by varying the ionization temperature. It has been found that there is a definite effect on the appearance of the CAD MS/MS spectra with ion internal energy. Further work is planned to see if this experimental procedure can be used to determine internal energy variations in ions formed by different methods.

A project has been started to interface a quadrupole to the front end of the MS50 to allow a variety of different MS/MS experiments. Initial experiments have been done using the quadrupole and lens system from the QT instrument with the thermal desorption probe. Work has also been started on the ion cyclotron resonance (ICR) instrument obtained from Y-12. Work to date has involved improvement of the electronic circuitry in order to get suitable performance from the instrument. Cell modifications are currently under way so that experiments using low energy positrons can be performed.

*(G. L. Glish, D. E. Goeringer, S. A. McLuckey, E. H. McBay)*

## ORGANIC SECONDARY ION MASS SPECTROMETRY

P. J. Todd

Two breakthroughs have occurred in our research into this area. First we have demonstrated that matrices can be employed to enhance secondary emission of protonated molecular ions ($MH^+$) from volatile organic compounds adsorbed onto or into a matrix. This permits a study of the relationship between organic secondary ion current and analyte concentration, since the latter follows either a Langmuir isotherm or Raoult's Law. Second, we have been able to demonstrate enhanced emission of secondary molecular ions ($M^+$) and their fragment ions by employing Lewis acid matrices. Such molecular ions can also be generated by electron impact ionization for which extensive data and theory exist on the energetics of formation and fragmentation. Application of this information to molecular secondary ionization yields insight into the characteristic internal energy distribution of secondary molecular ions. This information, which has been lacking to date, is invaluable in discerning the mechanism(s) of organic secondary ion emission.

Classically, the study of chemical reactions has involved the correlation of reactant properties with product properties. Imagine trying to elucidate a reaction mechanism in which no correlation could be made between the concentration of reactants and the concentration of products. This situation summarizes SIMS in general and organic SIMS in particular, where reactants are the condensed phase, analyte matrix, and primary ion and the products are secondary ions (and undetected neutrals). In organic SIMS, liquid matrices are employed to enhance secondary emission of protonated molecular ions from some involatile analyte dissolved in the matrix. Since the matrix evaporates and is sputtered away, the concentrations of both analyte and matrix are time dependent. We developed a method to introduce volatile analytes on the matrix surface so that under the condition of the experiment, the system appears as a vapor in equilibrium with a liquid. No matter how rapidly the matrix evaporates, in the absence of any introduced analyte, the standard state of unit mole fraction is maintained. Introduction of an analyte gas at an effective pressure, P, of about $10^{-7}$ torr implies a mole fraction of the analyte in the matrix of $10^{-7}$ at equilibrium if the vapor pressure of the pure analyte $P_0$ is 1 torr. This is a consequence of Raoult's Law. Our data indicate that such equilibrium is obtained about one minute after analyte introduction is commenced: for a series of ten volatile amines having vapor pressures of 0.01 to 30 torr at room temperature, a plot of secondary ion intensity vs the ratio of $P/P_0$ shows that all points fall on a single curve, independent of the amine's identity. This curve yields the functional dependence of secondary protonated molecular ion intensity on analyte mole fraction. This, of course, is what we wished to find in order to study the chemistry of molecular secondary ion emission.

To investigate the energetics of organic secondary emission, it is necessary to generate ions whose energy of formation and fragmentation are well-known. Molecular ions ($M^+$) are such species, and have been characterized by electron impact (EI) ionization studies. Enhanced secondary ion emission of such species was unknown, but it was known that secondary emission of protonated molecular ions ($MH^+$) from protic matrices was dependent upon the existence of the solvated ion in the condensed phase matrix. By analogy, we felt that $M^+$ secondary ions could be generated from aprotic matrices in which solvated radical cations were known to exist. Certain arene/Lewis acids systems meet this criterion and have the added advantage that the chemistry of such systems is well-defined. Lewis acids such as $SbCl_3$ and $AlCl_3$ were employed thus as matrices and, in fact, we were able to demonstrate for the first time matrix enhanced secondary molecular ion emission. We then compared the relative abundance of fragment ions from SIMS and 70 eV EI spectra and found they were, surprisingly, identical. Because EI involves a vertical (i.e., Franck-Condon) ionization, these results implied that the origin of the secondary fragment ions was from vertical ionization of analyte molecules. The only sensible explanation invokes a charge-transfer mechanism between matrix ions (e.g., $AlCl_3^+$) and neutral analyte molecules which just happen to be sputtered in the same event as the matrix ion. This process is independent of the chemistry of the matrix and analyte, and indeed analyte fragment ions were detected even when the analyte molecular ion was not detected and known not to exist as an ion in the Lewis acid. However, consider the effect of an analyte molecular ion being ejected in the company of matrix neutrals or ions. No charge exchange occurs because the ionization energy of the arenes is some 4 eV less than the matrix neutral. Ejection of secondary molecular ions would reasonably be enhanced if the molecular ion existed on the matrix surface as an ion rather than as a neutral prior to the sputtering event. Such $M^+$ emission would thus be strongly dependent upon the arene-Lewis acid chemistry, which is, in fact, what is observed. The ability of the technique to distinguish between the two ionization processes is astonishing and unprecedented.

Sputtering phenomena have been known for some one hundred years or so, and there is still no unified theory concerning the phenomena. However, our approach to the study contributes information which has hitherto been lacking, and at present unavailable from any other source. Results from these experiments provide a unique opportunity to develop at least the theory of organic secondary ion emission.

*(P. J. Todd, C. P. Leibman, W. H. Christie)*

# 3. RADIOACTIVE MATERIALS ANALYSIS

**W. R. Laing**

The Radioactive Materials Analysis Section is composed of three groups: Radioactive Materials Analytical Laboratory, Transuranium Analytical Laboratory and Programs and Projects. A satellite laboratory is also operated within the Consolidated Fuel Reprocessing Facility. These groups provide support for many Laboratory programs, primarily those in the Chemical Technology, Fuel Recycle, Metals and Ceramics, and Operations Divisions. They also perform work for other federal and nonfederal agencies.

Development is an inherent activity and includes work on X-ray fluorescence, porosity and surface area measurements, mercury purification, testing of coatings for the nuclear industry, and the division's computer-based Data Management and Sample Transaction System.

## PROGRAMS AND PROJECTS

D. A. Costanzo

### X-Ray Fluorescence Development

Quantitative X-ray fluorescence (XRF) analysis for uranium and plutonium in highly radioactive solutions presents several unique problems. These problems include excitation of the uranium and plutonium by passive radiation in the sample (gamma, X-ray, alpha, and beta activity), the spontaneous emission of X-rays which results from internal conversion following the decay of radioactive isotopes (e.g., uranium L X-rays following the alpha decay of plutonium isotopes) and coping with the high degree of scattered radiation from the sample. The passive excitation and interference from internal conversion can be handled by appropriate analytical techniques; however, conventional XRF spectrometer designs cannot tolerate a very high degree of passive radiation from the sample. Therefore, the goal of this project is to provide an XRF system to monitor uranium and plutonium in highly radioactive feed solutions to a nuclear fuel reprocessing plant.

Investigations by C. J. Sparks, Jr. (ORNL Metals and Ceramics Division) led to the design of a wavelength dispersive XRF system which can be applied to the determination of uranium and plutonium (or most other elements) in highly gamma-active backgrounds. Due to the required operation of the system in a remote and inaccessible environment, a basic feature incorporated into the ORNL XRF spectrometer design is mechanical simplicity. The XRF system employs pyrolytic graphite for the X-ray optics which has the advantage of excellent radiation resistance and high diffraction efficiency (40% of incident power). In addition, the graphite diffraction crystal provides simultaneous diffraction of the entire energy region from $PuL_{\alpha}$ to $UL_{\alpha}$ with sufficient resolution (30 eV FWHM at 14 keV) to permit the use of a fixed-single crystal spectrometer. Therefore, a final *operational* system would have *no* moving parts after the initial alignment which simplifies instrument maintenance and enhances reliability; the constant geometry should also result in improved accuracy and precision.

The performance of the high-gamma XRF system is strongly dependent upon the focal spot dimensions (primarily the vertical dimension) obtained with the barrel monochromator which is used to focus exciting radiation on the sample. The original barrel design with singly-bent graphite (Johann optics) is limited to a minimum vertical dimension of approximately 2.5 mm, and could be improved significantly by using doubly-bent graphite. However, the expense of doubly-bent graphite would result in the barrel monochromator costing in excess of $50 K, therefore, lithium fluoride was considered as an alternative for barrel fabrication. Doubly-bent and ground (Johansson optics) lithium fluoride, LiF(200), would theoretically provide a vertical dimension of approximately 0.4 mm. This reduction in the focal area would yield at least a 10-20 fold increase in sensitivity with a 30 eV FWHM resolution.

Although the cost for a LiF(200) barrel monochromator is significantly less than for a graphite barrel, the lithium fluoride is degraded by extended exposure to high radiation and would require replacement periodically. (We predict annual replacement, but this depends upon accumulated exposure.) Graphite should last indefinitely in the high radiation environment and would be a one time expense.

Initial attempts to bend lithium fluoride to a small radius (less than 2.5 cm) were unsuccessful. However, with practice and refinement of technique, we have produced a 72 degree sector of an elliptical surface of rotation with central radii of 322.6 mm (12.7 in.) and 9.1 mm (0.36 in.) for the major and minor axes, respectively. The doubly-bent and ground LiF(200) crystal is approximately 2.5 cm in length. The goal is to bend a 72 degree sector that is 10 cm in length.

The cost for a lithium fluoride barrel turned out to be more than we initially expected because of design and fabrication of equipment to bend the crystal. Therefore, the performance factors for the barrel were re-evaluated with closer detail on absolute differences between doubly-bent and ground LiF(200) (Johannson optics) and doubly-bent graphite (Johann optics). This re-evaluation indicated that the lithium fluoride barrel would only perform 8 times better (translates to shorter analysis time) than the graphite barrel. The importance to this 8-fold difference in performance (sensitivity) is based upon the ratio of plutonium/uranium in the dissolver solutions for which the XRF system was designed. For ratios in excess of 0.1 (i.e., FBR fuel Pu/U approximately 0.25) the 8 X difference is not that critical; however, for ratios much less than 0.1 (LWR fuel Pu/U is less than 0.01) the difference in analysis time (15 min vs. 120 min for LWR solutions) may present problems in a reprocessing plant. At this time both lithium fluoride and graphite are viable options for the barrel monochromator.

Special arrangements are being discussed with the Carbon Products Division of Union Carbide for custom made monochromator grade (ZYA) graphite in an attempt to reduce the cost of a graphite barrel monochromator. Our proposal is that we provide the carving tools, which are graphite forms, to be used for the high temperature and high pressure press technique employed to produce the ZYA graphite. The graphite form we supply would include 4-5 sectors of an ellipsoidal surface on a single form. Therefore, all sectors required for a barrel monochromator could be produced in a single press.

During this reporting period capital funds (approx. $115 K) for the purchase of equipment to fabricate a prototype XRF system have been received. Equipment which has been purchased includes the computer system, multichannel analyzer, and high resolution position sensitive proportional counter. Specifications for a constant-potential 3 kW X-ray generator have been prepared and sent to vendors for a bid.

Most software for the acquisition, peak processing, and quantitation of XRF data has been completed. A special device handler program has been written which provides a communication link with the TN-7200 MCA on a serial line at speeds up to 4800 baud. The device handler, along with an application program, support bi-directional transfer of spectral data and transfer of control commands under either RT-11 or TSX-Plus operating systems. Integration of the software into a single menu driven application package remains to be completed.

Work through the completion of the current fiscal year will be directed towards the set-up, testing, and integration of this equipment and material into the prototype XRF system. Once the new X-ray generator is received, system performance will be re-evaluated. At that time the choice between lithium fluoride and graphite for a barrel monochromator will be made.

*(J. M. Keller)*

## Porosimetry of Particles

Particle analyses of HTGR graphite, sol gels, and fly ash have been made using the BET gas adsorption apparatus. Surface areas and pore size distributions were determined by nitrogen adsorption at 77°K. Complete adsorption-desorption isotherms were plotted. From these data other plots were prepared for the description of the surfaces and pore structures of the particles. The plots included BET diagrams, "T-plots", surface area distributions and pore volume distributions from the adsorption and desorption branches of their hysteresis loops. Calculations were made by the microprocessor to determine the "C" constant in the BET equation, the specific surface area of the particles, and the volume of gas adsorbed. The magnitude of "C" is a relative measure of heat of adsorption as compared to the heat of condensation of the gas on the adsorbent. The profiles of the hysteresis loops in the adsorption-desorption isotherms and "T-plots" have been used to describe the types of pore structures in the particles.

*(D. A. Lee)*

## Mercury Purification Facility

Analytical Chemistry Division has contracts with others to purify mercury metal for reuse. A new and improved facility has been built to process mercury more efficiently. The facility includes a closed system with counter-current acid extraction and water washing columns and a continuous flow vacuum drying column. The facility is isolated in a floor to ceiling hood to minimize exposure to mercury vapors. Impurities in the mercury are extracted with a 1N $HNO_3$ - 1$\underline{N}$ HF - 0.05 $\underline{M}$ $Hg(NO_3)_2$ solution. The product has less than 5 ppm total impurities. The traces of impurities remaining in the product are noble metals - principally Ag°.

A contract to supply Bendix Corporation with 4180 kg of purified mercury was completed. A similar contract has been made for next year. Additional mercury has been purified for Y-12 and other salvage operations. With the present facility it is possible to process 400 kg per day.

*(D. A. Lee)*

### Impurities in HTGR Graphite

Impurities in core and structural graphite for HTGR are known to cause catalytic oxidation of graphite by steam. To facilitate a program for the manufacture of structurally sound graphite for the HTGR, several graphite coupons selected from strategic positions in a large graphite billet have been quietly ignited in air at 820°C on platinum sheets. The patterns remaining in the ash residues have been photographed and selected areas of the ash have been analyzed by electron beam excited X-rays (SEM-EDX) and inductively coupled plasma (ICP). These analyses were used to help determine the source and mechanism of formation of the impurity inclusions so that a proposal may be made to prepare a superior grade of graphite for the HTGR.

*(D. A. Lee)*

### Radiochemical Analysis

Radiochemical analysis in the Radioactive Materials Analysis Section is concentrated in two areas, each with a different support function. The TRU (Bldg. 7920) laboratory is staffed 24 hours a day and provides data for process control of the Chemical Technology Divison's fuel dissolution studies and transuranium isotopes production. In addition, Operations Division relies heavily on the 24 hour service to aid in monitoring their waste processing facilities. Isotope product certification and general gamma-ray spectrometry analysis round out the TRU activities. The radiochemical laboratory at Bldg. 3019 offers special services in low energy beta analysis ($^3$H, $^{14}$C, $^{35}$S, $^{32}$P) and wet chemistry.

At TRU a concentrated effort has been made to improve the quality of the data being provided. With respect to gamma-ray spectrometry, the detector efficiencies were recalibrated; a new sample holder was constructed to eliminate geometry variances; a new, documented operating software was purchased for the ND6620; an extensive training program for analysts, shift supervisors, and control operators has increased confidence and independence and reduced errors; the magnetic tape unit has been re-installed for better data storage and documentation. All of the counting equipment at TRU was evaluated for accuracy, reliability and suitability. The reliability and ruggedness of the gross alpha counters were improved by converting to end window gas-flow proportional counters for high activity work. Direct reference to primary NBS standards was initiated. New procedures for instrumentation check out and prompt feedback on quality control results combined with the above actions has resulted in a measurable improvement in the quality of work being performed at TRU.

Also during the past year participation in the division's Quality Assurance program was begun, radiochemical standards were updated with NBS materials, and a review of basic concepts and calculations was conducted. These activities resulted in improved service to the Operations Division on a bio-engineering program for which we served as the certifying laboratory for the radioactive labeling material.

*(L. M. Rossberry)*

## RADIOACTIVE MATERIALS ANALYTICAL LABORATORY

D. A. Costanzo

The Radioactive Materials Analytical Laboratories (RMAL) provide support to various related Laboratory programs. The Consolidated Edison Uranium Solidification Program, Consolidated Fuel Reprocessing Program, hydrofracture waste disposal, isotopes production, Three Mile Island clean up, U/Pu gel microsphere production, and environmental monitoring are representative activities. Many Laboratory divisions are supported, including: Chemical Technology, Fuel Recycle, Physics, Environmental and Occupational Safety, Operations, Quality Assurance and Inspection, Health, and Plant and Equipment. Analysis of intermediate and high level radioactive samples that require glove box and/or hot cell operations are the primary services provided by the group; in addition, X-ray fluorescence, testing of coatings for application in nuclear facilities, and uranium ore concentrate analysis are also performed.

During this reporting period, approximately 2,000 samples were processed and 7,000 determinations were performed. A large number of highly radioactive samples were prepared for analysis by other sections within the division for isotopic analysis, mass spectrometry, gamma spectrometry, neutron activation and gas and ion chromatography. A variety of analytical techniques were employed at RMAL including:

potentiometric titrimetry, coulometry, X-ray fluorescence, alpha pulse height analysis, beta liquid scintillation, spectrophotometry, free acid and infra-red(IR) analysis for carbon.

Radiation tolerance, decontamination, and design basis accident (DBA) testing of coatings for application within nuclear facilities is provided to coating manufacturers, utilities, architect-engineering firms and testing agencies. Testing has been performed for 12 clients this year. Radiation tolerance testing is performed at the High Flux Isotope Reactor using the intense radiation emitted by the spent fuel elements. In the decontamination test, a mixture of gamma-emitting nuclides is applied to the surface of stainless steel panels or coated cement blocks and removed with various decontaminating solutions. In the DBA test, the coated specimens are exposed to an emergency coolant spray solution at elevated temperature. These tests conform to ASTM specifications.

For a number of years New Brunswick Laboratory has provided analytical chemistry services to the uranium mining and processing industry in the form of measurements of the uranium content of ore concentrates. Current contracts between shipper and receiver require that a certain percentage of samples be analyzed independently as well as those samples where shipper and receiver differ by more than 0.1 wt. %. The Department of Energy has determined that the New Brunswick Laboratory should not do this work since the work is not safeguards related. Thus, in mid-1984 the Radioactive Materials Analytical Laboratory was selected by two commercial uranium suppliers to provide the uranium referee analyses. The total number of samples expected to be analyzed is about 250 per year. The Davies-Gray potentiomentric method is being used. The relative standard deviation at the 95% confidence interval has been demonstrated to be 0.05% for samples containing 65-80 wt. % uranium.

Construction of an addition to the west end of Building 2026 was started in mid-May 1983 and was completed in August 1984. Occupancy of the addition should occur in January 1985 after several design and construction faults have been corrected. The addition contains a radiochemistry counting room ( 600 sq ft) on the first floor and three chemistry laboratories and an office on the second floor ( 2000 sq ft). Initial plans were to use the laboratories for radioactive operations in support of several proposed reactor fuel fabrication and reprocessing programs and to relieve the crowded conditions that have existed for the past several years in the radiochemistry and glove box laboratories in Building 2026. The radiochemistry operations in Building 3019B will be moved and consolidated with the operations in Building 2026. We are considering establishing a hazardous materials analytical laboratory in Building 2026.

*(R. D. Brooksbank and R. E. Jones)*

## Fuel Recycle Division Analytical Laboratory

The satellite service laboratory located in Building 7603 performed 2,921 analyses in support of process and engineering studies carried out by the Fuel Recycle Division (FRD). This was a 15% increase over the number of analyses performed the previous year. Types of chemical measurements included free acid, silver, chloride and carbonate by volumetry; uranium by spectrophotometry and volumetry; and $NO_x$ by spectrophotometry. Physical measurements included density, viscosity, foam break time (FBT), conductivity, and disengaging time. In addition, a number of special studies was carried out for six different groups within the FRD in support of the Integrated Equipment Test (IET).

A study was conducted to determine the uranium and nitric acid distribution coefficients in the IET solvent extraction system. The organic phase was 30% tributyl phosphate (TBP) - 70% normal paraffin hydrocarbons (NPH). Aqueous phases contained uranium varying in concentration from 50 to 250 mg/ml and nitric acid varying from 0.5$\underline{M}$ to 3.0$\underline{M}$. Extractions were performed using various organic to aqueous ratios and both phases were analyzed for uranium and acid before and after extraction. Distribution coefficients to be used in the design of a reprocessing plant were calculated using the resulting data.

Another study consisted of measuring the temperature, density, and conductivity of a series of IET samples and using a previously calculated model to predict the uranium and free acid concentrations.

The formation of foam in the solvent extraction system of IET was a continuing problem and several studies were undertaken to determine the effects of a number of variables upon the problem. Foam break time, defined as the time required for the foam formed when a sample was vigorously agitated for 30 seconds to break up and disappear, was measured and used as a standard of comparison. Variables studied were: 1) The presence of 10-1000 ppm of monobutyl phosphate and dibutyl phosphate in the TBP used to prepare the IET solvent. 2) The addition of a number of commercial anti-foaming agents to the IET solvent. 3) Sulfuric acid washes of various time periods of the NPH used to prepare the IET solvent. 4) Carbonate washes of the IET solvent. It was found that the phosphates and the commercial antifoaming agents decreased break times. Carbonate increased break time and sulfuric acid had no effect.

*(F. L. Layton)*

## TRANSURANIUM ANALYTICAL LABORATORY

J. L. Botts

The Transuranium Analytical Laboratory (TRU) continued to provide support to the Transuranium Production Program and the Solvent Extraction Test Facility (SETF) within the Chemical Technology Division and to the Operations Division in monitoring radioactive waste effluents. In support of these Laboratory programs approximately 36,000 determinations were made during this period.

Much effort this year was directed toward an orderly change of most of the personnel in the group and an update of the capabilities of the laboratory. Because of retirements and transfers, 70% of the lab personnel have been replaced by younger, inexperienced people. This change has prompted a large training-orientation program designed to fully acquaint and educate the personnel in the laboratory techniques and practices on handling and analysis of highly radioactive materials.

The updating of the instrumentation in TRU is still in progress. A computer controlled remote pipettor and titrator have been installed in the shielded hot cell. The pipettor was used to take aliquots of highly radioactive samples for dilutions and direct determinations. The titrator was used for the in-cell potentiometric determination of free acid in the TRU and SETF process solutions. The determination was made by titrating an aliquot of sample, containing sodium oxalate added to complex the hydrolyzable ions present, with standard sodium hydroxide solution. A new analyzer system for alpha spectrometry has been installed. This system allows the simultaneous analysis of four different samples. A low energy X-ray detector system has been calibrated and put into service. This system has the capability of plutonium isotopic analysis on SETF process solutions.

Radiometric measurements were made on actinide samples taken from a fuel pin irradiated in the Dounreay prototype Fast Reactor in Scotland. These measurements were part of the joint US-UK research program to study the behavior of selected higher actinides and to determine the cross sections of a number of higher actinide isotopes. These samples consisted of milligram quantities of oxides of curium, americum, plutonium, uranium and thorium.

*(J. M. Peele, R. A. Jones, C. C. Foust, M. R. Peters, R. T. Pack)*

# 4. BIO/ORGANIC ANALYSIS SECTION

M. R. Guerin

Each of the three groups in this section, Analytical Chromatography, Separations and Spectroscopy, and Special Projects, pursues fundamental and applied studies and supplies other Laboratory efforts with analytical services. During this past year analytical research has focused on separations, chromatographic detection, and chemical ionization mass spectrometry with a special emphasis on the utility of negative ion chemical ionization for both quantitation and isomeric discrimination. Applied research is related to the chemistry and toxicology of complex mixtures such as fossil fuels, cigarette smoke, and hazardous wastes. Increasing attention is being given to supplying organic analytical services in support of Laboratory programs. Technical support is also provided for the compilation of data and the review of issues related to synfuel technologies. The support for this section is provided primarily by the Department of Energy, the Department of Defense (Army), and the Department of Health and Human Services.

## SEPARATIONS AND SPECTROSCOPY

J. E. Caton

The Separations and Spectroscopy Group combines organic chemistry with analytical spectroscopy to achieve its research and services goals. Analytical research focuses on concepts and methods for the unambiguous isomeric identification of trace level organic chemicals in complex mixtures. Applied research addresses the identification and quantitation of biologically active constituents of mixtures. A special study being led by members of the group involves the detoxification of coal-derived crude oils by hydrotreatment and other methods. Increasing attention is being given to spectroscopic analytical services in support of Laboratory programs. Principal funding is provided by the Department of Energy and internal programs.

Chemical ionization mass spectrometry continues to receive primary attention in research aimed at isomeric discrimination. In a study involving more than forty polycyclic aromatic hydrocarbons (PAH) it was found that many isomeric PAH may be distinguished by electron capture negative chemical ionization processes. Using methane as the reagent gas to produce thermal electrons some PAH were observed to undergo ionization via electron capture to form molecular anions. Other PAH did not ionize and thus were not observed. For example, the potent carcinogen benzo(a)pyrene forms an anion under these conditions, whereas its relatively non-carcinogenic isomer benzo(e)pyrene does not ionize. When the electron affinity (EA) values of the compounds studied were compared, a threshold of about 0.5 eV was determined to be necessary for the electron capture process to occur. Because understanding the basis for the 0.5 eV threshold might allow it to be selectively altered to accommodate discrimination of specific isomeric pairs, a number of studies have been initiated to help elucidate the mechanism. Preliminary results of these studies indicate that at higher source pressures compounds which normally do not ionize in methane buffer gas do yield anions at mass M-1, where M is the mass of molecular ion. When ammonia was used as the buffer gas, the M-1 species were observed for the compounds with EA values <0.5 eV at all source pressures. A study using deuterated buffer gases, $CD_4$ or $ND_3$, revealed that two possible mechanisms may be occuring. For compounds with EA values >0.5 eV, direct electron capture was occuring, (equation 1, $k_1$ $k_2$), with no interaction with the buffer gas. For compounds with lower EA values, the buffer gas was participating in the gas phase reactions as indicated by complete deuterium substitution of the aromatic protons. This may suggest that after electron capture, the compounds with EA values <0.5 eV autodissociate to the neutral species unless the reagent can stabilize the species to form M-1 anions as shown in equation 2.

$$M + e^- \underset{k_2}{\overset{k_1}{\rightleftharpoons}} M^* \longrightarrow M^- \qquad (1)$$

$$M^* \xrightarrow{\text{Reagent}} (M-1)^- \qquad (2)$$

These observations suggest approaches to improve isomeric discrimination and aid in interpreting previously observed electron capture characteristics of organic compounds.

The relationship between negative chemical ionization mass spectrometry and electron capture detection for gas chromatographic analysis is being investigated. Many compounds of environmental and biological concern are commonly determined by electron capture. Early results confirm that negative chemical ionization mass spectrometry affords similar sensitivity with increased specificity. An observation of particular importance to section programs is that higher molecular weight PAH's are preferentially detected. It is anticipated that gas chromatography/negative chemical ionization mass spectrometry will become an increasingly important tool for analytical services and internal program support.

Funding has been acquired to purchase a Fourier Transform Mass Spectrometry (FTMS). The instrument, anticipated to be operational in the summer of 1985, will allow the study of ion cyclotron resonance for isomeric discrimination.

Capabilities for nuclear magnetic resonance (NMR) spectrometry have been upgraded this year. Our JEOL FX90Q NMR spectrometer has been modified to permit phase shifting and polarization transfer methods of pulsing. This involved some hardware modifications as well as the use of new software furnished by JEOL. The QUAT, INEPT and DEPT pulsing programs have been tested on a number of pure compounds. In the QUAT procedure, protonated carbon resonances are suppressed and quaternary carbon resonances are enhanced to permit visualization of only the quaternary carbon lines. With the coupled INEPT program (Incentive Nuclear Enhancement by Polarization Transfer) half of each doublet of the proton-coupled carbons is shifted 180° out of phase. This allows coupling patterns to be identified more easily, with the result that coupling constants can be measured more accurately. In addition, the signal-to-noise ratio is greatly enhanced, solvent peaks are suppressed, and the spectrum can be obtained in at least one-fifth the time required for a conventional coupled spectrum. The DEPT program (Distortionless Enhancement by Polarization Transfer) is a modification of the decoupled INEPT program which offers even more sophisticated information to be obtained from a sample. Three separate spectra are obtained which contain only CH, only $CH_2$, and only $CH_3$ carbon resonances. This allows the number of hydrogens attached to a carbon to be determined readily, without the use of proton-coupled experiments. This will be particularly useful for the study of complex mixtures, especially where the relative quantities of the various types of carbons are to be determined. In this report period, a spin simulation program has been used to accurately determine the $^{13}C$-NMR chemical shifts and coupling constants for a series of eight azaarene isomers of benzoquinoline.

An internal standard technique was developed for use with the diffuse reflectance technique in fourier transform infrared spectrometry. This approach which utilizes a reflecting medium (KBr) containing a constant amount of an internal standard ($K_3Fe(CN_6)$) for all samples in a series has shown very promising results for quantitation. Such an approach is especially applicable to functional group analysis of complex organic samples because diffuse reflectance is readily applied to difficult samples including intractable liquids, solids, and mixtures of liquids and solids.

Applied research on the chemical properties of coal-derived synfuels has continued. Studies of the polycyclic aromatic hydrocarbon (PAH) dermal tumorigens in coal oils have recently shown that two frequently reported tumorigens have been misidentified. Multiple isolation steps followed by high resolution gas chromatography and by mass spectrometry show that 3-methylcholanthrene (MCA) and 7,2-dimethylbenzanthracene (DMBA) are present (if at all) at 2 orders of magnitude lower concentrations than was previously reported. The identities of the interfering constituents and positive identification of other major tumorigens are being sought.

Research on refined syncrudes and on the toxicological characteristics of refining technologies has also continued. The vacuum gas oil (VGO, bp >343°C) fraction of crude coal liquids receives primary attention because both mutagenic and tumorigenic activity are concentrated in this fraction. Chemical treatments such as acid washing and pentane/furfural partition are found highly effective for removing mutagens from the VGO but not effective for removing tumorigens. Catalytic hydrogenation continued to be found the most effective means of eliminating both mutagens and tumorigens. Preliminary studies show that catalyst type is more important than reaction temperature and hydrogen partial pressure from removing benzo(a)pyrene. Three hydroaromatic derivatives of benzo(a)pyrene and two hydroaromatic derivatives of dibenz(a,h)anthracene have been synthesized to study the mechanism of tumorigen hydrogenation. A most interesting finding is that acid washing of the VGO before catalytic hydrogenation provides a much greater reduction in benzo(a)pyrene content than does hydrogenation of the untreated VGO.

Technical support efforts continued during the year. Much support was provided to the Fossil Energy Technology Environmental Program in the form of contributions to information data bases, reviewing environmental monitoring plans, preparing review documents, and aiding in program development plans. Other technical support was devoted to the ongoing Office of Health and Environment Research Programs for which a data base was prepared to aid in the selection of model compounds. Using this data base representatives from several DOE facilities (Pacific Northwest Laboratories, Argonne National Laboratory, Los Alamos Scientific Laboratory, Oak Ridge National Laboratory, and DOE) nominated a series of eleven compounds as possible benchmark chemicals to be used in fundamental chemical and biological studies relevant to the toxicological evaluation of energy-related processes.

Gas chromatography/mass spectrometry, nuclear magnetic resonance spectrometry and infrared spectrometry services in support of Laboratory-wide programs increased slightly during this year. Especially noteworthy in this increase was the development and adaption of approaches to use gas chromatography/mass spectrometry to quantitate various constituents in indoor air samples.

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## ANALYTICAL CHROMATOGRAPHY

W. H. Griest

The main activities of the Analytical Chromatography group are chromatographic methods development and applications associated with health and environmental issues. Funding is provided by the U.S. Department of Energy, the National Cancer Institute, the Department of Defense, the Environmental Protection Agency, and the Electric Power Research Institute. The ORNL Technology Transfer Program has funded the development of an improved chromatography detector. In-house organic analytical services have increased considerably in importance this year.

Basic chromatography research continues to focus upon improving the high resolution liquid chromatographic separation of complex mixtures. The primary emphasis has been on open tubular liquid chromatography (OTLC) and packed microcapillary high performance liquid chromatography (HPLC). Clogging problems encountered in the pretreatment of OTLC columns have been avoided by the use of an alternative: dynamically modified columns. In this procedure, a surfactant (cetyltrimethylammonium bromide) is added to the mobile phase for an untreated column. Dynamic adsorption/desorption of the surfactant by the glass results in a "stationary" phase which is moving, but at a velocity slower than that of the mobile phase. This results in a separation based on partition. Closely related polar organic compounds such as the B group vitamins have been separated by this technique.

Fused silica microcapillary columns (ca. 2m x 250 μm ID) have been slurry packed successfully with 3 μm bonded phase particles. The columns have yielded improved separations of polycyclic aromatic hydrocarbons (PAH). While the analysis times are relatively long, separations are good and the column capacity is not as limited as in OTLC. A He/Cd laser with output at 325 nm has been used with a low-volume flow cell for detection of separated components. Procedures have been devised for the low dead-volume connection of quartz flow cells to the column outlet. Detection limits of <1 pg have been obtained for certain dermal tumorigen PAH such as 3-methyl cholanthrene.

Developmental work in chromatography also included design modifications for improved operation of the helium ionization detector (HID). The HID has been coupled to a specially designed high voltage pulser and operating the detector has been operated in a fixed-frequency pulsed mode. Overall, a reduction in noise and background current levels was observed as compared to DC operation. It has also been determined that the pulsed detector can be used at higher voltages before the cell current breaks down. Since the response increases exponentially at higher voltages, operation under these conditions may provide significant increases in sensitivity. Detection limits for various gases are currently being determined so that the exact effects on sensitivity can be ascertained.

At certain frequency and duty cycle combinations, an inversion in signal polarity for the gases that normally provide a negative response has also been noted. The inverted peaks have an improved upper detection limit. Depending upon the analytical requirements, the response of the detector may be easily adjusted to cover a broad range simply by varying the pulse parameters. Additional electronic modifications which allow the HID to operate in a variable-frequency, constant-current mode have also been made. The response characteristics will be examined and the various modes of operation compared in the next year.

Applied research in the electrochemical determination of munitions components in water has been extended to linear scan and derivative mode linear scan voltammetry and also differential pulse voltammetry. These methods were found to allow rapid (30-70 sec.), direct (only degassing is required) estimation of munitions components in only 10 mL of water. One mg/L of TNT$^a$ in water can be easily measured by the linear scan derivative or differential pulse methods. Agreement of results for highly contaminated samples versus those determined by the resin sorption-HPLC method has been good (e.g., 80 mg/L of TNT$^a$ in pink water vs 80 mg/L by HPLC and 140 mg/L of NC$^a$ in process water vs 130 mg/L by HPLC). The voltammetric methods

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[*] Summer employee, 1984

[**] Retired

appear to have considerable potential for rapid, on-site or off-site screening of samples and for nearly real-time monitoring of process streams or fenceline ground waters for munitions.

The sorbent resin collection and HPLC/electrochemical detection procedures for the determination of munitions components in ground and surface waters have been validated in extensive laboratory and field studies. A substantial improvement was achieved in recoveries of nitroaliphatics over those reported in previous studies. However, recovery of TETRYL[a] remains a problem. Preliminary stability studies suggested that the munitions are stable for 4-6 weeks while stored on the resin, and considerably less stable in aqueous solution (original water sample) or in acetone (solvent eluate of resin). The HPLC separation of all eight munitions components (HMX, RDX, TETRYL, NG, TNT, 2,6-DNT, 2,4-DNT, and PETN)[a] was achieved by adding a small gradient to the HPLC elution procedure. A standard Operating Procedure was drafted and delivered to the U.S. Army Toxic and Hazardous Materials Agency.

Gas chromatographic methods development has focused upon the determination of volatile organic compounds in tobacco smoke, indoor air, and aqueous samples. Increasing need for the determination of highly volatile compounds in indoor air has led to the development of an improved trap design for collecting organic compounds as volatile as methylene chloride. Multi-sorbent designs (Tenax followed by silica gel and charcoal, or Tenax backed up with Carbopack and Ambersorb XE-340) have allowed the collection of as much as 60 L of indoor air without detectable breakthrough. The collected compounds are analyzed using thermal desorption gas chromatography (GC) with capillary column separation and selected ion monitoring mass spectroscopy for determining trace-level halocarbons, or flame ionization detection for measuring the more prevalent alkanes and aromatic hydrocarbons. Atmospheric moisture also collected in the traps has frozen out and caused plugging during cryogenic focusing in the GC. Further work is addressing this problem.

A micro-scale liquid-liquid extraction method has been devised for the routine determination of volatile priority pollutants in water at the 10 μg/L level. An aliquot of the pentane extract is split between dual parallel capillary GC columns with flame ionization and electron capture detectors. The much more lengthy and difficult purge-and-trap procedure is reserved for determinations at the 1 μg/L level.

A method for containing and collecting sidestream cigarette smoke, the major contributor to environmental tobacco smoke, has been devised. This method relies upon an existing smoking chamber design to contain the smoke but includes a special valving system for directing any selected portion of the sidestream smoke to a filter and Tenax trap without interrupting the normal flow of the smoke. Samples of the sidestream may be taken at any point during the cigarette smoking protocol. Gas phase compounds collected on Tenax are analyzed using thermal-desorption GC with multielement detection. Comparison of the organic gas phase composition of sidestream just before, during, and immediately after a mainstream puff showed all three samples to have similar composition, but the sample taken just after the mainstream puff was 2-4 times more concentrated. Large differences were noted in the mainstream organic gas phase deliveries of different commercial smoking products ranging from an ultra-low tar cigarette to a little cigar. However, the differences among their sidestream deliveries were much less pronounced.

The sequential HPLC/HPLC method for the determination of benzo(a)pyrene (BaP) in cigarette smoke total particulate matter (TPM) has been validated and applied to several commercial cigarettes. Normally, the TPM from only seven to twelve cigarettes is required, although for ultra-low tar brands, additional cigarettes must be smoked to produce the ca. 125-150 mg of TPM needed. The overall recovery of BaP, determined using a radio-labeled tracer, was 87 ± 7%. Accuracy and precision were demonstrated by standard addition experiments using the non-filtered high tar 2R1 and the filtered low tar 1R4F Kentucky Reference Cigarettes. Analysis of several domestic commercial brands of cigarettes showed mainstream BaP deliveries of 2.4 to 14.2 ng per cigarette under standard analytical smoking conditions. An experimental marijuana cigarette was found to deliver 20.9 ng of BaP per cigarette, while a "marijuana placebo" (solvent-extracted marijuana) delivered 40.6 ng per cigarette.

The demand for in-house organic analytical services has increased considerably this year.

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[a] HMX = cyclotetramethylenetetranitramine, RDX = hexahydro-1,3,5-trinitro-1,3,5-triazine, TETRYL = N-methyl-N,2,4,6-tetranitroaniline, NG = nitroglycerine, TNT = 2,4,6-trinitrotoluene, 2,6(or 2,4)-DNT = 2,6(or 2,4)-dinitrotoluene, PETN = pentaerythyryltetranitrate.

The sample load in FY 1984 consisted of 749 samples for volatile organics (9588 determinations), 679 samples for various extractable organics (6942 determinations), and 295 miscellaneous samples. In addition to these routine determinations, numerous non-routine measurements of volatile organic compounds in indoor air and of phosphate species in lead-phosphate glass leachates were conducted.

The facilities and procedures for service work are being upgraded to provide a more efficient operation. A sample receiving and preparation laboratory for the section was established to provide for the central acceptance, preparation, and archiving of samples submitted to the section for analysis of organic compounds. It has been equipped with various types of sample extraction devices, refrigerators and freezers, and a glove box for handling hazardous materials. Data acquisition, reduction, reporting, and archiving have been improved by the installation of an IBM PC/XT based chromatography data system (Nelson Analytical). This system collects and stores the entire chromatographic run, performs calculations, and reports data in the format used by the ACD data management syrtem. Direct connection to the ACD data management system is anticipated in early 1985.

Synthetic fuel research sample management in support of health and environmental studies continues as a group responsibility. The sample handling laboratory in building 3001 has been equipped for handling, homogenizing, and aliquotting drum quantities of samples.

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## SPECIAL PROJECTS

R. W. Holmberg

Studies of the chemical and physical properties of aerosols, inhalation exposure atmospheres, and the source materials for these atmospheres are emphasized in the Special Projects Group. Support is provided primarily by the National Cancer Institute for cigarette smoke studies, the National Institute of Drug Abuse (NIDA) for marijuana studies, and the Department of Defense for obscurant and inhalation exposure studies.

Field sampling has played an increasingly important role in the group's activities. A collection of portable instruments for a wide variety of airborne sampling protocols has been assembled and tested in field use. These include high volume air sampling pumps with both directional and non-directional sampling heads, large sorptive resin cartridges for volatiles collection, cascade impactors for particle sizing, low volume pumps for small gas and particle sample collection, and instrumental temperature, air flow rate and smoke concentration monitors. Much of this equipment was used on a field sampling expedition to Ft. Carson, Colorado. As part of a new project initiated with the Army, we are to collect and compare diesel exhaust samples from large military vehicles such as battle tanks, ammunition haulers and bulldozers. This study will provide a data base for a relative risk assessment of the health impact of conversion to shale-derived diesel fuel. Military vehicles at Ft. Carson, presently operating on conventional fuels, are scheduled to be converted to a shale derived source in 1985. The sampling trip was conducted in late September to obtain baseline data from vehicles operating on conventional fuels. Samples of exhaust from both dynamometer test facilities and field vehicles were collected. In addition, samples of aged exhaust present in vehicle repair bays were collected to provide a measure of the level of human exposure via this route.

A sampling trip was also made to the National Center for Toxicological Research (NCTR) laboratories in Pine Bluff, Arkansas, to obtain measurements of marijuana smoke exposure of rhesus monkeys as part of a cooperative project sponsored by NIDA. These initial experiments suggested that the animals retain about 50% of the psychoactive cannabinoid in the smoke to which they were exposed. This finding was in excellent agreement with blood carboxyhemoglobin levels in the animals, which also indicated that the animals retained about 50% of the carbon monoxide which they were offered for inhalation. A sampling trip was also made in support of red phosphorus/butyl rubber obscurant smoke exposures of rodents being conducted for the Army at the Illinois Institute of Technology Research Institute (IITRI). The results obtained from the samples collected indicated that the relative concentrations of polyphosphate chains and residual organic vapor levels in the exposure atmosphere smokes were very similar to those of smokes generated under more carefully controlled conditions at ORNL.

Projects supported by the National Institutes of Health are comprised of two components: The chemical characterization of tobacco and marijuana smoke, and bioassay instrumentation development. In studies supported by NIDA, the first characterizations of the smoke deliveries from marijuana cigarettes under standard, well defined smoking conditions have been performed. These studies indicated that the relative concentrations of cannabinoids in the smoke do not change with increasing puffing frequency. Placebo marijuana cigarettes, intended as controls for inhalation studies, delivered substantially more carbon monoxide and benzo(a)-pyrene than their marijuana counterparts. In tobacco smoke studies supported by the National Cancer Institute, we have continued to shift our emphasis toward characterization of cigarette smokes under more humanly relevant puffing conditions. We have begun studying the effect of puff volume and shape on the relative composition of cigarette smoke. Changes in puff shape do not appear to affect the deliveries of bulk constituents such as tar, nicotine, and carbon monoxide. The relative composition of some lesser constituents in the tar may, however, be altered as puff volume increases. As part of a joint project with the U.S. Department of Agriculture, a survey of smokes from cigarettes manufactured from Turkish tobaccos identified one tobacco type with an unusually high nicotine to tar ratio. This finding may be of use in lowering the level of tar smokers must take in to achieve a given level of nicotine exposure.

Instrumentation development during the past year has included a human smoke dosimeter and a computer controlled variable puff smoking machine. The smoke dosimeter is based on the simultaneous measurement of flow and aerosol particle concentration through a small cigarette holder. From this we derive a measure of the total amount of tobacco smoke being generated during puffing. Presently, the device uses analog electronics. Plans are being made to convert to online digital measurement and processing to improve calibration and read-out of data. The variable puff smoking machine has now been extensively tested. The puff volume and incremental flow rate are operator controlled so that a large variety of different puffing conditions, such as those found in human smoking, can be investigated.

An experimental obscurant source material (RPNO3), consisting of powders of red phosphorus and sodium nitrate bonded together with an epoxy resin, has been examined. Analyses for the major constituents of the matrix as well as for trace impurities are being performed. The obscurant aerosol formed from its combustion has been characterized and compared to that produced from red phosphorus-butyl rubber (RPBR) and white phosphorus-felt formulations studied earlier. We have found that the three phosphorus formulations give aerosols and atmospheres that are very similar when burned under similar conditions. The aerosol particle phase exists predominantly as a concentrated water solution of phosphoric acid. Anion exchange flow injection analyses show that this phosphoric acid is present as a complex mixture of condensed polymeric forms with monomeric ortho phosphoric acid being only a minor constituent. Chromatographic analyses and total organic carbon analyses indicate that organic materials are not incorporated into the smoke in appreciable quantities. Presently, the Department of Defense is sponsoring an inhalation toxicology experiment on the smoke from the RPBR formulation. This chemical study will aid them in deciding if toxicology studies need be carried out on the others.

We have continued an aerosol support program for a DOD sponsored inhalation toxicology investigation of the smoke from RPBR being carried out at the Illinois Institute of Technology Research Institute in Chicago, IL. In addition to supplying technical support for the use of a battery of extrusion combustion generators for the RPBR that were developed here, we have manufactured and supplied IITRI with the source material, hexane softened RPBR, for the smoke generation. In all fourteen shipments totaling 436 billets of RPBR have been shipped in the past year. Over the course of the toxicology experiment, we have supplied IITRI with 997 billets of RPBR, six generators, seven aerosol monitoring systems, and five flame-out detectors.

The Section's PDP11/23 computer has been moved to a small central room. Under an RT11/TSX+ operating system it is interfaced to seven terminals (including an IBM-PC). It is used for general scientific computing, report drafting, and particularly for graphic handling of experimental data obtained from laboratory instrumentation. The documentation of tobacco smoke analyses has been extensively computerized, enabling us to better track, validate, and analyze this extensive and growing data base.

*(G. M. Henderson, S. K. Holladay, R. W. Holmberg, R. A. Jenkins, J. H. Moneyhun, D. D. Pair, and T. M. Gayle*)*

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*Instrumentation and Controls Division

# 5. GENERAL AND ENVIRONMENTAL ANALYSIS

J. R. Stokely

The General and Environmental Analysis Section provides a diversity of analytical chemical services in support of Laboratory and Energy Systems programs. Activities within the section are organized into five groups: Chemical and Physical Analysis, Environmental Analysis, Low-Level Radiochemical Analysis, Activation Analysis, and Special Projects. The section performs routine analyses using established procedures and is involved in developmental activities to upgrade capabilities, to devise new methods of analysis, or to solve applied problems requiring analytical expertise. About 200,000 analyses are performed annually for a large number of programs and research projects.

Analytical chemistry support for environmental programs has continued to increase this year as Laboratory activities in environmental monitoring, acid rain research, and waste management have expanded. Radiological and nonradiological analyses are provided for these programs. It is anticipated that our support for these programs will expand, particularly for nonradiological analyses. Accordingly, we continue to improve capabilities by upgrading methodology and acquiring new instrumentation. An automated low-level beta counting system was put into routine operation this year, and a new total organic carbon analyzer is being procured to replace an old instrument. Experience is being obtained with a laboratory robotic system, and efforts are being made to utilize the system for routine laboratory operations. Acquisition of a new automated, atomic absorption spectrometer is planned.

## CHEMICAL AND PHYSICAL METHODS

J. H. Stewart, Jr.

The successful application of laboratory-scale chemistry to very large scale environmental radiological and toxic metal wastes was demonstrated at the Bechtel National Formerly-Used Niagara Falls Storage Site. Approximately 2 million gallons of contaminated waste liquid were treated, using the Oak Ridge National Laboratory (ORNL) procedure, and met New York State discharge criteria. That basic technology is now being modified and tested to permit treatment of additional storage sites.

A complete analytical characterization of uranium mill tailing ponds in Wyoming is now underway for the Nuclear Regulatory Commission (NRC). The NRC will use this study to identify potential contaminants in the large (100 acre or larger) ponds that currently store acidified process waters from the uranium recovery operations. We have completed a characterization of seven ponds at one mill, and the data are being evaluated by the NRC to permit planning for large-scale analytical surveys in early 1985.

Environmental monitoring of both ORNL surface and well waters has increased. We acquired a Hach spectrophotometer and packaged reagents to increase water analysis productivity and to significantly reduce cost of the chemical oxygen demand measurements. The Hach integral microprocessor establishes master calibration curves for some 60 parameters and computes final results in parts-per-million. We obtained a Zeiss Ultraphot II optical microscope from the Biology Division and now have a reliable service support optical microscopy capability. We also obtained components to establish a photographic enlarging and printing capability for rapid processing of our scanning and transmission electron microscope negatives. The PGT System 4 X-ray Analyzer became fully operational as a quantitative system and was immediately useful in identifying steel components, measuring thermocouple wires, analyzing thin films on ceramic substrates, and measuring the composition of glass.

Measurement of polychlorinated biphenyl compounds (PCB) has become a major analytical support activity. We monitor PCB in surface and well waters, in sediments, soils, residues, waste oils and organic liquids and in numerous types of tissue including frogs, turtles, and several species of fish. We have also surveyed PCB cleanup operations at ORNL for employee safety, and in materials from National Lead of Ohio and from a retired DOE machining site.

We provided the analytical support necessary to characterize all nonradiological process waste water streams at ORNL. The analyses will be used in the design of a pollution abatement facility for ORNL. Obtaining the necessary information required measurement of nearly 100 water quality parameters. We established a method for measuring asbestos fibers in water using transmission electron microscopy to meet one of these required parameters.

Evaluation and testing of the Zymark laboratory robotic system has involved developing hardware, software, and procedures for preparing liquid scintillation counting vials. Presently we are developing methods for performing colorimetric analyses, for extracting uranium from aqueous samples, and for preparing fluorimetric pellets for measurement of ppb concentrations of uranium.

Unusual analytical requests during this year became more frequent. We established a method for measuring iron (III) in iron (II) Fricke radiation dosimeters by direct optical spectrophotometry. The quality of a billion-ton deposit of pure $SiO_2$ was evaluated for possible commercial use. We established why uranium in ultra fine particles of $MgF_2$ slag could not be extracted in multiphase acid treatments. Referee methods for measuring iridium in precious metal scrap were provided. Perchlorate and dibutyl phosphate were measured in aqueous and in organic media. A method was developed for dissolution of $B_4C$. A new GC method was devised for determining 2-ethyl hexanol in LiBr heat transfer fluids. Aqueous leachates from cement encapsulation tests were analyzed for toxic organic materials.

We have significantly improved the method for quantitative determination of asbestos in building materials and have successfully performed comparison analyses on Research Triangle Institute reference materials. The ORNL-designed spectrometer was interfaced to a conventional X-ray diffraction analyzer to test our concept for simultaneously measuring chrysotile and amosite asbestos at the one percent level.

An interesting technique was developed for preparing human blood for simultaneous ICP multielement analysis, which may be useful in diagnosis of human health disorders. We then extended the method to human brain tissue. This has provided data which will be used in an effort to relate metallic concentrations in various lobes of the brain with observed neurological disorders. During the year the group reported a total of 134,564 individual analyses; metals analysis by simultaneous inductively coupled plasma (ICP) spectroscopy and anion measurements by ion chromatography provided a large percentage of the analyses.

*(J E. Attrill, B. Philpot, K. I. Webb, H. G. Davis, C. S. Alley, J. H. Hackney, W. F. Rogers, S. J. Bobrowski, N. P. Buddin,* H. W. Dunn, C. S. MacDougall, J. C. Price)*

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*On loan from ORGDP

## ENVIRONMENTAL ANALYSIS LABORATORY

N. M. Ferguson

The Environmental Analysis Laboratory (EAL) provides analytical support primarily for the Environmental Sciences Division (ESD) but it does a significant amount of work for other divisions and outside organizations. Support for other divisions increased this year and required analysis of a wide variety of materials.

We have been heavily involved with mercury analysis, for both new and continuing projects. Many industrial and municipal waste leachates, stream and water samples, sludges, charcoal filters, soils, fish, etc. were analyzed. An elaborate study of mercury levels in human hair was also conducted.

The Department of Environmental Management (DEM) continues to be heavily involved with the monitoring of effluents from ORNL at various discharge stations. During this report period several intensive sampling programs were completed and new ones continue to be initiated. These programs generated numerous samples. Most samples required special treatment (filtration, acid digestion, etc.) before analysis. All of the effluents were analyzed for Sb, As, Be, Cd, Cr, Cu, Fe, Pb, Ni, Se, Ag, Tl, and Zn. Total organic carbon, Kjeldahl nitrogen, ammonia, and total phosphorus were determined in most effluents. Many procedures were modified to obtain the desired very low concentration levels (ppt and ppb). Most of these projects were on very tight schedules and required fast turnaround time.

A very large portion of our work during this report period derived from the acid rain program in ESD, which is funded by The Electric Power Research Institute (EPRI), and encompasses many different projects. The atmospheric studies generated numerous throughfall, rain, and dry deposition samples from the Walker Branch Watershed for analyses for various cations (Ca, Mg, Na, K) and anions (Cl, $NO_3$, $PO_4$, $SO_4$). Organic acids (oxalic, citric, malic, formic, and acetic) and organic carbon were measured in several samples. A sizeable effort was expended to provide support for the project investigating the effects of acid rain on soils. This study produced large volumes of samples (soils, plants, waters, and soil leachates) at regular intervals for a variety of analyses (Al, Ca, Mg, Na, $NH_3$-N, K, Cl, $NO_3$-N, $PO_4$-P, $SO_4$, Kjeldahl N, and total P). The most recent project monitors the effects of acid rain on gradient streams in the Smoky Mountains for various cations and anions, with particular emphasis on aluminum. Monomeric and total aluminum were determined in many samples.

The Electric Power Research Institute in August 1984 approved a large environmental research project on the linkage between atmospheric deposition and nutrient cycling in forests. This includes the acid rain work. Oak Ridge National Laboratory was designated the prime contractor for this four-year acid rain project. During this report period the EAL has been preparing for the influx of samples which will begin early in 1985.

Fluoride, chloride, phosphate, nitrate, and sulfate were measured in thousands of samples without any major problems using ion chromatography during the past year. Several improvements were made in our ion chromatography procedures. A continuously regenerating (fiber) suppressor was procured for use with the organic acid columns. This suppressor eliminates the need to monitor the packed bed for depletion and improves peak resolution. A "high nitrate" column was obtained, thus making it possible to determine sulfate in 1% nitric acid. This column allows the elution of nitrate anion after sulfate.

A modification of the automatic sampler on the graphite furnace made it possible to analyze organic samples and increase productivity. Productivity was also increased by the acquisition of a second block digestor. There was an increasing need to determine Kjeldahl nitrogen and total phosphorus in a variety of samples (soil, vegetation, waters) for new and continuing programs. This second block digestor increased from 20 to 40 the number of samples that could be processed at a time for these analyses.

## LOW-LEVEL RADIOCHEMICAL ANALYSIS

### T. G. Scott

The Low-Level Radiochemical Analysis Group performed low-level radioanalyses for man-made and natural radionuclides in an ever-increasing variety of materials to support programs and projects conducted at ORNL and by outside organizations. During the past year, we supported various programs in the following proportions: environmental monitoring, 50%; environmental surveillance, 20%; research and development, 20%; and work for others, 10%.

The slight increase in the volume of work done for environmental monitoring was mainly due to projects conducted by the Department of Environmental Management. One project (Non-Rad Waste Water Treatment Project - NRWWTP) involved the determinations of gross alpha, gross beta, strontium-90, and gamma-emitting radionuclides in water sampled daily at nine discharge locations over several weeks. Another project (National Pollutant Discharge Elimination Systems - NPDES) required several groups of samples taken at selected discharge locations to be analyzed for gross alpha, gross beta, radium-226, and total radium.

Many organic waste materials were analyzed for a hazardous waste control group to determine the concentrations of gross alpha, gross beta, gross gamma, tritium, and carbon-14 activities. The purpose of this work was to determine if the waste materials met Environmental Protection Agency regulations for being shipped off-site for disposal or should they be held for future action. At best, most of the analyses on these materials were only cursory screening operations due to the nature of the samples, some of which had as many as four separate phases.

Researchers of the Environmental Sciences Division submitted several suites of brine water samples collected from test wells at the shale hydrofracture waste disposal site for the determinations of gross alpha, gross beta, strontium-90, technetium-99, tritium, cesium-137, cobalt-60, and transuranics. The radiochemical separations for strontium-90 and technetium-99 were complicated by the high concentrations of stable elements in the brine solutions (Ba, Ca, K, Mg, and Na ranged as high as 10 mg/mL and Sr as high as 1 mg/mL). Modifications of normal procedures were required to acquire data. Methods are being developed to improve our capabilities on samples of this nature.

Members of the Health and Safety Research Division submitted several hundred soil samples from cores taken at a St. Louis site where waste from a former uranium processing operation had been dumped. The object of this project was to provide engineers with the location of contaminated soil that was to be removed. Thorium-230 was the selected radionuclide used to measure the level of contamination. Our determinations of thorium-230 were simplified by the use of a new alpha/beta counting system.

Several projects that we supported required the development of procedures and/or methods to comply with requests. One project involved analyzing fish from local streams for technetium-99 content. The lack of a standard reference material similar to these samples made our work difficult, and the results were reported as tentative.

Another project dealt with the determination of the emanation rate of radon from concrete samples for the Charleston Naval Shipyard. A procedure was developed to process concrete disks and crushed concrete samples by coating some samples with epoxy and measuring all samples for radon exhalation. Each sample was sealed in a specially designed plastic container, the container was flushed with helium, and after an ingrowth period, the radon that emanated from the concrete was collected on silica gel at liquid nitrogen temperature. The "trapped" radon was subsequently measured by liquid scintillation counting.

During the year we responded to several episodes of varying degrees of urgency for the determinations of a variety of radionuclides. Sewage sludge from the Oak Ridge City sewage plant was found to be contaminated with radioactivity after some of the sludge had been spread on fields as fertilizer. We analyzed portions of the raw sludge and soil from the contaminated fields for strontium-90, uranium isotopes, and transuranics. Urine samples from ORNL personnel exposed to radioactive debris were analyzed on a rush basis for strontium-90 and plutonium isotopes, and various filter materials and smears taken at a waste-storage-tank site were analyzed for several radionuclides. All of these incidents required rapid response, superseded normal work, and usually necessitated overtime in order to deliver timely results.

## SPECIAL PROJECTS

J. S. Eldridge

The Special Projects Group (SPG) provides applied research and technical support in a variety of programs related to environmental surveillance, emergency response, and for inventories of precious metals and special nuclear materials.

Previous studies on the use of thyroids from vehicle-killed deer were extended to determine environmental pathways for radioiodine species. Earlier work had shown the presence of $^{125}$I (a 60-d. half-life, neutron-deficient radionuclide) in all deer thyroids collected on the Oak Ridge Reservation. Animals collected near the eastern boundary of Oak Ridge National Laboratory contained the highest concentrations of $^{125}$I in their thyroid glands. Since the prevailing winds are from west to east, it was speculated that ORNL was evidently the origin of the environmental $^{125}$I. A release of $^{125}$I near the end of 1983 resulted in elevated levels of radioiodine in deer thyroids. Charcoal samples used to monitor gaseous iodine releases from an ORNL stack were obtained for the period in late 1983 when the biological information indicated a gaseous release of iodine. In these charcoal samples, $^{125}$I concentrations equivalent to a 300 mBq (8 mCi) discharge were found. The $^{125}$I is not detected by the usual analytical procedure for radioiodine releases; therefore, the $^{125}$I discharge was detected as a direct result of a bioindicator measurement. The documented pathway analysis showing an airborne release coupled with the high levels of $^{125}$I in animals near ORNL in a downwind direction of the prevailing winds clearly demonstrated the importance of meteorological conditions on the patterns of airborne contaminants in the environment.

Iodine-129, an iodine isotope with a half-life of 16 million years has been determined by a direct gamma-ray spectrometric measurement in eight of fifty deer thyroid glands collected near ORNL. Usually, $^{129}$I determinations in thyroids from animals near nuclear facilities require special methods involving chemical separation of the iodine followed by neutron activation enhancement via $^{130}$I production. The eight thyroid glands contained $^{129}$I in concentrations ranging from 1 to 9 Bq/g (30 to 250 pCi/g) dry weight. By assuming an average stable iodine content of 2.3 mg iodine per gram of dried thyroid along with the specific activity of $^{129}$I (6.4 becquerels per microgram), an $^{129}$I to $^{127}$I atom ratio of $6.4 \times 10^{-4}$ was calculated for the animal with the highest $^{129}$I concentration. This result is about two orders of magnitude higher than those found in mule deer thyroids from the vicinity of the Idaho National Engineering Laboratory.

From these preliminary studies, it was concluded that there is an environmental pool of $^{129}$I in the vicinity of ORNL that is accessible to grazing animals that consume large quantities of grass. Further studies are planned to determine whether $^{129}$I reaches the environment by an airborne pathway.

Assistance to the DOE Radiological Assistance Program (DOE-RAP) continues to be provided by the SPG in the form of emergency drills as well as for actual emergencies. The State of Tennessee Division of Radiological Health requested assistance from DOE after it received a report of a missing well-logging source from a Crossville, Tennessee, firm. The source was described as a 6-in. long by 1 1/2-in. diameter stainless steel cylinder containing 3 curies of $^{241}$Am mixed with beryllium to produce neutrons for oil well-logging activities. The source was discovered missing when the transport vehicle arrived in Williamsburg, Kentucky, after departure from Crossville. It was presumed lost somewhere between those two cities. The vehicle passed through Oak Ridge on its trip. A search team directed by a SPG staff member was dispatched to Crossville to start a search in the east-bound direction, while the DOE mobile laboratory directed by another SPG staff member proceeded from Oak Ridge towards Williamsburg. Both teams used sodium iodide detectors with audible output for the detection system. The detectors were mounted outside the vehicles in such a fashion as to provide maximum sensitivity for the 60-keV gamma-rays of $^{241}$Am. The source was found lying about 3 feet from the pavement in the median strip of I-75 about 10 miles north of the Kentucky border. The source was found to be intact and was returned to Oak Ridge where it was returned to the owners.

Assistance was also provided in the operation of the DOE mobile laboratory during the two-day annual emergency exercise at the Watts Bar nuclear power plant. Mock emergency radiological measurements were provided at an emergency center established in Athens, Tennessee.

Nondestructive assay (NDA) measurements are performed in support of precious metals and special nuclear materials (SNM) inventories. A portable, cart-mounted intrinsic germanium X-ray detector system is used for both NDA applications. In the first case, a $^{57}$Co source exciter for X-ray fluorescence is used to produce characteristic X-rays from the precious metals, with the X-ray detector providing qualitative analysis information. In a typical application for the system, qualitative analyses are performed at the time of precious metal inventory audits at the respective storage vaults. It is possible to detect and sort precious metal alloys from lots of pure metal to correct labeling mistakes (a spool of wire labeled as platinum was found to be silver), and to detect base metal substitution (a large disc of metal 4-in. diameter by 1/4-in. thickness was found to be a base metal when a lot of platinum scrap was inventoried). The rapid and cost effective technique of metal identification via the source-excited X-ray emission is now a routine technique for inventory verification in conjunction with audits by the Laboratory Protection and Finance and Materials Divisions.

The portable X-ray detector system without the source exciter is used for measurement of low-energy X- and gamma-radiation from SNM at the time of annual or special audits. The measurement of characteristic X- or gamma-rays emitted from uranium, thorium, and plutonium nuclides serves as the basis for NDA procedures. The portability of the NDA system has proven to be a great asset in providing needed information to the Laboratory Protection Division for SNM inventory verifications. The system was taken recently to Building 7019 (where there is no electrical service) and operated with power from a portable electrical generator. Measurements were necessary to segregate a large quantity of nuclear fuel rods into thorium-bearing, natural uranium, and depleted uranium lots. Detection of the characteristic 186 keV gamma-ray from $^{235}$U was used for the NDA of uranium rods, and the 239 keV gamma-ray from the 10-h $^{212}$Pb daughter of the $^{232}$Th was used for the verification of the thorium-bearing rods. After the initial measurements and identification of thorium-bearing rods were made, the thorium rods could be segregated from the others, since they were only two feet in length and all uranium rods were four feet or greater.

*(N. A. Teasley)*

## ACTIVATION ANALYSIS

J. F. Emery

The Activation Analysis Group provides general neutron activation analysis (NAA) services for a number of Laboratory divisions and programs.

This year we determined Ag, Au, Ce, Cl, Co, Cs, Cu, Fe, Hg, I, $^{129}$I, K, Mn, Na, $O_2$, Sr, Ti, U, V, and Zn in alloys, ceramics, metals, single crystals, and soils for the Chemistry, Chemical Technology, Environmental and Occupational Safety, Health and Safety Research, Operations, Physics, and Solid State Divisions.

We have renewed efforts to obtain an addition to our High Flux Isotope Reactor (HFIR) NAA laboratory. The project will consolidate our ORR NAA facility with the NAA research facility at the HFIR. The radiochemistry laboratory at the HFIR will be enlarged, a new counting room will be constructed, and an office area will replace the present counting room. The new counting room will increase our usable space from 320 sq ft to 600 sq ft.

During a three month shutdown of the HFIR to replace the beryllium reflector, the incore section of the pneumatic tube was also replaced. Originally the lifetime of this section was to be 5 years, but mechanical tests indicated the lifetime could be extended to 8-1/2 years which coincides with the beryllium reflector changes. We have acquired high purity graphite rabbits to be used for extended irradiation times in the HFIR pneumatic tube. There is very little induced radioactivity in these rabbits after an hour irradiation. The rabbits are fabricated to our design by Poco Graphite, Inc.

We provide technical support to the Chemical Technology Division in a study, supported by the NRC, of the depositon of fission products from aerosols. Experiments have been performed to study the interaction of CsOH and CsI vapor with various materials, such as silver, manganese oxide, chromium oxide, iron, iron oxide, tin oxide, and zinc oxide, that might be produced in nuclear accidents.

Our work for others activities mostly consisted of determining Uranium and Thorium in semiconductors and semiconductor materials as more and more manufacturers are increasing their efforts to reduce the Uranium and Thorium in their products. The impetus for this is the soft error effect in computer memories caused by the ionizing alpha particles emitted by Uranium and Thorium and their daughters.

# 6. QUALITY ASSURANCE, SAFETY, AND TABULATION OF ANALYSES

W. R. Laing
P. L. Howell
C. S. MacDougall
A. L. Harrod
R. E. Jones

## QUALITY ASSURANCE

Responsibility for the quality of work done within the five sections of the Analytical Chemistry Division falls to the respective supervisors. Because of the diversity in types and scopes of work done in these sections, several means are used to assure quality. The annual quality assurance report (ORNL/CF-84/374) comprises a collection of statements from each group and/or laboratory, encompassing (1) scope of work, (2) quality assurance program and procedures, (3) quality assurance compliance information and data, and (4) standard reference materials and their sources. Included is a statement summarizing control samples prepared and distributed during the reporting period and the quality level obtained with these internal control (reference) standards. Table 6.1 shows the quality level for each of the participating laboratories within the division and compares the results with those from the previous year. Lists of the different control programs and the number of results reported for each are given in Tables 6.2 and 6.3. During 1984 control determinations increased by 16%; control programs increased 4%. Analytical work is summarized in Table 6.4.

During this year four internal audits were conducted, and there were five external audits of ACD programs. The division participated in Quality Assurance Week and conducted a customer survey. Six nonconformance reports were filed, and Quality Assurance Plans and Assessments were reviewed and revised. The quality control program was expanded, and quarterly reports prepared for distribution to management and customers. Special control solutions were prepared to meet unusual customer requirements. Quality problems were addressed in the areas of inductively coupled plasma spectroscopy, gamma spectrometry, acid measurement and commercially prepared standards. An improved mercury purification process was developed which exhibited better quality and performance. Backup was provided for certain critical tasks, and an alloy identification system was established to assist Inspection Engineering.

The Quality Assurance Coordinator (QAC) attended workshops on quality assurance assessments and on potential problem analysis. Assistance was given to the American Society for Testing and Materials (ASTM) and to the Institute for Nuclear Materials Management (INMM) in the preparation of analytical chemistry QA standards. An orientation program on QA was presented to new employees, and a quality assurance meeting for the four Energy Systems plants was held at ORNL.

Table 6.1 Distribution of control tests by service laboratory for October 1983 through September 1984

| Laboratory | Number of control results | | 1983 | 1984 |
|---|---|---|---|---|
| | Total | Outside fixed limits | Quality level | Quality level (%)[a] |
| Chemical and Physical Analysis | 2119 | 76 | 93% | 96% |
| Environmental Analysis Laboratory | 785 | 20 | 93% | 97% |
| [b]Low-Level Radiochemical Analysis | 132 | 23 | - | 83% |
| Radioactive Materials Analytical Laboratory | 837 | 22 | 98% | 97% |
| Transuranium Analytical Laboratory | 1627 | 84 | 96% | 95% |
| Total | 5500 | 225 | 95% | 96% |

[a] Quality level, % = $\frac{\text{no. results in control}}{\text{total no. of results}} \times 100$

[b] Began participating in control program 1st quarter 1984.

Table 6.2 Control samples prepared for October 1983 through September 1984

| Method | Number of control elements | Number of control solutions | Concentration range (mg/ml) |
|---|---|---|---|
| Atomic absorption spectrometry | 15 | 15 | 0.002-0.250 |
| Chemical separation/ liq. scintillation counting | 3 | 17 | 1.0E2 to E6 Bq/ml |
| Coulometry | 1 | 3 | 8-12 |
| Count, alpha | 1 | 4 | 0.03-0.05 |
| Fluorometry | 1 | 4 | 0.001-0.010 |
| Gamma-Spectrometry | 13 | 61 | 1.0 E1 to E7 Bq/ml |
| Gas Chromatography | 1 | 3 | 0.001 to 999 mg/l |
| Gravimetry | 3 | 12 | 2-75% |
| Gravimetry (Leco) | 2 | 4 | 100-800 µg/g |
| Ion chromatography | 7 | 4 | 0.001-0.060 |
| Ion selective electrode | 2 | 4 | 0.001-0.100 |
| Inductively coupled plasma (ICP) | 30 | 7 | 0.001-0.1500 |
| Infrared absorption spectrophotometry | 2 | 2 | 0.004-0.100 |
| Spectrophotometry | 8 | 11 | 0.0001-5.0 |
| Volumetric titrimetry | 13 | 34 | (acid)0.1 - 7 Normal, 2-120 |
| X-ray fluorescence spectrometry | 4 | 3 | 0.50-150 |
| Total | 106 | 188 | |

**Table 6.3 Distribution of control results by method for October 1983 through September 1984**

| Method of analysis | Number of control elements | Number of control results |
|---|---|---|
| Atomic absorption spectrometry | 15 | 365 |
| Chemical separation and liquid scintillation counting | 3 | 91 |
| Coulometry | 1 | 50 |
| Count, alpha | 1 | 368 |
| Fluorometry | 1 | 233 |
| Gamma spectrometry | 13 | 1242 |
| Gas chromatography | 1 | 1 |
| Gravimetry | 3 | 26 |
| Gravimetry (Leco) | 2 | 62 |
| Ion chromatography | 7 | 905 |
| Ion selective electrode | 2 | 4 |
| Inductively coupled plasma (ICP) | 30 | 1172 |
| Infrared absorption spectrophotometry | 2 | 13 |
| Spectrophotometry | 8 | 165 |
| Volumetric titrimetry | 13 | 718 |
| X-ray fluorescence | 4 | 85 |
|  | 106 | 5500 |

## SAFETY

The safety policy of the Analytical Chemistry Division is described in the following statements:

1. Conduct all activities with the lowest reasonable risk of personal injury or property loss due to preventable accidents.
2. Perform all work and maintain the working environment in accordance with laboratory safety regulations and designated national codes and standards.
3. Investigate all significant accidents, determine their causes, and take steps to prevent recurrence of similar accidents.
4. To improve employee awareness of safety while off the job as well as on the job.

The Analytical Chemistry Division has had only one (1) first aid case this year, no recordable injuries/illnesses, no lost workday cases, and to date, only three (3) off-the-job injuries. The employees have accumulated 2,644,009 workday hours since the last lost time accident in 1972. This record is possible because of the excellent safety awareness program within the division.

All division employees are requested to attend a safety meeting each quarter. During this reporting period, over 90% of the employees attended such a meeting.

The division conducted the following safety training and orientation workshop this year:

1. ACD Local Emergency Organization, Fire & CPR.
2. Health Physics Orientation, New Employees.
3. QA Orientation, New Employees.
4. ACD General Orientation Workshop. This included hazardous materials, radiation control, affirmative action and the division's open door program.

Quarterly safety audits were made by the division's safety committee and the division safety officer.

This division maintains a surveillance program that will reveal and correct conditions or deficiencies in the physical layout of equipment or activities which could result in injury to personnel or property damage.

## RADIATION SAFETY

During the year, three analytical chemistry facilities, Buildings 2026, 3019-B and 2011, have been reviewed by the Radioactive Operations Committee.

The goal of the division is to reduce radiation exposure to as low as reasonably achievable (ALARA). Five new laboratory employees were trained at Radioactive Materials Analytical Laboratory (RMAL) in hot cell, glove box and radiochemical procedures and techniques. They also received health physics training. All personnel at Transuranium Laboratory (TRU) and RMAL now wear personnel radiation monitors with audible indicators. New lead-lined glove boxes have been installed and are now in use at TRU. All new employees received a radiation safety orientation.

Other radiation-related safety activities for RMAL included installation of emergency lights in the radiochemical laboratories, crash bars on laboratory doors and automatic controls and switching devices for ventilation systems.

Radiation exposure averaged 580 mR/my at TRU and 160 mR/my at RMAL compared to the DOE maximum allowable exposure of 5000 mr/my.

The division is in compliance with the new Department of Transportation regulations regarding transfer of radioactive and hazardous materials between Oak Ridge installations.

Table 6.4 Summary of analytical work

| Organization | Number of results reported by | | | | | |
|---|---|---|---|---|---|---|
| | Mass and Emission Spectrometry | Transuranium Laboratory | Radioactive Materials Analytical Laboratory | Bio/Organic Analysis | General and Environmental Analysis | Total |
| ORNL Unit | | | | | | |
| Analytical Chemistry | 95 | | 115 | 73 | 7,548 | 7,831 |
| Biology | 110 | | 9 | 19 | 520 | 658 |
| Central Management | | | | | 6,695 | 6,695 |
| Chemical Technology | 4,199 | 19,554 | 3,597 | 60 | 11,637 | 39,047 |
| Chemistry | 181 | | | 22 | 2,201 | 2,404 |
| Energy | | | | | 912 | 912 |
| Engineering Physics | 30 | | | | 26 | 56 |
| Engineering Technology | 1,347 | | 171 | | 8,034 | 9,552 |
| Environmental and Occupational Safety | 105 | | | 590 | 15,601 | 16,296 |
| Environmental Sciences | 148 | | 55 | 1,156 | 124,703 | 126,062 |
| Finance and Materials | | | 82 | | 36 | 118 |
| Fuel Recycle | 155 | | 3,042 | | 602 | 3,799 |
| Fusion Energy | 334 | | | | 149 | 483 |
| Health | | | 10 | 8 | 568 | 586 |
| Health and Safety Research | 147 | | | 73 | 4,022 | 4,242 |
| Instrumentation and Controls | 15 | | | | 1,134 | 1,149 |
| Laboratory Protection | | | | | 12 | 12 |
| Metals and Ceramics | 5,340 | | 31 | 16 | 2,693 | 8,080 |
| Operations | 14,028 | 11,726 | 1,087 | | 3,298 | 30,139 |
| Physics | 95 | | 176 | | 218 | 489 |
| Plant and Equipment | | | | | 739 | 739 |
| Solid State | 503 | | | 48 | 1,000 | 1,551 |
| Others | | | | | | |
| Protective Coating Testing | | | 501 | | | 501 |
| K-25 and Y-12 | 3,578 | | 360 | | 6,116 | 10,054 |
| Miscellaneous | 942 | | 45 | 23 | 247 | 1,257 |
| Savannah River Laboratory | | | 155 | | | 155 |
| Work for Others | | 779 | 484 | 12 | 52 | 1,327 |
| TOTAL | 31,352 | 32,059 | 9,920 | 2,100 | 198,763 | 274,194 |

# 7. SUPPLEMENTARY ACTIVITIES

The division continues to maintain liaison with the academic community through its Advisory Committee, consultants, and various ORNL and ORAU programs. We also make facilities available for student and faculty research and training programs.

## ADVISORY COMMITTEE

The cycle for the division's Information Meeting has been extended to 18 months; the next meeting will be in July 1985. The 1984 Advisory Committee was composed of:

**J. W. Frazer**, 6767 Alisal Street, Pleasanton, CA.
**J. G. Grasselli**, Standard Oil Company, 4440 Warrensville Center Road, Cleveland, OH.
**M. V. Novotny**, Chemistry Department, Indiana University, Bloomington, IN.
**J. W. Taylor**, Chemistry Department, University of Wisconsin, Madison, WI.
**R. G. Wymer**, Chemical Technology Division, Oak Ridge National Laboratory, P.O. Box X, Oak Ridge, TN.

## CONSULTANTS

The following specialists were brought to ORNL on a short-term consulting basis this past year as part of our Seminar Program. Details of seminars are listed in Chapter 8.

**J. Harris**, University of Utah, Salt Lake City, UT.
**D. Johnson**, Environmental Sciences Division, ORNL, Oak Ridge, TN.
**N. R. Armstrong**, University of Arizona, Tucson, AZ.
**B. R. Kowalski**, University of Washington, Seattle, WA.
**R. J. Hessler**, Sea Coast Scientific, Stanford, CT.
**D. Gere**, Hewlett Packard Co., Avondale, PA.
**G. D. Owens**, Proctor and Gamble Co., Cincinnatti, OH.
**J. C. Giddings**, University of Utah, Salt Lake City, UT.

G. Mamantov and M. J. Sepaniak of the University of Tennessee continued working in the division as adjunct research participants this year.

## PARTICIPATION IN ORNL IN-HOURS PROGRAM

The following divisional people completed courses during the 1984 winter term of the Continuing Education Program for Scientific and Technical Personnel:

**Partial Differential Equations:** L. D. Hulett.
**Technical Writing:** M. P. May.
**MAPPER:** D. E. Goeringer.

The following courses were completed during the spring 1984 term:

**Selected Topics of DECsystem-10:** C. S. Alley.
**Introduction to FORTRAN:** S. K. Holladay.
**Intermediate FORTRAN:** S. K. Holladay.
**TELL-A-GRAF:** L. M. Roseberry.
**Artificial Intelligence:** W. D. Shults and H. H. Ross.

## STUDENT ASSIGNMENTS

B. A. Tomkins served as "Dean" for the division's student guests during 1984. Brisco Harvard, a predoctoral student from the University of Tennessee, continued his work in the Analytical Methodology Section as an ORAU graduate research participant.

The division maintains a subcontract with the University of Tennessee to enable advanced graduate students to participate in environmental analysis programs. Chris P. Leibman worked with P. J. Todd on organic SIMS studies. Also under this contract, C. N. Kettler worked with Mike Maskarinec to develop an electron capture detector for open tubular liquid chromatography.

The division was host to three ORAU graduate research participants this year. J. R. Parrish worked with Juel Emery performing neutron activation analysis at the HFIR reactor. B. L. Harvard continued his work in the Analytical Methodology Section. L. C. Baylor worked in the Bio/Organic Analysis Section with John Caton on developing an internal standardization technique for diffuse reflectance infrared Fourier transform (DRIFT).

## SUMMER PROGRAM

During the summer, Alison R. Kean, a representative of the Historically Black College and University Program, worked with Mike Maskarinec on high resolution chromatography development.

Greg Hurst, who was previously a CO-OP in the division, worked as a summer research intern with John Caton on the characterization of complex mixtures by chromatographic and spectroscopic methods.

## ORAU POSTGRADUATE RESEARCH TRAINING PROGRAM

G. S. Groenewold worked in the organic mass spectrometry program at Y-12.

W. G. Tong conducted postdoctoral research in laser analytical chemistry in the Analytical Methodology Section.

## SPECIAL AWARDS

W. R. Laing received an ASTM award for Editorial Excellence in July for recognition of his outstanding contribution to the editorial excellence of the standards promulgated by his committee.

J. H. Stewart received an ORNL Environmental Protection Award for collaborative Analytical Chemistry—Chemistry Division effort in developing a production scale method for silver recovery.

## 27th ORNL/DOE CONFERENCE ON ANALYTICAL CHEMISTRY IN ENERGY TECHNOLOGY

This year's conference was held at the Knoxville Hilton, October 1-4, 1984. The theme of the conference was "Analysis at the Energy Environment Interface". Attendance was 169, with representatives from foreign countries, academia, industrial institutions, and other government agencies. There were 18 exhibits (30 representatives) and 17 booths. Topics emphasized during the meeting included lasers, mass spectrometry, and nuclear and plasma spectroscopy.

A. L. Harrod was Arrangements Chairman, W. S. Lyon was Technical Program Chairman, and J. M. Holladay was Treasurer. Shirley Cates and Gail Vineyard were Conference Secretaries. Other conference committee members included B. R. Clark, W. R. Laing, L. N. Klatt, W. D. Shults, J. H. Stewart, and P. J. Todd.

## ANALYTICAL CHEMISTRY DIVISION SEMINARS AT ORNL

| Speaker | Title | Date |
|---|---|---|
| Harris, J.<br>Univ. of Utah, Salt Lake City, UT | "Laser Based Thermo-optical Methods for Trace Chemical Detection" | Jan. 19, 1984 |
| Johnson, D.<br>Environmental Sciences Division, ORNL | "Effects of Acid Rain Deposition of Forest Soils and Nutrient Cycling" | Apr. 25, 1984 |
| Armstrong, N. R.<br>Univ. of Arizona, Tucson, AZ | "Hydrogen Uptake and Release in Titanium Thin Films and Li/$SO_2$ Surface Chemistry Studies by Electron Spectroscopy, Rutherford Backscattering Spectroscopy and Quartz Crystal Microgravimetry" | May 3, 1984 |
| Kowalski, B. R.<br>Univ. of Washington, Seattle, WA | "Chemometrics and Process Analytical Chemistry" | June 4, 1984 |

| | | |
|---|---|---|
| Hessler, R. J.<br>Sea Coast<br>Scientific,<br>Stamford, CT | "A Laser Ionization Mass Analyzer" | Aug. 3, 1984 |
| Gere, D.<br>Hewlett Packard<br>Co.,<br>Avondale, PA | "Chromatography with Supercritical Carbon Dioxide" | Aug. 28, 1984 |
| Owens, G. D.<br>Proctor and<br>Gamble Co.,<br>Cincinnati, OH | "Applications of Robotics in Analytical Chemistry | Sep. 14, 1984 |
| Giddings, J. C.<br>Univ. of Utah,<br>Salt Lake City, UT | "Separation and Characterization of Macromolecules and Particles by Field-Flow Fractionation" | Nov. 15, 1984 |

## ADDITIONAL PROFESSIONAL ACTIVITIES

**ATTRILL, J. E.**

| | |
|---|---|
| Advisory Committee: | Subcommittee E-38.01 on Energy, ASTM |
| Member: | ASTM Committee E-38 on Resource Recovery<br>ORNL Emergency Oil Spill Response Team |
| Sample Coordinator: | ACD/Department of Environmental Management |

**BRAZELL-RAMSEY, R. S.**

| | |
|---|---|
| Chairman: | ACD Seminar Committee |

**BROOKSBANK, R. D.**

| | |
|---|---|
| Member: | ASTM Committee D-33, Protective Coating and lining Work for Power Generation Facilities |
| Division Assistant Representative: | Material Balance Area Representative for Nuclear Materials Management |
| Co-organizer and Session Chairman: | Seminar on Decontamination of Nuclear Facilities, ASTM D-33, May 1984 |

**BUCHANAN, M. V.**

| | |
|---|---|
| Member: | ORAU Traveling Lecture Program |

**CARTER, J. A.**

| | |
|---|---|
| Advisory Board: | *Analytical Chemistry* |
| Advisory Panel Member: | DOE/ISA Laboratory Advisory Group for Effluent Research (LACER) |
| Laboratory Coordinator: | ISPO Programs |
| Secretary: | Subcommittee C-5:05, Analytical Task Group (Reactor Grade Graphite), Committee C-5, Manufacturer of Carbon and Graphite Products, ASTM |

**CATON, J. E.**

| | |
|---|---|
| Member: | ACD Long Range Planning Committee |

**CHRISTIE, W. H.**
Member: ACD Long Range Planning Committee

**COSTANZO, D. A.**
Member: ASTM Committee C-26, Nuclear Fuel Cycle, and Subcommittee C-26:05, Test Methods
ACD Long Range Planning Committee

**DALE, J. M.**
Member: ACD Seminar Committee
ACD Solids/Surfaces Characterization Team

**DONOHUE, D. L.**
Lecturer: ORAU Traveling Lecture Program

**DYER, F. F.**
Member: ACD Seminar Committee

**ELDRIDGE, J. S.**
Member: ACD Long Range Planning Committee
Lab Committee 8 of the International Society Committee for the Methods of Air Sampling and Analysis
Steering Committee, Environmental Radiation Section of Health Physics Society

**EMERY, J. F.**
Division Representative: Laboratory Emergency Sample Coordinator
Division Computer Systems Security Officer

**FELDMAN, C.**
Fellow: American Society for Testing and Materials
Member: Committee E-2 on Emission Spectroscopy, ASTM, Subcommittees on Fundamental Methods, Editorial Practices, and Nomenclature
Scientific Editor: *Optics and Spectroscopy* (English translation of Russian journal, *Optika i Spektroskopita*)

**FERGUSON, N. M.**
Division Representative: Energy Systems Environmental Analysis Committee

**GLISH, G. L.**
Chairman: American Society for Mass Spectrometry History Committee
Lecturer: ORAU Traveling Lecture Program

**GOERINGER, D. E.**
Member: ACD Seminar Committee

**GRIEST, W. H.**
Consultant: Department of Energy - BES
Electric Power Research Institute
Environmental Protection Agency - IERL/TSO

| | |
|---|---|
| **GUERIN, M. R.** | |
| Consultant: | Department of Energy (OHER) |
| | Pacific Northwest Laboratory Program Review |
| | Complex Mixtures Program Chemistry Representative |
| | Analytical Methodology Program Panel |
| | National Institutes of Health |
| | NIDA Analytical Support of Drug Research Proposal Review Panel |
| | Department of Energy (FE) |
| | Pacific Northwest Laboratory Program Review |
| | International Research and Development Corporation Program Review |
| | Environmental Protection Agency |
| | Mutagenicity Sample Preparation Protocol Development Panel |
| | Division Representative |
| | DOE Chemical Defense Waste Program |
| **HIGGINS, C. E.** | |
| | ACD Ombudsman |
| **HOLMBERG, R. W.** | |
| Coordinator: | ACD Metric Coordinator |
| **HOWELL, P. L.** | |
| Coordinator: | ACD Quality Control Program |
| **HULETT, L. D.** | |
| Coordinator: | ACD Solids/Surfaces Characterization Team |
| **JENKINS, R. A.** | |
| Consultant: | National Institute of Drug Abuse - Exposure of Animals to Concentrated Smokes |
| Member: | ORNL Speaker's Bureau |
| **JONES, R. E.** | |
| Chairman: | Task Group on Methods of Coatings Analysis D-33 |
| Division Representative: | ACD Radiation Control Officer |
| | Respirator Trainer |
| Member: | ASTM Committee D-33, Protective Coating and Lining Work for Generation Facilities |
| **KELLER, J. M.** | |
| Member: | ACD Seminar Committee |
| | Digital Equipment Company Users Society (DECUS) |
| **KLATT, L. N.** | |
| Member: | Energy Systems, Ph. D. Recruiting Team |
| | ACD Long Range Planning Committee |
| | Planning Committee, 27th ORNL/DOE Conference on Analytical Chemistry in Energy Technology, Knoxville, TN, October 2-4, 1984 |
| Secretary: | ACS Division of Analytical Chemistry |

**LAING, W. R.**

| | |
|---|---|
| Chairman: | Subcommittee C26:05, Test Methods, Nuclear Fuel Cycle, ASTM |
| | ASTM Long Range Planning Committee |
| Coordinator: | ACD Quality Assurance Program |
| Fellow: | American Society for Testing and Materials |
| Member: | ASTM Committee D-33, Protective Coatings |
| | ASTM Committee on Technical Committee Operations |
| | ISO Technical Committee 85, Subcommittee 5 |
| | ORNL Pregrievance Committee |
| | Planning Committee, 27th ORNL/DOE Conference on Analytical Chemistry in Energy Technology, Knoxville, TN, October 2-4, 1984 |
| | INMM Group 5.1, Analytical Chemistry Laboratory Measurement Control |
| | Energy Systems-GAT Analytical Committee |
| Vice-chairman: | ASTM Committee C26, Nuclear Fuel Cycle |

**LYON, W. S.**

| | |
|---|---|
| Advisory Board: | *Analyst* |
| Chairman: | Honors & Awards Committee, Isotopes & Radiation Division, ANS |
| Fellow: | American Nuclear Society |
| Honorary Member: | Committee E-10, Nuclear Technology and Applications, ASTM |
| Member: | Organizing Committee, 7th International Conference on Modern Trends in Activation Analysis |
| | Scientific Committee 25 on Radiation Protection in the Use of Small Neutron Generators, NCRP |
| | Technical Program Committee, 5th International Conference on Nuclear Methods in Environmental and Energy Research, Mayaquez, Puerto Rico, April 2-7, 1984 |
| Regional Editor: | *Journal of Radioanalytical and Nuclear Chemistry* |
| Technical Program Chairman: | 27th ORNL/DOE Conference on Analytical Chemistry in Energy Technology, Knoxville, TN, Oct. 2-4, 1984 |

**MASKARINEC, M. P.**

| | |
|---|---|
| Member: | ASTM E-19 Committee on Chromatography |
| | ASTM D-34 on Waste Disposal |

**McKOWN, H. S.**

| | |
|---|---|
| Division Representative: | Interplant Computer Communications Committee |

**RAMSEY, J. M.**

| | |
|---|---|
| Member: | ACD Seminar Committee |

**ROSS, H. H.**

| | |
|---|---|
| Chairman: | ORNL Graduate Student Selection Panel |
| | ORNL Post Doctorate Selection Panel |
| Member: | ACD Seminar Committee |
| | Editorial Advisory Board, *Journal of Radioanalytical and Nuclear Chemistry* |
| | Fellowship Committee, ACS, Division of Analytical Chemistry |
| | ORNL Technology Utilization Committee |

**ROSSEEL, T. M.**

Member:
ACD Seminar Committee
Program Committee, 11th Annual FACSS Meeting
Scholarship Committee, Tennessee Valley Chapter, American Vacuum Society
ACD Solids/Surfaces Characterization Team

Chairman and Organizer:
Session on "Analysis and Characterization of Surfaces," 4th Annual Tennessee Valley Chapter, AVS Symposium, Knoxville, TN, May 1-3, 1984
Session on "New Directions in X-ray Analysis," 11th Annual FACSS Meeting, Philadelphia, PA, Sept. 16-21, 1984.

**SCOTT, T. G.**

Member:
Committee D-19, Water, ASTM, and Subcommittee 4, Methods of Radiochemical Analysis
Health Physics Society

**SHULTS, W. D.**

Consultant:
Energy Systems Career Planning Program

Member:
Advisory Committee, "Analytical Approach," *Analytical Chemistry*
Canvassing Committee for the ACS Award for Creative Advances in Environmental and Technology
Editorial Board, *Analytica Chimica Acta*
ORNL In-House Continuing Education Committee
Technical Services Committee, Energy Systems

Past President:
Sigma Xi RESA, Oak Ridge Chapter

**SMITH, D. H.**

Chairman
American Society of Mass Spectrometry Isotopes Ratio Committee

Coordinator:
ACD PhD Recruiting

Member:
ACD Long Range Planning Committee

Symposium Organizer:
Symposium on Frontiers in Isotope Ratio Measurements, 33rd Annual Conference on Mass Spectrometry and Applied Topics, San Diego, CA, May 1985

**STEWART, J. H., Jr.**

Division Representative:
ACD Environmental Protection Officer

Member:
ACD Solids/Surfaces Characterization Team Fluorescence Methods
Manuscript Review Board for *Analytical Chemistry*
Microbeam Analysis Society
Planning Committee, 27th ORNL/DOE Conference on Analytical Chemistry in Energy Technology, Knoxville, TN, October 2-4, 1984

Task Leader:
ASTM C-26.02 Nuclear Fuel Cycle Task: C-26.05 X-Ray
International Working Group "Analytical Standards of Minerals, Ores and Rocks"

**STOKELY, J. R.**

Coordinator:
ACD MS/BS Recruiting

**TODD, P. J.**
Member: Planning Committee, 27th Annual DOE/ORNL Conference on Analytical Chemistry in Energy Technology, Knoxville, TN, October 2-4, 1984
Coordinator: ACD Awards

**TOMKINS, B. A.**
Coordinator: ACD University Relations
ACD Family Day 1984

**WALKER, R. L.**
Advisory Panel Member: DOE/ISA Laboratory Advisory Group for Effluent Research (LAGER)

**WHITTEN, W. B.**
Lecturer: ORAU Traveling Lecture Program

**YOUNG, J. P.**
Fellow: American Association for the Advancement of Science

Materials analyzed this year have been Al, $SiO_2$, Ti, W, and polyimide encapsulating coatings. Our outside customers were Texas Instruments, DuPont, Cominco Electronic Materials, Harbison-Walker, IBM, and Varian.

Some other requests were for the determination of $O_2$ in sodium metal by the Air Force and radon emanation from concrete by the Navy.

*(F. F. Dyer, L. Robinson, and L. D. Bible)*

# 8. PRESENTATION OF RESEARCH RESULTS

As in past years, the division has actively responded to the changing priorities of the ORNL research effort by changing the emphasis of some of its own programs or instituting new studies. The increasing concern with energy - nuclear as well as non-nuclear - is reflected in the research results listed below. The multidisciplinary approach required in many such problems is indicated by the number of papers and talks coauthored by members of other ORNL divisions. Such persons are designated by an asterisk.

## PUBLICATIONS

### Contributions to Books, Proceedings, and Reports

| AUTHOR | AUTHOR(S) AND TITLE | PUBLISHED IN |
|---|---|---|
| Brooksbank, R. D. | Brooksbank, R. D.; Laing, W. R. "Radiation Tolerance Testing of Organic Materials" | *Trans. of 1984 Annu. Meet., Am. Nucl. Soc.* Vol. 46, p. 363, 1984 |
| Buchanan, M. V. | Buchanan, M. V.; Olerich, G. "Differentiation of Isomeric Polycyclic Aromatics Using Negative Chemical Ionization" | *Proc., 32nd Annu. Conf. on Mass Spectrom. and Allied Topics*, San Antonio, 1984, p. 65 |
| Carter, J. A. | Glish, G. L.; McLuckey, S. A.; Carter, J. A. "Mass Spectrometry/Mass Spectrometry as a Screening Method for Detecting Explosives Vapors" | *Proc., 25th Annu. Conf., INMM*, Columbus, OH, July 15-18, 1984, p. 216 |
| Christie, W. H. | Christie, W. H.; Taylor, D. H.;* Eby, R. E. "SIMS Study of Compositional Changes Observed in $PuO_2$ Heat Source Cladding Alloy" | *Proc., 32nd Annu. Conf. on Mass Spectrom. and Allied Topics*, San Antonio, TX, May 27-June 1, 1984, p. 614 |
|  | Christie, W. H.; Taylor, D. H.;* Eby, R. E.; Pavone, D.* "SIMS Study of Compositional Changes Observed in a $PuO_2$ Heat Source Cladding Alloy" | *Analytical Spectroscopy*, W. S. Lyon, ed., Elsevier Science Publishers, Amsterdam, 1984, p. 167 |
| Dale, J. M. | Hulett, L. D.; Dale, J. M.; Miller, P. D.;* Moak, C. D.;* Pendyala, S.;* Triftshauser, W.;* Howell, R. H.;* Alvarez, R. A.* "The Generation of Monoenergetic Positrons" | *Positron Scattering in Gases*, John W. Humberston and M. R. C. McDowell, eds., Plenum Press, 1984, p. 195 |
|  | Hulett, L. D.; Dale, J. M.; Pendyala, S.* "The Generation of Monoenergetic Positrons and Some Potential Applications in Materials Science" | *Materials Science Forum*, Vol. 2, Trans. Tech. Publ., Switzerland, 1984, p. 133 |

| AUTHOR | AUTHOR(S) AND TITLE | PUBLISHED IN |
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| Dale, J. M. | Rosseel, T. M.; Dale, J. M.; Hulett, L. D.; Krause, H. F.;* Raman, S.;* Vane, C. R.;* Young, J. P. "Heavy-Ion-Induced X-ray Satellite Emission as a Chemical Probe" | *Analytical Spectroscopy*, W. S. Lyon, ed., Elsevier Science Publishers, Amsterdam, 1984, p. 249 |
| Donohue, D. L. | Donohue, D. L.; Smith, D. H.; Young, J. P.; Ramsey, J. M. "Isotopic Bias Effects in Resonance Ionization Mass Spectrometry" | *Resonance Ionization Spectroscopy 1984*, G. S. Hurst and M. G. Payne, eds., The Institute of Physics, Boston, 1984, p. 83 |
|  | Young, J. P.; Donohue, D. L.; Smith, D. H. "Assessment of Resonance Ionization Mass Spectrometry for Analytical Chemistry and Spectroscopy" | *Resonance Ionization Spectroscopy 1984*, G. S. Hurst and M. G. Payne, eds., The Institute of Physics, Boston, 1984, p. 127 |
|  | Young, J. P.; Donohue, D. L.; Smith, D. H. "Resonance Ionization Mass Spectrometry" | *Proc., 35th Pittsburgh Conf. on Anal. Chem. and Appl. Spectrosc.*, Atlantic City, NJ, March 5-9, 1984, p. 294 |
|  | Donohue, D. L.; Young, J. P.; Smith, D. H. "Isotopic Measurements of Uranium and Plutonium by Resonance Ionization" | *Analytical Spectroscopy*, W. S. Lyon, ed., Elsevier Science Publishers, Amsterdam, 1984, p. 143 |
| Eby, R. E. | Christie, W. H.; Taylor, D. H.;* Eby, R. E. "SIMS Study of Compositional Changes Observed in $PuO_2$ Heat Source Cladding Alloy" | *Proc., 32nd Annu. Conf. on Mass Spectrom. and Allied Topics*, San Antonio, TX, May 27-June 1, 1984, p. 614 |
|  | Christie, W. H.; Taylor, D. H.;* Eby, R. E.; Pavone, D.* "SIMS Study of Compositional Changes Observed in a $PuO_2$ Heat Source Cladding Alloy" | *Analytical Spectroscopy*, W. S. Lyon, ed., Elsevier Science Publishers, Amsterdam, 1984, p. 167 |
| Eldridge, J. S. | Stegnar, P.;* Eldridge, J. S.; Teasley, N. A.; T. W. Oakes* "Environmental Applications for an Intrinsic Germanium Well Detector" | *Analytical Spectroscopy*, W. S. Lyon, ed., Elsevier Sci. Pub., Amsterdam, 1984, p. 313 |
| Glish, G. L. | Glish, G. L.; McLuckey, S. A.; Carter, J. A. "Mass Spectrometry/Mass Spectrometry as a Screening Method for Detecting Explosive Vapors" | *Proc., 25th Annu. Conf., INMM*, Columbus, OH, July 15-18, 1984, p. 216 |

| AUTHOR | AUTHOR(S) AND TITLE | PUBLISHED IN |
|---|---|---|
| Glish, G. L. | Glish, G. L.; Goeringer, D. E.; McLuckey, S. A. "Internal Energy Effects in Low Energy CID" | *Proc., 32nd Annu. Conf. on Mass Spectrom. and Allied Topics*, San Antonio, TX, May 27-June 1, 1984, p. 719 |
| | Goeringer, D. E.; Glish, G. L. "Tandem Quadrupole/Time-of-Flight Instrument for MS/MS Studies" | *Proc., 32nd Annu. Conf. on Mass Spectrom. and Allied Topics*, San June 1, 1984, p. 521 |
| Goeringer, D. E. | Glish, G. L.; Goeringer, D. E.; McLuckey, S. A. "Internal Energy Effects in Low Energy CID" | *Proc., 32nd Annu. Conf. on Mass Spectrom. and Allied Topics*, San Antonio, TX, May 27-June 1, 1984, p. 719 |
| | Goeringer, D. E.; Glish, G. L. "Tandem Quadrupole/Time-of-Flight Instrument for MS/MS Studies" | *Proc., 32nd Annu. Conf. on Mass Spectrom. and Allied Topics*, San Antonio, TX, May 27-June 1, 1984, p. 521 |
| Griest, W. H. | Ho, C.-h.; Ma, C. Y.; Griest, W. H.; Guerin, M. R. "Preparative Scale Organic Extraction and Chemical/Biological Analysis of Stack and ESP Hopper Ash" | ACS Preprints, *Div. Environ. Chem.* 24(2), 1984, p. 68 |
| Guerin, M. R. | Ho, C.-h.; Ma, C. Y.; Griest, W. H.; Guerin, M. R. "Preparative Scale Organic Extraction and Chemical/Biological Analysis of Stack and ESP Hopper Ash" | ACS Preprints, *Div. Environ. Chem.* 24(2), 1984, p. 68 |
| Ho, C.-h. | Ho, C.-h.; Ma, C. Y.; Griest, W. H.; Guerin, M. R. "Preparative Scale Organic Extraction and Chemical/Biological Analysis of Stack and ESP Hopper Ash" | ACS Preprints, *Div. Environ. Chem.* 24(2), 1984, p. 68 |
| Hulett, L. D. | Hulett, L. D.; Dale, J. M.; Pendyala, S.* "The Generation of Monoenergetic Positrons and Some Potential Applications in Materials Science" | *Materials Science Forum* Vol. 2, Trans. Tech. Publ., Switzerland, 1984, p. 133 |
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| | Rosseel, T. M.; Dale, J. M.; Hulett, L. D.; Krause, H. F.;* Raman, S.;* Vane, C. R.;* Young, J. P. "Heavy-Ion-Induced X-ray Satellite Emission as a Chemical Probe" | *Analytical Spectroscopy*, W. S. Lyon, ed., Elsevier Science Publishers, Amsterdam, 1984, p. 294 |

| AUTHOR | AUTHOR(S) AND TITLE | PUBLISHED IN |
|---|---|---|
| Laing, W. R. | Brooksbank, R. D.; Laing, W. R. "Radiation Tolerance Testing of Organic Materials" | *Trans. of 1984 Annu. Meet., Am. Nucl. Soc.* Vol. 46, p. 363, 1984 |
| Lyon, W. S. | Lyon, W. S. (ed.) *Analytical Spectroscopy* | Elsevier Science Publishers, Amsterdam, Netherlands, 1984 |
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| McLuckey, S. A. | Glish, G. L.; McLuckey, S. A.; Carter, J. A. "Mass Spectrometry/Mass Spectronmetry as a Screening Method for Detecting Explosive Vapors" | *Proc., 25th Annu. Conf., INMM*, Columbus, OH, July 15-18, 1984, p. 216 |
| | McLuckey, S. A.; van der Zande, W.;* de Bruijn, D. P.;* Los, J.;* Kistemaker, P. G.* "Scattering in Dissociative Collisions of Ionized Acetone with Helium" | *Proc., 32nd Annu. Conf. on Mass Spectrom. and Allied Topics*, San Antonio, TX, May 27-June 1, 1984, p. 715 |
| | Glish, G. L.; Goeringer, D. E.; McLuckey, S. A. "Internal Energy Effects in Low Energy CID" | *Proc., 32nd Annu. Conf. on Mass Spectrom. and Allied Topics*, San Antonio, TX, May 27-June 1, 1984, p. 719 |
| Olerich, G. | Buchanan, M. V.; Olerich, G. "Differentiation of Isomeric Polycyclic Aromatics Using Negative Chemical Ionization" | *Proc., 32nd Annu. Conf. on Mass Spectrom. and Allied Topics*, San Antonio, TX, 1984, p. 65 |
| Ramsey, J. M. | Donohue, D. L.; Smith, D. H.; Young, J. P.; Ramsey, J. M. "Isotopic Bias Effects in Resonance Ionization Mass Spectrometry" | *Resonance Ionization Spectroscopy 1984*, G. S. Hurst and M. G. Payne, eds., The Institute of Physics, Boston, 1984, p. 83 |
| Rosseel, T. M. | Rosseel, T. M.; Dale, J. M. Hulett, L. D.; Krause, H. F.;* Raman, S.;* Vane, C. R.;* Young, J. P. "Heavy-Ion-Induced X-ray Satellite Emission as a Chemical Probe" | *Analytical Spectroscopy*, W. S. Lyon, ed., Elsevier Science Publishers, 1984, p. 294 |
| Shaw, R. W. | Shaw, R. W.; Howell, H. E.;* Mamantov, G;* Wehry, E. L.* "Matrix-Isolation Photoacoustic Spectroscopy" | *Analytical Spectroscopy*, W. S. Lyon, ed., Elsevier Science Publishers, Amsterdam, 1984, p. 37 |

| AUTHOR | AUTHOR(S) AND TITLE | PUBLISHED IN |
|---|---|---|
| Smith, D. H. | Donohue, D. L.; Smith, D. H.; Young, J. P.; Ramsey, J. M. "Isotopic Bias Effects in Resonance Ionization Mass Spectrometry" | *Resonance Ionization Spectroscopy 1984*, G. S. Hurst and M. G. Payne, eds., The Institute of Physics, Boston, 1984, p. 83 |
| | Young, J. P.; Donohue, D. L.; Smith, D. H. "Assessment of Resonance Ionization Mass Spectrometry for Analytical Chemistry and Spectroscopy" | *Resonance Ionization Spectroscopy 1984*, G. S. Hurst and M. G. Payne, eds., The Institute of Physics, Boston, 1984, p. 127 |
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| | Bayne, C. K.;* Smith, D. H. "A New Method for Estimating Isotopic Ratios from Pulse-Counting Mass Spectrometric Data" | *Proc., 32nd Annu. Conf. on Mass Spectrom. and Allied Topics*, San Antonio, TX, May 27-June 1, 1984, p. 406 |
| | Donohue, D. L.; Young, J. P.; Smith, D. H. "Isotopic Measurements of Uranium and Plutonium by Resonance Ionization" | *Analytical Spectroscopy*, W. S. Lyon, ed., Elsevier Science Publishers, Amsterdam, 1984, p. 143 |
| Teasley, N. A. | Stegnar, P.;* Eldridge, J. S.; Teasley, N. A.; T. W. Oakes* "Environmental Applications for an Intrinsic Germanium Well Detector" | *Analytical Spectroscopy*, W. S. Lyon, ed., Elsevier Sci. Pub., Amsterdam, 1984, p. 313 |
| Todd, P. J. | Groenewold, G. S.;* Todd, P. J. "Detection/Identification of Volatile Organic Phosphonates and Phosphates by SIMS" | *Proc., 32nd Annu. Conf. on Mass Spectrom. and Allied Topics*, San Antonio, TX, May 27-June 1, 1984, p. 12 |
| | Todd, P. J.; Groenewold, G. S.* "Gas Phase Molecular SIMS" | *Proc., 32nd Annu. Conf. on Mass Spectrom. and Allied Topics*, San Antonio, TX, May 27-June 1, 1984, p. 247 |

| AUTHOR | AUTHOR(S) AND TITLE | PUBLISHED IN |
|---|---|---|
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## ORAL PRESENTATIONS

As in previous years, staff members have made presentations at local, national, and, in a few instances, international meetings. The papers covered a wide variety of subjects, reflecting the division's broad spectrum of activities.

### At Meetings of Professional Societies, Conferences, and the Like

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| Andrews, M. C.* | Andrews, M. C.;* McDaniel, F. D.;* Duggan, J. L.;* Mehta, R.;* Miller, P. D.;* Krause, H;* Pepmiller, P. L.;* Rosseel, T. M.; Rayburn, L. A.;* Lapicki, G.* "L Shell X-ray Production Cross Sections in Nd, Gd, Ho, Yb, Au and Pb for Incident 25 MeV Carbon and 32 MeV Oxygen Ions" | 8th Conf. on the Application of Accelerators in Res. and Ind., Denton, TX, Nov. 12-14, 1984 |
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| Attrill, J. E. | Attrill, J. E.; Stewart, J. H.; Rogers, W. F.; Webb, K. I. "Problems and Remedies in the Measurement of PCB in Environmental Materials" | Am. Chem. Soc. Reg. Meet., Raleigh, NC, Oct. 24-26, 1984 |
| Aziz, M. J.* | Aziz, M. J.;* Lowndes, D. H.;* Jellison, G. E.;* White, C. W.;* Thompson, M.O.;* Peercy, P. S.;* Christie, W. H. "A Search for Solute Drag" | Annu. Meet. Mater. Res. Soc., Boston, MA, Nov. 26-30, 1984 |
| Aziz, M. J.* | Aziz, M. J.;* Nygren, E.;* Christie, W. H.; White, C. W.* "Measurement of Low-Temperature Self-Diffusion in Intrinsic Silicon" | Annu. Meet. Mater. Res. Soc., Symp. on Diffusion and Gettering in Semiconductors, Boston, MA, Nov. 26-30, 1984 |
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| Buchanan, M. V. | Buchanan, M. V.; Guerin, M. R. "Development of Analytical Methods for the Unambiguous Identification of Organics at the Isomeric Level" | OHER Meas. Sci. Workshop, Battelle Seattle Conf. Ctr., Seattle, WA, May 14-17 1984 |
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| | Christie, W. H.; Taylor, D. H.;* Eby, R. E. Pavone, D.* "SIMS Study of Compositional Changes Observed in a $PuO_2$ Heat Source Cladding Alloy" | 4th Annu. Tenn. Valley Chapt., Am. Vac. Soc., Knoxville, TN, May 1-2, 1984 |
| | Christie, W. H. "SIMS Study of Compositional Changes Observed in a $PuO_2$ Heat Source Cladding Alloy" | Anal. Chem. Div. Inf. Meet., Oak Ridge Nat. Lab., Oak Ridge, TN, Jan. 30-Feb. 1, 1984 |
| | Christie, W. H.; Donohue, D. L.; Goeringer, D. E.; McKown, H. S. "Resonance Ionization Mass Spectrometry for Ultra Trace Analysis" | ISA-LAGER Committee, Washington, DC, Oct. 24, 1984 |

| SPEAKER | AUTHOR(S) AND TITLE | PRESENTED AT |
|---|---|---|
| Costanzo, D. A. | Costanzo, D. A. "Analytical Chemistry Development for CFRP" | CFRP Task Rev., Oak Ridge Nat. Lab., Oak Ridge, TN, March 13, 1984 |
| | Costanzo, D. A. "Analytical Chemistry Development for CFRP" | USDOE/UKAEA Inf. Exchange Meet., Oak Ridge, TN, March 26, 1984 |
| | Costanzo, D. A. "Analytical Assistance to the Hydrofracture Waste Disposal Program" | Energy Systems, Goodyear Atomic Corp. Anal. Services Meet., Oak Ridge, TN, Nov. 13, 1984 |
| Donohue, D. L. | Donohue, D. L.; Smith, D. H.; Young, J. P.; Christie, W. H. "Isotopic Bias Effects in Resonance Ionization Mass Spectrometry" | 2nd Int. Conf. on Resonance Ionization Spectrom. (RIS), Knoxville, TN, April 20, 1984 |
| | Donohue, D. L.; Smith, D. H.; Young, J. P. Christie, W. H.; Goeringer, D. E. "Resonance Ionization Mass Spectrometry Using a Commercial Ion Microprobe Mass Analyzers" | Invited, 187th Nat. Am. Chem. Soc. Meet., Philadelphia, PA, Aug. 26-31, 1984 |
| | Donohue, D. L.; Christie, W. H.; Goeringer, D. E.; McKown, H. S. "Ultra-Sensitive Isotopic Analysis by Resonance Ionization Mass Spectrometry" | Invited, Int. Conf. on Lasers '84, San Francisco, CA, Nov. 26-30, 1984 |
| | Donohue, D. L.; Young, J. P.; Smith, D. H. "Current Status of Inorganic Mass Spectrometry Research" | BES Chemical Sciences Site Review, Oak Ridge, Nat. Lab., Oak Ridge, TN, Mar. 28, 1984 |
| | Donohue, D. L. "New Directions in Resonance Ionization Mass Spectrometry" | Anal. Chem. Div. Inf. Meet., Oak Ridge Nat. Lab., Oak Ridge, TN, Jan. 30-Feb. 1, 1984 |
| | Donohue, D. L.; Christie, W. H.; Goeringer, D. E. "Measurement of Uranium and Plutonium Isotope Ratios by RIMS" | ISA-LAGER Committee, Washington, DC, Apr. 11, 1984 |
| | Donohue, D. L.; Smith, D. H.; McKown, H. S.; Carter, J. A. "A Proposed Vent Gas Monitor for $UF_6$" | Portsmouth Gaseous Diffusion Plant, Portsmouth, OH, Oct. 10, 1984 |
| Eldridge, J. S. | Eldridge, J. S. "The Use of Bioindicator Organisms and Specialized Analysis Techniques in a Program of Environmental Surveillance" | 29th Annu. Meet. Health Physics Soc., New Orleans, LA, June 3-8, 1984 |

| SPEAKER | AUTHOR(S) AND TITLE | PRESENTED AT |
|---|---|---|
| Ferguson, M. M. | Ferguson, M. M. "Automation in the Determination of Anions" | UCCND-GAT Anal. Comm. Meet., Oak Ridge Nat. Lab, Oak Ridge, TN, March 20, 1984 |
| | Ferguson, M. M. "Data Management and Quality Control" | EPRI Pre-Project Rev., Oak Ridge Nat. Lab., Oak Ridge, TN, April 10, 1984 |
| Francis, C. W.* | Francis, C. W.;* Maskarinec, M. P.; Goyert, J. C.* "A Laboratory Extraction Method to Simulate Co-Disposal of Solid Waste in Landfills" | ASTM Symp. on Solid and Hazardous Waste Testing, Arlington, VA, May 2-4, 1984 |
| Glish, G. L. | Glish, G. L.; Goeringer, D. E. "Internal Energy Effects in Low Energy CID" | 32nd Annu. Conf. on Mass Spectrom. and Allied Topics, San Antonio, TX, May 27-June 1, 1984 |
| | Glish, G. L.; McLuckey, S. A.; Carter, J. A. "Mass Spectrometry/Mass Spectrometry as a Screening Method for Detecting Explosive Vapors" | 25th Annu. Meet., INMM, Columbus, OH, July 15-18, 1984 |
| | Glish, G. L.; Todd, P. J. "The Effect of Cationizing Species on MS/MS Spectra" | Anal. Chem. Div. Inf. Meet., Oak Ridge Nat. Lab., Oak Ridge, TN, Jan. 30-Feb. 1, 1984 |
| | Glish, G. L.; Goeringer, D. E.; McLuckey, S. A. "Current Status of Research in Organic Mass Spectrometry" | BES Chemical Sciences Site Review, Oak Ridge Nat. Lab., Oak Ridge, TN, March 28, 1984 |
| Goeringer, D. E. | Goeringer, D. E.; Glish, G. L. "Tandem Quadrupole/Time-of-Flight Instrument for MS/MS Studies" | 35th Pittsburgh Conf. on Anal. Chem. and Applied Spectrosc., Atlantic City, NJ, March 5-9, 1984 |
| | Goeringer, D. E.; Glish, G. L. "Tandem Quadrupole/Time-of-Flight Instrument for MS/MS Studies" | 32nd Annu. Conf. on Mass Spectrom. and Allied Topics, San Antonio, TX, May 27-June 1, 1984 |
| | Goeringer, D. E.; Glish, G. L. "A Tandem Quadrupole/Time-of-Flight Instrument for MS/MS Studies" | Anal. Chem. Div. Inf. Meet., Oak Ridge Nat. Lab., Oak Ridge, TN, Jan. 30-Feb. 1, 1984 |

| SPEAKER | AUTHOR(S) AND TITLE | PRESENTED AT |
|---|---|---|
| Griest, W. H. | Griest, W. H.; Tomkins, B. A.; MacDougall, C. S.; Harris, L. A.* "Characterization of Carbonaceous Matter Emitted to the Atmosphere" | Anal. Chem. Div. Inf. Meet., Oak Ridge Nat. Lab., Oak Ridge, TN, Jan. 31, 1984 |
|  | Griest, W. H. "Microscopic Characterization of Stack Ash Particles from Coal Combustion" | Univ. of Tenn. Knoxville, TN, May 11, 1984 |
| Groenewold, G. S.* | Groenewold, G. S.;* Todd, P. J. "Enhancement of Secondary Radical Cation Emission Molten $SbCl_3$" | 32nd Annu. Conf. on Mass Spectrom. and Allied Topics, San Antonio, TX, May 27-June 1, 1984 |
|  | Groenewold, G S.;* Todd, P. J. "Detection/Identification of Volatile Organic Phosphonates by SIMS" | 32nd Annu. Conf. on Mass Spectrom. and Allied Topics, San Antonio, TX, May 27-June 1, 1984 |
| Guerin, M. R. | Guerin, M. R. "Chemical Implications of Model Compound Selection Exercises" | OHER Meas. Sci. Workshop, Battelle Seattle Conf. Ctr., Seattle, WA, May 14-17, 1984 |
|  | Guerin, M. R.; Griest, W. H.; Ho, C.-h.; Smith, L. H.;* Epler, J. L.;* Witschi, H. R.* "Comparative Toxicological and Chemical Properties of Fuels Developed from Coal, Shale, or Petroleum" | Spring Nat. Meet. of the Am. Inst. of Chem. Eng., Anaheim, CA, May 20-23, 1984 |
|  | Guerin, M. R.; Griest, W. H.; Ho, C.-h.; Smith, L. H.;* Epler, J. L.;* Witschi, H. R.* "Comparative Toxicological and Chemical Properties of Fuels Developed from Coal, Shale, or Petroleum: Coal Liquids Update" | Dir. Lique. Prog. Contractor's Proj. Rev. Conf., Albuquerque, NM, Oct. 17-18, 1984 |
| Haire, R. G.* | Haire, R. G.;* Young, J. P.; Peterson, J. R.* "Absorption Spectrophotometric and X-ray Diffraction Studies of $AmI_3$ and $CmI_3$ as a Function of Temperature and Pressure" | PAC Chem. '84, Honolulu, HI, Dec. 16-21, 1984 |
| Higgins, C. E. | Higgins, C. E.; Griest, W. H.; Guerin, M. R. "Methodology for the Sampling and Analyses of Sidestream and Mainstream Smokes" | 38th Tobacco Chemist's Res. Conf., Atlanta, GA, Nov. 5-8, 1984 |
| Ho, C.-h. | Ho, C.-h.; Ma, C. Y.; Griest, W. H.; Guerin, M. R. "Preparative Scale Organic Extraction and Chemical/Biological Analysis of Stack and ESP Hopper Ash" | Am. Chem. Soc., Philadelphia, PA, Aug. 26-31, 1984 |

| SPEAKER | AUTHOR(S) AND TITLE | PRESENTED AT |
|---|---|---|
| Holmberg, R. W. | Holmberg, R. W.; Brazell-Ramsey, R. S.; Moneyhun, J. H. "Chemistry of Phosphorus Obscurant Smokes" | 2nd Biennu. Environ. and Health Effects of Smokes and Obscurants Workshop, Pacific Northwest Lab., Richland, WA, July 18-19, 1984 |
| Hulett, L. D. | Hulett, L. D. "Applications of Monoenergetic Positrons in Chemistry and Physics" | Invited, Chem. Dept., Univ. of Illinois, Urbana, IL, Mar. 29, 1984 |
|  | Hulett, L. D.; Dale, J. M.; Pendyala, S.* "Prospects for Positron Microprobe and Positron Microscopy Techniques" | Invited, 4th Annu. Am. Vac. Soc. Symp., Knoxville, TN, May 1-3, 1984 |
|  | Hulett, L. D. "Positron Spectroscopy, an Overview" | Gordon Conf. on Elec. Spectrosc., Wolfeboro, NH, July 16-21, 1984 |
| James, R. B.* | James, R. B.;* Narayan, J.;* Christie, W. H. Holland, O. W.;* Wood, R. F.* "Pulsed $CO_2$ Laser Annealing of Silicon" | Annu. Meet. Mater. Res. Soc., Boston, MA, Nov. 26-30, 1984 |
| Jenkins, R. A. | Jenkins, R. A.; Holmberg, R. W.; Gayle, T. M.;* Holladay, S. K.; Allin, G. W.* "A Variable Puff Parameter Smoking Machine for Smoke Composition Studies" | 38th Tobacco Chemist's Res. Conf., Atlanta, GA, Nov. 5-8, 1984 |
| Keller, J. M. | Keller, J. M. "Application of Fundamental Parameter Technique to the Analysis of U and Pu in Dissolver Solutions" | CFRP Tech. Sem., Oak Ridge Nat. Lab., Oak Ridge, TN, Jan. 31, 1984 |
|  | Keller, J. M. "An X-ray Fluorescence Spectrometer for Determination of U and Pu in Dissolver Solutions" | USDOE/UKAEA Inf. Exchange Meet., Oak Ridge Nat. Lab., Oak Ridge, TN, March 26, 1984 |
|  | Keller, J. M. "X-ray Fluorescence for Highly Radioactive Samples" | ORNL/SRL Inf. Exchange Meet., Oak Ridge Nat. Lab., Oak Ridge, TN, Aug. 5, 1984 |
| Klatt, L. N. | Klatt, L. N.; Harward, B. L.;* Mamantov, G.* "Multipass Spectroelectrochemistry in Molten Salts" | Am. Chem. Soc., Philadelphia, PA, Aug. 27-31, 1984 |
| Laing, W. R. | Laing, W. R. "The ACD Quality Assurance Program" | UCCND-GAT Anal. Comm. Meet., Oak Ridge Nat. Lab., Oak Ridge, TN, March 20, 1984 |

| SPEAKER | AUTHOR(S) AND TITLE | PRESENTED AT |
|---|---|---|
| Ma, C. Y. | Ma, C. Y.; Ho, C.-h.; Caton, J. E.; Guerin, M. R. "Identification and Determination of Benzoquinolines in Crude Synthetic Oils" | 36th Southeastern Reg. Meet., Am. Chem. Soc., Raleigh, NC, Oct. 24-26, 1984 |
| Maskarinec, M. P. | Maskarinec, M. P.; Sepaniak, M. J.;* Vargo, J. D.* "Developments in Open Tubular Liquid Chromatography" | Anal. Chem. Div. Inf. Meet., Oak Ridge Nat. Lab., Oak Ridge, TN, Jan. 31, 1984 |
|  | Maskarinec, M. P.; Vargo, J. D.;* Sepaniak, M. J.* "Developments in Column Technology for Open Tubular Liquid Chromatography" | 1984 Pittsburgh Conf., Atlantic City, NJ, Mar. 5-9, 1984 |
|  | Maskarinec, M. P.; Sepaniak, M. J.;* Balchunas, A. S.;* Vargo, J. D.* "Liquid Chromatography in Open Tubes" | Symp. on Advanced Anal. Concepts for the Chem. Lab., Knoxville, TN. Apr. 12-13, 1984 |
|  | Maskarinec, M. P.; Manning, D. L.; Harvey, R. W.; Griest, W. H., Tomkins, B. A. "Determination of Munitions Components by HPLC/Electrochemical Detection" | Int. Symp. on Advances on Chromatogr., New York, NY, Apr. 16-18, 1984 |
|  | Maskarinec, M. P.; Francis, C. W.;* Goyert, J. C.* "Mobility of Organic and Inorganic Constituents from Coal Combustion and Conversion Wastes Under Co-Disposal Condition" | 1984 Annu. Meet. Mater. Res. Soc. Symp. M: Coal Combus. and Conversion Solid Wastes: Charac., Utiliz. and Disposal, Boston, MA, Nov. 26-30 1984 |
| McLuckey, S. A. | McLuckey, S. A.; van der Zande, W.;* de Bruijn, D. P.;* Los, J.;* Kistemaker, P. G.* "Scattering in Dissociative Collisions of Ionized Acetone with Helium" | 32nd Annu. Conf. on Mass Spectrom. and Allied Topics, San Antonio, TX, May 27-June 1, 1984 |
| Peterson, J. R.* | Peterson, J. R.;* Haire, R. G.;* Young, J. P. Ensor, D. D.* "Investigations of the Solid State Properties of Transcurium Halides: How Absorption Spectrophotometry Makes it Possible" | Invited, PAC Chem. '84, Honolulu, HI, Dec. 16-21, 1984 |
| Ramsey, J. M. | Ramsey, J. M. "Chemical Analysis Using Phase Conjugate Optical Techniques" | Invited, Dept. of Chemistry, Univ. of of North Carolina Chapel Hill, NC, Apr. 17, 1984 |
|  | Ramsey, J. M. "Spectroscopic Instrumentation Developments at ORNL" | 2nd Workshop on Remote Monitoring and Dosimetry, Knoxville, TN, Apr. 9, 1984 |

| SPEAKER | AUTHOR(S) AND TILE | PRESENTED AT |
|---|---|---|
| Ramsey, J. M. | Ramsey, J. M.; Whitten, W. B. "Phase Conjugate Optics and Atomic Spectroscopy" | Invited, 11th Annu. Fed. of Anal. Chem. and Spectrosc. Soc. Meet., Philadelphia, PA, Sept. 16-21, 1984 |
| | Ramsey, J. M. "Passively Controlled High-Resolution Dye Lasers: A New Spectroscopic Source?" | Invited, Univ. of Illinois, Dept. of Chemistry, Urbana, IL, Dec. 7, 1984 |
| | Ramsey, J. M.; W. B. Whitten "High Resolution Spectroscopy with a Self-Scanning CW Dye Laser" | 1984 Pittsburg Conf. and Expo. on Anal. Chem. and Applied Spectrosc., Atlantic City, NJ, Mar. 5-9, 1984 |
| | Ramsey, J. M. "New Laser Based Spectro-Chemical Measurements" | Invited, The Upjohn Company, Kalamazoo, MI, June 18, 1984 |
| | Ramsey, J. M.; Whitten, W. B. "A Narrow-Bandwidth Self-Scanning CW Dye Laser: Analytical Applications" | Anal. Chem. Div. Inf. Meet., Oak Ridge Nat. Lab., Oak Ridge, TN, Jan. 30-Feb. 1, 1984 |
| Ramsey, R. S. | Brazell-Ramsey, R. S.; Holmberg, R. W.; Moneyhun, J. H. "The Chemical Characterization of Phosphorus Aerosols" | Anal. Chem. Div. Inf. Meet., Oak Ridge Nat. Lab., Oak Ridge, TN, Jan. 31, 1984 |
| | Brazell-Ramsey, R. S. "Flow Injection Analysis: Principles and Applications" | UCCND-GAT Anal. Meet., Oak Ridge Nat. Lab., Oak Ridge, TN, Mar. 20, 1984 |
| | Brazell-Ramsey, R. S. "Pulsed Helium Ionization Detection" | 2nd DOE Workshop on Portable Instrum. and Dosim., Knoxville, TN, Apr. 9-11, 1984 |
| | Brazell-Ramsey, R. S.; Todd, R. A.* "A New Design for Helium Ionization Detection" | Int. Symp. on Advances in Chromatogr., New York, NY, Apr. 16-18, 1984 |
| | Brazell-Ramsey, R. S. "Pulsed Helium Ionization Detection for Trace Analysis" | Energy Systems-GAT Anal. Meet., Portsmouth, OH, July 17, 1984 |

| SPEAKER | AUTHOR(S) AND TITLE | PRESENTED AT |
|---|---|---|
| Roseberry, L. M. | Roseberry, L. M.; Dyer, F. F. "Instrumental Neutron Activation Analysis in the Measurement of Trace Element Distribution at Two Coal Conversion Plants" | Fifth Int. Conf. Nucl. Methods in Environ. and Energy Res., Mayaguez, Puerto Rico, Apr. 1-6, 1984 |
| Ross, H. H. | Alley, C. S.; Ross, H. H. "Accuracy and Precision Performance Parameters of a Laboratory Robot System" | 27th Conf. on Anal. Chem. in Energy Technol., Knoxville, TN, Oct. 2-4, 1984 |
| Rosseel, T. M. | Rosseel, T. M.; Dale, J. M.; Hulett, L. D.; Krause, H. F.;* Pepmiller, P. L.;* Raman, S.;* Vane, C. R.;* Young, J. P. "Application of Heavy-Ion Induced X-ray Satellite Emission (HIXSE) to Transition Metal Alloys and Compounds" | 4th Annu. Am. Vac. Soc. Symp., Knoxville, TN, May 1-3, 1984 |
|  | Rosseel, T. M.; Dale, J. M.; Hulett, L. D.; Krause, H. F.;* Pepmiller, P. L.;* Raman, S.;* Vane, C. R.;* Young, J. P. "Chemical Analysis of Materials Using Heavy-Ion-Induced X-ray Satellite Emission (HIXSE)" | Invited, 11th Annu. Fed. of Anal. Chem. and Spectrosc. Soc. Meet., Philadelphia, PA, Sept. 16-21, 1984 |
|  | Rosseel, T. M.; Dale, J. M.; Hulett, L. D.; Krause, H. F.;* Pepmiller, P. L.;* Raman, S.;* Vane, C. R.;* Young, J. P. "Effect of Chemical Environment on the L and M Heavy-Ion-Induced X-ray Satellite Emission (HIXSE) Spectra" | 8th Conf. on the Application of Accelerators in Res. and Ind., Denton, TX. Nov. 12-14, 1984 |
|  | Rosseel, T. M.; Dale, J. M.; Das Gupta, A.;* Hulett, L. D.; Krause, H. F.;* Liu, C. T.;* Raman, S.;* Vane, C. R.;* Young, J. P. "Development of Heavy-Ion Induced X-ray Satellite Emission (HIXSE) as a Materials Probe" | 1984 Anal. Chem. Div. Inf. Meet., Oak Ridge Nat. Lab., Oak Ridge, TN, Jan 30-Feb. 1, 1984 |
|  | Rosseel, T. M.; Dale, J. M.; Hulett, L. D.; Young, J. P. "Heavy-Ion Induced X-ray Satellite Emission" | DOE Chemical Sciences Site Review, Oak Ridge Nat. Lab., Oak Ridge, TN, Mar. 27, 1984 |
| Shults, W. D. | Shults, W. D.; Roseberry, L. M. "The Matrix Scientist in a Matrix Organization" | Am. Chem. Soc. Reg. Meet., Great Lakes Section, Kalamazoo, MI, May 23, 1984 |
| Stewart, J. H. | Stewart, J. H. "Urgent Need for Nationally Certified Reference Standards for ICP Analyses" | ASTM C-26 Nucl. Fuel Cycle Comm. (ICP Task Group), San Diego, CA, Jan. 22-26, 1984 |

| SPEAKER | AUTHOR(S) AND TITLE | PRESENTED AT |
|---|---|---|
| Stewart, J. H. | Stewart, J. H. "Status of Nationally Certified Standards for ICP Spectroscopy" | ASTM C26.05 Inductively Coupled Plasma Working Comm., Colorado Springs, CO, Aug. 1, 1984 |
| | Stewart, J. H.; Dunn, H. W. "The Quantitative Measurement of Chrysotile Asbestos in Environmental Materials at the One-Percent Concentration Level" | Am. Chem. Soc. Reg. Meet., Raleigh, NC, Oct. 24-26, 1984 |
| Todd, P. J. | Todd, P. J.; Groenewold, G. S.* "Gas Phase Molecular SIMS" | 32nd Annu. Conf. on Mass Spectrom. and Allied Topics, San Antonio, TX, May 27-June 1, 1984 |
| | Todd, P. J.; Groenewold, G. S.;* Leibman, C. P.* "Matrices and Molecular SIMS" | 27th Conf. on Anal. Chem. in Energy Technol., Knoxville, TN, Oct. 2-4, 1984 |
| | Todd, P. J. "Molecular Secondary Ion Mass Spectrometry (SIMS): Matrix Effects" | Anal. Chem. Div. Inf. Meet., Oak Ridge Nat. Lab., Oak Ridge, TN, Jan. 30-Feb. 1, 1984 |
| Tomkins, B. A. | Tomkins, B. A.; Higgins, C. E.; Manning, D. L.; Griest, W. H., Caton, J. E. "Methods for the Determination of Particulate Organic Matter from Coal Combustion" | 187th Am. Chem. Soc. Nat. Meet., St. Louis, MO, Apr. 8-13, 1984 |
| | Maskarinec, M. P.; Francis, C. W.;* Goyert, J. C.;* Tomkins, B. A. "Leaching of PAH from Industrial Wastes with Municipal Waste Leachate" | 9th Int. Symp. on Polynuc. Arom. Hydrocarbons, Battelle, Columbus, OH, Oct. 30-Nov. 1, 1984 |
| | Tomkins, B. A.; Reagan, R. R.; Buchanan, M. V. Olerich, G.; Griest, W. H.; Caton, J. E. "The Isolation, Identification, and Quantitation of the Four- and Five-Ring Dermal Tumorigen PAH on Petroleum Crude Oils and Distillate Fractions Using Normal-Phase Isolation HPLC and GC/MS in the Single-Ion Monitoring Mode" | 9th Int. Symp. on Polynuc. Arom. Hydrocarbons, Battelle, Columbus, OH, Oct. 30-Nov. 1, 1984 |
| Vane, C. R. * | Vane, C. R.;* Hulett, L. D., Jr.; Kahane, S.;* Kallne, E.;* Kallne, J.;* McDaniel, F. D.;* Milner, W. T.;* Nakajima, T.;* Nestor, C. W.;* O'Kelley, G. D.;* Raman, S.;* Rosseel, T. M.; Slaughter, G. G.;* Stelson, P. H.;* Varghese, S. L.;* Walkiewicz, T. A.;* Young, J. P. "Environmental Effects in High Resolution X-ray Satellite Spectra Induced by Heavy Ions" | Invited, 11th Annu. Fed. of Anal. Chem. and Spectrosc. Soc., Philadelphia, PA, Sept. 16-21, 1984 |

| SPEAKER | AUTHOR(S) AND TITLE | PRESENTED AT |
|---|---|---|
| Whitten, W. B. | Whitten, W. B.; Ramsey, J. M. "Holographic Tuning of CW Dye Lasers" | Invited, DOE Workshop, "Advanced Laser Tech. for Chem. Measurements" Brookhaven National Laboratory, Upton, NY, Oct. 23-25, 1984 |
|  | Whitten, W. B.; Ramsey, J. M. "Phase Conjugate Feedback" | Invited, Research Committee, Oak Ridge Nat. Lab., Oak Ridge, TN, Feb. 1, 1984 |
|  | Whitten, W. B.; Ramsey, J. M. "Laser Spectrometry" | DOE Chemical Sciences Site Review, Oak Ridge Nat. Lab., Oak Ridge, TN, Mar. 27, 1984 |
| Young, J. P. | Young, J. P.; Donohue, D. L.; Smith, D. H. "Assessment of Resonance Ionization Mass Spectrometry for Analytical Chemistry and Spectroscopy" | Invited, 2nd Int. Symp. on Spectrosc. and Its Applications, Knoxville, TN, Apr. 16-20, 1984 |
|  | Young, J. P.; Donohue, D. L.; Smith, D. H. "Factors Affecting the Analytical Usefulness of Resource Ionization Mass Spectrometry" | Invited, DOE Workshop, "Advanced Laser Technol. for Chem. Measurements", Brookhaven Nat. Lab., Upton, NY, Oct. 23-25, 1984 |
|  | Young, J. P.; Donohue, D. L.; Smith, D. H. "Assessment of RIMS for Analytical Chemistry and Spectroscopy" | 1984 Pittsburg Conf. and Expo. on Anal. Chem. and Applied Spectrosc., Atlantic City, NJ, Mar. 5-9, 1984 |

## ARTICLES REVIEWED OR REFEREED FOR PERIODICALS

| | *Anal. Chem.* | *Anal. Chim. Acta* | *Health Physics* | *J. Radioanal. Nucl. Chem.* | *J. Radioanal. Nucl. Chem. Lett.* | *ORNL Reports* | *Optics Letters* | *Proposals* | *Others* | *Total* |
|---|---|---|---|---|---|---|---|---|---|---|
| Bostick, D. A. | | | | | | | | | 2 | 2 |
| Buchanan, M. V. | 1 | | | | | | | 1 | 1 | 3 |
| Carter, J. A. | 2 | | | | | | | 2 | | 4 |
| Caton, J. E. | | | | | | | | | 1 | 1 |
| Christie, W. H. | | | | | | | | 2 | | 2 |
| Clark, B. R. | | | | | | | | 1 | 1 | 2 |
| Costanzo, D. A. | | | | | | 2 | | | | 2 |
| Dale, J. M. | | | | | | | | 1 | | 1 |
| Donohue, D. L. | 2 | | | | | | | | | 2 |
| Eldridge, J. S. | | | 2 | | | 1 | | | | 3 |
| Feldman, C. | 3 | | | | | | | | | 3 |
| Glish, G. L. | 2 | | | | | | | 2 | | 4 |
| Griest, W. H. | 2 | | | | | | | 1 | 2 | 5 |
| Guerin, M. R. | | | | | | | | 1 | 11 | 12 |
| Holmberg, R. W. | | | | | | | | 1 | 1 | 2 |
| Hulett, L. D. | | | | | | | | | 3 | 3 |
| Jenkins, R. A. | | | | | | | | 2 | 1 | 3 |
| Klatt, L. N. | 2 | | | | | | | 2 | 1 | 5 |
| Lee, D. A. | | | | | | | | | 2 | 2 |
| Lyon, W. S. | | 2 | | 3 | 4 | | | 2 | 2 | 13 |
| Maskarinec, M. P. | 4 | | | | | | | 2 | | 6 |
| Ramsey, J. M. | 2 | | | | | | 1 | 3 | | 6 |
| Ramsey, R. S. | 2 | | | | | | | | 1 | 3 |
| Ross, H. H. | | | | | | | | 10 | | 10 |
| Shaw, R. W. | 2 | | | | | | | 2 | | 4 |
| Shults, W. D. | 2 | | | | | | | 2 | | 4 |
| Tomkins, B. A. | 8 | | | | | | | 2 | 1 | 11 |
| Whitten, W. B. | | | | | | | 3 | 3 | | 6 |
| Young, J. P. | 3 | | | | | | | 2 | 1 | 6 |
| Total | 37 | 2 | 2 | 3 | 4 | 3 | 4 | 44 | 31 | 130 |

# DIVISIONAL MAN-POWER AND FINANCIAL SUMMARY
## FY 1984

| Source | $K | PY |
|---|---|---|
| **DOE programs** | | |
| Health and Environmental Research | 779 | 6.1 |
| Basic Energy Sciences | 1,328 | 10.0 |
| Nuclear Energy | 301 | 2.2 |
| Fossil Energy | 515 | 4.5 |
| Safeguards and Security | 152 | 1.1 |
| Total DOE programs | 3,075 | 23.9 |
| **Work for others - federal agencies** | | |
| Department of Defense (U.S. Army) | 549 | 5.8 |
| National Cancer Institute | 404 | 4.3 |
| Department of State (IAEA) | 55 | 0.6 |
| National Institute on Drug Abuse | 164 | 1.6 |
| Miscellaneous | 17 | 0.2 |
| Total work for others - federal agencies | 1,189 | 12.5 |
| **Work for others - nonfederal agencies** | | |
| Electric Power Research Institute | 38 | 0.3 |
| Protective Coating Companies | 92 | 1.0 |
| DuPont (SRL) | 86 | 0.9 |
| Sandia | 247 | 2.6 |
| Bechtel National, Inc. | 64 | 0.7 |
| Miscellaneous | 84 | 0.9 |
| Total work for others - nonfederal agencies | 611 | 6.4 |
| **Support/Services** | | |
| ORNL divisions | 4,173* | 67.5 |
| Other clients | 951* | 15.7 |
| | 5,124* | 83.2 |
| Total financial plans | 9,999 | 126.0 |

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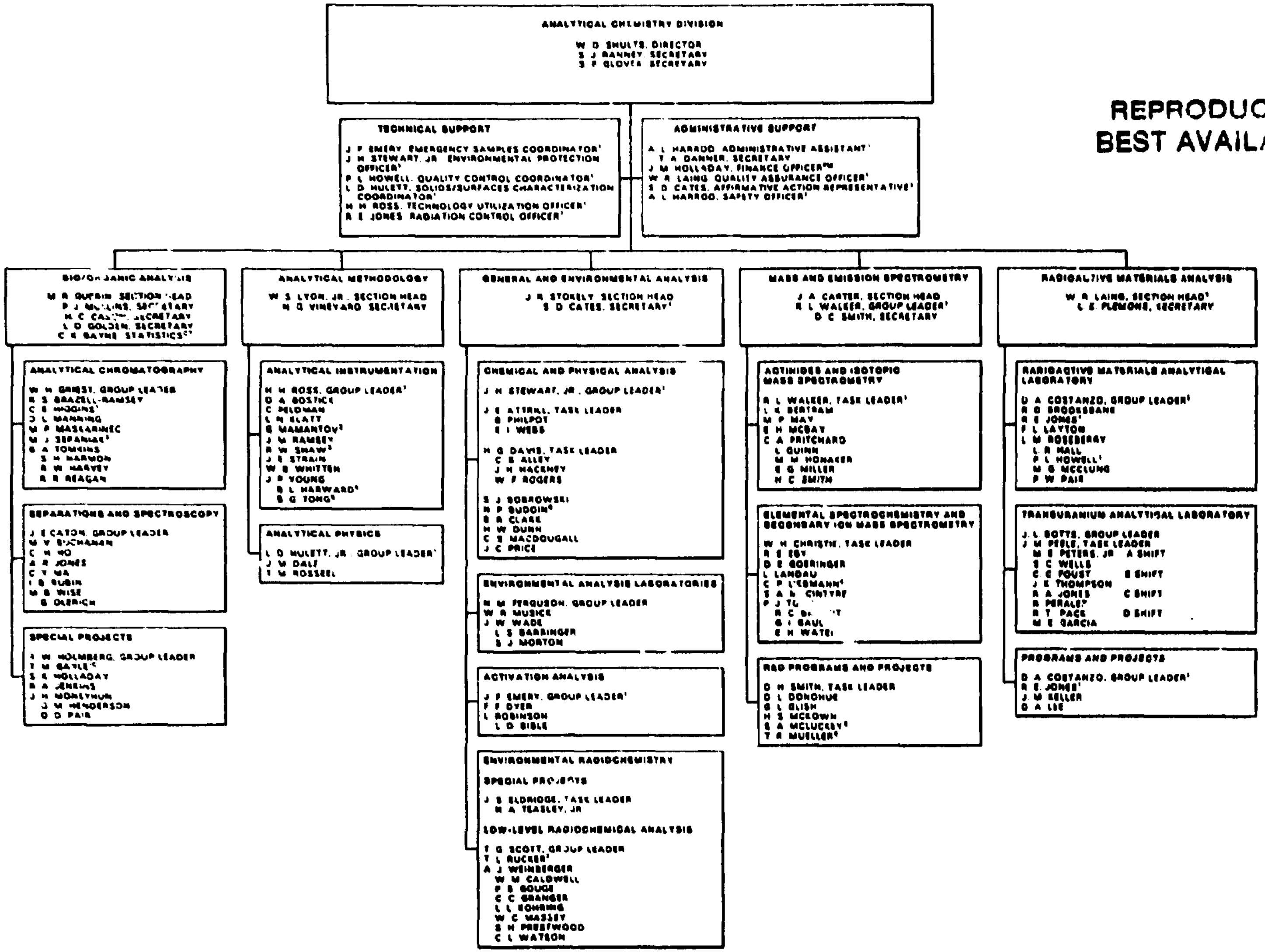
*Does not reflect Laboratory general and administration expense or general plant services.

DECEMBER 31, 1984



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