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**Analytical Chemistry Division
Annual Progress Report
for Period Ending December 31, 1987**



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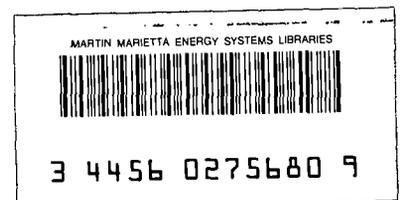
**ANALYTICAL CHEMISTRY DIVISION
ANNUAL PROGRESS REPORT**

For Period Ending December 31, 1987

W. D. Shults, Director

Date Published - May 1988

OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee 37831
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INTRODUCTION

W. D. Shults, Director

The Analytical Chemistry Division of Oak Ridge National Laboratory (ORNL) is a large and diversified organization. As such, it serves a multitude of functions for a clientele that exists both in and outside ORNL. These functions fall into the following general categories:

1. **Analytical Research, Development, and Implementation.** 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; however, attention is also given to advancing the analytical sciences themselves. This program is composed of medium- to long-term projects and is supported primarily by the DOE. Fundamental R&D constituted approximately 23% of our effort in FY 1987.

2. **Programmatic Research, Development, and Utilization.** The division carries out a wide variety of chemical work that typically involves analytical research and/or development plus the utilization of 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 constituted about 28% of our work in FY 1987.

3. **Technical Support.** The division performs chemical and physicochemical

analyses of virtually all types. The development of methodology is an inherent part of this activity because of the variety of analytical problems that arise in a multiprogram institution like ORNL. Consultation, collaboration, and special projects are involved. Much of this work is short-term in nature and comes from other divisions and programs within ORNL, but a significant fraction originates outside of ORNL and involves the use of talent and/or facilities in which the division is particularly strong or unique. This type of work accounted for approximately 49% of our effort in FY 1987.

The Analytical Chemistry Division is organized into four major sections, each of which may carry out any of the three types of work previously mentioned. Chapters 1 through 4 of this report highlight progress within the four sections during the period January 1 to December 31, 1987. A brief discussion of the division's role in two sizable environmental programs is given in Chapter 5. Information about quality assurance, safety, and training programs is presented in Chapter 6, along with a tabulation of analyses rendered. Publications, oral presentations, professional activities, educational programs, and seminars are cited in Chapters 7 and 8. Approximately 55 articles, 37 proceedings publications, and 33 reports were published, and 166 oral presentations were given during this reporting period. Some 435,711 determinations were performed, with an overall quality factor of 96.5%.

HIGHLIGHTS

Professor Georges Guiochon and six of his students and post-docs joined the division this year, as part of the University of Tennessee/ORNL Distinguished Scientist Program. The research of the ORNL group focuses on three areas: nonlinear chromatography, the sources of error in chromatographic measurements, and the development of nonselective detectors for high performance liquid chromatography.

Micellar electrokinetic capillary chromatography (MECC) has been evaluated for its utility as a tool for separating nucleic acid constituents. It has been found capable of resolving normal and modified deoxyribonucleosides with efficiencies easily exceeding 100,000 theoretical plates. At low solute concentrations (nmol/mL), efficiencies of 320,000 theoretical plates (corresponding to 500,000 plates/m) have been realized. Sequence- and modification-selective separations of oligonucleotides have also been achieved.

The ability of supercritical fluid chromatography (SFC) to separate polar, thermally labile compounds such as nucleic acid constituents and explosives is being determined. Several nucleic bases including adenine and methylated analogs and the ribonucleoside adenosine have been mobilized and separated in underivatized forms using a cyano-phase packed column and supercritical carbon dioxide programmed from 20 to 35 MPa (200 to 350 atm) at 150°C. Several explosives, including tetranitroaniline (Tetryl) and trimethylenetrinitramine (RDX), have been chromatographed by SFC for the first time.

In collaboration with Y-12 Plant personnel, we developed an advanced version of our atmospheric sampling MS/MS explosives detector. This version is highly automated. It samples the air

continuously, monitoring for explosive molecules with one MS stage. If the signal exceeds a certain threshold level, the instrument obtains the MS/MS spectrum and alarms if the spectrum indicates positive identification of explosive vapors. Research in this area has turned to the coupling of our glow discharge ionization source with an ion trap mass spectrometer.

A project was initiated this year to develop our glow discharge ionization source into an interface for liquid chromatography/mass spectrometry. Feasibility has been demonstrated. Emphasis has shifted to optimization of the source and electrode geometries to maximize sensitivity.

We have found that glow discharge ionization mass spectrometry can be used for the direct analysis of volatile organics in water and soils. This is done by monitoring purge air or headspace at flow rates of 1-2 mL/sec. Initial results indicate that this technique coupled with MS/MS is promising as a rapid method for EPA-specified pollutants.

Our PCB analytical capability has quadrupled through purchase of a Sigma 300 automated gas chromatograph which is operated in conjunction with the new Fisher grinder and SOXTEC rapid organic extraction system. The SOXTEC has proved so valuable that a lengthy set of experiments, to demonstrate equivalency with the EPA SW 846 Soxhlet method, has been completed and an approved petition for ORNL use of the method for environmental compliance specimens has been submitted to EPA Region IV. The new method reduces the lapsed time for specimen extraction from 17 to 2 hours!

A database system was developed for performing all calculations, quality

control checks, and identifications in the PCB/pesticides component of the Contract Laboratory Program (CLP) protocol. Calibration data and results are automatically compiled. The system affords considerable savings in time. It has attracted interest by other laboratories that utilize CLP procedures.

A number of FTMS techniques have been developed for the characterization of high molecular weight components (200-1000 daltons) in complex mixtures. Molecular weight profiles can be generated, components can be separated by molecular weight, and identifications can be made by the exact mass of the ions. This powerful methodology has been applied to the characterization of tobacco smoke particulates.

Laser ionization Fourier transform mass spectrometry (FTMS) has been investigated as a means of generating ions from nonvolatile compounds. Negative ions corresponding to specific nucleic bases, sugars, and phosphate groups in nucleotides have been generated with a Nd:YAG laser. The negative ion spectrum provides more information about bases in a nucleotide than does the positive ion spectrum. A pulsed dye laser has been acquired to further these studies.

We have continued to study negative ion processes that may be useful for identification and/or isomeric differentiation of polynuclear aromatic hydrocarbons. It has been shown that negative ions are produced by three different means: electron-molecule, ion-molecule, and wall-assisted reactions. Oxygen participates in unusual wall-assisted reactions that are followed by electron capture. These reactions are especially useful for isomeric differentiation.

In a joint effort with the National Institute of Mental Health, we have

developed a secondary ion MS/MS and begun to explore its utility for obtaining organic ion micrographs. Images of liquid samples dispersed across a grid have been obtained with spatial resolution of less than 0.5 mm. These are the first such images to be reported.

For measurement of the nuclear properties of promethium, a unique lanthanide with no stable isotopes, it is necessary to correct for the (isobaric) samarium content of samples. Resonance ionization mass spectrometry (RIMS) has been shown to circumvent this problem. Promethium can be selectively ionized using flashlamp-pumped rhodamine 6G dye laser focused into the plume of atoms from a hot rhenium filament in the source region of a single magnetic stage mass spectrometer. The filament is loaded with 100 and 600 ng of promethium and samarium, respectively. An elemental selectivity factor of at least 1000:1 is estimated for promethium over samarium. This single-laser method should find use as a convenient method for isotopic analysis of promethium in the presence of its daughter, samarium.

The Oak Ridge Electron Linear Accelerator (ORELA) provides photoneutrons by bombarding a tantalum target with 150 MeV electrons, which produces gamma bremsstrahlung. A facility has been built for insertion behind the tantalum target to capture the excess gamma energy and convert it to slow positrons. Calculations indicate that the excess gamma flux of the ORELA will produce about 10^8 slow positrons per second. Provisions have been made to capture the positrons in a Penning trap and disperse them over long time periods, approximately equal to the dead time of the accelerator. Construction of the facility components, which include the solenoids, Penning trap, and pulsing electronics is now 100% complete.

The lutetium double spike technique for tank volume calibration has been verified as a valuable safeguards method by British Nuclear Fuels personnel. They report precisions of 0.04% for volume determinations of plutonium holding tanks. This success, coupled with ours, has resulted in plans to evaluate the technique in collaboration with Savannah River Laboratory personnel. One of their tanks will be used for evaluation under conditions reflecting a typical working environment.

Work on a fiber-scintillator radioactivity detector for beta-emitting isotopes was completed this year. The flow-cell detector has good detection efficiency when 0.1 mm diameter fibers are used. Use of scintillator in fiber form provides improved light collection as well as improved operating characteristics.

The air-acetylene burner is commonly used as an atomization source for elemental analysis with various spectroscopic methods. The technique exhibits excellent sensitivity for trace elements in water, typically in the parts per billion range or better. The spectral resolution, however, is limited to about 5 GHz because spectral lines are broadened by collisions in the atmospheric pressure flame. We have developed a low-pressure interface that allows a hundred-fold improvement in resolution while retaining the sensitivity and convenience of flame atomization.

All neutron activation analysis work at Oak Ridge Research Reactor was discontinued this year. All laboratory apparatus, the ND 6620 spectrometer and its associated counting equipment, etc. were moved to our new General Purpose Neutron Activation Analysis Facility (GPNAAF) at the HFIR. The Pneumatic Tube System Mark II (PTS MK-II) incore section was installed in June in preparation for the hydrostatic test of the HFIR. The PTS MK-II Programmable Gould

Controller is presently being tested. By the time the HFIR begins operation, the GPNAAF will be completely operational.

1. ANALYTICAL SPECTROSCOPY

J. A. Carter

The Analytical Spectroscopy Section is composed of four groups: Laser Spectroscopy and Instrumentation, Actinides and Isotopic Mass Spectrometry, Spectroscopic R&D Programs, and Inorganic and Secondary Ion Mass Spectrometry. Special research and development projects in radiochemistry are also carried out in the section. The R&D efforts in each group are directed toward enhancing analytical capabilities through new discoveries and developing a clearer understanding of fundamental physical processes that can be exploited into new instruments or analytical approaches for solving energy-related problems. Many projects toward the overall goal are described in the technical summaries; some outstanding developments for the year are: a) a low-pressure interface in laser spectroscopy work that improved resolution and sensitivity by two orders of magnitude; b) laser based refractive index instrumentation schemes with potential for chromatographic detection; c) an atmospheric sampling glow discharge ionization source (patent applied for in 1987) coupled with a MS/MS instrument for detection of high explosives; d) ion-trap mass spectrometry (ITMS) as an efficient trace organic environmental monitor. Constructions include: a) a high resolution HIXSE spectrometer, b) a slow positron source facility at ORELA; c) improved organic SIMS instrumentation that provides ion micrographs for the first time; and d) modifications to the VG 354E mass spectrometer to obtain more precise rare gas isotope ratios.

About 50% of the section's research and development support is provided by the Division of Chemical Sciences of the Office of Energy Research, U.S. Department of Energy. This effort, broad in nature, provides the technical base from which current and future spectroscopic needs are addressed. Another 30% of the section's R&D funding is about equally distributed between the DOE Offices of Safeguards and Security, International Security Affairs, and Health and Environmental Research. The projects for these funding sources are generally short range and of an applied nature. The balance of our effort is funded by Laboratory divisions and programs, various interagency agreements (FAA, ISPO/State, and IAEA), and other work-for-others contracts.

LASER SPECTROSCOPY AND INSTRUMENTATION

J. M. Ramsey

This group performs research in the field of laser-based analytical measurements. Areas of activity include very high-resolution laser spectroscopy of atoms and the use of photorefractive optical elements in optical instruments and rotational spectroscopy. The group also develops measurement schemes and designs instrumentation for specific measurement problems.

Low-Pressure Laser Spectroscopy with Flame Atomization

The air-acetylene burner is commonly used as an atomization source for elemental analysis with various spectroscopic methods, ranging from flame emission to sophisticated laser-based techniques. The latter include laser-induced fluorescence, laser-enhanced ionization, polarization saturation spectroscopy, and degenerate four-wave mixing. All of these techniques exhibit excellent sensitivity for trace elements in water, typically in the parts per billion range or better. The spectral resolution, however, is limited to about 5 GHz because the spectral lines are broadened by collisions in the atmospheric pressure flame. We have developed a low-pressure interface that allows a hundred-fold improvement in resolution while retaining the sensitivity and convenience of flame atomization.

In our apparatus, flame gases containing the atomized sample expand through a small orifice into a chamber that is evacuated by a mechanical roughing pump. The pressure within the chamber is about 0.2 Torr (26 pascal). Quartz windows provide optical access to the chamber for Doppler-free laser spectroscopy.

We have tested the apparatus by studying the spectrum of the sodium D lines, using a type of saturation spectroscopy. We have been able to separate hyperfine transitions and crossover resonances with a resolution of better than 50 MHz. A recognizable hyperfine spectrum can be obtained with as little as 100 ppb of sodium.

Effusive beam spectra such as these are distorted somewhat by velocity-changing collision pedestals and crossover resonances. To overcome these problems we have recently made modifications to the low pressure cell that permit measurements under supersonic expansion conditions. The atoms from the flame depart from the expansion orifice in a collision-free environment. Furthermore, because of their straight line trajectories, we can optically select only those atoms moving perpendicular to the laser beam. The result is a considerably improved spectrum. For the sodium D_1 line, for example, three of five crossover resonances and essentially all of the pedestals are eliminated.

(W. B. Whitten, T. G. Nolan,* L. B. Koutny,** J. M. Ramsey)

*ORAU Postdoctoral Research Associate

**Summer Student

Degenerate Four-Wave Mixing Spectroscopy

Work is continuing on degenerate four-wave mixing (dfwm) studies for elemental spectroscopy in flames. One problem encountered in these experiments is that the input laser beams are severely distorted within the flame by thermal refractive index gradients. These distorted input beams produce a signal beam which is equally distorted and

results in a reduced measured signal. We have recently found a novel way to compensate for the thermal distortions of the input beams by using a phase-conjugate reflector to generate the second pump beam. An input probe beam is phase conjugated in dfwm only if the two pump beams are phase conjugates of one another. This phase-conjugate condition does not exist in the conventional approach to dfwm because of the distortions produced in the flame. Strong spatial filtering is used to discriminate against background signals in these experiments; thus any distortion of the signal beam reduces the measured signal.

We and others have found that a single crystal of barium titanate can be used to create a phase-conjugate reflection of a laser beam and automatically correct for optical distortions along its path, with a response time of tenths of a second. The process of optical phase conjugation produces a beam that is exactly counterpropagating to an input beam, even when aberrations are present. Thus, we have been able to use this passive barium titanate phase conjugator to generate phase-conjugate pump beams in our dfwm experiments. The phase-conjugate pump beams are generated by first passing one pump beam through the flame and then phase conjugating this beam with the barium titanate crystal. The barium titanate phase conjugator automatically compensates for static and slowly varying distortions within the flame. We are presently studying the degree of improvement that can be obtained in this way and working to increase the response time of the crystal.

The technique of dfwm spectroscopy is a Doppler-free form of spectroscopy similar to the saturation spectroscopy used in the study discussed above and thus also benefits from reduction of sample pressure. With the low-pressure flame interface

described above, the D_2 lines of sodium were observed with a linewidth of 130 MHz, which is narrower than the linewidth obtainable in an ambient pressure flame because of reduced collisional broadening. Studies are being carried out to compare the trade-offs between high-resolution and sensitivity.

(T. G. Nolan,* P. R. Blazewicz,* W. B. Whitten, J. M. Ramsey)

*ORAU Postdoctoral Research Associate

Refractive Index Measurement Techniques

We are investigating possible schemes for measuring small changes in refractive index (RI). The methods have potential application in chromatography and flow-injection analysis.

The first scheme is based on a two-wave mixing phenomenon in photorefractive crystals. The intersection of two laser beams in a single-domain crystal of barium titanate creates a periodic modulation of its refractive index. This photorefractive effect is caused by light-induced migration and separation of charge in the crystal, which produces internal static electric fields. A spatial modulation of the refractive index is produced via the linear electro-optic effect. An interesting phenomenon which occurs in this process is that the two beams emerge from the crystal with a different relative intensity than when they entered it. This energy transfer is the result of a spatial phase shift of $\pi/2$ between the intensity interference pattern and the refractive index pattern.

Two-wave mixing is used as a refractive index detector by placing a liquid flow cell in the path of one of the laser beams. Injection of solutions of varying

refractive index into a flowing solvent stream will give a change in the path length of light and, consequently, its phase. A sudden change of the phase of one of the beams reduces the coupling efficiency of the beams in the barium titanate crystal; i.e., the $\pi/2$ phase shift is destroyed. This change in phase is measured as a change in intensity of one of the beams emerging from the crystal. Slow changes in phase are not seen by this detector because the charges in the crystal migrate to preserve the $\pi/2$ phase mismatch condition. The response time of the index grating is such that it cannot respond to fast changes in phase but can adjust for slow changes produced by thermal drift or changes in the mobile phase when gradient elution is used.

Preliminary investigations using a 2-milliwatt helium neon laser yielded detection limits on the order of 10^{-5} $\Delta RI/cm$. The response of the detector to solvent gradients improves with faster crystal response times. Flat baselines are obtained for solvent gradients of 4×10^{-5} $DRI \text{ min}^{-1} \text{ cm}^{-1}$, which corresponds to a linear gradient from 50%-50% MeOH-H₂O to 80%-20% MeOH-H₂O in ten minutes. Crystal response time increases with higher laser power; the above values were obtained with a 2 mw laser. The orientation of the barium titanate crystal axis also affects the response time; faster response times are obtained when the c-axis points in the direction of fringe movement. Peak shapes obtained using this technique depend on the response time of the crystal compared to the residence time of the peak in the detector. Slow crystal response times compared to peak residence times give good peak shape integrity, while fast relative crystal response times lead to tailing or even double peaks.

The characteristics of the instrument response have been modeled. It is assumed

that grating build-up and motion follows an exponential response, whereas the interference fringes move instantaneously with a phase change in one of the two crossing beams. The different time responses lead to a time-dependent phase shift between the interference pattern and the refractive index grating, which results in a change in two-wave mixing efficiency, and, hence, our signal. This model does not accurately predict the signal amplitude for phase changes greater than 2π but is still in qualitative agreement.

A similar approach to the measurement of optical path length changes makes use of a double beam interferometer. The two beams generated at a beam splitter are fed into a photorefractive barium titanate crystal, which generates a time-reversed beam for each input beam. The output is measured where the two beams recombine at the beam splitter. According to Stokes' Theorem, the two beams should subtract, giving zero background intensity. Changes in phase in one arm of the interferometer due to a refractive index change will give rise to a signal due to the slow response time of the barium titanate phase conjugator.

Our experiments have revealed a large source of noise in this type of measurement that was not due to phase fluctuations between the beam paths of the interferometer. The noise may result from instabilities in the phase conjugation efficiency in the crystal. It is suspected that the noise results from spontaneous two-wave mixing competing with the phase conjugation process. Further investigations will be necessary to determine whether this technique has any merit.

A somewhat different concept for the measurement of refractive index makes use of the dependence of laser longitudinal mode frequencies on the optical path

length of the cavity. Two orthogonal modes will oscillate in a laser gain medium. The laser cavity will include a calcite beam displacer to separate the two modes. A sample cell can now be made to exclusively affect one of the laser modes. The cavity length for that mode will depend upon the refractive index of the sample. The resulting shift in frequency can be measured relative to the other oscillating mode by heterodyning them at a detector and determining the beat frequency. This approach has a fundamental limit of detection of $\approx 10^{-16} \Delta RI/cm$. A practical limit of detection is thought to be better than $10^{-10} \Delta RI/cm$.

The proposed laser system has a HeNe gas mixture enclosed in a tube 24 cm in length. The tube terminates in non-polarizing antireflective windows. A flat mirror is placed at one end of the tube. As light exits the other end, it passes through the calcite crystal. Each of these beams is reflected from a spherical mirror and thereby induced to lase. The two beams share the same gain medium but are spatially separated outside that gain medium. A sample cell holding a solution is introduced into one of the two beams.

We are modifying a commercial HeNe tube to use for our gain medium. Testing has shown what ranges of pressure and discharge currents provide high gain. We are currently in the process of replacing one of the laser's original mirrors with an antireflection coated window.

(T. G. Nolan,* J. A. Howell,** C. C. Chang,*** W. B. Whitten, J. M. Ramsey)

*ORAU Postdoctoral Research Associate

**On sabbatical, Earlham College,
Richmond, IN

***ORAU Summer Student

Microwave Spectroscopy

Microwave spectroscopy is being investigated as a method for studying isotopic abundances of molecular species. Isotopic substitution in a molecule generally changes the rotational constants sufficiently to shift a rotational transition by a large amount on the microwave frequency scale. Thus, microwave determinations of molecular species are isotopically selective and generally free of interference from other species.

Along these lines, we have studied several microwave transitions of carbonyl sulfide and nitrogen dioxide in our vacuum beam apparatus. The microwaves resonate in a Fabry-Perot cavity consisting of parallel copper mirrors inside the apparatus. The gaseous sample is introduced into the chamber via a pulsed valve. The pulsed sample modulates the microwave power in the cavity, allowing sensitive lock-in detection of microwave absorption. Next we will search for transitions due to minor isotopic components of the species.

More recently we have begun investigations of microwave-optical double resonance in nitrogen dioxide. Such schemes should permit more sensitive optical detection of microwave absorption. The microwaves induce a rotational transition in the electronic ground state of the molecule, and a laser is then used to excite a rotational state-selective transition to an excited electronic state. Changes in the fluorescence induced by the laser, or in the polarization of the propagating laser beam, occur when the microwave and optical transitions share a common rotational energy level.

The nitrogen dioxide is contained in a waveguide cell fitted with glass windows on the ends. Changes in polarization of the exciting laser may be detected by placing the cell between crossed polarizers and detecting light leak-through with a photomultiplier. To date, such attempts have been made using a pulsed Nd:YAG-pumped dye laser and a high-resolution CW ring dye laser. Additional experiments will search for changes in fluorescence.

(W. B. Whitten, P. R. Blazewicz,* J. M. Ramsey)

*ORAU Postdoctoral Research Associate

Heat Pump Fluid Measurement Systems

The development of instrumentation for the in-situ real-time measurement of absorbent concentration in operating heat pump systems has continued. Work performed this year was aimed at meeting three objectives. The first was to characterize the improved fiber optic refractometer measurement system for LiBr-H₂O machines. The second objective was to measure improvements in absorber efficiency in the presence of absorption additives. The final objective was to continue development of an in-situ real-time sensor suitable for NH₃-H₂O absorption heat pump systems.

A major practical problem with previous versions of the refractometer was the difficulty of installing the fiber optic sensors on the heat exchange tubes. This problem was finally solved by fabricating a fiber optic mount that maintains alignment of the exposed portion of the fiber optic. Two small Teflon blocks attached to either end of a stainless steel brace are now snapped onto a heat exchange tube. The blocks contain a hole to receive and to hold the stripped fiber optic parallel to

the heat exchange tube. Use of the new mounts has considerably reduced the time required to install the optical fibers within the absorption vessel. Fibers can be removed and replaced within a couple of hours without breakage. Because the alignment of the sensor is fixed, variability of the calibration curve from run-to-run has been reduced.

The new electronic and optical components improved both the short and long term stability of the fiber optic refractometer. The use of individual light-emitting-diodes (LED) as sources resulted in a more uniform and equivalent illumination of the optical fibers. The variation in LED intensity, monitored by reference photodiodes that measured the back-scattered light from each LED, was less than 0.1% over a 3-month period.

Absorption experiments to determine the reproducibility of the experimental data were completed. Experimental conditions, after the system stabilized, were 50% LiBr recirculating at approximately 100 mL/min, coolant water temperature of 18°C flowing at approximately 2.5 L/min through the absorber heat exchange tubes, 40°C sump temperature, and 300 watts heat input. A consistent decrease in LiBr concentration was observed from heat exchange tubes 1 through 3, followed by an increase in LiBr concentration on tube 4. The concentration of LiBr on tubes 2, 3 and 4 was $0.988 \pm .003$, $0.953 \pm .024$ and $0.987 \pm .014$, respectively, relative to the LiBr concentration present on tube 1. The mean heat recovery for six runs was $88 \pm 5\%$.

These results were compared with those obtained in the presence of various concentrations of 2-ethylhexanol. This particular absorption additive has been described in the literature as increasing absorber efficiency by 10 to 20%. Data from experiments in which the initial additive concentration was $\leq 0.03\%$

indicated that most of the 2-ethylhexanol is removed by the degassing procedure before the absorption experiment is initiated. Less dilution of LiBr was observed on the heat exchange tubes in these absorption runs than observed without the additive, indicating that absorption was not occurring. At initial concentrations of 2-ethylhexanol greater than 0.3%, measurable quantities of the additive were collected at the end of the absorption experiments. Even at this higher additive concentration, little or no absorption was observed. Variations in the method of addition of the 2-ethylhexanol to the absorption vessel, in the concentration of LiBr charge, and in the solution degassing procedures did not improve results. The vessel was tested several times to ensure that there was no measurable in-leakage of noncondensable gases during the course of the absorption experiments that might alter the performance of the additive.

Failure to observe an improvement in the heat absorption of our miniabsorber might be attributed to construction differences between it and equipment described in the literature. Copper heat exchange tubes were used in absorption experiments in which 2-ethylhexanol was observed to improve the absorption efficiency. The heat exchange tubes in the miniabsorber are made of stainless steel, which may alter the LiBr-metal surface interaction such that the additive would have no effect. A second possibility is that the absorber and generator are normally separate vessels in most absorption heat pump equipment; both functions are combined as a single unit in the miniabsorber. The rapid boiling and erratic pressure changes resulting from this type of absorber configuration may negate the effect of an additive. Because of these differences in experimental apparatus, further testing of additives

will be delayed until a new absorption machine, more closely matching the systems in which the previous additive experiments were performed, is available.

A measurement system is also being developed for ammonia-water absorption equipment. Because the refractive index of ammonia-water solutions is much less than LiBr-H₂O fluids, the geometry of the fiber optic sensor must be sharply curved to accommodate the different measurement range. A more intense light source and/or a more sensitive photodetector must be used to compensate for the less effective light transmission of this sensor. A small high-pressure vessel has been assembled to study the characteristics of the sensor over the 2-50% ammonia concentration range. A diode laser has been received and its drive circuit fabricated. The diode laser will be used to illuminate the ammonia probe.

(D. T. Bostick, L. N. Klatt, A. Fong*)

*ORAU summer student

Counterfeit Deterrence and Detection

A new program, funded as a Work-For-Others contract from the Department of Treasury, Bureau of Engraving and Printing (BEP) was initiated this year. The objective of this program is the development of concepts and the requisite instrumentation for the detection of counterfeit documents.

The original goal was to develop an instrument for the detection and encoding of a specific tag added to currency. Good progress was made in meeting this objective; however, during a meeting of the various BEP contractors it became apparent that the specific tag concept for which we were designing instrumentation

could be easily defeated by a counterfeiter. As a result, our involvement changed from strictly an instrumentation development program to a basic research program. We have developed alternate counterfeit deterrence concepts and have proved technical feasibility of one of the concepts. This concept has been received by BEP staff with considerable interest, and we expect to receive funding to continue its development. The ultimate goal is to provide a technology base for the Department of Treasury so it can implement new methods of counterfeit deterrence on rather short notice.

A secondary part of the work involves serving as adversaries to ideas presented by other contractors as possible deterrence concepts. We have reviewed an interesting concept presented by another group and showed that their concept could be easily defeated.

(L. N. Klatt, J. M. Dale, R. E. Valiga,
J. M. Ramsey)

ACTINIDES AND ISOTOPIC MASS SPECTROMETRY

R. L. Walker

This group performs research and development in mass spectrometry of solids and gases with emphasis on actinides and fission products. The group also develops and improves measurement schemes for precise isotope ratio analyses of a wide range of stable isotopes enriched by the electromagnetic process. Other activities include analytical support for a variety of Laboratory research programs.

Development of the NBS Beryllium Isotope Standard Reference Material

The National Bureau of Standards
(K. G. W. Inn, J. D. Fassett and B. M.

Coursey), in conjunction with the Oak Ridge National Laboratory (ORNL) and the Accelerator Mass Spectrometry (AMS) community, is in the process of developing a beryllium isotopic solution Standard Reference Material (SRM). The beryllium isotopic SRM will serve as the primary measurement standard for AMS studies of important geoscience fields such as plate tectonics, terrestrial accumulation of cosmic dust, meteorite irradiation histories, soil erosion, terrestrial magnetic reversals, solar radiation cycle, and sediment and ice accumulation rates.

Since there was no existing beryllium isotopic SRM nor any high purity beryllium-10, several mass spectrometric methods were employed for the isotopic concentration determinations in an attempt to evaluate the isotopic discrimination effects in the measurements. The atom counting techniques employed were resonance ionization (RIMS), secondary ionization (SIMS), inductively coupled plasma, and thermal mass spectrometries. The RIMS and SIMS results were the most reliable and were used to certify the beryllium isotopic material (± 5 percent). The certified beryllium material was then measured by radioactivity (liquid-scintillation counting) and finally diluted to a $^{10}\text{Be}/^9\text{Be}$ atomic ratio of approximately 3×10^{-11} . The diluted material is being measured in an interlaboratory AMS comparison exercise and will be issued as a SRM in the near future.

(R. L. Walker and S. Raman*)

*Physics Division

Safeguards-Related Projects

The lutetium double spike technique, reported in last year's annual report

(ORNL-6357), has been verified as a valuable safeguards method by British Nuclear Fuels personnel. They report precisions of 0.04% for volume determinations of plutonium holding tanks. This success, coupled with ours, has resulted in plans being developed to evaluate the technique in collaboration with Savannah River Laboratory personnel. One of their tanks will be used for evaluation under conditions more nearly reflecting a typical working environment than we have encountered heretofore.

Evaluation of a Finnigan-MAT THQ quadrupole mass spectrometer has been completed. It is being considered for use as an on-site inspection device by the International Atomic Energy Agency to help verify conformance with the arms limitation treaty. The experiment, set up in consultation with IAEA and staff members of the West German reprocessing plant in Karlsruhe, revealed far more serious shortcomings in sample preparation and filament loading than in purely mass spectrometric operation. We recommended a more thorough evaluation under more controlled conditions. Although it seemed that the THQ was capable of providing results of sufficient quality, this was not proven in the experiment. A few doubts remained that demand resolution before extensive installation of such instruments can be contemplated.

(M. P. May, R. L. Walker, D. H. Smith, J. A. Carter)

Isotope Ratio Mass Spectrometry

The feasibility of using the VG354E mass spectrometer for isotope ratio measurements on gases was successfully demonstrated. Measurements of the major isotope ratios of krypton and xenon were made with errors of less than 20 ppm on the pure gases.

Multi-collector (MC) detection offers the possibility of improving measurement precision with small samples and of minimizing analysis time. Use of the VG354E for high precision analysis of light elements by MC detection is not always possible because of the large (550 mm) dispersion of the instrument. We developed a sequential mass measurement method for the isotopic analysis of calcium. Samples from an ion-exchange enrichment program were analyzed with a precision of about 0.2% rel. std. dev. for the ratio 40/44.

Limitations on the MC detection technique for high precision work are the determination of relative gains of the detectors and the stability of these gains with time and temperature. The stability of the magnetic field is also critical. We have deduced, and in some instances employed, ways in which to minimize the adverse aspects of MC measurements so that very precise measurements can be made.

We have evaluated the analysis of tin (Sn) by single filament, thermal ionization mass spectrometry. For this effort pulse counting detection is required to obtain enough sensitivity, since tin is difficult to ionize thermally (I.P. 7.3 eV) and most tin compounds suitable for filament loading are volatile at low temperatures.

We first established and optimized our operating parameters by adding extra gain capability to the sweep amplifier. This allows us to sweep the 10 isotopes of normal tin (112, 114, 115, 116, 117, 118, 119, 120, 122, and 124) plus the 126 mass from fuel burnup production. This span of 15 mass units necessitates a steady signal. A nominal rhenium filament loading of $\approx 1 \mu\text{g}$ runs at $1400^\circ \leq$ and magnet settings of voltage = 10, amp = 5. This $1 \mu\text{g}$ filament loading is stabilized and enhanced by adding first a very small

quantity of silica gel slurry to the filament, followed by the 1 μg tin and then 1 μl 0.75N H_3PO_4 . Using this procedure, count rates $>1\text{E}5$ could be maintained for 18 or 19 runs. Comparing our isotopic measurements to the International Union of Pure and Applied Chemistry (IUPAC) values, we found excellent agreement for masses 112, 114, 116, 118, 122 and 124. Interferences at 115 and 119 are most likely due to In^+ and RhO^+ species, respectively. Mass 117 problems are minimal and could generally be controlled by altering operating conditions. We found that we could run a minimum of 0.2 μg tin. Our ability to run small amounts of tin can be applied to fuel burnup studies, isotope abundances and cross section measurements.

(T. R. Mueller, D. H. Smith, R. L. Walker)

Mass Spectrometric Projects and Support

We continued to provide analytical support for the transuranium production, stable isotopes, and radioactive isotopes sales programs. The isotopic analysis of gadolinium and europium in support of the production of ^{153}Gd was continued. Gas analysis continued to be an important activity of the group. Most of the work was done in support of the Y-12 plant. We also provided unique gas analyses in support of research and sales programs. Samples were analyzed for gas composition as well as isotopic content of the special element of interest (e.g., hydrogen, carbon, sulfur, krypton, and xenon).

We have been working with members of the Physics Division on a long-term actinides research program. This year we concluded analyses of specimens from the second fuel pin that had been irradiated for 90 full-power days in the Dounreay Fast Breeder Reactor. This program will continue over

the next year or so with the analysis of the No. 4 fuel pin after it has been irradiated for more than 180 full-power days.

Support was continued to a long list of projects this past year. These included measurements of low-level plutonium in Y-12 sewer discharges, isotopic and concentration analysis of uranium and plutonium in waste streams for environmental monitoring, certification analysis of uranium in a variety of sample types for Y-12, thorium in iridium used for space power probes, thorium and uranium in rocks and salt brines for waste isolation research (disequilibrium studies), thorium and uranium analyses in zircoloy cladding material, and isotopic analyses supporting site cleanup programs such as Formerly Utilized Sites Remedial Action Program (FUSRAP) and Environmental Restoration and Facilities Upgrade (ERFU).

(L. K. Bertram, R. M. Coleman, R. J. Hydzik, M. P. May, T. R. Mueller, C. A. Pritchard, L. Guinn, M. M. Honaker, E. G. Miller)

SPECTROSCOPIC R&D PROGRAMS

D. H. Smith

This group performs research in a wide variety of areas. The analytical utility of positrons is being investigated. Heavy-ion induced X-ray satellite emission (HIXSE) is being developed as an analytical tool. The application of laser ionization to isotope ratio mass spectrometry is being pursued. Organic mass spectrometry, especially MS/MS, represents a significant part of our effort. In all of these areas, both basic and applied research is carried out with a view to expanding the field and enhancing the arsenal of analytical chemistry.

Resonance Ionization Mass Spectrometry

For any measurement of the nuclear properties (e.g., half-life, reaction cross section, etc.) of promethium, a unique lanthanide with no stable isotopes, it is necessary to correct for the (isobaric) samarium content of samples. Resonance ionization mass spectrometry (RIMS) has been shown to circumvent this problem. Promethium was selectively ionized using a flashlamp-pumped rhodamine 6G dye laser focused into the plume of atoms from a hot rhenium filament in the source region of a single magnetic stage mass spectrometer. A small charcoal fragment in the filament had been loaded with 100 and 600 ng of promethium and samarium, respectively. The two strongest promethium resonance ionization lines in the R6G range (580 - 614 nm) occur at 584.6 and 585.0 nm. The 584.6 nm line is one nanometer from the nearest samarium lines and an elemental selectivity factor of at least 1000:1 is estimated for promethium over samarium. Strong resonance ionization lines of samarium are found nearby at 587.55 and 587.8 nm in the same dye range. Due to this proximity of promethium and samarium lines within the range of a powerful and long-lived laser dye, this single-laser method should find use as a convenient method for isotopic analysis of promethium in the presence of its daughter, samarium. These results have been accepted for publication in Analytical Chemistry.

These resonance ionizations are thought to be 1+1+1 (i.e., three-photon) processes. Each ionization pathway begins with an electronic transition from a low lying state, followed by an additional bound-bound transition, and finally excitation to the continuum. The photons required for each step are all available within the 3 cm^{-1} spectral bandwidth of the dye laser. For the 584.6 nm promethium

line used above, the first step is from the ground state to 17105 cm^{-1} .

Promethium and samarium resonance ionization spectra have also been recorded for the coumarin 522 dye range (528 - 560 nm). A total of 45 promethium and 40 samarium resonances have been observed.

A new magnetic sector mass spectrometer has been constructed in our laser laboratory at ORNL. This instrument is identical to the spectrometer used for past RIMS research at Y-12, with the exception that it is controlled by either the usual PDP-11 computer or an IBM PC. The former is useful for mass scans and makes use of the mass spectrometric software developed over the past decade. The PC controls the mass spectrometer, scans our dye laser, and logs data for RIMS optical scans. The proximity of the new mass spectrometer to several types of lasers (Nd:YAG-pumped dye laser, copper vapor laser-pumped dye laser, flashlamp-pumped dye laser, and stabilized single-frequency ring dye lasers) will allow many new RIMS investigations to be initiated.

(L. K. Bertram, R. W. Shaw, D. H. Smith, J. P. Young)

Organic Mass Spectrometry

The missions of this group are to perform basic research in the use of various types of particle interactions for the study of polyatomic molecules, with particular emphasis on enhancing analytical capabilities and to apply these findings to difficult or nonroutine problems. All of the current projects involve the use of one or more forms of mass spectrometry as the analytical tool and most employ mass spectrometry/mass spectrometry (MS/MS). Major efforts in

applications projects have been made in explosives detector development and in the chemical ionization and MS/MS analysis of geoporphyrins. Significant progress in more general research projects has been made in ion trap chemical ionization and MS/MS, the characterization of the atmospheric sampling glow discharge ionization source, the evaluation of a glow discharge interface for liquid chromatography/mass spectrometry (with the Organic Chemistry Section), in the construction of instruments to study the dissociative charge exchange of polyatomic molecules, and the interactions of positrons with organic compounds.

Over the last two and one-half years this group has developed an explosives detector based on an atmospheric sampling glow discharge ionization source (patent applied for in June 1987) coupled with a unique MS/MS instrument constructed in-house. The prototype consists of a quadrupole mass filter as the first stage mass spectrometer and a time-of-flight instrument as the second stage mass spectrometer. A 180° electric sector is incorporated into the time-of-flight portion of the instrument. During 1987, a second explosives detector, based on the prototype but without the electric sector, was constructed in cooperation with E. R. Rogers, T. T. Adams, and E. R. Flanagan of the Y-12 Plant Laboratory. Considerable effort went into making the new explosives detector very simple from the operational standpoint. This required the new instrument to be highly automated. The instrument continuously samples the air. Using the first stage mass spectrometer, a series of m/z values corresponding to ions formed in the ion source from explosive molecules are monitored. If a signal is observed that exceeds a threshold level, the instrument automatically obtains the MS/MS spectrum and sets off an alarm if the

appropriate fragment ions are detected. A simple system for sampling letters and packages was also developed for the new instrument, consisting of a hand-held vacuum cleaner and a sampling tube containing quartz wool. Air from around the package is drawn through the sampling tube (and over the quartz wool) by the vacuum cleaner. The sampling tube is then removed from the vacuum cleaner and is inserted into a heated block attached to the inlet of the ion source. The explosive molecules that are adsorbed on the quartz wool then thermally desorb and are drawn into the ion source. The new instrument will shortly be delivered to another site in the Y-12 Plant for routine use.

Work has also proceeded, in parallel with the construction of the instrument for Y-12, in research to improve the detection of explosives. This work focusses on the use of the Ion Trap Mass Spectrometer (ITMS) as the MS/MS instrument. The ITMS has several features that make it potentially superior as the analyzer for ions formed in the glow discharge source to the present beam-type instrument, viz. the quadrupole/time-of-flight spectrometer. The ITMS is a much smaller device and operates at a much higher background pressure thereby reducing pumping requirements. Further, the ITMS is a more efficient MS/MS instrument in that it can allow MS/MS to be performed on a much greater percentage of parent ions. This feature is a result of the fact that the ITMS is an ion trapping instrument in which the time spent trapping and storing the parent ions can be much greater than the time of MS/MS analysis. The fragment ion mass resolution afforded by the ITMS is also much better than that available from the present beam instrument. This feature improves the specificity of the analysis.

A major question to be addressed in the use of an ITMS with the atmospheric sampling glow discharge ionization source is the efficiency with which ions can be injected into the ITMS. We have constructed, assembled, and vacuum-tested the hardware necessary to make the first tests. The instrument is built within a 6" cube and sits on a wheeled cart that holds the roughing pumps. We await the delivery of a new ITMS, which is anticipated early in 1988, for the electronics necessary for the next step in this work.

A significant effort has been devoted to the chemical ionization and MS/MS analysis of porphyrins, particularly geoporphyrins. Porphyrins are an important class of biological compounds, and the geoporphyrins are particularly important as biomarkers in oil exploration. The molecular weight distribution and structural composition of the porphyrins in a petroleum sample can reveal important information regarding an oil field. Mass spectrometry is the method of choice for obtaining the molecular weight distribution, but little structural information is available from electron impact ionization. Other, less sensitive, methods such as NMR are therefore used to obtain this information. It is highly desirable to be able to maximize the structural information obtainable from mass spectrometry. MS/MS is an obvious possibility, but porphyrin molecular ions are very stable and do not fragment extensively using kiloelectron-volt collision-energy MS/MS. Structural information is limited to the R groups attached to the porphyrin macrocycle. It is important, however, to know which R groups are attached to each pyrrolic unit of the macrocycle. It has been reported that in certain types of chemical ionization (CI) that a porphyrin is reduced to the porphyrinogen. In hydrogen CI an

ion is observed corresponding to $(M+6H)^+$ and in ammonia CI an ion is observed corresponding to $(M+7H)^+$. Whenever these ions are observed, intense lower mass ions are also observed that correspond to the pyrrolic fragments. It has been speculated that these fragments come from the porphyrinogen. Several groups have noted this observation but report that the formation of these ions is highly irreproducible. We have studied the CIMS of several porphyrins using a variety of reagent gases and have established conditions under which the porphyrinogen ions are readily formed. The porphyrinogen ions are much less stable than the corresponding porphyrin ions and fragment readily in the MS/MS experiment. The major fragmentations are successive losses of pyrrolic units. From these studies we have been able to deduce the mechanism for porphyrinogen formation in high pressure CI sources and to devise a scheme to obtain the necessary structural information for porphyrins using MS/MS methods. We have also demonstrated that the pyrrolic fragments do come from the porphyrinogen.

In related work, we have also begun to evaluate the ITMS as an instrument for the analysis of porphyrins. The ITMS is much more sensitive than the sector instrument used for the bulk of the CI work on porphyrins. We have not, however, been able to form the porphyrinogen ions observed in the high pressure CI source using chemical ionization as performed with the ion trap. There are important differences in the two approaches to performing CI. The lack of reduction to porphyrinogen in the ITMS has important implications in elucidating the mechanism for reduction in the high pressure CI source of the sector instrument. Nevertheless, we have made some interesting observations with the ITMS

concerning the low collision energy MS/MS of protonated porphyrins and porphyrin molecular ions. We are currently testing other possible means for forming porphyrinogens in the ITMS.

A number of other studies have been performed focussing directly on important variables in the performance of CI and MS/MS in the ITMS. Many of these studies grew out of work performed for Martin Marietta Denver Aerospace, shortly after the ITMS was installed, in evaluating the ITMS for use on the proposed space station. The ITMS affords much greater control over the identity of the reagent ions for CI than is possible in a conventional high pressure ion source. For example, by one or more of several means, one or several species of ions can be selectively stored to serve as reagent ion(s) for a sample molecule in the ITMS. Much less control is possible in conventional CI. In conventional methane chemical ionization, both CH_5^+ and C_2H_5^+ are formed and serve as reagent ions. The CI mass spectrum therefore reflects products of the reactions of both ions. In the ITMS these ions can be isolated so that the ion/molecule reactions of each reagent ion can be studied separately. This capability has led us to a number of interesting observations that reveal some of the mechanisms likely to contribute to conventional CI mass spectra. CI studies on the ITMS in conjunction with MS/MS have also revealed cases where the sites of protonation are a function of the reagent ion, which leads to isomeric ions being formed from a single sample compound.

The atmospheric sampling glow discharge ionization source has been further characterized during this year, and a number of compounds have been tested with it. A number of vapors of flammable liquids has been tested and mass spectra and MS/MS spectra have been acquired.

Included among these are various alcohols, ketones, gasoline, kerosene, jet fuel, perfumes, and lighter fluid. The liquid mixtures were also characterized by GC/MS using 70 eV electron impact ionization.

A project was undertaken to evaluate the glow discharge ionization approach as an interface for a liquid chromatograph in collaboration with R.S. Ramsey of the Organic Chemistry Section. Preliminary work has been promising. Initially, difficulties were encountered in maintaining a stable discharge when large doses of solvent were introduced into the ion source. These problems have largely been eliminated, and studies are currently under way to optimize the ion source and electrode geometries for best sensitivity.

The construction of two new instruments has begun during this year for two promising new areas of research. One instrument is being built in collaboration with D. E. Goeringer, and the other is being assembled in collaboration with D. L. Donohue and L. D. Hulett. The former instrument is being constructed to study the dissociative charge exchange reaction for polyatomic ions and consists of a first stage magnetic sector followed by a collision cell, deflector plates, a flight tube, and a time- and position-sensitive detector. Time- and position-sensitive detection will allow for determination of the masses of the fragments along with the kinetic energy released in the dissociation. This method allows us to study the dissociation of neutral molecules resulting from charge transfer or electron detachment. These reactions have large cross sections and may therefore prove to be very useful analytically in MS/MS. We have recently modified the VG ZAB mass spectrometer to perform neutralization-reionization mass spectrometry (NRMS). The first step of this experiment is the dissociative charge

exchange reaction, and the second step involves collisional reionization of the products. NRMS will be useful to give supporting information regarding the dissociative charge exchange results obtained using the new instrument.

A time-of-flight mass spectrometer is being constructed to be added to the ORELA positron facility described below. The objective is to study the ionic products resulting from the interaction of slow positrons with organic molecules. We are interested in the mechanism(s) of ionization and the dependence of ionization probability on the nature of the molecule. Whether the ionization of polyatomic ions by slow positrons will ever be useful is unknown, but it could possibly be a soft and highly selective ionization method for some molecules.

(G. L. Glish, S. A. McLuckey, H. S. McKown, G. J. Van Berkel,* K. G. Asano, E. H. McBay, B. C. Grant)

*Postdoctoral fellow

Heavy-Ion-Induced X-Ray Satellite Emission

In the diverse field of materials science, analytical methods are needed not only to identify the elemental composition of a sample, but also to characterize the chemical state of the constituents and to provide insight into their chemical bonding. Heavy-ion-induced X-ray satellite emission (HIXSE), an X-ray fluorescence technique that uses energetic heavy ions to excite intense, chemically-sensitive satellite lines, is being evaluated for this role.

In order to take advantage of HIXSE's chemical sensitivity, a high-efficiency, high-resolution, von Hamos X-ray spectrometer is being constructed. The

spectrometer has been designed for an energy range of 0.5 to 12 keV (using several Bragg crystals) and a resolution of 0.04%. A multichannel, position-sensitive proportional counter (PSPC) with an energy bandwidth of 10% of the central X-ray and vertical focusing optics combine to provide high efficiency. A 10 cm x 10 cm Bragg crystal, with a radius of curvature of 25.4 cm, and the PSPC, with an active length of 10 cm are mounted on prealigned, parallel, dove-tail tracks in a stainless steel, high vacuum chamber.

The centerpiece of the von Hamos spectrometer is the linear PSPC. This detector can be viewed simply as a real-time photographic plate; i.e., the position of the dispersed X-ray on the PSPC is translated, using resistance-capacitance position encoding, in real time, as an equivalent energy on a multichannel analyzer. For optimal performance, the PSPC must have good spatial resolution and high conversion efficiency over a wide range of soft X-rays. The 150 μm spatial resolution of the model 1100X (Ordela, Oak Ridge, TN) is sufficient for the spectrometer design. In addition, its 8 mm height (20% smaller than most commercially available PSPC detectors) improves the overall instrumental resolution by eliminating a greater fraction of the image-broadened wings. The smaller detector height and, consequently, reduced efficiency is compensated for by using a high-efficiency conversion gas (Xe/CO_2) at high pressure 2E5 Pa (1500 torr). The high conversion efficiency gas also mitigates parallax. The conversion efficiency of the 1100X using a 250 μm Be window is greater than 80% between 5-9 keV but falls off to \approx 20% at 2.5 keV because of absorption by the Be window.

The performance of the new 1100X PSPC was examined by measuring its detection

efficiency for, and resolution of, Mo L (2.3 keV) and Ti K (4.5 keV) X-rays, produced via electron bombardment. The instrumental resolution of ≈ 1.2 eV for the Mo L and ≈ 2.1 eV for the Ti K X-rays was judged to be within the design specifications. As expected, the efficiency of the 1100X for Ti K X-rays was excellent, while the efficiency for Mo L X-rays clearly indicated the need for an absorption correction factor when comparing the relative intensities of different transitions. The mitigation of parallax minimized the need for precise alignment of the detector with respect to the central ray of the source-crystal-detector.

The spectrometer vacuum chamber has also been modified to accept a retractable energy-dispersive Si(Li) detector. This detector will be used for measuring cross-sections and determining α/β branching ratios. In addition, it may also be used for low-resolution analysis of X-rays produced by particles other than heavy-ions (e. g., photons, positrons and electrons).

(T. M. Rosseel)

ORELA Slow Positron Facility

The Oak Ridge Electron Linear Accelerator (ORELA) provides photoneutrons by bombarding a tantalum target with 150 MeV electrons, which produces gamma bremsstrahlung. About 15% of the gamma energy is irretrievably lost through forward scattering beyond the target. A facility has been built for insertion behind the tantalum target to capture the excess gamma energy and convert it to slow positrons.

Annealed tungsten vanes, mounted in a water cooled enclosure, will intercept the gamma beam and produce high energy positrons and electrons through pair formation. The positrons are thermalized,

and some of them escape with energies ranging from 0-2 eV. Tungsten plays two roles in this process: the high mass of its nuclei promotes pair formation, and it has a property known as the negative work function for positrons. Thermalized positrons are in a higher state of potential energy inside the tungsten than on the outside; thus the thermalized positrons are spontaneously emitted from the tungsten, provided they can diffuse to within the escape distance, about 100 nm, of the surface. The majority are annihilated before they can escape, but about 0.1% reach the surface and are expelled. Calculations indicate that the excess gamma flux of the ORELA will produce about 10^8 slow positrons per second.

The ORELA is a pulsed accelerator, having repetition rates variable from 30-1000 hz. Pulse durations are about 40 nanoseconds. Slow positron emission is effectively instantaneous and tracks the accelerator pulses. An extraction tube, placed immediately in front of the tungsten vanes, is driven negative during the burst of slow positron emission, accelerating them down the axis of an evacuated magnetic solenoid. The solenoidal field guides the positrons to an adjacent experiment room, about 10 meters away, which is shielded from the gamma flashes of the accelerator. The slow positrons will be delivered in bursts of about 10^5 during the 40 nanosecond cycle, followed by dead times of 1 ms or longer. This rate will be too fast for most digital counting techniques, so provisions have been made to capture the positrons in a Penning trap and disperse them over longer time periods, approximately equal to the dead times.

Construction of the facility components, which include the solenoids, Penning trap, and pulsing electronics is

now 100% complete. The target room solenoid, extraction tube, and tungsten vanes are being installed. Shielding of the target room solenoid, such that neutron and gamma radiation are blocked from the experiment room, is a major problem that must be overcome.

The slow positron beam will be utilized in many projects of analytical chemistry research, involving both gases and solids. The first experiments to be done will be the 'soft ionization' of gaseous molecules to produce ions for mass spectral measurements. Ordinary methods of ionization, such as bombardment by electrons or photons, often leave the ions in excited states which causes unpredictable fragmentation processes. When slow positrons attach to molecules, ionization does not occur until after the molecule has time to adjust to the presence of the extra positive charge. The relaxation processes that occur after the electron is removed are not as violent.

(L. D. Hulet, Jr., T. A. Lewis,* D. L. Donohue, S. Pendyala,** K. B. Lim***)

 *Instrumentation and Controls Division
 **On sabbatical, State University of New York, Fredonia
 ***GLCA/ACM student, Coe College

INORGANIC AND SECONDARY ION MASS SPECTROMETRY

W. H. Christie

This group conducts research in both organic and inorganic secondary ion mass spectrometry (SIMS) and provides specialized analyses that require the extreme sensitivity of the SIMS technique.

Inorganic SIMS

In a collaborative effort with workers at both Sandia and Lawrence Livermore National Laboratories, we have used SIMS analysis to study the interaction of high-power pulsed microwave radiation with arsenic ion-implanted silicon wafers. Using SIMS to profile the implanted arsenic, we have shown that microwave pulses can melt the near-surface region of the material for pulse powers exceeding a threshold energy of about 1.1 MW. Inhomogeneities in the microwave beam produced a silicon surface with adjacent smooth and rippled areas. The arsenic profile in the smooth region is approximately the same as the arsenic profile in unirradiated samples. Arsenic diffuses rapidly in liquid silicon. SIMS measurements showed that the smooth regions of the surface did not melt since no redistribution (diffusion) of the arsenic was noted in this region. The arsenic profile in the interfacial region between the smooth and rippled areas is different from that in the arsenic-implanted sample; however, the observed redistribution is not consistent with the diffusion of arsenic in liquid silicon. Instead, it appears that some material has been redeposited on the surface to a thickness of about 60 nm. In the heavily damaged regions we found that most of the arsenic atoms (> 96%) have been lost by vaporization of the surface. For arsenic atoms that did not evaporate from the surface, the depth profile in the near-surface region is approximately uniform, which is consistent with the diffusion of arsenic in liquid silicon.

Our next concern was to identify the material that had been deposited on the target surface. SIMS analysis showed the presence of F, Cl, Al, Mg, and B in the

damaged region and the area adjacent to it. These elements were not observed in the as-implanted samples. The presence of fluorine and chlorine are believed to indicate a breakdown of the Freon-12 backfill gas. The presence of aluminum, magnesium, and boron shows that the plasma formed during irradiation causes the ablation of the Macor used to support the samples in the center of the waveguide.

In conclusion, the results of SIMS analysis show that melting and vaporization of the near-surface region is possible using pulsed microwave radiation. The usefulness of this technique as a means to remove ion implantation damage and to electrically activate implanted dopants will require that the problems associated with the inhomogeneous energy deposition and subsequent damage be overcome.

(W. H. Christie and R. E. Valiga)

Organic SIMS

Distributions of various compounds within biological systems play an important role in the understanding of how such systems work. For example, the high concentration of DNA in cell nuclei provides clear evidence that DNA performs some function with regard to genetics. There are other types of pharmaceutical, medical, and general biological problems in which knowledge of if and where particular chemicals and their metabolites are localized would be invaluable. Localization in many cases need not be, and in many cases is not, confined to particular organelles within cells, but rather to particular tissue areas of individual organs. One analytical method which may prove useful in providing information about such distributions is SIMS.

A conceptually simple approach is to raster a finely focussed primary ion beam of keV energy ions back and forth across a tissue sample and plot the intensity of secondary ions with particular m/z values emitted from each position on the sample. This technique works well with inorganic samples, and yields ion micrographs with up to 40 nm spatial resolution. It does not work well for biological samples because such samples are inherently complex mixtures, and at each m/z value, there will be secondary ions with the same nominal molecular weight (isobaric interference) as well as ions with the same exact mass as the ion of analytical interest, but with a different structure than that characteristic of the analyte of interest (isomeric interference). To solve these problems, a special organic SIMS instrument was constructed at the National Institute of Mental Health that employs MS/MS, a technique that can eliminate such interferences. While the concept is simple, execution is not, and the instrument was not capable of providing ion micrographs.

This machine was redesigned and reconstructed at ORNL and organic ion micrographs have been obtained. At present, these images are of liquid samples dispersed across a grid, and show a spatial resolution of less than 0.5 μ m. Nonetheless, these images are the first of their kind. They provide proof of principle. Additionally, spatial distribution of fragment ion intensities show that many of the fragment ions which are observed in organic SIMS spectra arise from locations where liquid sample is depleted.

A new secondary ion source for this instrument has been constructed and installed and improves the transmission by a factor of about 1000. Other improvements,

including redesign of the primary ion source, promise to provide submicron spatial resolution and improved secondary ion yield.

(P. J. Todd, C. P. Leibman,* D. H. Friar)

*Predoctoral Student, University of Tennessee, Knoxville

Sputter Atomization/Resonance Ionization Mass Spectrometry

We have continued to evaluate the combination of sputter atomization and resonance ionization mass spectrometry (SA/RIMS) as a technique for high sensitivity materials analysis. Pulsed laser ionization is an attractive process for coupling with ion beam sputtering because the ionization process can be saturated if the laser beam is sufficiently powerful. In addition, when pulsed sputtering is utilized, the spatial overlap between the sputtered plume and laser beam can approach unity. Therefore, efficient laser post-ionization of the sputtered neutral fraction presents the possibility for enhanced sensitivity.

Previous results with the uranium-containing samples U metal, UO_2 , and U_3O_8 , indicated that the density of ground-state U atoms sputtered from such samples is quite sensitive to oxygen content and surface contamination. To demonstrate and quantify the effect of sample matrix on atomic neutral yield, a set of depth profile measurements using SA/RIMS was made on a thin film UO_2/U metal sample. Careful determination of the sputter yield (U atoms ejected/incident ion) for Ar^+ on U metal and UO_2 indicated that, in the absence of any matrix effects on sputtered atomic neutral yields, the U^+ photoion signal from UO_2 would be about 3 times smaller than that from U metal. However, the photoion

signal for U^+ from the UO_2 layer was a factor of 44 less than that from the U metal indicating that the ground-state U atom fraction was strongly influenced by a matrix effect.

A set of SA/RIMS pulse sputtering experiments demonstrating the effect of surface contamination on atomic neutral yields was also performed with U metal. The experimental results indicated that the ground-state U atom fraction from sputtered U metal is also sensitive to a matrix effect due to chemisorbed surface impurities and suggested that oxide formation on U metal is responsible for this matrix effect.

A detailed consideration of extraction efficiency, cw vs. pulse-sputtering, and spatial overlap between sputtered atoms and the photon field enabled us to estimate, within approximately an order of magnitude, the number of ground-state U atoms sputtered relative to the number of secondary U^+ ions ejected, (U^0/U^+), for cw, Ar^+ ion sputtering. The ratios for U metal and UO_2 are estimated to be 3×10^3 and 6×10^1 , respectively. Although the observed matrix effects on atomic neutral yield in these experiments are attributed to an increase in the secondary ion or molecular neutral yield, the possibility exists that the population of a low-lying excited atomic state is being increased in those instances. However, in either case the matrix effects result in reduction of the number of ground-state uranium atoms and concomitant complications for quantitative analytical measurements. The absolute efficiency for SA/RIMS of U metal, the number of U^+ photoions detected/U atoms sputtered, using our experimental setup was estimated to be 9×10^{-4} . Further analysis revealed this rather low efficiency to be due to the low transmission/detection efficiency ($\sim 1.3 \times 10^{-3}$) for photoions in our experiments.

A number of chemical reducing agents was introduced into the sputtering region in an attempt to increase the yield of sputtered atomic neutrals from oxidized forms of uranium. Three approaches were used: 1) cesium ions were employed as a sputtering beam, 2) benzene and anhydrous ammonia were impinged on the sputter targets via a capillary jet during Ar^+ sputtering, and finally, 3) Al, Ti, Zr, and C were used as substrates upon which particulate samples were dispersed prior to sputtering. During these studies, the effect on the yield of sputtered neutral U atoms was observed by monitoring the U^+ photoion signal. These experiments can be summarized simply by reporting that none of the approaches resulted in enhanced yields of sputtered U atoms from oxygen containing samples.

At one time high efficiency, laser post ionization of ion-sputtered samples was thought to offer the possibility for quantitative surface analysis without matrix effects. Studies performed under ultra-high vacuum conditions using inert gas sputtering of metallic targets showed that in some cases quantitative analyses could be accomplished without matrix effects. Unfortunately, these laboratory conditions do not exist for the analysis of all samples. The industrially generated, particulate emissions for many reactive metals of environmental significance are often not found in the metallic state; they form a variety of compounds through reaction with the environment. This work clearly indicates that the population of desorbed neutrals above such sample surfaces may not always be directly representative of its composition. The possibility that the desorbed neutral fraction can be partitioned amongst a variety of species will complicate the task of relating laser-generated ion abundances to the actual composition of the sputtered surface.

(D. E. Goeringer and W. H. Christie)

RADIOCHEMICAL RESEARCH AND DEVELOPMENT

H. H. Ross

Work on the previously described fiber-scintillator radiation detector has been successfully completed. The flow-cell radioactivity detector which we have developed (1) has shown good detection efficiency for most beta emitting isotopes when 0.1 mm diameter fibers were used. The use of the scintillator in fiber form allows improved light collection and denser packing than can be obtained with scintillator particles of the same diameter. The fiber cell has also demonstrated improved compound retention characteristics and cell back-pressure (better by about a factor of 50 over conventional devices).

The experimental efficiency obtained from the fiber cell was very close to the theoretical geometric efficiency predicted by computer modeling of the detector system. However, major deviations from the model by tritium suggest that light absorption and scattering is also a major efficiency parameter. Thus, trying to improve the geometry of a cell by using ever smaller particle sizes will not improve efficiency if the pulse-height response decreases due to photon scatter. Our conclusion, therefore, is that efforts aimed at further improvements in efficiency should be aimed at the development of better scintillator photon yields.

One measurement study that is not easily possible with either the scintillator fibers or the conventional powdered scintillators is the independent evaluation of scattering parameters in a heterogeneous detector cell. This is because there is no easy way to change either scattering or geometry without affecting both variables. To make such measurements, a different approach is needed. We carried out, therefore, a

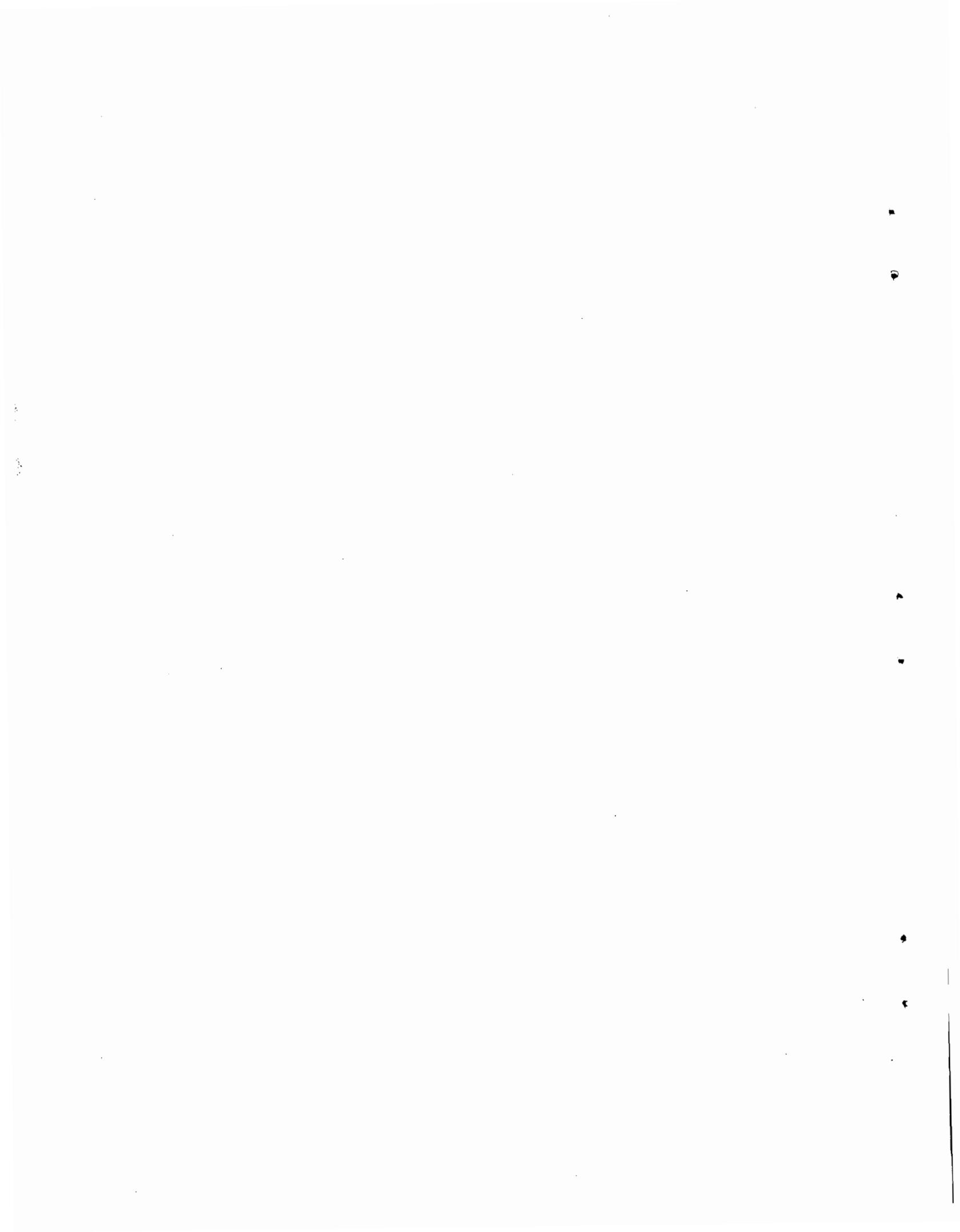
separate study designed to isolate the efficiency variables of heterogeneous detector cells. An unusual aspect of this work was the fabrication of a new type of polystyrene scintillator bead in different sizes and with uniform and smooth surface characteristics. The results of the study vividly demonstrate why, because of multiple photon scattering effects, it is futile to try to improve detection efficiency of low-energy beta emitters with ever decreasing sizes of conventional powder scintillators. The observed effects also explain why reduced efficiencies are frequently seen with very small particle size scintillators in spite of greatly improved geometry.

During the course of all of this work, our experimental work was guided by predictions from a computer model of the heterogeneous detector system (2,3). The program that we developed uses a Monte Carlo type of approach to model the geometric efficiency of a given flow-cell design. In our simulation program, the origin and direction of the radiation is generated randomly; the efficiency is determined by calculating the distance it travels in the liquid before reaching the scintillator. The process is then repeated for multiple points and directions. Our algorithm simplifies the traditional Monte Carlo technique of exhaustively following each beta event by incorporating known absorption relationships of beta spectra. The result is that our simulation can run effectively on a personal computer rather than a mainframe unit that is usually employed.

(H. H. Ross and T. L. Rucker)

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2. RADIOACTIVE MATERIALS ANALYSIS

J. R. Stokely

The Radioactive Materials Analysis Section supports a large number of Laboratory projects by providing radiochemical analysis, neutron activation analysis, and general analysis of radioactive materials. Development work consists of special projects to upgrade analytical capabilities or to solve chemical analysis problems for Laboratory projects. Small research activities are maintained to study advanced techniques for neutron activation analysis and methods for environmental surveillance of radionuclides.

Over the last year, there have been several significant changes in some of the work performed by the section. The shutdown of ORNL reactors caused a major curtailment of neutron activation analysis service work and analytical work associated with the transuranium element production program. New work was started to investigate the possible use of cold neutron sources for neutron activation analysis. Also, a major new project to characterize the contents of radioactive waste storage tanks was started. The latter project will involve considerable analytical development to devise special techniques for determination of toxic and hazardous inorganic and organic constituents in highly radioactive solutions and sludges. Environmental surveillance activities at ORNL and the DOE Site Survey Program continued to require large numbers of low level radiochemical analyses.

LOW-LEVEL RADIOCHEMICAL ANALYSIS

J. W. Wade

The Low-Level Radiochemical Analysis Group provides analytical support and services for several divisions and programs at ORNL and for others outside the DOE sponsored facilities. Some 20,000 determinations were performed this year on water, soil, sediment, air filters, and other types of environmental matrices.

Increases in our workload originated from several programs. For the DOE Site Survey Program, we have completed work on the Rocky Flats, Pantex, and Livermore/Sandia sites; we have begun work on the Argonne site. The majority of

samples were soil and water, and most of the analyses were gross alpha, gross beta, gamma scans, and isotopic uranium. We continued to provide daily analyses for the Process Treatment Plant's Zeolite Column Program. The treatment facility processes about 75% of the waste water generated by the Laboratory before it is discharged into White Oak Creek. We provide gross beta and strontium determinations that indicate "breakthroughs" of strontium from the columns. We have continued to provide support for various programs within the Department of Environmental Management on a daily basis. (Many of these samples have required us to work overtime to meet deadlines).

During the past year, we received several samples from the Nuclear Regulatory Commission for the determination of ^{210}Pb , total radium, and ^{230}Th . These samples were taken from an area where uranium milling had been done, and they contained excessive amounts of alkaline salts that interfered with our standard ^{210}Pb procedure. Several documented procedures were tried before we gave up and developed our own. The samples were evaporated to near dryness and made 1 M in HCl. Bismuth carrier was added and the samples were passed through an anion exchange column that had been converted to the chloride form. The ^{210}Bi (daughter of ^{210}Pb) was eluted with 1.5 M H_2SO_4 and further purified by precipitating any residual lead as the sulfate. The bismuth was then precipitated as the hydroxide and counted on a low background beta counter. Standards and spikes were satisfactorily processed along with the samples. We plan to test this procedure further and then add it to our bench manual.

We also measured isotopic uranium in two groups of samples for the Army's Toxic and Hazardous Materials Agency (USATHAMA). Both groups of samples were of groundwaters from an area where uranium had been processed in the past. There was some concern that uranium had percolated through the soil and contaminated the local groundwater. The second of these two groups of samples was from deep wells, where the water resembled a brine. We found that the ^{234}U activity in these samples was about a factor of two higher than that expected in nature. Consultation with other ACD staff confirmed that these were not unexpected results for deep water brines. The abnormally elevated ^{234}U isotopic ratio in deep water brines is thought to result from a geological phenomenon associated with the decay of ^{238}U . Uranium-234 is a product of the

decay of ^{238}U . The ^{238}U decay to ^{234}U involves formation of the intermediates ^{234}Th and ^{234}Pa . If hydrological and geological conditions are such that selective leaching of ^{234}Th or ^{234}Pa from uranium in soils and rocks takes place, these deep water brines will accumulate the long-lived ^{234}U daughter. This accumulation of ^{234}U causes uranium in the brines to have a higher than normal ^{234}U isotopic content.

We have completed documentation of standard operating procedures (SOP's) dealing with: sample receiving; sample log-in; sample preparation; frequency of spikes, duplicates, and standards; routing of paper to get the final report; initial setup of counting systems; and information about standards and where they should be stored and the information recorded. A form is placed in each folder that will be used to document deviations from these SOP's or routine sample analysis procedures. We have begun work to update our bench manuals so that they contain or reference EPA procedures whenever possible. Future plans are to remodel our existing sample receiving room and adjoining office and convert those rooms to a combined sample receiving/chain-of-custody room.

We have converted to a new radium counting system and calculations are now performed on an IBM PC. The counting is being done on a Tennelec LB4000, 12 detector counting system. The use of this system has allowed us to increase our throughput of samples by a factor of three. The system is controlled by an IBM PC which eliminates hand calculations and allows customer reports to be generated. The LB4000 allows us to use a ^{133}Ba tracer and count its beta emissions at the same time that the alpha emissions of the radium are measured. The "crosstalk" between the alpha and beta regions is less

than 0.02% for the amount of barium spike that we intend to use.

A new apparatus for tritium distillations was procured and installed. We have begun to use the EPA method for tritium in drinking water for all aqueous samples. This method requires distillation of the sample followed by liquid scintillation counting in an appropriate scintillator solution. We were limited to one distillation at a time prior to the installation of this new apparatus. The apparatus consists of a self-contained refrigerated pump, thermostatically controlled heating mantles and water cooled condensers. We now have a train of six distillation flasks that are cooled by the refrigerated pump.

(D. L. Dihel, L. D. Bible, M. T. Davis, S. F. Glover, P. S. Gouge, C. C. Granger, R. D. Johnson, G. C. Keyes,* S. H. Prestwood)

*Co-op student, Mississippi State University

RADIOCHEMICAL AND ACTIVATION ANALYSIS

J. F. Emery

The Radiochemical and Activation Analysis Group provides general neutron activation analysis (NAA) services for a number of Laboratory divisions and programs, in addition to radioisotope assay, gamma-ray spectrometry services, and technical assistance.

The High Flux Isotope Reactor (HFIR) has been down all of 1987; the Oak Ridge Research Reactor (ORR) was shut down the latter part of March. We have provided U and Th determinations at the part per billion level in semiconductor materials by irradiating these materials overnight in

graphite rabbits in the ORR pneumatic tube. (An overnight irradiation at the ORR is approximately equivalent to a one hour irradiation in the HFIR.) Other materials analyzed by NAA include Nb for Ta, W, and other trace impurities, brine solutions for Au (gold solubility studies); Co flux monitor wires; and Hf metal. Since the HFIR shutdown, radioisotope assays have been limited to ^{153}Gd , ^{147}Pm , $^{119\text{m}}\text{Sn}$, and ^{169}Yb , ^{85}Kr ampoules, and ^{244}Cm - ^{13}C gamma-ray sources. There were also a large number of HFIR surveillance flux monitors assayed for ^{54}Mn , ^{58}Co , ^{60}Co and $^{93\text{m}}\text{Nb}$.

Our Work-For-Others (WFO) activities, consisting mostly of NAA determinations of semiconductor materials, have virtually ceased since the shutdown of the reactors. There are three main reasons for this: (1) reactor shutdown forcing the vendors to find other assay methods, e.g. glow discharge mass spectrometry, (2) vendors perfecting their purification processes (we have been analyzing these materials for at least four years) and, (3) semiconductor manufacturers developing techniques, software-hardware and barrier layers to minimize the alpha particle effect. Other WFO activities consisted of analyzing concrete and concrete components for ^{222}Rn emanation rates, U, Th and ^{40}K (U. S. Navy) and soil samples for ^{226}Ra (U. S. Bureau of Mines).

Some of the highlights of 1987 include the following: continued work on the fuel scanning system, upgrade of the neutron generator facility, phaseout of the NAA laboratory at the ORR following the move to and setup of the new activation analysis facility at the HFIR, and conceptualization and planning for a low temperature isotopic neutron source.

The 14-MeV neutron generator facility at building 2011 once again is being maintained and operated by ACD personnel. The facility was reviewed by the

Accelerators and Radiation Source Review Committee and the Office of Operational Safety. The results of this review were favorable, and all recommendations of the committee are being implemented. Additionally, the data collection and reduction procedures at the facility are being updated. State-of-the-art electronics have been purchased with funds provided by the Metals and Ceramics Division. These new electronics will allow direct data transmission to a personal computer (PC) where results can be calculated automatically and transferred electronically into ANALIS.

The ORR has been shut down permanently. Consequently, all neutron activation analysis work at this facility has been discontinued. The laboratory apparatus, the ND 6620 and its associated counting equipment, etc., is being moved to the new General Purpose Neutron Activation Analysis Facility (GPNAAF) at the HFIR. The ND 6620 is being configured to provide ease of maintenance, and all detectors are being recalibrated to ensure the most accurate results possible. The Pneumatic Tube System Mark II (PTS MK-II) incore section was installed in June in preparation for the hydrostatic test of the HFIR. The PTS MK-II Programmable Gould Controller is presently being tested; and by the time the HFIR begins operation, the GPNAAF will be completely operational.

(F. F. Dyer, L. Robinson, N. A. Teasley)

Use of Low-Temperature Neutron Sources for Analytical Measurements

Reports in the literature indicate that neutrons moderated to liquid hydrogen temperatures and conducted through neutron guides to a remote site can be used for neutron capture prompt gamma ray analysis (CGA). Use of this technique results in

considerable gain in measurement sensitivity for chemical element determination relative to ambient temperature measurements.¹ We have begun an evaluation of possible analytical uses of isotopic neutron sources whose neutrons are moderated to cryogenic temperatures. Two systems have been identified that appear to have useful applications each in distinctively different ways. Both are variations of common neutron source designs except that the neutrons have lower energies.

The first system would make use of (α, n) sources, such as $^{241}\text{Am-Be}$. It would consist of a cryostat containing liquid deuterium, the external surface of which would be covered with the neutron source. We estimate that about 50-100 sources, each containing 1-2 grams of ^{241}Am would be necessary. A neutron reflector (probably beryllium) would be placed outside the sources. A gamma-ray shield would be placed outside the reflector. An opening would be provided through the shield, reflector, source layer, and inner cryostat to a chamber inside the cryogen where a sample might be placed for CGA. It is expected that with proper design, this system would yield a neutron flux of near $10^7 \text{ n cm}^{-2} \text{ s}^{-1}$ and that a Ge gamma detector might be placed close enough to the sample so that the measurement sensitivity might rival that of some reactor-based CGA systems. An invention report describing this system is being prepared and will be given to the ORNL Patent Office.

The second system consists of a cryostat containing a cryogen and ^{252}Cf and is being studied in cooperation with C. D. West and others in the Engineering Technology Division. Of principal interest in this system is the magnitude of the low-temperature neutron flux that can be extracted from the source with

neutron beam guides. It is expected that the neutron flux in the guide might exceed $10^8 \text{ n cm}^{-2} \text{ s}^{-1}$. It is thought that the flux in such beams would also be useful for CGA measurements as well as neutron scattering studies. If the system could be designed and built quickly, knowledge gained from its study would probably provide some beneficial input to cryostat design for the Advanced Neutron Source which is being designed at this time. An invention report for this system is also being submitted to the ORNL Patent Office. A proposal for seed money to design the system has been prepared.

(F. F. Dyer and L. Robinson)

Automation of Fission Track Counting

Riley² has reported that fission track counting with a carefully calibrated automated image-counting system results in considerable improvement in precision and accuracy over other methods of estimating track densities in the measurement of uranium by the fission track method. In preparing to resume operation with the HFIR, we have begun an effort to assemble an automated counting system patterned after the one used by Riley. For this purpose, a Nikon Optiphot microscope was purchased. With the addition of a stage for the microscope that is driven by a computer-controlled stepping motor, camera (charge coupled device), and image analysis software, the system will be ready for use. We expect to complete assembly of the system sometime within the next year.

(F. F. Dyer)

Assay of ^{153}Gd Photon Bone Scanning Pellet

An assay of ^{153}Gd in a pellet of Gd_2O_3 used for bone scanning was completed for

the Isotopes Section of the ORNL Operations Division using both non-destructive and destructive measurements with gamma spectrometry. At the time it was prepared the pellet was believed to contain $3.7 \times 10^{10} \text{ Bq}$ (1.0 Ci) of ^{153}Gd (February 2, 1987), but tests at New England Nuclear indicated the pellet contained only about $2.59 \times 10^{10} \text{ Bq}$ (700 mCi). Following the measurements described here, further examinations of the records of the source material from which the pellet was prepared revealed that the pellet activity was lower than originally believed. The pellet was returned to ORNL and assayed. Gamma spectra of the pellet were acquired with the pellet placed at a distance of 1247 cm from the end cap of a Ge gamma detector. No attenuator or collimator was used. A spectrum of a $4.70 \times 10^7 \text{ Bq}$ (1.27 mCi) standard of ^{153}Gd was also acquired at that counting geometry. Subsequently, the pellet was dissolved in a few mL of 10 M HNO_3 , and the solution was diluted to 100 mL. Aliquots of 0.500 and 0.200 mL were taken and counted at the 1247 cm geometry. Smaller aliquots were also taken and counted at conventional counting geometries. All results agreed well and confirmed the low value found by New England Nuclear.

As part of these measurements, attempts were made to determine the counting efficiency of the Ge detector at the 1247 cm geometry by counting low-level standards at source-to-detector end cap distances of 30 and 45 cm and then correcting the results to the 1247 cm counting geometry. Corrections were made for distance by the inverse square function and for gamma attenuation in air. Results obtained by this procedure yielded counting efficiencies that were approximately 20 percent lower than values obtained with the high-level ^{153}Gd

standard. The reason for this result is unknown, and further study of this problem is planned.

(F. F. Dyer and L. Robinson)

Assistance to Rocky Mountain Arsenal Project

Assistance is being given to a project that seeks to determine which analytical methods are suitable, under field conditions, for measurement of toxic chemicals in soil and water. The project, administered by the Organic Chemistry Section, is being undertaken for the US Army's Toxic and Hazardous Materials Agency (USATHAMA). The Army is in the process of decommissioning the Rocky Mountain Arsenal, and field-usable methods will be needed during the cleanup to measure toxic chemicals at contaminated sites. The efforts of this investigator have been to select for evaluation methods that show promise for determining the toxic inorganic elements copper, arsenic, cadmium, mercury, and lead. After considerable review of various instruments and methods, it was decided to evaluate a portable atomic absorption spectrophotometer for the lower concentrations of the elements, a portable X-ray fluorescence system for higher concentrations, and a system made by Jerome Instrument Company that measures mercury by allowing it to absorb on a gold film with a consequent increase in electrical resistance. Efforts to evaluate these systems are about to begin.

(F. F. Dyer)

Special Projects

Recent establishment of Canada Geese colonies on the Oak Ridge Reservation has suggested the possibility of off-site

transport of radioactivity by such migratory waterfowl. A program to investigate the magnitude of such transport has been established using field observations and detailed analyses of geese observed to reside on contaminated ponds at ORNL. The first study involved two pair of geese observed to be making nesting plans near Pond 3524 with an estimated residence time of two weeks prior to sacrifice. Autopsy samples were taken of bone, muscle, liver, feces, and crop contents from all geese. High-resolution gamma-spectrometry and radiochemical determinations were used in the detailed analyses. Control geese from off-site reservoirs were obtained from local taxidermists; those birds were analyzed in a manner similar to that of the study suite. Radioactivity levels in the control geese were extremely low (probably due to global fallout from previous atmospheric weapons tests).

Results from the detailed analyses of autopsy samples showed that Canada Geese residing on the contaminated pond at ORNL contain muscle concentrations of ^{137}Cs ranging from 50 to 225 Bq/g and bone concentrations of ^{90}Sr ranging from 15 to 45 Bq/g. From the detailed examination of the crop contents and fecal elimination, it appears that the geese obtain a significant portion of their diet from bottom sediments.

Autoradiographic studies showed generalized distribution of radioactivity within bone and associated marrow. This finding is consistent with the quantitative results that show concentrations of ^{137}Cs in soft tissue somewhat similar to those of ^{90}Sr in bone from the same bird. (The autoradiograms do not distinguish between ^{137}Cs and ^{90}Sr).

(J. S. Eldridge and B. D. Keele)

**Specialized Techniques for Radioactivity
Surveys: Autoradiography on Biological
Specimens**

Conventional radioassay procedures for biological specimens give quantitative values for specific radionuclide concentrations. Both destructive and non-destructive procedures yield concentrations that are averaged for the mass of the sample aliquot. In low-level work, several grams of specimen usually constitute a sample aliquot. In many instances, it is desirable to determine the distribution of radionuclides within the specimen or aliquot. A useful technique for delineating the distribution is autoradiography (first applied in the 1890's to investigate the phenomenon later known as radioactivity). Application of the autoradiographic technique for general purposes is simple enough: place the specimen containing radioactivity in close proximity to a photographic emulsion for a time sufficient to produce visible images at the sites of radioactivity. The darkening of the emulsion by ionizing radiation occurs in a fashion similar to that produced by visible light. With single-emulsion X-ray film and macro-sized specimens, beta radiation is considerably more effective than alpha or gamma radiation in producing the images.

Many of the current applications of bio-indicators for environmental monitoring need qualitative distribution information. One such application is the use of rabbit bones (as well as some deer specimens) as indicators for ^{90}Sr environmental pools. It is known that ingestion of ^{90}Sr by animals results in an increased uptake of the radionuclide at the sites of bone growth (joints and ends of long bones). By examination of autoradiograms of rabbit femurs, it is possible to determine whether chronic or acute ingestions of ^{90}Sr has

occurred. We have observed both types of ingestions by rabbits trapped on the ORNL Solid Waste Storage Areas (SWSAs).

The classical technique is changed in our applications by two modifications: sample preparation and exposure technique. Because our samples are biological, we have to prepare a flat surface for apposition to the emulsion side of the film. This is accomplished by sawing the frozen bone sample mounted on a wooden holder with a miniature band saw housed in a simple glove box. The flat bone (with bone dust removed by scraping) is placed in apposition to the X-ray emulsion (separated by a thin polyethylene film) in a darkroom. A special cassette is used to hold the film and sample. The cassette is then placed between two plywood boards that serve to clamp and hold the materials in place. The entire assembly is placed in a freezer and kept frozen during the exposure period. After an exposure period determined by the activity level, the film is removed and developed. Because the specimens are preserved intact by freezing, reexposure is simply done by use of a second film. Exposure periods from 70 hours to 30 days have been used during the current studies. We are currently using Kodak GBX No-screen X-ray film for the autoradiograms. Successful autoradiograms have been prepared that show ^{90}Sr distributions in bones of rabbits, deer, and geese as well as in a fern plant.

(J. S. Eldridge and B. D. Keele).

**Radiological Surveys for DOE-TWRA
Deer Hunts.**

Deer harvested by hunters during the annual DOE-Tennessee Wildlife Resources Agency managed hunts on the Oak Ridge

Reservations are surveyed for radioactive contamination to ensure that appropriate regulatory guidelines concerning radiation exposure are followed. Soft tissue radionuclide concentrations are estimated by performing a quantitative measure of ^{137}Cs (along with a check for other gamma-emitting nuclides) in a sample of liver from each deer. These measurements are conducted in a "near real-time" mode at a field laboratory at the checking station. Simultaneously with the liver assay, a screening measurement is made on a specimen of bone from the leg of each deer to detect possible ^{90}Sr contamination.

The deer harvest for 1985 was 926 animals from five separate hunts. Seven animals were retained due to ^{90}Sr contamination detected by the bone screening procedure. The maximum ^{137}Cs concentration was 110 mBq/g (3 pCi/g), and only 10 animals exceeded 40 mBq/g (1 pCi/g). The 1986 hunts were conducted as the 1985 ones with a few small changes in the hunting areas and a new location for the checking station. The ^{137}Cs contamination levels continued to be quite low and acceptable for the total harvest of 660 deer. However, the retention of deer from the hunters due to ^{90}Sr contamination showed a significant increase to a total of 29 animals. Examination of database information concerning kill locations showed a significant inverse correlation of contaminated deer with the distance from former SWSAs at ORNL. For the current 1987 hunts, a scheduling change was made so that only four separate hunts were planned (two archery and two gun hunts). After the first three 1987 hunts (two archery and one gun hunt), 400 deer had been harvested along with a total retention of 19 animals—all due to ^{90}Sr in bone.

Following the 1986 hunts, an attempt was made to use other bioindicator organisms to help elucidate the exposure pathway for

^{90}Sr to the deer. Rabbits residing in the SWSA areas have a limited home range and a diet not unlike that of deer. By trapping rabbits at specific locations near and within the SWSAs it was possible to detect locations containing vegetation with ^{90}Sr unaccompanied by ^{137}Cs . It is likely that the important source of ^{90}Sr for the deer contamination is vegetation as opposed to a drinking water source.

(J. S. Eldridge and B. D. Keele)

Radioiodine Studies in Bio-indicator Organisms

Previous quantitative determinations of iodine radionuclides in thyroid glands from "bio-indicators" have established the presence of three radioisotopes of iodine in the environment of the Oak Ridge Reservation. The most surprising of these is ^{125}I , a neutron-deficient radionuclide that is no longer made at ORNL. Earlier studies have shown that ^{125}I is released from the low level waste (LLW) system following disposal by chemists utilizing the material for organic syntheses of nuclear medicine products. Levels of this 60-day half-life radionuclide dropped considerably during the period when a storage tank in the LLW system was on stand-by. Levels increased dramatically in deer thyroids following the tank's return to service. Another interesting finding is the presence of ^{131}I , an 8-day fission product nuclide. Deer thyroids as well as two geese thyroids have shown the presence of ^{131}I . Since all reactors at ORNL have been shut-down for many months, it is unlikely that the ^{131}I was produced here. It is probable that the material was purchased for synthesis programs similar to those using ^{125}I and released by the same pathway.

Deer retained from hunters due to ^{90}Sr contamination are examined for the presence of radiiodines in their thyroid tissues. A strong correlation has been shown in these deer between the presence of ^{129}I , a 16-million year half-life nuclide, and the presence of elevated levels of ^{90}Sr in the same animals. This correlation suggests a different source term for the ^{129}I than for the ^{125}I and ^{131}I . These preliminary findings will form a basis for further studies.

(J. S. Eldridge and B. D. Keele)

INORGANIC AND PHYSICAL ANALYSIS

D. A. Costanzo

The Inorganic and Physical Analysis Group continues to provide analyses of intermediate and high-level radioactive materials that require glove box or hot cell procedures in the High-Radiation-Level Analytical Laboratory (HRLAL), Building 2026. Services were provided to: the Chemical Technology Division for the ^{233}U solidification and Three Mile Island cleanup programs; the Operations, Environmental Compliance and Health Protection Divisions for low-level waste processing and monitoring programs; the Operations Division for product certification of isotopes; and the Fuel Recycle Division for breeder fuel reprocessing studies. In addition, the group continues to perform irradiation, decontamination, and design basic accident testing on coatings and related materials for nuclear applications for outside customers. During the reporting period, HRLAL processed approximately 2,400 samples and performed 5,350 analyses.

A major effort continued in support of the Chemical Technology Division

Radiochemical Processing Plant (RPP) at Building 3019 where conversion of ^{233}U bearing solutions to solid-oxide form for safe, long-term storage is being carried out. The RPP has prepared for the Canadian Atomic Energy Commission several shipments of high-purity ^{233}U oxide that required chemical and physical characterization. Rapid turnaround was required for most process control samples; two- to eight-hour turnaround was provided by HRLAL and the Transuranium Analytical Laboratory at Building 7920. Most of the process samples were for uranium, thorium, free acid, and alpha pulse-height analyses. Oxide measurements included particle size, surface area, oxygen to metal ratio, carbon, nitrogen, and uranium. The RPP sample load represented approximately two-thirds of all analyses performed at HRLAL.

Another major source of samples continued to be low-level radioactive waste and environmental monitoring activities. Generally, when the radioactivity level of waste or environmental samples is above allowable limits for processing by other groups in the division, the samples are submitted to HRLAL for analysis. If the required measurement technique does not exist at HRLAL, the sample may be prepared or treated at HRLAL and then diluted to an acceptable level of activity for analysis by other groups. Low-level radioactive waste samples, liquid and sludge, from the Melton Valley Storage Tanks have required hot cell, as well as glove box operation for sample preparation. The samples are essentially a high-pH, sodium nitrate (2-5M), salt solution with smaller concentrations of potassium, calcium, and magnesium cations, and sulfate, chloride, fluoride, and phosphate cations. The principle radionuclides are ^{90}Sr , ^{137}Cs , and ^{60}Co . Analyses of the low-level

radioactive waste samples included total and dissolved solids and specific gravity. Physical and chemical measurements required for characterization of waste samples are specific gravity, total and dissolved solids, alkalinity, pH, anions, cations, gross activities, alpha emitters, gamma emitters, tritium, ^{14}C , and ^{90}Sr . Of particular importance are the EPA toxic metals and transuranium nuclides.

(C. J. Baggett, H. J. Hall, L. R. Hall, R. E. Jones, M. G. McClung, M. E. Peters)

X-Ray Fluorescence Development

The development of XRF as an analytical technique applicable to fuel reprocessing facilities was concluded after the new prototype was evaluated. This project has been discontinued due to lack of funding. A final report was issued.

Several candidates for a cell window were compared for acceptable transmission of uranium and plutonium L X-rays, resistance to chemical corrosion, susceptibility to radiation damage, and physical strength. Of the various materials compared, vitreous carbon (glassy carbon) best meets the necessary requirements for a suitable cell window. Vitreous carbon has excellent chemical resistance (e.g., less than 1% weight loss when exposed to 92% nitric acid at 25°C for one month), is resistant to radiation damage, has acceptable physical strength, and has a low mass attenuation coefficient for the uranium ($0.945 \text{ cm}^2/\text{g}$ at 13.6 keV) and plutonium ($0.839 \text{ cm}^2/\text{g}$ at 14.3 keV) L X-rays. Approximately 51% of uranium L radiation was observed with a 2mm vitreous carbon window in front of a sample positioned with a 45 degree incident and take-off angle (the 45 degree angle yields an actual window thickness of 2.83mm),

which compares well to the predicted result of 56.5%. Similar results were observed for SrK α (a surrogate for PuL α), with 54% experimental versus 58.7% calculated. Predicted results indicate a loss of less than 25% for a 1mm thick window.

(J. M. Keller and J. M. Dale*)

*Analytical Spectroscopy Section

ICP Installation at 2026

A Perkin-Elmer Inductively Coupled Plasma Atomic Emission Spectrometer (ICP), Model 6500, will be used for the analysis of alpha and low level beta/gamma contaminated samples. The Model 6500 was chosen for this application because its modular design permits physical separation between the scanning monochromator and the ICP Torch box.

One of the glovebox laboratories (Room 138), was converted to a laboratory for the ICP and AA during this reporting period. Conversion included removal of the contaminated tile floor, covering the concrete floor with several coats of epoxy paint, painting the walls and laboratory furniture, and installation of required utilities.

The stainless steel enclosure for the ICP, designed and fabricated by Gem City Engineering Company, was delivered and installed in Room 138 during July. Installation of the ICP torchbox in the enclosure and optical alignment between the plasma and monochromator have been completed. Due to the strike, there was a delay in coupling the enclosure to the building exhaust system; however, supervisors in the sheet metal shop completed the fabrication and installation of the exhaust duct during September.

After installation of the exhaust duct, it was discovered that a sufficient flow could not be achieved over the plasma exhaust to allow safe operation of the system. Therefore, a supplemental exhaust blower was ordered and installed on the plasma exhaust line to correct the problem.

(J. M. Keller and C. J. Baggett)

Software Development

Lotus 1-2-3 worksheet files were developed to handle calculations for gross-alpha, gross-beta, tritium, free-acid, ^{90}Sr , and ^{90}Y analyses. These spreadsheets have been incorporated into a menu-driven application, which provides easy access by the users. Additional spreadsheets will be developed as the needs arise and will be incorporated into the same menu driver.

Software was developed to plot gamma spectra taken by a Nucleus, Inc. Personal Computer Analyzer (PCA) board, using a Hewlett-Packard plotter. The PCA board is an IBM PC-based multichannel analyzer. The plotting program reads the file generated by the PCA software directly and labels the plot with spectra parameters such as live time, real time, date taken, counts full scale, and energy calibration parameters.

(J. M. Keller)

Atomic Absorption Spectrometer Installation at 2026

The new Perkin-Elmer Model 5100 Atomic Absorption System for the analysis of alpha and low-level beta/gamma contaminated samples was received and installed by Perkin-Elmer representatives during July for temporary cold operation. It includes Zeeman background correction, a graphite

furnace, autosamplers for both the AA and graphite furnace, and the MHS-10 mercury/hydride system for the AA module. The plans are to contain the Zeeman graphite furnace module in a custom enclosure for the analysis of alpha and low-level beta/gamma contaminated samples. Specifications have been prepared, and the enclosure is currently out for bids. An additional \$25,000 was obtained from the Waste Management Section of the Operations Division for the design and fabrication of the enclosure.

(J. M. Keller and C. J. Baggett)

Upgrade of Radiochemical Capabilities at HRLAL

Two multichannel analyzers (MCAs) were installed and put into service. The first MCA, a Nuclear Data system obtained from the Operations Division, consists of a ND66 with numerous options to support stand-alone operation, a high-efficiency NaI detector and shielding. The second MCA, a Nucleus Personal Computer Analyzer (PCA-4000), was installed in a new IBM-AT personal computer.

Other counting equipment that has been obtained at this time includes a Tennelec TC-256 alpha spectrometer, and a Nucleus Model 5030 Alpha/Beta Spectrometer. The Model 5030 provides alpha spectrometry capabilities similar to the TC-256, but can also be used for high resolution beta spectrometry up to 1 MeV. For general beta spectrometry, an anthracene scintillation crystal beta spectrometer was installed.

A new high-purity germanium detector system was purchased from Tennelec, Inc. and has been installed and calibrated for use in gamma spectrometry. The germanium detector has an efficiency of 23% relative

to a 7.62 x 7.62 cm (3 x 3 in.) NaI detector and has a 1.9 keV FWHM resolution at 1.33 MeV. A Nuclear Data Micro MCA was purchased to replace a unit that had been borrowed. The new analyzer allows input from up to 4 ADCs. It is interfaced to an existing IBM PC/AT for data acquisition and display control and for data reduction. The new analyzer is used in connection with the new germanium detector and with an existing Ge(Li) detector for performing gamma-ray spectrometry, thus enhancing the capabilities of our facility.

New NMC and Tennelec instrumentation has been ordered to upgrade our gross alpha/beta counting and alpha spectrometry capabilities. When installed the new alpha spectrometry equipment will allow a faster sample throughput. With this equipment we can do initial screening of samples for segregation according to activity level. This will help prevent contamination of low background counters.

Operation, calibration and documentation procedures are being reestablished for the counting room. A technician is being trained to perform routine counting and quality control work.

(T. L. Rucker and J. M. Keller)

Nuclear Coatings Test Program

The workload for the testing of protective coatings for nuclear applications has been decreased during this reporting period because of the High Flux Isotope Reactor shutdown on November 14, 1986. However, we were able to use some of the spent fuel elements through June 1987. We did radiation stability, decontamination or design basis accident (DBA) testing for the following customers: Carboline, D. M. Alger, Ameron Corp., Cleveland Electric, Keeler and Long, Vimasco Corp. and Isotron Corp.

The autoclave (DBA testing) is being upgraded and computerized by the Instrumentation and Controls Division. We expect this work to be completed by the time the reactor restarts. The workload should increase considerably when this happens.

(R. E. Jones)

Facility Upgrade and Maintenance

Since spring 1986, a number of major facility upgrades have been completed to improve the reliability and safe operation of the HRLAL facility ventilation system and to provide a safe working environment. In 1986, a solid-state emergency power switching control center, dry-type 480-KVA electrical transformer, and 480-KVA emergency generator were installed; these replaced equipment that was originally installed when the facility was constructed in 1963. Failure of the old equipment has resulted in the loss of ventilation and evacuation of the building on numerous occasions during the past several years. The new equipment was installed during off-hours without interruption of analytical chemistry operations or release of activity to the facility.

Upgrading of the facilities continued in 1987. A solid state fire alarm control panel was installed replacing the original, which was one of a kind at the Laboratory. Battery-powered emergency lights and eyewash stations were added in all laboratories, and crash-bars were installed on all laboratory and emergency exit doors.

The Engineering Division has completed an update of the electrical engineering drawings for the heating, ventilation and air conditioning (HVAC) system. The new

drawings represent "as built" systems and include numerous changes that have been made in the HVAC system.

A laboratory engineering study is underway to install a doubly-contained, pressure testable, low-level radioactive drain system in Building 2026 to serve the hot cells and support laboratories. The existing radioactive drain does not meet the new guidelines for radioactive drain systems; it is neither double-walled or pressure testable.

We are trying to make hot cell and support facilities that are not needed by the division available for use by other divisions at the Laboratory at a cost sufficient to cover utilities, maintenance, repair, waste, etc. The mock-up hot cell and decontamination room have been decontaminated, and repairs and maintenance performed on the master-slave manipulators. Both facilities have been used extensively by the Chemical Technology Division for leaching studies of contaminated concrete specimens from Three Mile Island and for engineering studies for treatment of liquid low-level radioactive waste. Extensive decontamination of two hot cells is underway. One cell will be used by Metals and Ceramics Division for a long-term project to study the release of fission products from irradiated High-Temperature Gas-Cooled Reactor fuel, and the other cell is to be used by Chemical Technology Division.

(C. J. Baggett, H. J. Hall, L. R. Hall, J. M. Keller, R. E. Jones, M. G. McClung, M. E. Peters)

Building 3019B Upgrade

The Analytical Chemistry Hot Cell Facility in Building 3019B was identified in 1986 by the Environmental Restoration

and Facility Upgrade Program (ERFU) as a surplus contaminated facility. Decontamination and upgrading of the facility has been divided into four major phases: (1) cleanup of the operating area, (2) removal of radioactive waste and equipment from the hot cells, (3) cleanup of the access area, and (4) final decontamination and preparation of the facility for standby operation and surveillance.

Phase 1, cleanup of the operating area and phase 3, cleanup of the access area are essentially complete. The contaminated radiochemical hoods, laboratory work benches, emission spectrometer in the operating area have been removed and sent to the burial ground. Also, all utilities and services, with the exception of electrical power, lighting and ventilation have been removed to the point of entry to the operating area.

Phase 2, removal of radioactive waste and equipment from the hot cells, has been delayed due to inadequate ventilation. Exhaust ducts with a control damper were installed between the operating and access area to improve and regulate the differential pressure between the area. Also an air supply duct was installed and connected to the Chemical Technology Division HVAC system in the Radiochemical Processing Plant, Building 3019, to provide improved heating and air conditioning. The hot cell masterslave manipulators, intercell conveyor, and storage cell transfer cart have been repaired and are operational. The viewing windows of the storage cell and three of the six operating cells have been cleaned to permit viewing for removal of waste. A shielded cask, top loading/bottom emptying type, has been obtained to transfer the hot cell, solid waste to waste storage area. Plans to wash down the interior of

the hot cells in an attempt to remove loose contamination and to lower the background radiation to an acceptable level for personnel entry into the cells have been canceled because the hot cell radioactive drain system is out of service. Removal of waste from the hot cells and final facility preparation for standby operation should be completed by the end of calendar year 1988.

(H. J. Hall, L. R. Hall, R. E. Jones)

Fuel Recycle Division Analytical Support Laboratory

The analytical support laboratory located at the Fuel Recycle Division, Building 7601, performed 5,500 analyses on 2,150 samples in support of process and engineering studies for the Consolidated Fuel Reprocessing Program (CFRP). This sample load represents approximately a ten percent increase over the previous annual reporting period.

Analytical support was provided to three major experimental processing campaigns to demonstrate prototypical equipment for processing breeder-type fuel. Campaigns included demonstrations of the centrifugal contactors, automated operation and process control of the entire solvent extraction system, and an environmental test chamber to study the effect of humidity and acid on materials in a simulated, remote processing environment. Each of the campaigns operated for approximately 100 hours continuously, and required around-the-clock analytical coverage to provide measurements for process control and to verify in-line process control instrumentation. Both physical and chemical analyses were required and included conductivity, density, interfacial surface tension, viscosity, foam break time, solvent equilibration tests, free acid, uranium,

magnesium, zirconium, chlorine, oxides of nitrogen and tributyl phosphate.

In early fall 1987, the Fuel Recycle Division initiated a new program to demonstrate the CFRP prototype equipment for processing uranium-bearing materials produced by the Atomic Vaporization Laser Isotopic Separation (AVLIS) process. Due to the classified nature of the program, the analytical support laboratory has been provided with intrusion alarms, and other measures have been initiated to secure classified samples and data.

(P. W. Pair)

Radiation Safety

During the year, Building 2026 was reviewed and approved by the Radioactive Operations Committee.

The Analytical Chemistry Division has continued to strive to comply with ALARA (as low as reasonably achievable) goals. Three groups within the Radioactive Materials Analysis Section had an average radiation exposure of 182 mR/my compared to DOE maximum allowable exposure of 5000 mR/my. This represents an average reduction of about 10 percent over the previous year. Exposures have continued to decrease for the last six years. The same groups mentioned above wear the visual dosimeters and exposure is recorded daily. They also wear the new Los Alamos rad alarm audible chirper and the Panasonic supplemental dosimeter, which detects neutrons as well as beta/gamma radiation.

(R. E. Jones)

TRANSURANIUM ANALYTICAL LABORATORY

J. L. Botts

The Transuranium Analytical Laboratory continued to support the Transuranium Production Program in the Chemical Technology Division and the monitoring of radioactive waste effluents for the Operations Division. A total of 55,000 analytical determinations were made during 1987 (44,500 for the Operations Division and 10,500 for the Chemical Technology Division).

The group provided support for TRU Campaign '67 which was completed in March of 1987. Thirteen irradiated curium targets were processed yielding picogram to milligram quantities of transcurium isotopes. Approximately 7,000 determinations were made in support of this campaign.

A new method has been proposed by the International Standards Organization (ISO) for testing and assessing the ease of decontamination on coated surfaces. It was the purpose of these tests to decontaminate coated panels by the presently accepted ASTM method (D4256) and to test similar panels by the new ISO method. Test panels of nine different coatings were supplied by various vendors from the coating materials industry. Over 200 panels were tested for ease of decontamination. In the ASTM method, test panels were contaminated with a mixed tracer of ^{95}Nb , ^{95}Zr , ^{106}Ru , ^{137}Cs , and ^{144}Ce . Duplicate aliquots of the contaminant solution were air dried on the panels and the gamma activity measured. The panels were then decontaminated by immersion in a water bath at 25°C for 10 minutes. The bath was stirred vigorously. A second bath contained a solution of oxalic acid, sodium fluoride, and hydrogen peroxide. The panels were finally immersed in a bath of the same compositions at 85°C .

The panels were air dried after each bath and the adhering gamma activity was measured. All radioactivity measurements were made on a Nuclear Data 9900 data acquisition system that was equipped with a Ge(Li) detector. In the new method proposed by ISO, a contaminant solution of ^{60}Co and ^{137}Cs was used for the tests. The test panels were placed in special holders, as described in the method, and 1 ml of tracer solution was added to each panel. The contact time for the first series of tests was two hours followed by a second series of seven days. The excess tracer solution was removed and the panels placed in a water bath at 23°C for 2.5 minutes. The bath was equipped with a stirrer set at 100 rpm and again a special holder was used to position the panels in the bath. The panels were then rinsed in a bath of fresh H_2O for 10 seconds, dried, and the adhering gamma activity measured.

Parameters that were considered in comparing the two methods include: ease of decontamination, necessary equipment, time required for analysis, availability of the tracer, reproducibility of results, and cost per analysis. It was not the objective of these tests to evaluate the two methods, but to provide essential data to be used by ASTM and the coating materials industry in formulating an acceptable method for testing the ease of decontaminating coating materials. It was our observation, however, that the new proposed method was much easier to use and resulted in comparable decontamination of test coatings.

The Nuclear Data 9900 Analyzer System (ND 9900) is being used both for the gross alpha determinations and for alpha spectrometry. The ND 9900 and alpha spectrometry package along with Canberra surface barrier detectors enable us to complete these two determinations simultaneously. In the past, it was

necessary to perform these separately using a gas proportional counter and a multichannel analyzer. The ND 9900 system calculates the gross alpha and integrates each component peak, reducing the time for analysis and the possibility of error. One problem encountered with this system is peak distortion due to excessive solids on the sample plate after evaporation. The proportional counters will therefore still be used for samples with a high salt content.

A method was developed for use in tracing and determining sources of leaks in the waste line system on the ORNL site. An Aminco SPF125 spectrophotofluorimeter was set up to measure the concentration of the dyes fluorescein and rhodamine B in aqueous solutions. The concentration is determined by measuring the intensity of the fluorescence after excitation. The method was recently used to measure the fluorescein present in samples taken from White Oak Creek after an ethylene glycol spill from a cooling tower line.

Fifteen fuel pins, which were irradiated in the Dounreay Reactor, were analyzed for actinides and samples prepared for the determination of their isotopic composition. The actinide elements were determined by gross alpha counting and alpha spectrometry. Chemical separations by resin column and solvent extraction were made when necessary to determine the concentration of actinides and to isolate particular actinides for mass determinations.

(C. Cook, G. L. Herndon, R. A. Jones, R. T. Pack, R. Peacher,* J. M. Peele, R. Peralez, C. R. Cooper, I. de Leon)

*On loan from Research Reactor Division

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3. INORGANIC CHEMISTRY

W. R. Laing

The Inorganic Chemistry Section provides analytical services and support to a number of Laboratory, DOE and outside contractor programs. The DOE Environmental Survey Program made up the largest segment of work this year; twenty people from the section were involved in the analysis of water, soil and sediments for toxic metals, asbestos and other hazardous materials. Stringent quality assurance requirements were imposed on chain-of-custody, storage, analytical procedures, quality control and records.

Other programs to which the section contributed included EPRI Acid Rain, Remedial Action, and compliance with the National Pollution and Discharge Elimination System and the Resource Conservation and Recovery Act. Assistance was also given to the NRC.

Improved procedures were developed for PCB extraction, sample dissolution and analysis of ceramics. New equipment included a flow injection system, microwave ovens, an X-ray diffraction instrument and an extraction system.

ENVIRONMENTAL ANALYSIS LABORATORY

N. M. Ferguson

The Environmental Analysis Laboratory (EAL) provides analytical support for the Environmental Sciences Division (ESD) primarily, and also does a significant amount of work for other ORNL divisions. Approximately 90,000 analytical results were reported during this period. One person was added to the staff and many overtime hours were worked.

The ESD acid rain program, funded by the Electric Power Research Institute (EPRI) continued to generate the largest number of samples. The project sites (Whiteface Mountain, the Great Smoky Mountains, and Cowetta) generated large numbers of throughfall, rain, dry deposition, vegetation, and soil samples. All water samples were analyzed for Na, Ca, Mg, K, Al, SO_4 , PO_4 , Cl, NO_3 , total phosphorus,

and total Kjeldahl nitrogen. All soil and vegetation samples were analyzed for Ca, Mg, K, P, and total Kjeldahl nitrogen.

We supplied large amounts of technical support for the DOE environmental survey. Quarterly instrument detection limits for As, Se, Pb, Tl, Hg, K, Cd, Be, Cr, Ag, Ni, and Na were measured. Quarterly performance evaluation samples from EPA for the Contract Laboratory Performance Evaluation Program were analyzed. Four DOE sites (Rocky Flats, Pantex, Livermore, and Argonne) generated 200 aqueous samples and 350 solid samples. Mercury was determined using a cold vapor atomic absorption method. Sodium and potassium were measured using flame atomic absorption methods, and all other metals were measured using a Perkin-Elmer 5000 graphite furnace with Zeeman background correction.

Work for Health, Health and Safety Research, and Environmental and Occupational Safety Divisions increased

substantially this year. Weekly, biweekly, and monthly effluent samples were analyzed to comply with National Pollution and Discharge Elimination System (NPDES) requirements. Similarly, groundwater samples were analyzed quarterly to comply with Resource Conservation and Recovery Act (RCRA) requirements. Numerous blood, urine, and air filters were analyzed for the Industrial Hygiene groups.

A major effort was required to determine mercury for continuing and new programs for ESD and other divisions. We monitored the uptake of mercury in fish from soils and water, and measured elemental and total mercury in water samples for a new Remedial Action Program (RAP). Aqueous slurries were prepared from fish muscle, and we measured organic and total mercury to determine the species of mercury in fish. Biological monitoring of East Fork Poplar Creek generated hundreds of fish for mercury determinations.

A new Lachat automated ion analyzer was placed in operation in EAL this year. Initially, problems were encountered with procedures, software, and hardware. Currently, the analyzer is performing well. The analyzer utilizes EPA approved methods. Using the flow injection technique, the analyzer is capable of measuring four analytes simultaneously in a single sample aliquot. It determines nitrate-nitrogen and phosphorus at the 1 ppb and 2 ppb levels respectively.

We purchased a new microwave oven (CEM Model MDS-81D) for preparing samples for analysis. A closed vessel microwave technique was used to prepare fish tissue (freeze dried and fresh) for metal analyses. We measured 13 elements (Cr, Li, Zn, Cd, Pb, Cu, Ni, Be, Tl, Ag, Sb, Se, As) in the fish tissue. Zinc and copper were measured by flame atomic absorption. All other elements were measured by graphite furnace atomic absorption. The method is

fast, simple, and uses only HNO_3 , which is ideal for graphite furnace work.

The number of ion chromatographic analyses increased considerably during this year. Thousands of determinations (chloride, phosphate, nitrate, sulfate) were made each month on aqueous samples (soil leachates, rain, stem flow, streams) using the Model 2110 "i" Dionex ion-chromatograph (equipped with automatic sampler). A new Dionex ion-chromatograph (Model 4000 "i") was placed in operation early this year. Later an automatic sampler was added to the instrument. This instrument was used extensively to determine low concentrations ($<0.1 \mu\text{g/mL}$) of nitrates and sulfate in NaOH and K_2CO_3 solutions. It also measured organic acids (citric, formic, malic, acetic, propionic) in acid rain samples.

We have investigated, modified, and placed in use several new procedures this year. These include: a method to determine phosphorus in particulate matter collected on glass fiber filters; an improved extraction technique for measuring low ppb (<10) Cd in urine; a method to determine nitrogen ($<0.01\%$) in oil samples; and an improved method (block digester-Kjeltec auto analyzer) to determine nitrogen in soils.

(L. S. Barringer, T. L. Baxter, A. R. Herndon, P. L. Howell, J. D. Mayton, S. A. MacIntyre, M. L. Moore, S. J. Morton, W. R. Musick, J. Summers, C. L. Watson)

CHEMICAL AND PHYSICAL ANALYSIS

J. H. Stewart

ORNL was selected as one of the three DOE facilities that support the DOE-EPA Site Evaluation Program. This has resulted in large blocks of specimens, received within 10 day sampling intervals,

which require meeting stringent EPA holding times. The regulations also require substantial increases in quality assurance measurements, a totally new documentation system, and the addition of several parameters never required before, such as hexavalent chromium, asbestos, cyanide and phenols. During the year we received approximately 2000 specimens requiring 28,000 measurements from Rocky Flats, Lawrence Livermore/Sandia, Pantex, and Argonne National Laboratory. These were in addition to the normal support role for ORNL programs, which totaled 7500 specimens and 81,000 measurements. The quality level remained above 95%, despite the increased work load. The Performance Evaluation test specimens, required for participation in the Site Survey Program, were analyzed and reported on schedule each quarter. ORNL consistently scored in the top levels among the participating facilities.

During this year, the new ANALIS data management system was tested and placed in full operation. Among other features, we can now enter, store, and generate graphic data from the hundreds of internal quality control specimens analyzed each year. In the past, these data have not been retrievable for use in program management quality control.

With strong ACD management support, funding was provided for a state-of-the-art scanning X-ray diffractometer, two new optical microscopes for solid specimen asbestos quantitation, and a personal computer with a special capability for connecting it to a low cost X-ray fluorescence analyzer. The diffractometer makes possible rapid specimen throughput, lower detection limits, and lattice parameter measurements, all at reduced cost. The instrument provides an outstanding color graphics display of data that can be directly used by customers in their reports. The entire laboratory area

has been renovated, cleaned, painted and is a credit to the division.

Our PCB analytical capability has quadrupled through purchase of a Sigma 300 automated gas chromatograph which is operated in conjunction with the new Fisher grinder and SOXTEC rapid organic extraction system. The SOXTEC has proved so valuable that a lengthy set of experiments, to demonstrate equivalency with the EPA SW 846 Soxhlet method, has been completed and an approved petition for ORNL use of the method for environmental compliance specimens has been submitted to EPA Region IV. The new method reduces the lapsed time for specimen extraction from 17 to 2 hours!

The ORNL Program support, as well as Site Survey, requested measurements rarely performed at ORNL. Thus, modification of existing procedures or development of new ones was required. These included new methodology for determination of Pt and Rh in (Pt,Rh+x) alloys; measurements of Eu-Ti-Zn-Al oxide ceramics and Cu-Ti-Al alloys, a dissolution technique for $K_3(Nb_xTa_{1-x})_2O_5$ crystals prior to ICP analysis, and determination of Ho in $KCaF_3 \cdot Ho$ crystals weighing only 5-10 mg. Other modified analytical techniques included a pyrohydrolysis method for nitrogen in sintered silicon nitride and silicon carbide, separations and measurement of traces of Zr in concentrated uranyl nitrate solutions by ICP, and a technique for determining traces of organic and inorganic phosphorus compounds in aqueous solutions exposed to tributylphosphate extractant. We also participated in a safety analysis of an iridium cyanide etching facility. From this study, a less hazardous procedure was developed and recommended to the operating personnel.

Support for NRC continued as uranium mill decommissioning in the western states

accelerated. We performed analyses for PCB, anions, alkalinity, fluorimetric uranium, and anion/cation balances. Metals were determined by ICP. The specimens included water from aquifers, impoundment fluids (pH2 H₂SO₄), soils, and sediments. The Savannah River requests for support included scanning electron microscopy, X-ray fluorescence elemental measurements, optical microscopic studies of dust particles, X-ray diffractometer patterns for compound identification, and particle size measurements. A set of powdered vegetation specimens was analyzed for uranium, as an NRC followup to the 1986 Sequoyah Nuclear Fuel release of a UF₆ cylinder. The uranium concentrations averaged 40 ng/g.

(D. J. Batiste, S. J. Bobrowski, P. V. Ditavong, C. Feldman, J. H. Hackney, R. L. Holmes, C. S. MacDougall, N. E. Owen, B. Philpot, J. C. Price, W. F. Rogers, J. K. Thompson, K. I. Webb, K. S. Whaley)

4. ORGANIC CHEMISTRY

M. R. Guerin

This section carries out analytical and applied research, methods development, and analytical services. Analytical research currently emphasizes spectroscopic and separations techniques for the identification and quantitation of trace organic chemicals in natural media. Spectroscopic research continues to focus on Fourier transform mass spectrometry (FTMS) and gas-phase ion-molecule chemistry. Separations work has been extended to include fundamental mechanisms of chromatography with the addition of Dr. Georges Guiochon and his staff through the University of Tennessee/ORNL Distinguished Scientist Program. Programs involving the generation or characterization of experimental and natural atmospheres continue. Materials under study include military smokes and obscurants, environmental tobacco smoke, airborne military chemical agents, and small rocket exhausts. Environmental analytical chemistry is receiving increased attention with new programs addressing field-portable analytical technologies, quality assurance issues, and advanced environmental analytical instrumentation. Increased attention is also being given to biomolecule identification and quantitation in support of the DOE/OHER initiative in DNA-adducts for molecular dosimetry. The section now provides fully qualified environmental organic analytical services in support of ORNL regulatory compliance and research programs dealing with site surveys and waste characterization.

ORGANIC SPECTROSCOPY

M. V. Buchanan

Research in the Organic Spectroscopy Group focuses on the development of techniques for the unambiguous identification of organic chemicals. Progress has been made in a number of areas this year. These include, 1) studies of negative ion processes for the identification of polycyclic aromatics by

mass spectrometry, 2) the generation and detection of normal and modified nucleotides and oligonucleotides by laser desorption Fourier transform mass spectrometry (FTMS), 3) FTMS characterization of higher molecular weight constituents in tobacco smoke tar, 4) refinement of our multimode ionization detector, and 5) development of mass spectrometry-based techniques for the detection of trace organics in air and the chemical characterization of combustion products from colored smoke grenades. The group

also provides specialized spectroscopic support to other programs in the Organic Chemistry Section and to other groups within the Laboratory.

Negative Ion Processes for Identification of Polycyclic Aromatics

Mechanisms responsible for negative ion formation from polycyclic aromatic hydrocarbons (PAH), methyl-substituted PAH, and aromatic amines have been studied using FTMS and by conducting doping experiments in a high pressure chemical ionization source on a quadrupole mass spectrometer. These experiments have focused upon understanding the formation of unusual ions that appear in the spectra of PAH, which may be used to differentiate isomeric compounds. From these experiments, it has been shown that negative ions are produced by three different means: electron-molecule, ion-molecule, and wall-assisted reactions. We have shown previously that electron capture reactions may be used to differentiate isomeric polycyclic aromatic hydrocarbons on the basis of relative electron affinity values. In addition, ionic species, produced from trace impurities in the reagent gas (such as water or oxygen), are involved in ion-molecule reactions which yield products such as $[M-H]^-$ or $[M+O-H]^-$ ions. These ions are not particularly useful for isomeric differentiation, however. In contrast, oxygen has been found to participate in unusual wall-assisted reactions that are followed by electron capture. These reactions are useful for the differentiation of methyl-substituted PAH. For example, 7,12-dimethylbenz(a)-anthracene, a carcinogen, can be distinguished readily from 3,9-dimethylbenz(a)anthracene, a noncarcinogenic isomer. These reactions may also be used

to provide isomeric information regarding aromatic amines and methyl-substituted aromatic amines.

(M. V. Buchanan and E. A. Stemmler*)

*Postdoctoral Research Associate

Laser Desorption FTMS for Investigation of DNA Constituents

Laser ionization FTMS has been investigated as a means of generating ions from nonvolatile compounds, such as normal and modified nucleotides. Using the 1060 nm fundamental line from the Nd:YAG laser, negative ions corresponding to the specific nucleic bases, sugars, and phosphate groups present in the nucleotides are generated. It was found that the negative ion spectrum yielded greater information on the bases present in the nucleotide than the positive ion spectrum. Collision-induced dissociation of negative ions containing the nucleic base produces abundant daughter ion formation, rather than simple electron detachment, which is often encountered in the collisional activation of negative ions. A series of methyl-substituted nucleotides, as well as several cyclic adducts of nucleotides, have been characterized by collisional activation. These spectra yield information related to the position and nature of the modification to the nucleic base.

Molecular ions from oligonucleotides are difficult to generate due to the ease of fragmentation of the glycosidic bond. However, sequence-specific information has been obtained from dimeric and trimeric nucleotides. Further investigations into the generation of molecular ions from higher oligomers are currently in progress.

A pulsed dye laser was recently received and is being interfaced to the FTMS. The existing Nd:YAG laser will be used both to pump the dye laser and to desorb and ionize nonvolatiles. The dye laser will provide capabilities for multiphoton ionization and photodissociation processes, which will be used to probe the structure of the ions and neutrals generated by laser desorption.

(R. L. Hettich)

FTMS Characterization of Tobacco Tar Constituents

FTMS has also been used in studies of tobacco smoke particulates. Emphasis has been on the development of techniques for the characterization of higher molecular weight constituents in the range of 200 to 1,000 daltons. Software was written for the generation of molecular weight profiles using direct sample introduction via the heated solids probe. Soft ionization methods combined with medium to high resolution allows the components in the mixture to be separated on the basis of their molecular weights. Identification of constituents is based on the exact mass of the ions; this enables determination of molecular formulae and, whenever possible, collision induced dissociation spectra. In the latter case, the generated spectrum is compared with that from an authentic standard. The new direct probe software allows both positive and negative ion spectra to be generated from the same sample. Additionally, the probe temperature is automatically ramped and a series of spectra is stored, allowing a complete temperature profile of the tar sample to be generated. Finally, accurate control of the sample heating and vaporization by the computer system provides sufficient reproducibility to enable

quantitative measurements to be performed when internal standards are included in the sample.

Another software program has been devised to allow enhanced MS/MS capabilities with the FTMS, especially for the analysis of complex mixtures, and includes parent ion and neutral loss scans. With this program, one has the option of extracting neutral loss information or storing the entire daughter ion spectrum. Additional options and features include automatic background spectrum subtraction, constant excitation energy for all parent ions, and a relative threshold for the rejection of parent ions below a selected abundance level.

(M. B. Wise)

Improvements in the Multimode Ionization Detector

Work has continued this year on the refinement of the multimode ionization detector, MMID. The first generation detector was investigated with respect to fundamental parameters affecting response in the three basic operating modes: electron capture, electron emission, and selective. Parameters studied include reagent gas composition, reagent gas flow rate, temperature, and applied cell voltage. Good linearity and detection limits were obtained using argon as the reagent gas in both the electron capture and emission modes, comparing favorably with a flame ionization detector.

A second generation MMID was built this year and initial experiments are under way. This detector differs from the original MMID in that the detector is operated at a constant reduced pressure, and its response can be changed simply by altering the detection pulse intervals.

Longer pulse intervals allow thermalization of electrons, enhancing electron capture reactions; shorter pulse intervals result in more energetic electrons, enhancing electron emission reactions. This new concept makes it much easier for the operator to switch between the three detection modes and increases the versatility of the MMID as well. A patent disclosure is in process.

(M. V. Buchanan, M. B. Wise, and J. W. Haas*)

*Postdoctoral Research Associate

Monitoring of Military Chemical Agents in Air

A discharge ionization source was designed and tested for the mass spectrometric monitoring of military chemical agents in air. The source is based on the concept employed by G. L. Glish and S. A. McLuckey of the ACD for explosives monitoring. The design is unique, however, in that a concentric geometry was devised to maximize contact of the air sampled with the discharge region. Also, the source is optimized for positive ion detection rather than negative ion detection. An ultimate detection limit for the chemical agent simulant dimethylmethylphosphonate of 50 parts per trillion (molar concentration) in air was achieved using the source interfaced to a Finnigan 3200 quadrupole mass spectrometer.

We find that the discharge ionization mass spectrometer can also be used for the direct analysis of volatile organics in water and soil samples. This is done by monitoring the headspace of a sample or by purging samples with the air flowing into the ionization source. Using headspace analysis, linear calibration curves for

benzene in water were easily generated in the range of 10-100 ppb. The level of benzene in an actual sample provided a result within 10% of the true value. Purging the samples with air at a flow rate of 1-2 mL/sec provides approximately an order of magnitude increase in sensitivity and also decreases by a factor of 5 to 10 the time required for quantitative measurements. Linear calibration curves for benzene and trichloroethylene have been generated down to low ppb levels. An especially important feature of this ionization source design is that it yields minimal water cluster ions. These experiments indicate that this method coupled with an MS/MS system is extremely promising as a rapid analytical method for EPA specified pollutants.

An ion trap mass spectrometer (ITSM) has been ordered as part of a study related to our previous project for the detection of chemical agents by mass spectrometry. In the first phase of this project, a feasibility study was undertaken to evaluate the practicality of interfacing a tandem mass spectrometer (MS/MS) to an automated thermal desorption unit for the rapid analysis of trace organics absorbed on sorbant traps. This involved both a survey of the literature for references to prior work and the evaluation of commercially available thermal desorption units and tandem mass spectrometer. No references for the direct thermal desorption of organics into an MS/MS were found. A review of commercially available automated thermal desorption units revealed that none met the requirements of this project. Finally, a head-to-head laboratory evaluation of two triple quadrupole-based MS/MS units and an ion trap mass spectrometer (ITMS) showed that only the ITMS could meet the sensitivity

requirements of this project. The second phase of this project is presently under way and includes the design and fabrication of an automated thermal desorption unit and the interfacing of this unit to the ITMS. A design for the thermal desorption unit has been formulated, and it is being constructed.

(M. B. Wise, J. M. McMahon, and M. V. Buchanan)

Chemical Characterization of Products from Signal Smoke Grenades

In another project for the Department of Defense, four colored smoke mixes used in signal grenades were studied to determine whether chemical transformation occurs upon detonation of the grenades. These smoke mixes, which included red, green, violet and yellow, are composed of one or more organic dyes, along with a number of inorganic components to aid in combustion. Dye standards and samples of the actual smoke mixes used in the manufacture of the smoke grenades were characterized by chromatographic and spectroscopic techniques, including GC, HPLC, HPTLC, mass spectrometry (GC/MS and FTMS), ^1H and ^{13}C NMR, GC/FTIR and diffuse reflectance FTIR, and UV/vis. Tents were used as combustion and sampling chambers for the grenades. Vapor phase constituents were collected on Tenax and XAD sorbant traps. Particulates were collected on filters, and glass trays placed on the floor of the tent were used to collect fallout particles. Samples for particle size distribution were also taken using an eight-stage cascade impactor. The particulates were analyzed by the same methods used for the smoke mixes, and the volatiles were analyzed by GC/MS.

(C. Y. Ma and M. V. Buchanan)

Special Spectroscopic Studies

A number of special spectroscopic studies were also performed this year, including GC/MS and FTMS. One study involved the analysis of air samples, taken by researchers in the Health and Safety Research Division, by thermal desorption GC/MS to evaluate levels of targeted organics in indoor air. Thermal desorption GC/MS was also used in a number of studies originating in other programs within the Organic Chemistry Section. A number of special samples were run on the FTMS, taking advantage of its unique capabilities. These included the analysis of material on a filter from Three Mile Island using laser desorption FTMS, the determination of a targeted compound in a kidney extract for researchers at ORAU Medical, and a study of multiply-charged species for Health and Safety Research. To improve our capabilities in periodic assistance with infrared studies, a Nicolet Fourier transform infrared (FTIR) spectrometer was installed recently. This instrument is capable of obtaining conventional transmission and absorption spectra, as well as diffuse reflectance and attenuated total reflectance spectra. A major feature of this instrument is the ability to obtain infrared spectra of compounds in mixtures using a gas chromatographic interface.

(R. H. Ilgner, R. L. Hettich, M. B. Wise, and M. V. Buchanan)

SEPARATIONS AND SYNTHESIS

W. H. Griest

The main focus of this group is the development and application of organic chemical separations methods. Activities

include programmatic research and development for the Department of Energy and U.S. Army, and organic chemical separations and measurements as part of the section's analytical services for the Laboratory.

Separations and Synthesis Research and Development

Separations research in methodology for molecular dosimetry is supported by the U.S. DOE Office of Health and Environmental Research. Research this year has centered upon the development and evaluation of electrokinetic, liquid, and supercritical fluid chromatographic methods for the separation of trace quantities of analytes from mega amounts of structurally similar components in the physiological matrix for structural identification and measurement. Micellar electrokinetic capillary chromatography (MECC) has been evaluated for its utility as a tool for separating nucleic acid constituents. It has been found capable of resolving normal and modified deoxyribonucleosides with efficiencies easily exceeding 100,000 theoretical plates. At low solute concentrations (nmol/mL), efficiencies of 320,000 theoretical plates (corresponding to 500,000 plates/m) have been realized. Sequence- and modification-selective separations of oligonucleotides have also been achieved. Although on-column UV absorbance detection has very good absolute sensitivity (18 pg of deoxyguanosine at $S/N = 4$), the very small injection volumes (ca. 2 nL) render it insufficient for determining adducted species in physiological media. Alternate detection methods such as fluorescence are needed. The solute mass flux from the column under near-overloaded conditions was estimated to be 0.5 ng/sec, which is within the range of

on-line mass spectroscopy, but insufficient for collection and off-line spectroscopic characterization. MECC appears useful as a high resolution microanalytical technique where the volume of sample is extremely limited or localized.

Secondary equilibria in reverse phase liquid chromatography (RPLC) are being investigated as a means of enhancing selectivity and optimizing separations of adducted or modified species from normal DNA constituents. Capacity factors for a variety of compounds have been determined as a function of metal ion additive in the mobile phase. Differences in affinity based upon the type of cation, structure of the phosphate group in nucleotides, and base modification have been observed. Complexation of the solute molecules with the metal ions changes the electronic structure of the compounds and alters solute-solvent interactions. Alkali and alkaline earth metals bind primarily to phosphate groups, and transition metals also interact with purine bases. Pyrimidine dimers have been resolved from normal purines and pyrimidines in hydrolyzed samples of UV-irradiated DNA and will be identified structurally by Fourier transform MS in the Organic Spectroscopy Group. On-line MS detection and structural identification of adducted species isolated by liquid chromatography is being pursued in collaborative work with the Analytical Spectroscopy Section. MS/MS with an atmospheric sampling glow discharge source has been directly interfaced with HPLC, and spectra of nucleic bases have been recorded. The source tolerates a high solvent load and provides sensitivity in the positive ion mode comparable to other systems.

The ability of supercritical fluid chromatography (SFC) to separate polar, thermally labile compounds such as nucleic

acid constituents and explosives is being determined. Several nucleic bases including adenine and methylated analogs and the ribonucleoside adenosine have been mobilized and separated in underivatized forms using a cyano-phase packed column and supercritical carbon dioxide programmed from 20 to 35 MPa (200 to 350 atm) at 150°C. Several explosives including tetranitroaniline (Tetryl) and trimethylenetrinitramine (RDX), have been chromatographed by SFC for the first time. It is felt that directly coupled supercritical fluid extraction-SFC holds considerable advantages for the determination of explosive compounds, particularly those which do not possess a strong UV-absorbing chromophore and are not easily determined by HPLC.

An important part of this work has been the synthesis of adducted and modified DNA constituents to support the separations and structural identifications research in this section. An 8-bromo-2'-deoxyguanosine, a cyclic 1,N²-glyoxal-deoxyguanosine adduct, a bicyclic 1,N²-glyoxal-dimeric-2'-deoxyguanosine dinucleotide monophosphate adduct, two diastereoisomers of a cyclic 1,N²-acrolein-deoxyguanosine adduct, two dehydrated products of the previous adduct, and two diastereoisomers of 1,N²-crotonaldehyde-deoxyguanosine have been prepared, isolated, and structurally confirmed. The structures of some of these adducts are identical to those of cigarette smoke carcinogen-DNA adducts formed in-vivo. Cyclic dimers of thymine and uracil and the 4-6 uracil-thymine product also have been prepared for identification and quantification of UV irradiation-induced DNA lesions.

Development of other separation and detection methods include the establishment of a megabore capillary column gas chromatograph/argon microwave emission detector system with the assistance of the

Inorganic Chemistry Section. With this system we can make highly element-selective determinations of volatile and semivolatile compounds. For example, it has been used for the determination of methyl mercury in fish and water samples with a limit of detection of ca. 1 pg Hg injected. Instrumentation for the direct analysis of mixed wastes is being established. An HPLC has been installed in a contamination-zone hood and used for the determination of di(sec-butyl)phenylphosphonate in moderately radioactive samples.

(W. H. Griest, C.-h. Ho, R. S. Ramsey, K. H. Row,* R. Budhram,** V. Chan,[†] M. Decker,[†] C. Feldman,^{††} B. Dittmar,^{***} and D. Eaton^{**})

*UT Postdoctoral Research Associate

**OHER-HBCU Faculty Research Participant

***Student research participant

[†]GLCA student

^{††}GLCA student

Other Projects

Programmatic commitments have been completed for projects supported by the DOE Office of Fossil Energy and the U.S. Army Biomedical Research and Development Laboratory. Significant findings include the observation that the detoxifying ability of hydrogenation catalysts in two-stage coal liquefaction pilot plant operations can be maintained over time by increasing the severity of the hydro-treatment conditions. We also found that tumor promoting activity is important to the expression of the residual complete tumorigenicity in highly refined fuels. Finished fuels derived from coal and other synthetic sources are not likely to pose a

greater toxicological hazard than the analogous petroleum products. The fuel-related contamination in the atmosphere of military motor pool garages has been characterized extensively to aid in risk assessment and design of exposure conditions for animal toxicology studies.

Equipment and facility upgrading included the purchase of a Waters Model 600E HPLC with a model 490 programmable multiwavelength detector and a Suprex Model 200A supercritical fluid chromatograph with variable wavelength UV absorbance and flame ionization detectors for separations research. The Hazardous Materials Laboratory in building 2026B was expanded into a satellite separations laboratory to conduct separations research and to provide organic analytical services. Several chromatographs and additional staff have been relocated to 2026B. There we continue to do preanalytical preparation of hazardous materials such as mixed wastes and conduct routine GC and HPLC determinations. The DOE/OHER Synthetic Fuels Repository is maintaining its archival of natural and synthetic fuels samples, but the sample preparation lab in building 3001 was vacated because of the greatly decreased number of sample requests.

(W. H. Griest, C.-h. Ho, R. Merriweather, and B. A. Tomkins)

SPECIAL PROJECTS

R. A. Jenkins

The generation and chemical characterization of controlled and/or specialty atmospheres continues to be a major focus of this group. Activities have

now broadened to include environmental analytical chemistry, with a strong field-sampling and quality control/quality assurance component. In general, funding for the group remains highly issue-oriented, originating primarily from the Department of Defense (DOD), the National Institutes of Health and the Environmental Protection Agency.

Tobacco Smoke Studies

The characterization of environmental tobacco smoke (ETS) has been an important activity this year. To study the composition of ETS, it is necessary to have an atmosphere that can be easily sampled and, in the ideal, over which some measure of control can be maintained. We have deemphasized the use of small chambers in these studies, because their small size makes attainment of realistically low levels of ETS difficult. An existing office area (ca. 16 square meters with a 3 meter ceiling) has been converted into an experimental ETS room. The ventilation system has been modified so as to mimic more closely the air exchange rate typically encountered in modern office buildings (two - four air changes per hour). ETS is generated by using one or more smoking machines operating under controlled conditions and smoking reference cigarettes. Particulate concentrations are determined periodically using an automated reporting piezoelectric balance (TSI Model 5000) and can be determined empirically in real time using an ORNL-modified forward scattering light sensor. Carbon monoxide concentrations are currently determined using an electrochemical monitor and will be determined shortly using a newly acquired nondispersive infrared instrument. We have the capability of producing ETS

atmospheres with nicotine levels ranging from about $1 \mu\text{g}/\text{m}^3$ to about $300 \mu\text{g}/\text{m}^3$. Particulate concentrations as high as $1700 \mu\text{g}/\text{m}^3$ have been achieved.

A major focus of our efforts to characterize atmospheres of ETS has been the determination of selected volatile hydrocarbons and nitrogen-containing constituents. Samples of ETS vapor phase were collected using triple sorbent resin traps developed at ORNL. The samples were subjected to thermal desorption gas chromatograph/mass spectrometry (GC/MS) for tentative structural identification of the constituents and to GC for quantitative determination. Even at relatively high ETS particulate levels, concentrations of important vapor phase compounds were low. For example, at particle levels of $330 \mu\text{g}/\text{m}^3$ (a fairly smoky environment), concentrations of benzene, pyridine, and toluene were 16, 11 and $70 \mu\text{g}/\text{m}^3$, respectively.

We have received support from the Council for Tobacco Research to develop a personal sampling/analysis system to determine ambient nicotine using resin trapping/thermal desorption GC procedures. The method developed employs Tenax-GC as an absorptive resin, and uses triethylamine (TEA) added to the resin cartridge after sampling and prior to analysis to insure quantitative recovery of the nicotine during desorption. Recoveries of nicotine from the resin cartridges (using GC analysis with nitrogen specific detection) average more than 95% when the TEA is used. A number of in-house sampling and analysis validation studies show that this method produces results which are in agreement with those of other published methods. The limit of detection of the procedure is about $0.3 \mu\text{g}/\text{m}^3$, or a factor of 4 lower than other methods. Nearly 40 local restaurants were sampled unobtrusively by group staff members wearing a small

personal sampling pump and the resin cartridge. In general, ambient nicotine levels appeared to be distributed log-normally, with a geometric mean of $3.3 \mu\text{g}/\text{m}^3$. Many restaurants had nicotine levels near the detection limit of the analytical method, and there was a minimal correlation between the number of cigarettes observed being smoked and the ambient nicotine levels.

(R. A. Jenkins, C. E. Higgins, and C. V. Thompson)

Military Atmospheric Studies

One aspect of our aerosol technology program has been the generation of laboratory scale specialty atmospheres for characterization or toxicology testing. This past year, considerable effort has been applied to the development of a laboratory scale generator for a military biphasic obscurant aerosol. One phase is generated from a petroleum based liquid using evaporation/condensation technology previously developed in our laboratory. The other phase is generated from finely divided solid material which is deagglomerated and dispersed using a small jet mill. The two aerosol clouds are mixed at ambient temperature and delivered to a chamber for characterization or biological study. Analyses of particle sizes using cascade impaction have indicated that the liquid droplets by themselves have a mass median diameter (MMD) of $0.5 \mu\text{m}$, while the solids have an MMD of $2.5 \mu\text{m}$. The combined aerosol has an MMD of ca. $1.0 \mu\text{m}$. Chemical characterization studies are expected to begin soon.

An important new project involves characterizing the atmospheres generated from the firing of small rocket motors

used in hand-held anti-tank weapons systems. The purpose of this effort is both to provide a data base for health risk assessment and to input projections of exhaust products into an improved computer model. We are to determine concentrations of both major and minor vapor and particle phase constituents. Since many of the potential species are reactive, we are using real-time monitoring systems to determine levels of particulates, ammonia, oxides of nitrogen, hydrogen cyanide, carbon monoxide and dioxide, and hydrogen chloride. Analytical methods validation experiments are being performed using free burns of small quantities of the candidate propellants in a small chamber at ORNL. Samples for actual characterization will be collected from firings of test motors in a 19.6 m³ chamber at the Army's Signature Characterization Facility (ASCF) at Redstone Arsenal in Huntsville, Alabama. A sampling trip was made to the ASCF in late August. Real time analysis of the exhaust atmospheres produced from the firings of 75 g test motors of the first propellant indicated that levels of CO in the chamber reached 340 ppm, particulates (about half of which were lead), about 75 mg/m³, and oxides of nitrogen, about 3.5 ppm. GC/MS analysis of low level vapor and particle phase constituents is currently in progress.

Airborne contaminant characterization issues have also been addressed for the Army's Office of the Program Manager for Chemical Munitions. Analytical methods are required to protect the occupational health of workers involved in the demilitarization of nerve and blister agents. Because of the relatively high toxicity of these compounds, extremely high sensitivity is required. Much of our work was conducted with agent simulants, compounds which are structurally similar to the agents themselves, but of lesser toxicity. This

work is currently on hold, pending approval by DOE for use of environmental quantities of the actual agents at ORNL. Progress made when the project was active included identifying promising sampling media and monitoring instrumentation and developing quality assurance procedures.

(R. A. Jenkins, J. H. Moneyhun, T. M. Gayle,* R. L. Moody, C. V. Thompson, and M. P. Maskarinec)

*Instrumentation and Controls Division

Field Sampling Studies

Sampling airborne materials under field conditions continues to be an important activity of this group. Evaluations of the personal sampling system for ambient nicotine described above were conducted by initially sampling various workplaces at the X-10 site, including offices, shop rest areas, common areas, and laboratories. In addition, a comparative study of this system with other analytical methods for ambient nicotine was conducted at a large ETS chamber located in the R. J. Reynold's research complex in Winston-Salem, NC. Rocket exhaust characterization experiments were performed at Redstone Arsenal in Alabama. A particularly important aspect of the field sampling effort is the support to various bioassay laboratories in the conduct of inhalation exposure studies of animals. In August, a major sampling trip was made to the USDA's Denver Wildlife Research Center, which is exposing animals to phosphorous smoke aerosols generated using ORNL developed systems. Samples were acquired to determine the physical and chemical characteristics of these atmospheres. Of special interest was a finding that suggests that the organic

vapor phase composition of the aerosol is influenced by geographical elevation. Another sampling trip was made to the marijuana smoke exposure study being conducted at the National Center for Toxicological Research (NCTR). NCTR is using ORNL modified exposure systems to dose rhesus monkeys with a controlled amount of smoke. The purpose of the trip was to determine the amount of that atmosphere to which the animals are exposed and the fraction of the smoke which they retain. Results of analyses for the psychoactive delta-9 tetrahydrocannabinol indicate that the animals retain slightly more than half of the smoke which they are offered.

(R. A. Jenkins, J. H. Moneyhun, C. V. Thompson, R. L. Moody, and T. M. Gayle*)

*Instrumentation and Controls Division

Environmental Studies

The other major component of group activities is environmental analytical chemistry. A study of the preanalytical holding times of soil and water samples is in progress and an assessment of field portable analytical methods has been performed. An interagency task force has been formed for the purpose of improving the various quality assurance procedures associated with environmental analyses.

The study of preanalytical holding times has focused primarily on volatile organic compounds in water and soil. For water samples, it was found that containerization with no headspace prevents the loss of these analytes due to volatilization. The sample degradation which occurs is either by chemical reaction or by microbial action. Many of the analytes were stable for as long as one year. Those analytes

which did degrade could be stabilized by addition of HCl to the sample. Thus, the preanalytical holding times could be extended well beyond the currently allowed 10 days without compromising the results. A survey was made which indicated that extension of the holding time for these samples to 28 days would save about \$3.5 M annually on a nationwide basis by easing field sampling logistics and permitting laboratories to make more cost effective use of analytical facilities. For volatile organics in soils, it was shown that the containers currently used were totally inappropriate with the sample integrity compromised even after three days. Losses were due both to volatilization and to microbial and chemical action. At this time, no totally suitable container has been found, and no storage condition is completely appropriate. A limited number of semivolatile organics have also been studied. In general, it appears that the primary source of integrity loss is microbial activity.

In the area of field portable instrumentation, an assessment has been made of available technology suitable for use in the field, and implementation of these methodologies is beginning. For metals, the most appropriate screening technique appears to be X-ray fluorescence. Specific metals such as mercury and arsenic can be measured at lower levels using portable monitors combined with field sample preparation. For volatile organics, portable gas chromatographs should allow analysis in the field without compromising either throughput or analytical performance. For semivolatile organics, sample preparation is the key issue requiring further development. Several portable instruments have been identified which appear sufficiently rugged to be fieldable if the

sample preparation problems can be overcome.

(M. P. Maskarinec, R. A. Jenkins, R. L. Moody, D. D. Pair, M. E. Garcia, and L. H. Johnson)

Quality Assurance and Lab Upgrading

Quality assurance has become extremely important within analytical chemistry. This is a result of increasing realization that data must not only be of high quality, but must meet the objectives of the program generating the data. This may include the ability of the data to be used in litigation. As a result, many different approaches to quality assurance in analytical chemistry have evolved. These approaches are not often mutually exclusive, but frequently do produce redundancies and increased analytical cost without the intended increased data quality. An interagency (DOE, EPA, and DOD) task force has been established to address these issues and to attempt to bring about a unified approach to quality assurance that will improve data quality in an efficient and economical manner. Initially, the focus is on data associated with waste site characterization and remedial action - where large numbers of samples are analyzed annually and where litigation is often involved. Sixteen general areas of quality assurance have been differentiated, and the best current approach to each identified. A recommended system for implementing these improvements is now before the task force.

We continue to upgrade our analytical capabilities. In the general area of air monitoring equipment, we have acquired a Dycor Quadrapole Mass Analyzer and are acquiring a Tapered Element Oscillating Microbalance (TEOM). The latter will give

us the ability to monitor particle mass concentrations down to a few micrograms per cubic meter, similarly to the automated piezoelectric microbalance that we already use. However, the TEOM can be calibrated using actual mass standards, a capability possessed by no other commercially available instrument of this type. We also have acquired real time electrochemical monitors for hydrogen cyanide and ammonia, and nondispersive infrared monitors for carbon monoxide and carbon dioxide. In the area of chromatographic instruments, we have acquired a Tracor Model 540 GC with a gas sampling valve and a flame photometric detector, a Scentor portable GC, and two thermal desorption units. Our data handling capabilities have also been upgraded with the purchase of a stand alone chromatographic integrator and a Maxima chromatography workstation.

(M. P. Maskarinec, R. L. Moody, R. A. Jenkins, and T. M. Gayle*)

*Instrumentation and Controls Division

ORGANIC ANALYSIS

J. E. Caton

The Organic Analysis Group emphasizes the quantitative determination of trace organic components in a wide variety of sample matrices. These include ground water, discharge water, soil, sludge, tissue, solid waste, and commercial products. Significant progress has been made in expanding laboratory capabilities, adding hardware, and increasing efficiency. However, the demands for environmental organic analyses have been expanding at a rate which often exceeds our progress in upgrading the

capabilities. This effort supports Laboratory programs and work for others programs as well as studies and surveys being carried out on a national scale by the Department of Energy and the Department of Defense. Funding is derived entirely from the programs which this effort supports.

Improvements in Preparation and Analysis

Sample preparation capabilities were greatly enhanced by the addition of both hardware, person-power, and laboratory space. Hardware upgrades include the addition of continuous liquid-liquid extractors, three ultrasonic devices for extraction of solids and a preparative gel permeation chromatograph, which can automatically inject and collect up to 23 samples. Our capacity for sample preparation increased substantially through cooperation with the Transuranium Analytical Laboratory at the TRU facility. This group was equipped to prepare soil and sludge samples for the determination of semivolatiles and pesticide/PCB's. Even though such samples have a 10-day preparation holding time, the efforts at the TRU facility made possible the timely preparation of two large sample consignments (144 soil samples received between August 8 and August 17, 1987, and 93 soil samples received between November 11 and November 23, 1987). The preparation laboratories in 4500S and 2026B have also been expanded. The facilities in 2026B are dedicated to mixed wastes, leaching-type preparation, and special preparations/analysis (such as high explosives and mixed waste volatiles). The original facility in 4500S is a general preparation laboratory with good preparation capabilities (including continuous liquid-liquid extraction, separatory funnel

extraction, soxhlet extraction, ultrasonic extraction, preparative chromatography, and tissue preparation). This laboratory specializes in the preparation of water samples during periods when large numbers of samples must be prepared. When all of this preparation capability is available and functioning, approximately 100 samples per week can be processed for the determination of semivolatile organic compounds, pesticides, and PCB's. As a continuing capability, approximately 30 samples per week can be prepared.

The ability to analyze prepared samples for pesticides and PCB's was also improved this year. Two full-time professionals and a technician are now dedicated to the effort. Auto samplers are functioning for both the quantitation and confirmation gas chromatographs. However, the greatest area of improvement has been the utilization and upgrading of the data system. Data from both the quantitation and confirmation columns are collected by the Nelson Analytical Chromatography Data System. Subsequently, these data are transferred to a data base system where all calculations, quality control checks, and confirmed identifications are printed on specific forms after verification. This system eliminates manual calculation and compilation of quality control information, and automatically compiles calibration data and results. Consequently, sample throughput is increased. The gas chromatography effort has many general functions in addition to pesticide and PCB determinations. For example, during the past year hundreds of aqueous samples were analyzed for ethylene glycol in support of laboratory operations. Other functions include the analysis of aqueous samples for solvents and other organics, the characterization of solvents, and high resolution profiling of unknown organic mixtures.

The capabilities of the gas chromatography/mass spectrometry effort were also enhanced this year by the addition of an advanced personal computer system which accepts information directly from the instruments used to determine volatile organic compounds. Then, after verification by the analyst, completed results forms can be printed. Here again, much manual manipulation of data has been eliminated and sample throughput has been aided. An example of the capacity to analyze volatile organics occurred in June 1987, when 189 volatile samples were received during a 10-day period. Analytical data for all samples were generated within the required 10-day holding time. However, sample volumes of this magnitude stress both personnel and equipment and the continuing capacity for volatile organic analyses is 40 samples per week.

(W. M. Caldwell, R. M. Edwards, G. S. Fleming, L. D. Golden, S. H. Harmon, R. W. Harvey, G. M. Henderson, J. A. Hayden, C. A. Treese, C. D. Varnadore, and J. E. Caton)

ORNL/UTK DISTINGUISHED SCIENTIST PROGRAM

G. A. Guiochon

The research work conducted by the group at ORNL deals with three main topics, the behavior of large concentration signals in chromatographic columns (nonlinear chromatography), the sources of error in chromatographic measurements and the development of nonselective detectors for HPLC.

Nonlinear Chromatography

A model for the behavior of large concentration signals in chromatographic columns has been developed. It is based on the use of mass balance equations for each of the compounds involved, either the components of a mixture or the constituents of the mobile phase. It is only possible to assume that the weak solvent is not adsorbed, by a proper choice of the adsorption convention. The model we have developed is an equilibrium model. It uses the equilibrium isotherms of the compounds involved (competitive isotherms for all n -compound problems for $n > 1$) to take into account the variation of their interaction with the stationary phase with increasing concentration. However, it is a major improvement of the ideal model because it can simulate the finite character of the column efficiency. We have written a program to solve the system of nonlinear partial differential equations obtained.

We have mainly investigated the following problems: elution of a single compound by a pure mobile phase (1-compound problem), elution of a mixture of two components by a pure mobile phase, elution of a single compound by a binary solvent, displacement of a single compound (2-compound problems) and displacement of a binary mixture (3-compound problem).

Currently, we use this model to study the influence of the various experimental conditions on the separation of a binary mixture by liquid chromatography, on the separate yield of the two compounds at various degrees of purity and on their production. Of special importance is the

observation that the productions of the two compounds are at a maximum for very different sets of values of the experimental parameters, so in practice, the optimum experimental conditions will be very different depending upon whether one is interested in the first eluted compound or in the second. We also find from our work that many controversies found in the literature are essentially the result of too broad conclusions derived from insufficient data. The column efficiency, for example, is a very important factor in the production of pure compounds when they are extracted from mixtures where they are dilute or very dilute. On the other hand, it has much less influence on either the yield or the production of purified compounds derived from technical products, when trace or minor impurities have to be extracted.

The influence of the relative composition of the mixture, the sample size, the column efficiency, the differences between the equilibrium isotherms of the two compounds (relative retention and ratio of the curvature of their isotherms) have also been studied.

Displacement chromatography is another popular mode of implementation of liquid chromatography which is especially attractive for preparative applications. We are using the same model of nonlinear chromatography to investigate the influence of the experimental conditions on the yield and production of the compounds prepared by this method. In displacement chromatography, the feed is injected in a solution of a weak solvent. Then a steady flow of a compound more strongly adsorbed than any component of the mixture under investigation is pumped through the column. The competitive adsorption process leads to the formation of adjacent bands moving at the same velocity. There is rather little interference between these bands.

As a consequence, however, when the displacement train has left the column, it has to be purged, cleaned and conditioned before a second batch can be processed. This takes time and although the production achieved by displacement chromatography is larger for one batch than the one achieved with elution, it is not clear whether, or under which conditions, either one of the two methods gives a larger or a less expensive production than the other one. This problem is one of our topics of research and will be actively pursued in 1988.

The results given by simulations have to always be tested by comparison with experimental results. This is another of our important activities. We are determining equilibrium isotherms and generating overloaded elution and displacement chromatograms using mixtures of compounds for which we know the competitive isotherms and can carry out precise numerical simulations. This permits a quantitative comparison. We are also studying various packing materials for liquid chromatography, in order to investigate the variability of equilibrium isotherms and compare their performance in preparative applications on a theoretically sound basis.

(S. Ghodbane,* A. Katti,** and M. T. Aubel*)

*Postdoctoral Research Associate

**Predoctoral student, University of Tennessee

Precision in Chromatography

We are studying the sources of errors in the determination of peak areas (quantitative analysis), retention times and peak efficiency. The work is

done experimentally. Various simulation programs are used to sort out the various contributions of the equipment and the measurement process. Column efficiencies have been measured by countless authors who have derived various, sometimes contradictory or even inconsistent, conclusions. It turns out that the precision of most methods is just insufficient to support many of these conclusions. In a paper in preparation we shall recommend a procedure which is more precise and accurate than the more popular ones.

(J. V. H. Schudel**)

Nonselective Detection in HPLC

We are studying the properties of HPLC detectors based on the nebulization of the column eluent in a stream of gas, followed by the vaporization of the solvent. The cloud of nonvolatile particulate is detected by the amount of light scattered when it flows through an intense light beam. Provided the nonvolatile content of the solvent is small enough, the detection limits are about one order of magnitude better than those of a refractive index detector. We are investigating the influence of the nature of the solvent (surface tension, viscosity, density) and of the solute on the response factor. We are also building different versions of the detector, using different nebulizers, sources of light, etc.

(M. T. Aubel,* and M. F. Righezza*)

*Postdoctoral Research Associate

**Predoctoral student, University of
Tennessee

5. ORNL ENVIRONMENTAL PROGRAMS

B. R. Clark

Environmental analysis has rapidly moved into a predominant position in the analytical support function of the division. The sheer number of samples as well as the increased demand for more stringent quality control in sample handling and analysis has required the participation of many.

Two large programs, the national DOE environmental survey and the in-house remedial action program, continued to make up the bulk of analytical work in the environmental areas. Activity in the survey increased substantially, while work in-house changed focus during the last year.

DOE ENVIRONMENTAL SURVEY

In early 1986 the DOE initiated a nation-wide survey of DOE sites to assess environmental contamination problems at each facility. The program has evolved rapidly in order to complete the survey within approximately a two and a half year time frame. The ACD has been a participant from the beginning and has actively supported the survey in many ways. Along with other participating laboratories, this division does extensive chemical analyses, helps develop special methods, reviews sampling and analysis plans and helps prepare reports.

ORNL conducted sampling and analysis campaigns at three sites this past year. These sites were the Pantex plant at Amarillo, Texas, the Lawrence Livermore and Sandia laboratories at Livermore, California, and Argonne National Laboratory at Chicago, Illinois.

Approximately 1550 samples were received from Pantex last June. Since Pantex is a final assembly plant for nuclear weapons,

very little radiochemical contamination exists and the focus was on environmental contamination from chemicals and explosives. This fact is reflected in the sample distribution which was about 750 samples each for organic and inorganic trace contaminants and only 33 samples for radiochemicals. Some 165 samples were for analysis of various conventional explosives that have been handled at the site. All Pantex samples have been analyzed and the sampling and analysis report is being prepared for an early 1988 publication date.

Lawrence Livermore National Laboratory, in conjunction with the Sandia-Livermore facility nearby, were surveyed in August. About 1500 samples were received for analysis which included 530 for organics, 490 for inorganics and 450 for radiochemicals. Most analyses have been completed and some samples were sent to other participating laboratories because of capacity overflow here.

Argonne National Laboratory was surveyed during the first three weeks of

November. About 1100 samples were generated - 500 for organic contaminants, 450 for inorganics and 120 for radiochemicals. A second phase of well sampling and installation is being conducted by Battelle, Columbus.

The survey has had a great impact on the analytical services operations of the division. Most affected was the organic analysis group which, several times, received hundreds of samples over only a few days interval. Very short holding times for some classes of analyses required a campaign effort by the staff. In addition, the survey adopted the EPA Contract Laboratory Program analytical protocols, so both the organic and inorganic sections had to accommodate a significant increase in the number of quality control samples and, especially, the amount of paper work required.

The impact of the survey on the division's service operations has been positive in several respects. The need to define and document standard operating procedures was underscored. The division now has these mostly in place. Nearly all environmental analyses require strict adherence to well-documented standard operating procedures and standard analytical methods in order to produce results acceptable to regulatory agencies. In general, the survey has helped the division increase both awareness and practice of more extensive QA/QC measures.

REMEDIAL ACTION PROGRAM

ORNL's in-house environmental restoration program took several turns which have affected this division's role. Most significant was the decision to use an outside contractor to conduct the remedial investigations and feasibility studies (RI/FS) for most of the X-10 complex. This decision meant a decreased role for the

division in performing analyses, but a new role in serving as advisor and QC overseer with respect to analytical services contracted to outside laboratories.

Certain components of the RI/FS program have been retained in-house. The division's support role remains intact in those areas and will actually include a significant increase in water quality analyses as more wells are installed. The principal remaining activities are those involving characterization of the low level waste system, including the hydrofracture site, some pits and trenches and portions of solid waste storage areas.

Several remedial action technology demonstration projects are in progress and/or planned. These usually include the division in a planning as well as analytical support role. Most recently the division has performed critical analyses on samples from a demonstration experiment of in-situ vitrification, viewed as a possible alternative to the stabilization of radionuclides buried in trenches.

The dispositions of unknown materials in inactive storage tanks is a crucial remedial action problem which has involved the division in a major way from the start. The disposal plan revolves around some choice of solidification and shallow land burial, but analytical results are crucial to the decision making. All the inactive tank contents are radioactive with unknown hazardous chemical components. Liquid phases are saturated with dissolved salts, principally, sodium nitrate, making trace chemical analyses a unique problem, unapproachable by standard procedures. The division will have a very important task this next year in providing analyses of these difficult matrices.

6. QUALITY ASSURANCE, SAFETY, AND TRAINING

QUALITY ASSURANCE/QUALITY CONTROL

W. R. Laing
P. L. Howell*

The quality control program comprised over 13,000 analyses in 1987--with an overall quality level of 96.5%. This includes over 2500 results submitted in an external blind inorganic control program. Six groups in the division were audited internally. Laboratories participating in analyses for the DOE Environmental Site Survey were audited five times by external auditors. These audits examined all aspects of these laboratories' operations including sample tracking, sample handling, laboratory operations, technical methods, documentation, and defensibility of data generated. Other laboratories in the division were audited four times by teams from outside ORNL. The Quality Assurance Coordinator and Quality Assurance Specialist participated in three audits outside the division.

Quality problems were addressed in a number of areas, including gamma counting, radon analyses, uranium measurements, and mercury preservation and determinations. A vendor of quality control and standard solutions was examined by ORNL, Y-12, K-25 and Portsmouth analytical laboratories and was found to be a suitable supplier of many control and standard materials for inorganic analyses. This has been particularly useful in light of EPA's recent inability to supply adequate amounts of independent QC solutions, as they had in the past.

The conversion of ACD's data management system to AnaLIS, using a new divisional to computer presented many problems in retrieval of statistical data for reporting quality control results. A joint effort of quality and programming staff members provided resolution of most problems and a suitable format for generating reports for external (blind) control data. Similar ideas will be applied to internally generated controls and data during 1988.

Improvements in overall laboratory quality assurance operations were made because of customer requirements for NQA-Level 1 (top level) quality assurance application and documentation. Methods for one program have undergone extensive review and revision to encompass provisions for quality control/assurance. Others will be revised and written during the coming months for other programs. Standard operating procedures were written for laboratory operations such as sample receiving, tracking, custody and instrument instructions.

The DOE Environmental Site Survey Program involves extensive organic, inorganic and radiochemical analyses. All are performed under extensive quality assurance protocols. Inorganic and organic laboratories performing analyses for the project are required to participate in a quarterly performance evaluation administered in conjunction with EPA's Contract Laboratory Program for superfund projects. These quarterly samples assure ongoing laboratory compliance with required protocols for documentation and analysis.

Quarterly performance evaluation (PE) sample sets were completed by groups in the division who perform analyses for the Environmental Survey. The average score for inorganics laboratories was 94.5%, and the organics laboratories average was 77%. Laboratories participating in the program will be required to complete the PE samples, for the duration of the sampling and analysis project.

*Quality Department

SAFETY PROGRAM

A. L. Harrod
R. E. Jones

The Analytical Chemistry Division continued to earn superior performance ratings during 1987. Notable achievements during this reporting period include:

1. Creation of a new program for training and documentation in reactor and nonreactor nuclear facilities. M. P. May was appointed to the new position of Training Coordinator.

2. Established emergency response squads for all facilities in the division and have documentation on file.

3. Initial stage of computerizing all training records for division personnel, which will be maintained at both the division office and at each facility.

4. Health and Environmental Training Courses for ACD staff - May 1987.

5. CPR course for Emergency Personnel - July 1987.

6. Annual drill to test the response of the local emergency personnel and building occupants - September 1987.

On December 4, 1987, the division received the Distinguished Safety Performance Award, in recognition of 15 years without a disabling injury.

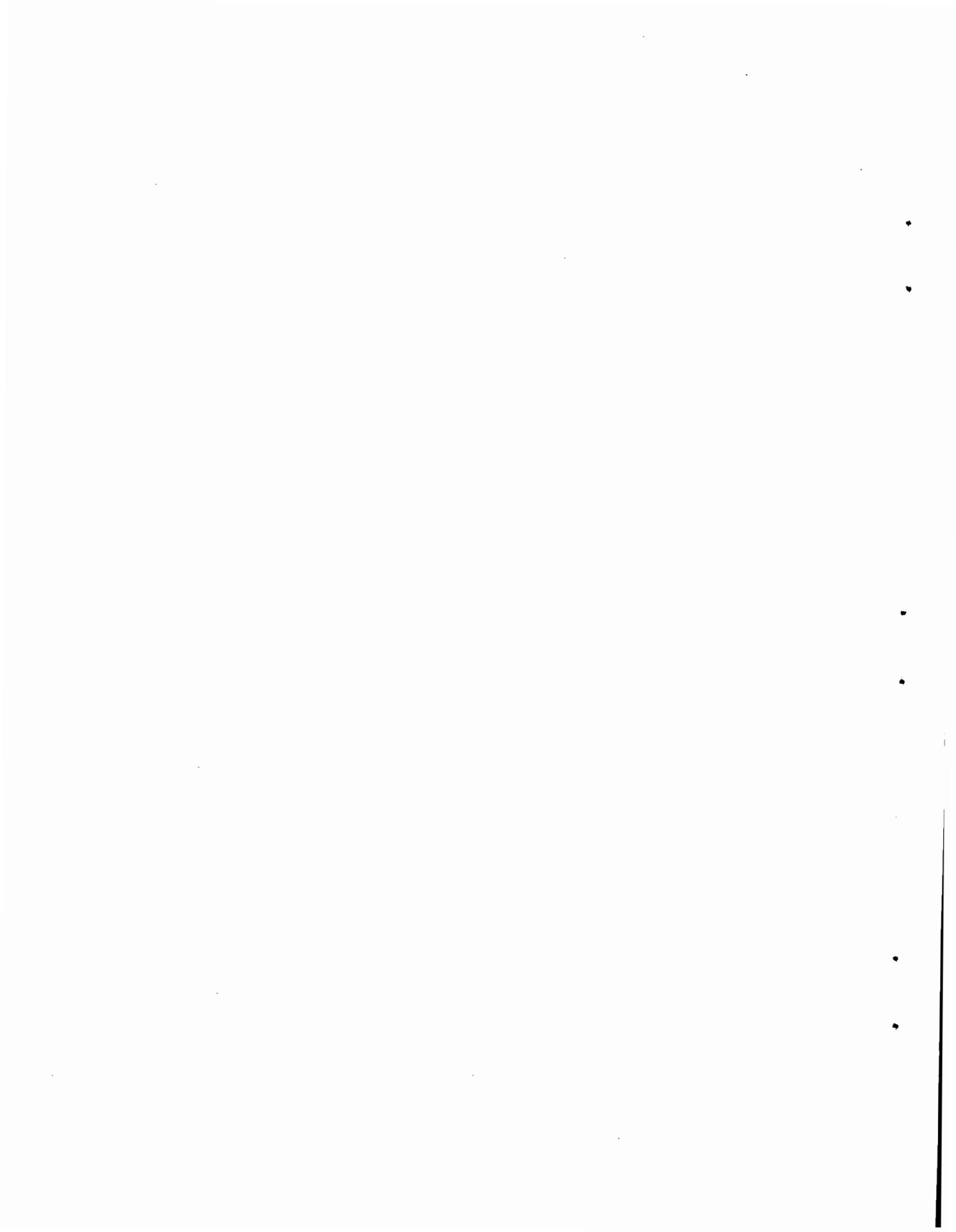
TRAINING PROGRAM

M. P. May

The effort this year has focused on improving training programs and establishing a record keeping system for our two nonreactor nuclear facilities in Bldg. 2026 and the Transuranium Pilot Plant (TPP). Training documentation files now exist at both facilities and in the division office. Copies of training records will be maintained at all three locations. Computer files have been set up using dBase III to store and manage all the training documentation, including employees, their facilities, exam scores, date taken, instructor, courses available, etc. We are still in the process of entering all past records while keeping the ongoing training records current. John Keller has written two programs to aid in maintaining and using the training data. The first is used by the training coordinator to edit and printout individual training records by facility. The second is used primarily by the division and facility offices to examine and/or print current individual records. This allows for a quick check without having to shuffle through paper files.

In training program development we have worked with the Training Resources Section of the Environmental Compliance & Health Protection Division (ECHP) to design a Basic Radiation Safety and Environmental Health Training Course for our nonreactor nuclear facility (NNF) workers. It is a day-long course given by a trainer and a certified health physicist (both from ECHP) and our facility manager, culminating with a written exam. We have held two such training sessions. Our goal is to train all division employees to the same basic level. More specific and/or

rigorous training will occur at the facility level. We are in the process of upgrading facility training to meet DOE regulations for content and to include written exams. We hope to soon put "Basic Emergency Alarms and Evacuation Systems at 2026" onto a video for training purposes. We are also updating our training for craft personnel, visiting scientists and experimenters, visitors and other non-permanent facility workers. Division-wide, all groups now have emergency evacuation squads and have been through a training drill or actual evacuation. The division has also purchased new video equipment and several commercially produced videos for training. At present we are preparing for an early 1988 DOE Audit.



7. SUPPLEMENTARY ACTIVITIES

The division maintains 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 1987 Advisory Committee was composed of:

- R. G. Cooks, Chemistry Department, Purdue University, West Lafayette, IN.
- J. W. Frazer, P. O. Box 1417, Tuolumne, CA.
- J. C. Giddings, Department of Chemistry, University of Utah, Salt Lake City, UT.
- E. D. Pellizzari, Research Triangle Institute, Research Triangle Park, NC.
- L. B. Rogers, Chemistry Department, University of Georgia, Athens, GA.
- R. G. Wymer, Chemical Technology Division, Oak Ridge National Laboratory, Oak Ridge, TN.

CONSULTANTS

The following experts served on a short-term consulting basis this past year.

- C. Magee, RCA, David Sanoff Research Center, Princeton, NJ.
- P. Pollard, AERE Harwell, Didcot, Ohon, UK.
- W. H. Pirkle, University of Illinois, Urbana, IL.
- R. M. Lindstrom, National Bureau of Standards, Gaithersburg, MD.
- E. Mahler, E. I. DuPont de Nemours and Company, Wilmington, DE.
- R. J. Wamack, Oak Ridge National Laboratory, Oak Ridge, TN.
- P. W. Bohn, University of Illinois, Urbana, IL.
- R. W. Linton, University of North Carolina, Chapel Hill, NC.
- C. Hatton, UCLA/USOC, Los Angeles, CA.
- T. Whitaker, Pacific Northwest Laboratory, Richland, WA.
- W. M. Bass, III, University of Tennessee, Knoxville, TN.
- J. W. Taylor, University of Wisconsin, Madison, WI.
- A.O.C. Nier, University of Minnesota, Minneapolis, MI.

The following experts served on a long-term basis this year.

- G. Mamantov, University of Tennessee, Knoxville, TN.
- K. D. Cook, University of Tennessee, Knoxville, TN.
- M. J. Sepaniak, University of Tennessee, Knoxville, TN.
- R. W. Holmberg, retired, Oak Ridge, TN.
- A. G. Marshall, Ohio State University, Columbus, OH.
- J. W. Taylor, University of Wisconsin, Madison, WI

EDUCATION PROGRAMS

Debra Bostick served as University Relations Coordinator and "dean" for student guests during 1987.

OAK RIDGE ASSOCIATED UNIVERSITIES PROGRAMS

Postgraduate Research Programs. Perry R. Blazewicz (Yale University) joined the Analytical Spectroscopy Section to do research in laser and microwave spectroscopy with Mike Ramsey and Bill Whitten.

Thomas G. Nolan (University of Wyoming) is continuing his research with Mike Ramsey investigating analytical applications of degenerate four-wave mixing and the development of novel liquid chromatography detectors.

Michelle Buchanan has two post-docs working with her in the Organic Chemistry Section: John W. Haas (University of Massachusetts) and Elizabeth A. Stemmler (Indiana University). Both are investigating the analytical characteristics of the multimode ionization detector.

ORAU Graduate Student Research Program. Chris P. Liebman, who previously worked through a subcontract with the University of Tennessee, received an ORAU Fellowship and is continuing his predoctoral studies with P. J. Todd.

Lance Koutny, Cornell College, who previously worked in the division through the GLCA Program, continued his research as an ORAU graduate student participant. He worked with Bill Whitten on a low-pressure interface for high-resolution laser spectroscopy in an air/acetylene flame.

ORAU Faculty Research Program. Subrahmanyam Pendyala (SUNY, Fredonia) completed his sabbatical assignment and continued his research with Lester Hulet via a summer appointment. He worked on the magnetic field configurations and positron trajectories for the ORELA slow positron facility and participated in studies of epoxy-carbon fiber composite materials by use of positron lifetime spectroscopy.

J. A. Howell (Earlham College) is working with Mike Ramsey on laser spectroscopy research while on a sabbatical appointment.

ORAU Faculty Research Program for Historically Black Colleges and Universities. The division was host to two participants in the HBCU program, both of whom worked in the Organic Chemistry Section. Ronald S. Budhram (Tuskegee University) worked with Wayne Griest and C.-h. Ho on the synthesis of modified DNA bases and DNA adducts. David Eaton, (Fort Valley State College) worked with Wayne Griest on the separation of adducted nucleic acid bases, nucleosides, and nucleotides using supercritical fluid chromatography.

ORAU Summer Program. Anthony A. Bonanno (Clarkson University) worked with Bob Shaw, Jack Young, and Dave Smith to interface an IBM personal computer to a new resonance ionization mass spectrometer. Andrew Fong (Rhodes College) worked with Leon Klatt performing experiments to obtain heat and mass transfer data in liquid films using fiber optic technology. Carolyn Chang (Stanford University) worked with Mike Ramsey to help develop a refractive index detector for liquid chromatography based on two-wave mixing in photorefractive materials. Becky Dittmar (Angelo State University) worked with Rose Ramsey to aid in the evaluation of a new chromatographic detector.

SUMMER INTERN PROGRAM

The division had two students who participated in the summer intern program. A chemistry student, Kim P. Chen (University of North Carolina) worked with Joe Stewart and Cy Feldman in the Chemical and Physical Analysis Group. Kelli M. Trego (University of Tennessee) worked with Mike Ramsey in the Laser Spectroscopy and Instrumentation Group.

CO-OP PROGRAM

Co-Op Program. J. M. Cosgrove worked with Michelle Buchanan on multimode ionization detection and atmospheric pressure ionization mass spectrometry. G. C. Keyes worked with Jeff Wade in the low-level radiochemical analysis group, participating in various group projects.

UNIVERSITY OF TENNESSEE PROGRAMS

Distinguished Scientist Program. The Analytical Chemistry Division was successful in adding Dr. Georges Guiochon to the University of Tennessee/ORNL Distinguished Scientist Program. Five of his students are working at ORNL: Mark T. Aubel, a post-doc student from the University of Georgia; Samir Ghodbane, an Algerian citizen, who is a post-doc from Georgetown University; Michel F. Righezza, a French citizen, also a post-doc from Georgetown University; and two predoctoral graduate students: Jennifer Schudel and Anita Katti.

Science Alliance Research Program. Luis E. Cuellar from El Salvador is a student at the University of Tennessee; he worked with Lester Hulett measuring positron lifetime spectra of composite materials and other solids.

Postgraduate Research Program. Gary J. VanBerkel (Washington State University) is working with Dave Smith pursuing research and development projects in the field of mass spectrometry/mass spectrometry.

Kyung H. Row (Korea Advanced Institute of Science and Technology) is continuing his work with Wayne Griest in the Organic Chemistry Section, studying the feasibility of using analytical-scale separations data to design preparative-scale separations conditions for the isolation of biopolymers related to biotechnology and to molecular dosimetry.

INTERNATIONAL ATOMIC ENERGY AGENCY PROGRAM

The division was host to an IAEA Fellow, Solaiman A. Tarafdar, from Bangladesh, for a six-month period. He was assigned to Jim Eldridge to assist in development activities related to the measurement of low-levels of radionuclides in environmental materials.

GREAT LAKES COLLEGES ASSOCIATION PROGRAM

Kheng B. Lim, a Malaysian citizen from COE College, worked with Lester Hulett on measuring positron lifetime spectra of composite materials and other solids.

A student from Dennison University, Vivian Chan, worked with Rose Ramsey studying the chromatographic separation and detection of DNA components. Vivian is originally from Hong Kong.

Margaret Decker, Earlham College, spent her summer assignment with Wayne Griest working in chromatographic separation and detection of DNA components and explosives by supercritical fluid chromatography.

ORNL/DOE CONFERENCE ON ANALYTICAL CHEMISTRY IN ENERGY TECHNOLOGY

The 30th annual conference was held at the Knoxville Hilton Hotel, October 20-22, 1987. The theme of the conference was "Back to the Future." Attendance was 265, with representatives from foreign countries, academia, industrial institutions, DOE contractors, and other government agencies. There were 16 exhibits (43 representatives) and 18 booths. Major topics included nuclear technology, environmental measurements, mass spectrometry, spectroscopy, actinide chemistry, and a look to the future.

A. L. Harrod was Arrangements Chairman, W. R. Laing was Technical Program Chairman, and S. D. Wright was Treasurer. P. M. Trentham and L. D. Golden were Conference Secretaries. Other conference committee members were C. E. Coffey (Savannah River Laboratory), F. F. Dyer, N. M. Ferguson, G. L. Glish, W. H. Griest, W. D. Shults, and W. B. Whitten.

SEMINAR PROGRAM

<u>Speaker</u>	<u>Title</u>	<u>Date</u>
Magee, C. RCA, David Sarnoff Res. Center, Princeton, NJ	"Materials Characterization Using Ion Beams"	April 2, 1987
Pollard, P. AERE Harwell, Didcot, Oxon, UK	"Photoacoustic Spectrophotometric Analysis of Environmental Actinides"	June 16, 1987
Pirkle, W. H. Univ. of Illinois, Urbana, IL	"Advances in Chiral Recognition: from Chromatography to Synthesis"	August 3, 1987
Lindstrom, R. M. NBS, Gaithersburg, MD	"Elemental Analysis Using Neutron Capture Prompt Gamma Rays"	August 12, 1987
Mahler, E. E.I. DuPont de Nemours and Co., Wilmington, DE	"Artificial Intelligence in DuPont-- A Pragmatic Approach"	Sept. 15, 1987
Warmack, R. J. ORNL, Oak Ridge, TN	"Scanning Tunneling Microscopy"	Sept. 17, 1987

Bohn, P. W. Univ. of Illinois Urbana, IL	"Absorption and Scattering: Thin Film Analogs of Classical Experiments"	Sept. 24, 1987
Linton, R. W. Univ. of N. Carolina, Chapel Hill, NC	"Three-Dimensional Materials Characterization Using Microprobe Mass Spectrometry"	Oct. 12, 1987
Hatton, C. UCLA/USOC, Los Angeles, CA	"Drug Testing in Sports"	Oct. 15, 1987
Whitaker, T. Pacific Northwest Laboratory, Richland, WA	"High Resolution Resonance Ionization Mass Spectrometry"	Oct. 23, 1987
Bass, W. M., III Univ. of Tennessee, Knoxville, TN	"Recent Forensic Cases"	Nov. 4, 1987

AWARDS AND HONORS

Two Martin Marietta Energy Systems Inventors Awards were given to the division at Awards Night, May 8, 1987. Gary L. Glish and Scott A. McLuckey received the award for the conceptualization and development of an advanced atmospheric-pressure ionization source for mass spectrometry. Leon N. Klatt, James E. Strain, and Debra A. Bostick received the award for the successful development of an invention entitled "Method for Measurement of Refrigerant/Absorbent Concentration in Heat Absorption Machines."

Joel A. Carter received a Martin Marietta Energy Systems Technical Achievement Award for exemplary technical leadership and many significant achievements that have placed ORNL among the nation's leaders in analytical mass spectrometry.

The division has established a Distinguished Service Award to be given to deserving permanent staff members of ACD. Jack Moneyhun received the first such award recognizing his technical work both inside and outside the Laboratory as well as high creativity, high standards, persistence, and ready willingness to help others. The award consists of a plaque and a monetary award. Hank McKown received the second ACD Distinguished Service Award for a lifetime of contributions to the work of the division, both research and service.

Gary L. Glish has been elected to the Vice Presidency of the American Society of Mass Spectrometry.

Mike Maskarinec has been asked to serve on a national task force of six experts for the Environmental Protection Agency and the Department of the Army. The task force is to improve QA/QC for the two agencies in the areas of data management, quality assurance, quality control, and methods development.

Joel Carter has been nominated as an observer to an Advisory Group Meeting on Quality of Safeguards Analytical Services, sponsored by the International Atomic Energy Agency.

Michelle Buchanan has been invited to speak in the Samuel M. McElvain Seminar Series at her alma mater, the University of Wisconsin. This special series promotes informal interaction between university people and distinguished guests from both academia and industry.

Warner Christie was a featured speaker at the dedication of the Center for High Performance MS/MS at the University of Tennessee.

Rose Ramsey has been elected Program Chairman of the East Tennessee Chromatography Discussion Group.

Four people in the division have received Significant Event Awards. Scott McLuckey and Gary Glish received this award for inventing a unique ion source for analytical mass spectrometry. Mike Ramsey and Bill Whitten received the award in recognition of their research in degenerate four-wave mixing.

The division received a Certificate of Accomplishment, recognizing 15 years of safe operation without a lost-time accident.

Michelle Buchanan's book on Fourier Transform Mass Spectrometry was published as one of the "symposium-based books" series of the American Chemical Society. It is the first book that deals exclusively with FTMS.

Bill Laing has been elected chairman of ASTM Committee C-26 on Nuclear Fuel Cycle.

Mike Ramsey was invited to Venezuela to teach a course on Laser Spectroscopy at the Universidad de Los Andes. His trip will be sponsored by the Venezuela Council for Scientific Humanistic and Technological Development.

PATENTS

Rose Ramsey and Richard Todd (I&C Division) received two patents this year, one for a pulsed helium ionization detector and one for a closed-loop pulsed helium ionization detection system.

ADDITIONAL PROFESSIONAL ACTIVITIES

BUCHANAN, M. V.

Editorial Board:	<u>Biomedical and Environmental Mass Spectrometry</u>
Chairman:	Environmental Applications Group of the American Society for Mass Spectrometry
Consultant:	National Research Council of Canada: National Center for FTMS
Advisory Board:	National Science Foundation, Biological Centers Program
Councilor:	East Tennessee Mass Spectrometry Discussion Group

CARTER, J. A.

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

COSTANZO, D. A.

Member:	ASTM Committee C-26, Nuclear Fuel Cycle, and Subcommittee C-26:05, Test Methods
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DYER, F. F.

Member: Planning Committee, 30th Analytical Chemistry in Energy
Technology Conference

ELDRIDGE, J. S.

Board Member: Environmental Radiation Section of the Health Physics Society
Member: Oak Ridge Reservation -- Resource Management Organization:
Environmental Surveillance
Lab Committee 8 of the International Society Committee for the
Methods of Air Sampling and Analysis

EMERY, J. F.

Division Laboratory Emergency Sample Coordinator
Representative: Division Computer Systems Security Officer

FELDMAN, C.

Fellow: American Society for Testing and Materials
Member: ASTM Committee E-2 on Emission Spectroscopy, Subcommittees on
Fundamental Methods, Editorial Practices, and Nomenclature

FERGUSON, N. M.

Division Energy Systems Environmental Analysis Committee
Representative:
Member: Planning Committee, 30th Analytical Chemistry in Energy
Technology Conference

GLISH, G. L.

Chairman: Ion Physics and Instrumentation Committee, American Society for
Mass Spectrometry
Lecturer: ORAU Traveling Lecture Program
Member: Planning Committee, 30th Analytical Chemistry in Energy
Technology Conference

GRIEST, W. H.

Consultant: Department of Energy - SBIR Review
Department of Energy - BES
Electric Power Research Institute
Environmental Protection Agency - IERL/TSO
Member: Planning Committee, 30th Analytical Chemistry in Energy
Technology Conference

GUERIN, M. R.

Member: National Academy of Sciences Committee on Multimedia Approaches
to Pollution Control
University of Kentucky Tobacco and Health Research Institute
Advisory Board
Chairman: National Cancer Institute Program Project Review Committee on
Experimental Tobacco Carcinogenesis
Consultant: SBIR Reviews, DOE
SBIR Reviews, NCI
NAS, Passive Smoking Report
PHS, Passive Smoking Report
Organizer: DOE Workshop on Analytical Approaches to DNA Adduct Measurement

HIGGINS, C. E.

ACD Ombudsman

HOWELL, P. L

Coordinator: ACD Quality Assurance Program
 Member: ASTM Committee C-26 Nuclear Fuel Cycle
 Publicity: American Society for Quality Control, Tennessee Section
 Chairman:

ILGNER, R. H.

Consultant: Fourth Judicial District Court, Monroe, LA

JENKINS, R. A.

Consultant: Federal Trade Commission: Standardized Smoking Practices
 National Institute on Drug Abuse - SBIR Reviews

JONES, R. E.

Division: ACD Radiation Control Officer
 Representative: Respirator Trainer
 Alternate Division Safety Officer
 Member: ASTM Committee D-33, Protective Coating and Lining Work for
 Generation Facilities

KLATT, L. N.

Member: Energy Systems, Ph.D. Recruiting Team
 Secretary: ACS Division of Analytical Chemistry
 ACD Seminar Committee

LAING, W. R.

Chairman: ASTM Subcommittee C-26:05, Test Methods, Nuclear Fuel Cycle
 Vice-Chairman: ASTM Committee C-26, Nuclear Fuel Cycle
 Coordinator: ACD Quality Assurance Program
 ACD Energy Conservation Program
 Fellow: American Society for Testing and Materials
 Member: ASTM Committee D-33, Protective Coatings
 ASTM Committee D-34, Waste Disposal
 ISO Technical Committee 85, Subcommittee 5
 ORNL Pregrievance Committee
 INMM Group 5.1, Analytical Chemistry Laboratory Measurement
 Control
 Technical Program: 30th Analytical Chemistry in Energy Technology Conference
 Chairman:

MA, C. Y.

Coordinator: ACD Family Day Tour

MASKARINEC, M. P.

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

MCLUCKEY, S. A.

Chairman: ACD Seminar Committee
 Organizer: Environmental Applications Interest Group Workshop, American
 Society for Mass Spectrometry Annual Meeting, Denver, CO

RAMSEY, J. M.

Member: ORNL Exploratory Studies Proposal Review Committee
 ACS Program Advisory Committee, Analytical Chemistry Division
 Editorial Advisory Board, Progress in Analytical Spectroscopy

RAMSEY, R. S.

Member: ACD PhD Recruiter

ROSS, H. H.

Faculty Member: Dept. of Chemistry, University of Tennessee, Knoxville (Adjunct Professor, Science Alliance)

Advisory Board: Journal of Radioanalytical and Nuclear Chemistry

Advisory Committee: International Conference on Liquid Scintillation Science and Technology

Member: ACS Fellowship Committee, Division of Analytical Chemistry
Executive Committee: Martin Marietta Energy Systems Inventors Forum

Judge: International Science Fair, Knoxville (Chemistry)

Consultant: Piper & Marbury, Washington, DC

ROSSEEL, T. M.

Treasurer: American Vacuum Society (AVS) Tennessee Valley Chapter

Chairman: AVS, Tennessee Valley Chapter, Scholarship Committee

Member: AVS, 35th National Symposium, Local Organizing Committee

SHULTS, W. D.

Member: Advisory Board, ACS Publications
Board of Visitors, Chemistry Department, University of Tennessee
Science Alliance Faculty Awards Committee, University of Tennessee

Advisory Committee, MMES Travel Department

Organizing Committee, Second Karlsruhe Conference on Analytical Chemistry in Nuclear Technology

Chairman: ORNL Division/Program Directors' Caucus

ORNL PIP Committee On Expert Systems

SMITH, D. H.

Member: ORNL PIP Committee on Expert Systems

STEWART, J. H., Jr.

Division Representative: ACD Environmental Protection Officer

Member: ASTM Committee D-34 Waste Disposal
International Working Group, "Analytical Standards of Minerals, Ores, and Rocks"

ACD Coordinator: Manuscript Review Board for Analytical Chemistry
Technician Advisory Group

STOKELY, J. R.

Coordinator: ACD MS/BS Recruiting

Chairman: DOE Site Survey RAD Committee

TODD, P. J.

Coordinator: ACD Awards

Member: ORAU Traveling Lecture Program

ASMS Solids and Surface Analysis Interest Group

TOMKINS, B. A.

Member: ACD Seminar Committee

WALKER, R. L.

Advisory Panel Member: DOE/ISA Laboratory Advisory Group for Effluent Research (LAGER)

WHITTEN, W. B.

Member: Planning Committee, 30th on Analytical Chemistry in Energy
Technology Conference

WISE, M. B.

Chairman: ACD Seminar Committee
Member: ACD Safety Committee
ORAU Traveling Lecture Program

YOUNG, J. P.

Fellow: American Association for the Advancement of Science
Division Graduate Fellow Selection Panel, ORNL
Representative:

8. PRESENTATION OF RESEARCH RESULTS

As in past years, the division has actively responded to the evolving priorities of the ORNL research effort by changing the emphasis of some of its own programs or instituting new studies. Subjects of major concern include nuclear and nonnuclear energy, new instrumentation and its application, and environmental problems such as monitoring and clean up at ORNL and elsewhere. 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, TITLE, AND WHERE PUBLISHED
Buchanan, M. V.	Wise, M. B.; Buchanan, M. V.; Hettich, R. L., "Analysis of Environmental Tobacco Smoke Using FTMS," in <i>FT/ICR Newsletter</i> , Spring Issue 1987, Ohio State University, April 1987, p.1.
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- Feldman, C. Feldman, C., *Report of Foreign Travel of C. Feldman, Research Staff Member, Analytical Chemistry Division, (Italy)*, ORNL/FTR-2701, October 6, 1987.
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- Francis, C. W.;* Maskarinec, M. P., *Leaching of Metals from Alkaline Wastes by Municipal Waste Leachate*, ORNL/TM-10050, March 1987.
- Manning, D. L.; Maskarinec, M. P., *Analysis of Nitroguanidine in Aqueous Solutions by HPLC with Electrochemical Detection and Voltammetry*, ORNL/TM-10295, April 1987.
- Maskarinec, M. P.; Manning, D. L.; Harvey, R. W., *Application of XAD-4 Solid Sorbent and HPLC with Electrochemical Detection to the Analysis of Phenols in Water*, ORNL/TM-10309, June 1987.
- Maskarinec, M. P.; Manning, D. L., *Application of XAD-4 Solid Sorbent to the Collection of Pesticides from Water Samples*, ORNL/TM-10527, October 1987.
- Maskarinec, M. P.; Holladay S. K., *Quality Assurance/Quality Control in Waste Site Characterization and Remedial Action*, ORNL/TM-10600, October 1987.
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- McKown, H. S. McKown, H. S., *Report of Foreign Travel by Henry S. McKown, Staff Member of Analytical Chemistry Division, (Austria and West Germany)*, ORNL/FTR-2570, June 5, 1987.

- McKown, H. S. Smith, D. H.; McKown, H. S.; Carter, J. A., *ISPO Task A-143, Final Report of an Evaluation of the Finnigan-MAT THQ Mass Spectrometer as an On-Site Inspection Instrument (ISPO-285)*, ORNL/TM-10627, November 1987.
- Todd, P. J.; McKown, H. S.; Smith, D. H., *Installation and Operation of the Ion Optical Program Simion*, ORNL/TM-10409, May 1987.
- Smith, D. H.; McKown, H. S.; Carter, J. A., *ISPO Task A-143: Final Report of an Evaluation of the Finnigan-MAT THQ Mass Spectrometer as an on-site Inspection Instrument ISPO-285*, ORNL/TM-10627, December 1987.
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- McLuckey, S. A., *Report of Foreign Travel of Scott A. McLuckey, Research Staff, Analytical Chemistry Division, (Spain, Belgium, and The Netherlands)*, ORNL/FTR-2719, October 19, 1987.
- Rosseel, T. M. Rosseel, T. M.; Dale, J. M.; Hulett, L. D.; Pepmiller, P. L.;* Vane, C. R.;* Walkiewicz, T. A.;* Young, J. P., "Advances in High-Resolution Studies of the Chemical Effects in the Molybdenum L Heavy-Ion Induced X-ray Satellite Emission (HIXSE) Spectra," *Physics Division Progress Report for Period Ending Sept. 30, 1986*, ORNL-6326, March 1987.
- Smith, D. H. Smith, D. H., *Report of Foreign Travel by David H. Smith, Chemist, Analytical Chemistry Division, (Austria)*, ORNL/FTR-2603, July 10, 1987.
- Todd, P. J.; McKown, H. S.; Smith, D. H., *Installation and Operation of the Ion Optical Program SIMION*, ORNL/TM-10409, May 1987.
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- Stokely, J. R. Stokely, J. R., *Analytical Chemistry Division Summary Report: Radioactive Materials Analysis Section*, ORNL/CF-87/29, January 1, 1987.
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- Todd, P. J. Todd, P. J., *Report of Foreign Travel by Peter J. Todd, Research Associate, Analytical Chemistry Division*, (France, FRG, Wales), ORNL/FTR-2705, October 6, 1987.
- Todd, P. J.; McKown, H. S.; Smith, D. H., *Installation and Operation of the Ion Optical Program Simion*, ORNL/TM-10409, May 1987.
- Walker, R. L. Goeringer, D. E.; Christie, W. H.; Bertram, L. K.; Walker, R. L.; Carter, J. A., *Ion Microprobe Mass Spectrometry Using Sputtering Atomization and Resonance Ionization: Final Report*, ORNL/TM-10643, December 1987.
- Wise, M. B. Maskarinec, M. P.; Wise, M. B.; Buchanan, M. V., *Technology Assessment for the Determination of Chemical Agent Vapors in Demilitarization Facilities*, ORNL/TM-10346, January 1987.
- Young, J. P. Rosseel, T. M.; Dale, J. M.; Hulett, L. D.; Pepmiller, P. L.;* Vane, C. R.;* Walkiewicz, T. A.;* Young, J. P., "Advances in High-Resolution Studies of the Chemical Effects in the Molybdenum L Heavy-Ion Induced X-ray Satellite Emission (HIXSE) Spectra," *Physics Division Progress Report for Period Ending Sept. 30, 1986*, ORNL-6326, March 1987.

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.

SPEAKER	AUTHORS, MEETING, AND DATE
Asano, K. G.	Asano, K. G.; Glish, G. L.; McLuckey, S. A.; McKown, H. S., "The Rapid Analysis of Trace Organics in Air by Mass Spectrometry/Mass Spectrometry," 17th Annual Symposium on the Analytical Chemistry of Pollutants, Jekyll Island, GA, May 18-20, 1987.
	Asano, K. G.; McLuckey, S. A.; Glish, G. L.; McKown, H. S., "The Analysis of Gaseous Organics in Ambient Air by Glow Discharge Ionization Coupled with Mass Spectrometry/Mass Spectrometry," 30th Analytical Chemistry in Energy Technology Conference, Knoxville, TN, October 20-22, 1987.
Bostick, D. T.	Bostick, D. T.; Klatt, L. N.; Perez-Blanco, H.,* "The Fiber Optic Refrigeration Cycle Monitor," 1987 Technical Symposium Southeast on Optics, Electro-Optics, and Sensors, Orlando, FL, May 17-22, 1987.
Buchanan, M. V.	Buchanan, M. V.; Rubin, I. B.; Stemmler, E. A., "Negative Ion CI Studies of Nitrogen-Substituted Aromatics by GC/MS and FTMS," 35th American Society for Mass Spectrometry Conference, Denver, CO, May 24-29, 1987.
	Buchanan, M. V.; Ma, C. Y.; Moneyhun, J. H.; Guerin, M. R., "Chemical Characterization and Toxicological Evaluation of Airborne Mixtures - Smoke Generation and Characterization. Part 2. Colored Smoke Grenades," U.S. Army Biomedical Research and Development Laboratory Contractors' Review Meeting, Fort Detrick, Frederick, MD, September 15-16, 1987.
	Buchanan, M. V., "Negative Ion Processes for Structural Information," McElvain Lecture, Chemistry Department, University of Wisconsin-Madison, Madison, WI, October 1, 1987 (invited).

- Buchanan, M. V. Buchanan, M. V., "Gas Phase Processes for the Isomeric Identification of Trace Organics Using Mass Spectrometry," U.S. Department of Energy, Office of Health and Environmental Research Site Review, Oak Ridge National Laboratory, Oak Ridge, TN, October 15-16, 1987.
- Wise, M. B.; Buchanan, M. V.; Haas, J. W., "Multimode Ionization Detector for Gas Chromatography and Electron Affinity Estimation," U.S. Department of Energy, Office of Health and Environmental Research Site Review, Oak Ridge National Laboratory, Oak Ridge, TN, October 15-16, 1987.
- Carter, J. A. Carter, J. A.; Walker, R. L.; Smith, D. H.; May, M. P.; Hebble, T. L.,* "Independent Fissile Inventory Verification in a Large Tank Employing Lutetium Double Spikes," Institute of Nuclear Materials Management Annual Meeting, Newport Beach, CA, July 12-16, 1987.
- Carter, J. A., "Detection of Explosives by MS/MS," National Security Information Exchange Conference, Idaho National Engineering Laboratory, Idaho Falls, ID, October 14, 1987.
- Carter, J. A., "Mass Spectrometry," 30th Analytical Chemistry in Energy Technology Conference, Knoxville, TN, October 20-22, 1987.
- Christie, W. H. Christie, W. H.; Goeringer, D. E.; Valiga, R. E., "Sputter Atomization Resonance Ionization Mass Spectrometry," Analytical Chemistry Division Information Meeting, Oak Ridge National Laboratory, Oak Ridge, TN, February 18-19, 1987.
- Christie, W. H., "Mass Spectrometry Laboratory," DOE-BES Chemical Sciences Site Review, Oak Ridge National Laboratory, Oak Ridge, TN, March 30-31, 1987.
- Christie, W. H., "Mass Spectrometry at Oak Ridge: Past Present, and Future," University of Tennessee, Knoxville Hertel Event, Knoxville, TN, November 12-13, 1987.

- Christie, W. H. Christie, W. H.; Goeringer, D. E., "Investigation of Matrix Effects on the Neutral Fractions Ejected from Ion-Bombarded, Uranium-Containing Solids having Resonance Ionization Mass Spectrometry," Sixth International Conference on Secondary Ion Mass Spectrometry, Paris, France, September 13-18, 1987.
- Costanzo, D. A. Costanzo, D. A.; Peretz, F. J.,* "Methodology for the Characterization of Low-Level Liquid Radioactive Wastes at the Oak Ridge National Laboratory," Oak Ridge Model Conference, Oak Ridge, TN, October 13-16, 1987.
- Dale, J. M. Dale, J. M.; Hulett, L. D., Jr.; Rosseel, T. M.; Fellers, J. L.* "Positron Annihilation Spectroscopy," 194th National Meeting of the American Chemical Society, New Orleans, LA, August 30-September 4, 1987.
- Datz, S.* Datz, S.;* Hippler, R.;* Andersen, L. H.;* Dittner, P. F.;* Knudsen, H.;* Krause, H. F.;* Miller, P. D.;* Pepmiller, P. L.;* Rosseel, T. M.; Stolterfoht, N.;* Yamazaki, Y.;* Vane, C. R.,* "Electron Transfer Processes in Collisions of Highly Charged Energetic (0.1 to 1.0 MeV/Nucleon) Ions with Helium Atoms," U.S.-Japan Cooperative Science Programme, Joint Seminar on Interactions of Highly Ionized Atoms Produced in Heavy Ion Collisions, Kobe, Japan, March 16-20, 1987.
- Donohue, D. L. Donohue, D. L.; Fiedler, R.;* Swietly, H.;* Deron, S.,* "Characterization and Use of a Four-Isotope Tracer for Internally Calibrated Isotope Dilution Mass Spectrometry of Uranium and Plutonium," 4th Meeting on Nuclear Analytical Methods, Dresden, DDR, May 4-8, 1987.
- Donohue, D. L., "Laser-Based Resonance Ionization Mass Spectrometry of U and Pu," European Institute for Transuranium Elements, Karlsruhe, DDR, February 12, 1987 (invited).
- Dyer, F. F. Dyer, F. F.; Emery, J. F.; Robinson, L.; Teasley, N. A., "Design and Use of the ORNL HFIR Pneumatic Tube Irradiation System," 2nd International Workshop Activation Analysis With Short-Lived Nuclides," Vienna, Austria, September 21-24, 1987.

- Dyer, F. F. Dyer, F. F., "Use of Spreadsheet and Terminal-Emulation Software in Gamma-Ray Spectroscopy," 1987 Winter Meeting of the American Nuclear Society, Los Angeles, CA, November 15-19, 1987.
- Dyer, F. F., "Automated Gamma-Ray Scanner for ORR Fuel Elements," Analytical Chemistry Division Information Meeting, Oak Ridge National Laboratory, Oak Ridge, TN, February 18-20, 1987.
- Dyer, F. F., "Facilities of ORNL for Neutron Activation Analysis and Gamma Ray Spectroscopy," International Atomic Energy Agency at Seibersdorf, Austria, September 25, 1987.
- Dyer, F. F., "Facilities of ORNL for Neutron Activation Analysis and Gamma Ray Spectroscopy," Institute Laue-Langeuin, Grenoble, France, October 2, 1987.
- Dyer, F. F., "Facilities of ORNL for Neutron Activation Analysis and Gamma Ray Spectroscopy," Institute for Nuclear Science, Gent, Belgium, October 5, 1987.
- Dyer, F. F., "Facilities of ORNL for Neutron Activation Analysis and Gamma Ray Spectroscopy," Nuclear Research Center, Julich, FRG, October 8, 1987.
- Eldridge, J. S. Eldridge, J. S.; Oakes, T. W.,* "Transport of Strontium-90 from Waste Disposal Sites by White-Tailed Deer," Fourth Annual Meeting of the Southeastern Regional Health Physics Society Chapters, Williamsburg, VA, May 7-9, 1987.
- Eldridge, J. S.; Keele, B. D.; Oakes, T. W.,* "Transport of Radioactivity from Contaminated Ponds by Migratory Waterfowl," Health Physics Society Meeting, Salt Lake City, UT, July 7-11, 1987.
- Feldman, C. Feldman, C., "Purification of Wastewater From the Beneficiation of Pitchblende Ores: Removal of Hazardous Chemical and Radioactive Impurities," 6th International Conference on Chemistry for Protection of the Environment, Torino, Italy, September 15-18, 1987.

Glish, G. L.

Glish, G. L.; McLuckey, S. A., "Experiments with a Novel Hybrid MS/MS Instrument," Analytical Chemistry Division Information Meeting, Oak Ridge National Laboratory, Oak Ridge, TN, February 18-19, 1987.

Glish, G. L.; McLuckey, S. A.; McKown, H. S., "A Tandem Quadrupole/Time-of-Flight Mass Spectrometer for Application to Environmental Analyses," Pittsburgh Conference and Exhibition, Atlantic City, NJ, March 9-13, 1987.

Glish, G. L., "Recent Developments in Applications of MS/MS," Symposium on Applications of Hybrid Techniques in Mass Spectrometry, sponsored by Spectroscopy Society of Pittsburgh, Pittsburgh, PA, March 21, 1987 (invited).

Glish, G. L.; McLuckey, S. A.; Asano, K. G.; McBay, E. H., "Organic Mass Spectrometry," DOE-BES Chemical Sciences Site Review, Oak Ridge National Laboratory, Oak Ridge, TN, March 30-31, 1987.

Glish, G. L., "A New Dimension in Mass Spectrometry: Mass Spectrometry/Mass Spectrometry," University of North Carolina, Asheville, NC, (ORAU Traveling Lecture Program), April 8, 1987 (invited).

Glish, G. L., "A New Dimension in Mass Spectrometry: Mass Spectrometry/Mass Spectrometry," Chemistry Department, University of Puerto Rico, Mayaguez, April 30, 1987 (invited).

Glish, G. L.; McLuckey, S. A., "Ion Trap Mass Spectrometry in the United States Space Program," Finnigan MAT Users' Forum, Denver, CO, May 23, 1987 (invited).

Glish, G. L.; McLuckey, S. A., "Ion Trap Testing at Oak Ridge," Martin Marietta Space Station Contamination Monitoring Workshop, Denver, CO, May 24, 1987 (invited).

McLuckey, S. A.; Glish, G. L., "A New Atmosphere Sampling Ion Source," 35th American Society of Mass Spectrometry Conference, Denver, CO, May 24-29, 1987.

- Glish, G. L. Glish, G. L., "The Modern Mass Spectrometer: A Gas Phase Chemical Laboratory," ORNL Summer Symposium Series, ORNL, Oak Ridge, TN, August 11, 1987 (invited).
- Glish, G. L.; McLuckey, S. A., "Use of an Ion Trap for Mass Spectrometry/Mass Spectrometry," 30th Analytical Chemistry in Energy Technology Conference, Knoxville, TN, October 20-22, 1987.
- Glish, G. L., "A New Dimension in Mass Spectrometry: Mass Spectrometry/Mass Spectrometry," Chemistry Department, Western Kentucky University, Bowling Green, KY, October 2, 1987 (invited).
- Glish, G. L.; McLuckey, S. A.; Asano, K. G., "Environmental Monitoring Using a New Atmospheric Sampling Ion Source and MS/MS," 39th Southeast Regional Meeting of the American Chemical Society, Orlando, FL, November 4, 1987 (invited).
- Goeringer, D. E. Goeringer, D. E.; Christie, W. H.; Valiga, R. E., "Matrix Effects in Sputter Atomization/Resonance Ionization Mass Spectrometry of Uranium," 35th American Society of Mass Spectrometry Conference, Denver, CO, May 24-29, 1987.
- Goeringer, D. E.; Christie, W. H., "Sputter Atomization Resonance Ionization Mass Spectrometry," DOE-BES Chemical Sciences Site Review, Oak Ridge National Laboratory, Oak Ridge, TN, March 30-31, 1987.
- Goeringer, D. E.; Christie, W. H., "Resonance Ionization Mass Spectrometry Using Ion-Beam Sampling," Southwest Optics '87 Conference on Lasers in Materials Diagnostics, Albuquerque, NM, February 11-12, 1987.
- Griest, W. H. Griest, W. H.; Jenkins, R. A.; Ilgner, R. H.; Moneyhun, J. H.; Tomkins, B. A.; Gayle, T. M.;* Higgins, C. E.; Guerin, M. R., "Field Sampling and Analysis of Shale Oil-Derived Airborne Diesel Exhausts," U.S. Army Biomedical Research and Development Laboratory Contractors' Review Meeting, Fort Detrick, Frederick, MD, September 15-16, 1987.

- Griest, W. H. Griest, W. H.; Tomkins, B. A.; Ilgner, R. H.; Witschi, H. P.;* Smith, L. H.;* Higgins, C. E.; Guerin, M. R., "Synthetic and Alternate Fuels Characterization," U.S. Army Biomedical Research and Development Laboratory Contractors' Meeting, Fort Detrick, Frederick, MD, September 15-16, 1987.
- Griest, W. H.; Row, K. H.; Maskarinec, M. P., "Evaluation of Capillary Electrokinetic Separations of Normal and Modified Nucleic Acid Constituents," U.S. Department of Energy, Office of Health and Environmental Research Site Review, Oak Ridge National Laboratory, Oak Ridge, TN, October 15-16, 1987.
- Griest, W. H.; Row, K. H.; Ramsey, R. S.; Stemmler, E. A.; Buchanan, M. V., "Separation and Detection of Dideoxyribonucleosides," U.S. Department of Energy, Office of Health and Environmental Research Site Review, Oak Ridge National Laboratory, Oak Ridge, TN, October 15-16, 1987.
- Griest, W. H.; Row, K. H.; Maskarinec, M. P., "Evaluation of Micellar Electrokinetic Capillary Chromatography for the Separation and Detection of Normal and Modified Deoxyribonucleosides and Deoxyribonucleotides," 5th Symposium on Separation Science and Technology for Energy Applications, Knoxville, TN, October 26-29, 1987.
- Griest, W. H., "Electrochromatography of Biologically Important Compounds," 26th Annual Conference on the Practice of Chromatography, ASTM Committee E-19, Minneapolis, MN, October 6, 1987 (invited).
- Griest, W. H.; Row, K. H.; Maskarinec, M. P., "Separation of Modified Deoxyribonucleosides and Nucleotides by Micellar Electrokinetic Capillary Chromatography," 17th Ohio Valley Chromatography Symposium, Hueston Woods State Park, OH, June 24-26, 1987.
- Guerin, M. R. Guerin, M. R.; Ho, C.-h.; Buchanan, M. V., "Identification and Quantitation of Environmental Aryl Amines," 3rd International Conference on Carcinogenic and Mutagenic n-Substituted Aryl Compounds," Dearborn, MI, April 25-28, 1987 (invited).

- Guerin, M. R. Guerin, M. R.; Buchanan, M. V.; Wise, M. B.; Hettich, "Isomeric Identification and Selective Detection of Biologically Significant Organics," DOE Contractor's Meeting on Chemical Toxicity, Monterey, CA, June 23-26, 1987 (invited).
- Guiochon, G. A. Guiochon, G. A.; Katti, A. M., "Preparative Scale Liquid Chromatography," HPLC '87, Xith Symposium on Column Liquid Chromatography, Amsterdam, The Netherlands, June 28-July 3, 1987.
- Guiochon, G. A., "Simulation of the Elution and Separation of Overlapping Bands on Overloaded Liquid Chromatographic Columns," Eastern Analytical Symposium, New York, NY, September 13, 1987.
- Guiochon, G. A.; Ghodbane, S., "Theoretical Study of the Separation of Two Compounds in Non-Linear Chromatography," 5th Symposium on Separation Science and Technology Meeting, Knoxville, TN, October 26-29, 1987.
- Guiochon, G. A., "Preparative Scale Liquid Chromatography," Department of Chemical Engineering Seminar, University of Tennessee, Knoxville, TN, October 29, 1987 (invited).
- Guiochon, G. A., "Adsorption and Non-Linear Chromatography," Ecole Polytechnique Federale de Lausanne, Lausanne, Switzerland, October 9, 1987.
- Guiochon, G. A., "The Ideal Model in Non-Linear Chromatography," 5th International Symposium on the Separation of Peptides, Proteins, and Polynucleotides, Washington, DC, November 4, 1987.
- Guiochon, G. A., "Column Overloading in Chromatography," Plenary Lecture Expoquimia, Spain, November 9, 1987 (invited).
- Haas, J. W. Haas, J. W.; Buchanan, M. V.; Wise, M. B., "Characterization of Complex Mixtures Using a Multimode Ionization Gas Chromatographic Detector," 193rd National Meeting of the American Chemical Society, Denver, CO, April 5-10, 1987.

- Hettich, R. L. Hettich, R. L.; Buchanan, M. V., "The Identification and Characterization of Nucleotides by Laser Ionization Fourier Transform Mass Spectrometry," 35th American Society of Mass Spectrometry Conference, Denver, CO, May 24-29, 1987.
- Hettich, R. L., "Laser Ionization Fourier Transform Mass Spectrometry for the Characterization of Normal and Modified Nucleic Acid Constituents," U.S. Department of Energy, Office of Health and Environmental Research Site Review, Oak Ridge National Laboratory, Oak Ridge, TN, October 15-16, 1987.
- Hettich, R. L., "Examination of Glycerol Precursors for Platelet Activating Factor," U.S. Department of Energy, Office of Health and Environmental Research Site Review, Oak Ridge National Laboratory, Oak Ridge, TN, October 15-16, 1987.
- Hettich, R. L., "Characterization of Nucleotides and Oligonucleotides by Laser Ionization Fourier Transform Mass Spectrometry," 30th Analytical Chemistry in Energy Technology Conference, Knoxville, TN, October 20-22, 1987.
- Hettich, R. L.; Buchanan, M. V., "Combining Laser Technology with Fourier Transform Mass Spectrometry," U.S. Department of Energy Workshop, Advanced Laser Technology for Chemical Measurements, Gaithersburg, MD, November 4-6, 1987.
- Hettich, R. L.; Buchanan, M. V., "Laser Ionization Fourier Transform Mass Spectrometry for the Characterization of Nucleotides and Oligonucleotides," American Society of Mass Spectrometry Workshop on Mass Spectrometric Approaches to the Analysis of Biopolymers, Boston, MA, October 29-30, 1987.
- Higgins, C. E. Higgins, C. E.; Thompson, C. V.; Ilgner, R. H.; Jenkins, R. A.; Guerin, M. R., "Multicomponent Environmental Tobacco Smoke Analysis Using Triple Sorbent Traps and Thermal Desorption Gas Chromatography," 41st Tobacco Chemists' Research Conference, Greensboro, NC, October 5-7, 1987.

- Higgins, C. E. Jenkins, R. A.; Higgins, C. V.; Treese, C. A.; Guerin, M. R., "Organic Vapor Phase Composition of Sidestream and Environmental Tobacco Smoke from Cigarettes," 1987 EPA/APCA Symposium on Measurement of Toxic and Related Air Pollutants, Raleigh, NC, May 3-6, 1987.
- Ho, C.-h. Ho, C.-h.; Budhram, R.,* "Synthesis and Chemistry of Adducted Nucleic Acid Constituents," U.S. Department of Energy, Office of Health and Environmental Research Site Review, Oak Ridge National Laboratory, Oak Ridge, TN, October 15-16, 1987.
- Howell, P. L. Howell, P. L., "Evaluation of ISO/DIS 8690 Method for Decontamination of Radioactively Contaminated Surfaces," ASTM D-33 Meeting, Las Vegas, NV, April 27, 1987.
- Hulett, L. D., Jr. Hulett, L. D., Jr.; Dale, J. M.; Pendyala S.,* "A New Positron Source" DOE-BES Chemical Sciences Site Review, Oak Ridge National Laboratory, Oak Ridge, TN, March 30-31, 1987.
- Hulett, L. D., Jr.; Lewis, T. A.;* Pendyala, S.,* "Progress in the Construction of a Linac-Based Slow Positron Source," International Symposium on Positron Annihilation Studies of Fluids, University of Texas, Arlington, TX, June 8-12, 1987
- Hulett, L. D., Jr.; Pendyala, S.,* "Positron Spectroscopy Studies of Oxide Superconductors," Fall Meeting of the Metallurgical Society, Cincinnati, OH, October 12-14, 1987.
- Hulett, L. D., Jr.; Lewis, T. A.;* Pendyala, S.,* "Status of the High Intensity Slow Positron Source at ORNL," Workshop on Intense Positron Beams, Idaho National Engineering Laboratory, Idaho Falls, June 18-19, 1987.
- Hulett, L. D., Jr.; Pendyala, S., "The Feasibility of a Positron Microscope," Workshop on Intense Positron Beams, Idaho National Engineering Laboratory, Idaho Falls, ID, June 18-19, 1987.

- Jenkins, R. A. Jenkins, R. A.; Gayle, T. M.,* "Human Smoking Dosimeter for Behavioral Studies," Interagency Forum on Smoking and Health, Washington, DC, March 10, 1987 (invited).
- Jenkins, R. A.; Gayle, T. M.;* Guerin, M. R., "Chemical Characterization and Toxicological Evaluation of Airborne Mixtures: Analysis of M36 Propellants," U.S. Army Biomedical Research and Development Laboratory Contractors' Review Meeting, Fort Detrick, Frederick, MD, September 15-16, 1987.
- Jenkins, R. A.; Moneyhun, J. H.; Guerin, M. R., "Chemical Characterization and Toxicological Evaluation of Airborne Mixtures - Smoke Generation and Characterization. Part 1. Liquid/Solid Aerosol Generator," U.S. Army Biomedical Research and Development Laboratory Contractors' Review Meeting, Fort Detrick, Frederick, MD, September 15-16, 1987.
- Jenkins, R. A.; Lore, J. D.;* Moneyhun, J. H.; Rosee, R. H.,* "Problem Definition Study for Evaluating the Chemical and Toxicological Properties of the Combustion Products of Rifle and Gun Systems," U.S. Army Biomedical Research and Development Laboratory Contractors' Review Meeting, Fort Detrick, Frederick, MD, September 15-16, 1987.
- Jones, R. E. Jones, R. E.; Howell, P. L.; Peele, J. M., "An Evaluation of ISO/DIS Method 8690, Decontamination of Radioactively Contaminated Surfaces," ASTM D-33 Committee Meeting, Cocoa Beach, FL, October 27, 1987.
- Klatt, L. N. Klatt, L. N., "Real Time In-Situ Monitor for Absorption Heat Pump Systems," Analytical Chemistry Division Information Meeting, Oak Ridge National Laboratory, Oak Ridge, TN, February 18-19, 1987.
- Klatt, L. N.; Bostick, D. T.; Perez-Blanco, H.,* "Fiber Optic Refractometer for the Determination of Liquid Film Composition," 14th Annual Meeting of the Federation of Analytical Chemists and Spectroscopy Societies, Detroit, MI, October 4-9, 1987.
- Klatt, L. N. "Instrumentation," Savannah River Laboratory, Aiken, SC, December 18, 1987 (invited).

- Laing, W. R. Laing, W. R., "Quality Assurance - A Benefit or a Burden?" Pittsburgh Conference & Exposition on Analytical Chemistry & Applied Spectroscopy, Pittsburgh, PA, March 9-13, 1987.
- Laing, W. R., "DOE Environmental Site Survey Program," Energy Systems' 5-Plant Analytical Chemistry Forum, July 14, 1987.
- Leibman, C. P. Leibman, C. P.; Todd, P. J.; Mamantov, G.,* "SIMS and Chemistry of Substituted Aromatic/Sulfuric Acid Solutions," 35th American Society for Mass Spectrometry, Denver, CO, May 24-29, 1987.
- Leibman, C. P.; Todd, P. J.; Mamantov, G.,* "Organic Secondary Ion Emission from Substituted Aromatic/Concentrated Sulfuric Acid Solutions," Sixth International Conference on Secondary Ion Mass Spectrometry, Versailles, France, September 13-18, 1987.
- Lyon, W. S. Lyon, W. S., "Some Views of the Future from the Past," 30th Analytical Chemistry in Energy Technology Conference, Knoxville, TN, October 20-22, 1987.
- Lyon, W. S., "Neutron Activation Analysis as a Science Integrator," 30th Conference on Analytical Chemistry in Energy Technology, Knoxville, TN, October 20-22, 1987.
- Maskarinec, M. P. Maskarinec, M. P.; Goodin, J. E.; Moody, R. L., "A Data Base for Establishment of Preanalytical Holding Times," 3rd Annual Solid Waste Testing and Quality Assurance Symposium, Washington, DC, July 14-17, 1987.
- Merriweather, R.; Caldwell, W. M.; Maskarinec, M. P.; Caton, J. E., "The Determination of Chlorophenoxy Acid Herbicides by Liquid Chromatography Using Carbon-14 Tracers," 3rd Annual Solid Waste Testing and Quality Assurance Symposium, Washington, DC, July 14-17, 1987.
- Francis, C. W.;* Maskarinec, M. P., "Comparisons of Laboratory Waste Extracts with Field Leachates," 194th National Meeting of the American Chemical Society, New Orleans, LA, August 31-September 4, 1987.

- Maskarinec, M. P. Maskarinec, M. P.; Francis, C. W.,* "Problems in the Analysis of Solid Waste Extracts Derived from Leaching Tests," 194th National Meeting of the American Chemical Society, New Orleans, LA, August 31-September 4, 1987.
- Maskarinec, M. P.; Holladay, S. K.; Goodin, J. E.; Moody, R. L., "A Data Base for Establishment of Preanalytical Holding Times," EPA/CLP Organics Conference, New Orleans, LA, October 13-16, 1987.
- Maskarinec, M. P.; Moody, R. L.; "Storage and Preservation of Environmental Samples," 193rd National Meeting of the American Chemical Society, Denver, CO, April 4-8, 1987.
- May, M. P. May, M. P.; Carter, J. A.; Walker, R. L.; Smith, D. H., "Tank Calibration and Unknown Volume Determination by Isotope Dilution Mass Spectrometry," 30th Analytical Chemistry in Energy Technology Conference, Knoxville, TN, October 20-22, 1987.
- McDaniel, F. D.* Andrews, M. C.;* Duggan, J. L.;* Miller, P. D.;* Pepmiller, P. L.;* McDaniel, F. D.;* Krause, H. F.;* Rosseel, T. M.; Rayburn, L. A.;* Mehta, R.;* Lapicki, G.,* "L- and M-Shell Direct Ionization and Electron Capture for Highly Charged Carbon and Oxygen Ions," International Symposium on Ion Atom Collisions X, Bad Soden, Federal Republic of Germany, July 29-31, 1987.
- McLuckey, S. A. McLuckey, S. A.; Glish, G. L.; Kelley, P. E.,* "Collision Activated Decomposition of Negative Ions in an Ion Trap Mass Spectrometer," 35th American Society of Mass Spectrometry Conference, Denver, CO, May 24-29, 1987.
- McLuckey, S. A.; Glish, G. L.; DiDonato, G. C.;* Busch, K. L.,* "Effects of Site of Protonation on Low and High Energy Daughter Ion MS/MS Spectra of Phenothiazines," 35th Annual American Society of Mass Spectrometry Conference, Denver, CO, May 24-29, 1987.

- McLuckey, S. A. McLuckey, S. A.; Glish, G. L.; McKown, H. S., "Detection and Identification of Organic Explosives in Ambient Air," Analytical Chemistry Division Information Meeting, Oak Ridge National Laboratory, Oak Ridge, TN, February 18-19, 1987.
- McLuckey, S. A.; Glish, G. L.; McKown, H. S.; Asano, K. G., "Trace Analysis of Organics in Ambient Air," International Symposium on Applied Mass Spectrometry in the Health Sciences, Barcelona, Spain, September 28-30, 1987.
- McLuckey, S. A.; Glish, G. L., "High Sensitivity and High Specificity in Trace Analysis of Organics," University of Gent, Gent, Belgium, October 2, 1987.
- McLuckey, S. A.; Glish, F. L., "Fundamental and Practical Applications of MC/MS in Organic Mass Spectrometry," F.O.M. Institute for Atomic and Molecular Physics, Amsterdam, The Netherlands, October 6, 1987.
- McLuckey, S. A.; Glish, G. L.; McKown, H. S.; Asano, K. G., "Status of Explosives Detection at ORNL," FBI Conference on Explosives Detection, Quantico, VA, October 14, 1987.
- Mueller, T. R. Mueller, T. R.; Walker, R. L., "Isotopic Determination of Calcium by Thermal Ionization Using a VG 354E Mass Spectrometer," 35th Annual American Society of Mass Spectrometry Conference, Denver, May 24-29, 1987.
- Nolan, T. G. Nolan, T. G.; Ramsey, J. M.; Whitten, W. B., "Refractive Index Detector Based on Two-Wave Mixing in Photorefractive Materials," XXV Colloquium Spectroscopicum Internationale, Toronto, Ontario, Canada, June 21-26, 1987.
- Ramsey, J. M. Ramsey, J. M.; Whitten, W. B., "Spectrochemical Analysis Using Degenerate Four-Wave Mixing," Topical Meeting on Laser Applications to Chemical Analysis, Incline Village, NV, January 26-29, 1987.

Ramsey, J. M.

Nolan, T. G.; Ramsey, J. M.; Whitten, W. B., "Refractive Index Detector Based on Two-Wave Mixing in Photorefractive Materials," Topical Meeting on Laser Applications to Chemical Analysis, Incline Village, NV, January 26-29, 1987.

Ramsey, J. M.; Whitten, W. B., "Control of CW Dye Lasers Using Photorefractive Optical Elements," Analytical Chemistry Division Information Meeting, Oak Ridge National Laboratory, Oak Ridge, TN, February 18-19, 1987.

Ramsey, J. M.; Whitten, W. B.; Nolan, T. G., "Degenerate Four-Wave Mixing in an Analytical Flame," 1987 Pittsburgh Conference and Exposition on Analytical Chemistry, Atlantic City, NJ, March 10, 1987.

Ramsey, J. M.; Whitten, W. B.; Nolan, T. G., "Laser Spectroscopy" DOE-BES Chemical Sciences Site Review, Oak Ridge National Laboratory, Oak Ridge, TN, March 30-31, 1987.

Ramsey, J. M.; Whitten, W. B., "Laser Spectroscopy by Degenerate Four-Wave Mixing," 193rd National Meeting of the American Chemical Society, Denver, CO, April 7, 1987 (invited).

Ramsey, J. M., "Data Processing I: Correlation," 193rd National Meeting of the American Chemical Society, Denver, CO, April 7, 1987 (invited).

Ramsey, J. M., "Spectrochemical Analysis Using Laser Induced Gratings," Department of Chemistry, Bowling Green State University, Bowling Green, OH, April 24, 1987 (invited).

Ramsey, J. M.; Whitten, W. B., "Analytical Laser Spectroscopy Using Optical Phase Conjugation," XXV Colloquium Spectroscopicum Internationale, Toronto, Ontario, Canada, June 21-26, 1987 (invited).

Ramsey, J. M.; Whitten, W. B.; Nolan, T. G. "Applications of Laser-Induced Gratings to Chemical Measurements," 1987 Federation of Analytical Chemistry and Spectroscopy Society, Detroit, MI, October 4, 1987.

- Ramsey, J. M. Ramsey, J. M.; Whitten, W. B.; Nolan, T. G., Chang, C. C.,* "Laser-Based Measurements Using Photorefractive Optical Elements," Advanced Laser Technology for Chemical Measurements Workshop, Gaithersburg, MD, November 4-6, 1987.
- Ramsey, R. S. Ramsey, "A New Helium Discharge Ionization Detector for Gas Chromatography," 5th Symposium on Separation Science and Technology for Energy Applications," Knoxville, TN, October 26-30, 1987.
- Ramsey, R. S.; Asano, K. G.; Glish, G. L.; McLuckey, S. A., "Evaluation of Glow Discharge Ionization Source for LC/MS," U.S. Department of Energy, Office of Health and Environmental Research Site Review, Oak Ridge National Laboratory, Oak Ridge, TN, October 15-16, 1987.
- Ramsey, R. S., "Retention Modification of Nucleic Acid Constituents and Modified Analogs in Reverse Phase Liquid Chromatography," U.S. Department of Energy, Office of Health and Environmental Research Site Review, Oak Ridge National Laboratory, Oak Ridge, TN, October 15-16, 1987.
- Robinson, L. Robinson, L.; Dyer, F. F.; Hobbs, R.,* "An Automated Fuel Scanning System," 194th National Meeting of the American Chemical Society, New Orleans, LA, August 30-September 4, 1987.
- Ross, H. H. Ross, H. H., "Measurement of Radionuclides in the Environment via Cherenkov Radiation," 193rd National Meeting of the American Chemical Society, Denver, CO, April 5-10, 1987.
- Rucker, T. L.; Ross, H. H., "Computer Modeling of Fiber-Scintillator Flow-Cell Radiation Detector Geometry," 1987 Winter Meeting of the American Nuclear Society, Los Angeles, CA, November 15-19, 1987.
- Rosseel, T. M. Rosseel, T. M.; Dale, J. M.; Hulett, L. D.; Pepmiller, P. L.;* Vane, C. R.;* Walkiewicz, T. A.;* Young, J. P., "High Resolution Studies of the Chemical Effects in HIXSE Spectra," Analytical Chemistry Division Information Meeting, Oak Ridge National Laboratory, Oak Ridge, TN, February 18-19, 1987.

- Rosseel, T. M. Rosseel, T. M.; Young, J. P.; Dale, J. M., "Heavy-Ion Induced X-ray Satellite Emission," DOE-BES Chemical Sciences Site Review, Oak Ridge National Laboratory, Oak Ridge, TN, March 30-31, 1987.
- Rosseel, T. M.; Vane, C. R.;* Young, J. P., "High Resolution Studies of Chemical Effects in HIXSE Spectra," Federation of Analytical Chemistry and Spectroscopy Societies, Detroit, MI, October 4-9, 1987.
- Rucker, T. L. Rucker, T. L., "Scintillator-Fiber Flow-Cell Radiation Detector for Liquid Chromatography," Department of Chemistry, University of Tennessee, Knoxville, TN, April 24, 1987.
- Rucker, T. L.; Ross, H. H.; Schweitzer, G. K.,* "Scintillator-Fiber Flow-Cell Radiation Detector for Liquid Chromatography, 23rd Annual Conference on Bioassay, Analytical and Environmental Radiochemistry, Berkeley, CA, October 6-8, 1987.
- Shaw, R. W. Shaw, R. W.; Young, J. P.; Smith D. H., "Application of a High Repetition Rate Copper Vapor Laser to Uranium Resonance Ionization Mass Spectroscopy," Analytical Chemistry Division Information Meeting, Oak Ridge National Laboratory, Oak Ridge, TN, February 18-19, 1987.
- Shaw, R. W.; Young, J. P.; Smith, D. H., "Removal of the Sm/Pm Isobaric Interference by Resonance Ionization Mass Spectrometry," 1987 International Laser Science Conference, Atlantic City, NJ, November 1-5, 1987.
- Smith, D. H. Smith, D. H.; Walker, R. L.; Mueller, T. R., "High Precision Isotopic Analysis," DOE-BES Chemical Sciences Site Review, Oak Ridge National Laboratory, Oak Ridge, TN, March 30-31, 1987.
- Stemmler, E. A. Stemmler, E. A.; Buchanan, M. V., "Oxidation of Polycyclic Aromatic Hydrocarbons and Their Methyl Derivatives by Reactions Occurring in the Chemical Ionization Source," 30th Conference on Analytical Chemistry in Energy Technology, Knoxville, TN, October 20-22, 1987.

- Stemmler, E. A. Stemmler, E. A.; Buchanan, M. V., "Gas Phase and Surface-Catalyzed Reactions of Polycyclic Aromatics with Oxygen," U.S. Department of Energy, Office of Health and Environmental Research Site Review, Oak Ridge National Laboratory, Oak Ridge, TN, October 15, 16, 1987.
- Stewart, J. H. Stewart, J. H., Holmes, R. L., Rogers, W. F., "Evaluation of a Rapid Quantitative Organic Extraction System for Determining the Concentration of PCBs in Soil," Energy Systems' 5-Plant Analytical Chemistry Forum, Paducah, KY, July 14, 1987.
- Stewart, J. H., Holmes, R. L., Rogers, W. F., "Evaluation of a Rapid Quantitative Organic Extraction System for Determining the Concentration of PCBs in Soil," 39th Southeast Regional Meeting of the American Chemical Society, Orlando, FL, Nov. 3-6, 1987.
- Thompson, C. V. Thompson, C. V.; Higgins, C. E.; Jenkins, R. A., "Comparison of Personal Monitoring Systems for Ambient Nicotine," 41st Tobacco Chemists' Research Conference, Greensboro, NC, October 5-7, 1987.
- Todd, P. J. Todd, P. J. "Surface Concentration in Liquid SIMS," 35th Annual American Society for Mass Spectrometry, Denver, CO, May 24-29, 1987
- Todd, P. J.; Leibman, C. P.; Mamantov, G.,* "Organic SIMS," Analytical Chemistry Division Information Meeting, Oak Ridge National Laboratory, Oak Ridge, TN, February 18-19, 1987
- Todd, P. J., "Organic SIMS," DOE-BES Chemical Sciences Site Review, Oak Ridge National Laboratory, Oak Ridge, TN, March 30-31, 1987.
- Todd, P. J., "Liquid Secondary Ion Mass Spectrometry of Adsorbed Volatile Amines," Sixth International Conference on Secondary Ion Mass Spectrometry, (SIMS VI), Paris, France, September 13-18, 1987.
- Todd, P. J., "Quantitative Measurements in Liquid SIMS," University College of Swansea, Wales, UK, September 25, 1987 (invited).
- Todd, P. J., "Functional Relationships in Liquid SIMS," Idaho National Engineering Laboratory, Idaho Falls, ID, October 13, 1987 (invited).

- Tomkins, B. A. Tomkins, B. A.; Caton, J. E., "Preparation of Radioactive 'Mixed' Waste Samples for Measurement of RCRA Organic Compounds," 3rd Annual Solid Waste Testing and Quality Assurance Symposium, Washington, DC, July 14-17, 1987.
- Van Berkel, G. J. Van Berkel, G. J.; Glish, G. L.; McLuckey, S. A., "Geoporphyrin Structure Determination Using MS/MS," 194th National Meeting of the American Chemical Society, New Orleans, LA, August 30-September 4, 1987.
- Walker, R. L. Walker, R. L.; Mueller, T. R.; Smith, D. H. "Inorganic Mass Spectrometry," Analytical Chemistry Division Information Meeting, Oak Ridge National Laboratory, Oak Ridge, TN, February 18-19, 1987.
- Walker, R. L. "High Sensitivity Isotopic Analyses," DOE-BES Chemical Sciences Site Review, Oak Ridge National Laboratory, Oak Ridge, TN, March 30-31, 1987.
- Whitten, W. B. Whitten, W. B.; Ramsey, J. M., "CW Dye Laser with a Photorefractive Intracavity Mode Selector and Frequency Stabilizer," Topical Meeting on Laser Applications to Chemical Analysis, Incline Village, NV, January 26-29, 1987.
- Whitten, W. B., "Low-Pressure Laser Spectroscopy with Flame Atomization," Analytical Chemistry Information Meeting, Oak Ridge National Laboratory, Oak Ridge, TN, February 18-19, 1987.
- Whitten, W. B.; Koutny, L. B.;* Nolan, T. G.; Ramsey, J. M., "High Resolution Spectroscopy with Flame Atomization," 14th Annual Meeting of the Federation of Analytical Chemists Spectroscopy Societies, Detroit, MI, October 4-9, 1987.
- Whitten, W. B.; Ramsey, J. M.; Nolan, T. G.; Koutny, L. B.,* "High Resolution Atomic Spectroscopy," Advanced Laser Technology for Chemical Measurements Workshop, Gaithersburg, MD, November 4-6, 1987.

- Whitten, W. B. Whitten, W. B.; Ramsey, J. M., "Clinical Applicants of Microwave Spectroscopy/Isotope Ratios," Review of Office of Health and Environmental Research/Nuclear Medicine Research, Bethesda, MD, November 4, 1987.
- Wilmarth, W. R. Wilmarth, W. R.*; Begun, G. M.*; Haire, R. G.*; Young, J. P.; Peterson, J. R.* "Raman and Absorption Spectrophotometric Studies of the Trihalides of Promethium," 39th SE Regional American Chemical Society Meeting, Orlando, FL, November 4-6, 1987.
- Wise, M. B. Wise, M. B.; Buchanan, M. V., "Dual Cell FTMS - A Powerful Tool for Complex Mixture Analysis," 1987 Pittsburgh Conference and Exposition on Analytical Chemistry, Atlantic City, NJ, March 11, 1987 (invited).
- Wise, M. B., "Characterization of Constituents in Tobacco Smoke Using Fourier Transform Mass Spectrometry," 35th American Society for Mass Spectrometry Conference, Denver, CO, May 24-29, 1987.
- Wise, M. B.; Buchanan, M. V.; Guerin, M. R., "New Mass Spectrometric and Ionization Detection Technologies for Trace Organics," Chemical Research Development and Engineering Center Briefing, Edgewood, MD, September 23, 1987.
- Wise, M. B., "Glow Discharge Mass Spectrometry for Environmental Analysis," U.S. Department of Energy, Office of Health and Environmental Research Site Review, Oak Ridge National Laboratory, Oak Ridge, TN, October 15-16, 1987.
- Wise, M. B.; Buchanan, M. V.; Hettich, R. L., "Fourier Transform Mass Spectrometry (FTMS) at ORNL," U.S. Department of Energy, Office of Health and Environmental Research Site Review, Oak Ridge National Laboratory, Oak Ridge, TN, October 15-16, 1987.
- Wise, M. B.; Guerin, M. R., "Characterization of Higher Molecular Weight Constituents in Tobacco Smoke Tars Using Fourier Transform Mass Spectrometry," 41st Tobacco Chemists' Research Conference, Greensboro, NC, October 4-7, 1987.

- Young, J. P. Young, J. P., "Spectrophotometric Studies Applied to Transplutonium Chemistry," Analytical Chemistry Division Information Meeting, Oak Ridge National Laboratory, Oak Ridge, TN, February 18-19, 1987.
- Young, J. P.; Shaw, R. W.; Smith, D. H., "Resonance Ionization Mass Spectrometry," DOE-BES Chemical Sciences Site Review, Oak Ridge National Laboratory, Oak Ridge, TN, March 30-31, 1987.
- Young, J. P.; Haire, R. G.;* Peterson, J. R.,* "Spectrophotometric Study of Thermally Induced Structural Transformations of CfCl₃," 194th National Meeting of the American Chemical Society, New Orleans, LA, August 30-September 4, 1987.
- Young, J. P.; Shaw, R. W.; Smith, D. H., "Studies of Resonance Ionization Mass Spectrometry at Oak Ridge National Laboratory," 14th Annual Meeting of the Federation of Analytical Chemists Spectroscopy Society, Detroit, MI, October 4-9, 1987.
- Young, J. P.; Peterson, J. R.;* Haire, R. G.;* Wilmarth, W. R.,* "Spectrophotometric Studies of Microgram Amounts of Transplutonium Compounds," 30th Analytical Chemistry in Energy Technology Conference, Knoxville, TN, October 20-22, 1987.

ARTICLES REVIEWED OR REFEREED FOR PERIODICALS

	Anal. Chem.	Anal. Chim. Acta	Fuel	Health Phys.	Int. J. Appl. Radia. & Isot.	Int. J. Mass Spectrom.	Radiochim. Acta	Optics Lett.	Proposals	Other	Total
Buchanan, M. V.	5		3						17	1	26
Carter, J. A.	2								1		3
Caton, J. E.									1		1
Christie, W. H.									1		1
Dyer, F. F.		1									1
Eldridge, J. S.				3							3
Feldman, C.	4								2	1	7
Glish, G. L.	2					2			2	1	7
Goeringer, D. E.		1							1		2
Griest, W. H.	1								4	1	6
Guerin, M. R.									2	1	3
Hettich, R. L.										1	1
Ho, C.-h.									1		1
Hulett, L. D.									2		2
Jenkins, R. A.	1										1
Lyon, W. S.		1								3	4
Maskarinec, M. P.	1								1	2	4
McLuckey, S. A.									2	1	3
Ramsey, J. M.								2	7		9
Ramsey, R. S.	4								1		5
Robinson, L.							2				2
Ross, H. H.	1				2				1		4
Rosseel, T. M.									1	1	2
Shaw, R. W.									7	1	8
Smith, D. H.									3	3	6
Stemmler, E. A.	1										1
Todd, P. J.	1								3	1	5
Tomkins, B. A.	7									3	10
Walker, R. L.									1		1
Whitten, W. B.									1		1
Wise, M. B.									1		1
Young, J. P.	1							1	3		5
Total	31	3	3	3	4	2	3	3	66	18	136

SUMMARY OF ANALYTICAL WORK

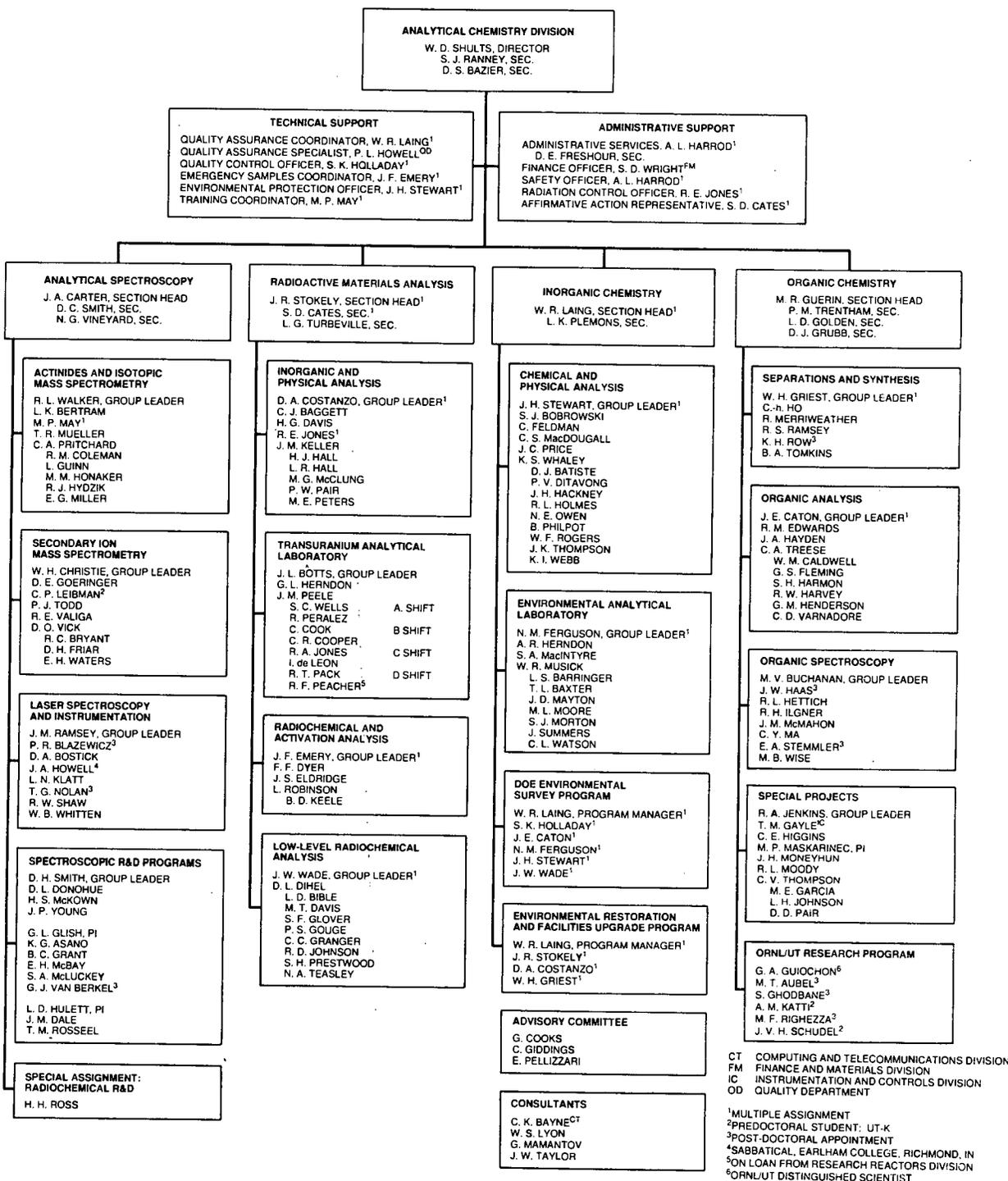
Organization	Number of results reported by				Total
	Analytical Spectroscopy	Radioactive Materials Analysis	Organic Chemistry	Inorganic Chemistry	
ORNL Unit					
Analytical Chemistry	345	4,032	1,304	1,609	7,290
Biology	174		635		809
Central Management		729		757	1,486
Chemical Technology	3,896	20,326	5,756	8,956	38,934
Chemistry	251	40		853	1,144
Energy		274	6,112	2,612	8,998
Engineering Physics	75	34		225	334
Engineering Technology	1,143	34	635	353	2,165
Environmental Compliance and Health Protection	126	9,792	12,089	10,731	32,738
Environmental Sciences		2,496	12,104	114,949	129,549
Fuel Recycle	1,185	10,850		368	12,403
Fusion Energy	320	113		339	772
Health and Safety Research	168	8,110	392	1,814	10,484
Metals and Ceramics	3,962	214		1,423	5,599
Operations	9,721	47,847	13,024	2,045	72,637
Physics		202			202
Plant and Equipment	164	152		453	769
Research Reactor		434		326	760
Solid State	237		24	124	385
Others					
DOE Environmental Survey Program		2,424	77,072	12,350	91,846
K-25 and Y-12	3,272	554	860		4,686
Miscellaneous	1,208	393		713	2,314
Nuclear & Engineering Tech.				7,262	7,262
ORAU			189		189
Savannah River Laboratory	487				487
Work for Others	<u>1,439</u>	<u>30</u>			<u>1,469</u>
TOTAL	28,173	109,080	130,196	168,262	435,711

DIVISIONAL MANPOWER AND FINANCIAL SUMMARY
FY 1987

Source	\$K	PY ^a
DOE programs		
Energy Research		
Basic Energy Sciences	1,724	11.0
Health and Environmental Research	563	3.3
Environmental Site Survey	706	4.9
Safeguards and Security	605	4.3
Nuclear Energy	193	1.3
Fossil Energy	71	0.5
Miscellaneous	<u>515</u>	<u>3.5</u>
Total DOE Programs	4,377	28.9
Work for others - federal agencies		
Department of Defense	1,285	7.9
National Cancer Institute	309	2.4
Environmental Protection Agency	211	1.5
Bureau of Engraving and Printing	205	1.4
Federal Aviation Administration	143	0.9
National Institute on Drug Abuse	82	0.3
Miscellaneous	<u>173</u>	<u>1.1</u>
Total WFO - federal agencies	2,407	15.5
Work for others - nonfederal agencies		
Council for Tobacco Research	318	2.6
Protective Coating Companies	45	0.0
Uranium Ore Testing Companies	20	0.0
Miscellaneous	<u>64</u>	<u>0.4</u>
Total WFO - nonfederal agencies	447	3.0
Support/Services		
ORNL Divisions/Programs	5,061	68.3
Other Clients	<u>1,802</u>	<u>24.3</u>
Total Support/Services	<u>6,863^b</u>	<u>92.6</u>
TOTAL FINANCIAL PLANS	14,094	140.0

^aPerson years

^bDoes not reflect Laboratory general and administrative expense or general plant services.



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38.	B. L. Burks	79.	E. H. Krieg	139.	W. B. Whitten
9-41.	J. A. Carter	80.	W. R. Laing	140.	R. E. Valiga
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