ORNL-6547
Distribution Category UC-4

Contract No. DE-AC05-840R21400

ORNL--6547

DE89 013736

ANALYTICAL CHEMISTRY DIVISION ANNUAL PROGRESS REPORT

For Period Ending December 31, 1988

W. D. Shults, Director

Date Published - May 1988

OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee 37831
operated by
MARTIN MARIETTA ENERGY SYSTEMS, INC.
for the
DEPARTMENT OF ENERGY

CONTENTS

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 of 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 19.8% of our effort in FY 1988.

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 constitutes about 25.1% of our work in FY 1988.

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. This type of work accounted for approximately 55.1% of our effort in FY 1988.

The Analytical Chemistry Division is organized into four major sections, each of which may carry out any of the three types of work mentioned above. Chapters 1 through 4 of this report highlight progress within the four sections during the period January 1 to December 31, 1988. A brief discussion of the division's role in an especially important environmental program 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 44 articles, 32 proceedings, and 2[illegible] reports were published, and 153 oral presentations were given during this reporting period. Some 405,542 determinations were performed.

HIGHLIGHTS

Characterization of the contents of nuclear wastes--liquids and sludges in storage tanks--has become a very important challenge at ORNL and other DOE sites. We devoted considerable effort to the modification of EPA SW-846 methodology so as to purge volatile organics and extract semivolatile organics from highly radioactive samples, and thence be able to determine the regulatory organic compounds in them. Surrogate and matrix spike recoveries have been acceptable and the separated materials can be analyzed in conventional organic analysis facilities. Concurrently, an inductively coupled plasma emission spectrometer and an atomic absorption spectrometer were installed in custom enclosures in our High Radiation Level Analytical Facility in order to provide capabilities for elemental analysis of radioactive materials. These systems have been used to characterize liquids and sludges from numerous storage tanks around the Laboratory, to classify the wastes as RCRA, TRU, or "mixed", and to evaluate options for disposal. Radiochemical analyses were also performed on these waste materials.

We have continued to study the detection of organic vapors (e.g. explosives) via atmospheric sampling glow discharge ionization (ASGDI) plus MS/MS analysis. Much interest has developed in this work, as indicated by the fact that the Finnigan Corporation was granted a license to market our ASGDI. Our research has turned to the use of ion trap mass spectrometry (ITMS) for this application. Ion traps are well suited for monitoring since they are relatively small and have good MS/MS efficiency. We were the first to show ion injection from a high pressure source directly into an ion trap. We were also first in performing laser desorption ionization in an ITMS.

We have developed methodology for the rapid identification and quantitation of chemical agents in air. The system consists of an automated thermal desorption unit with a 16-port autosampler, interfaced to a Finnigan ion trap mass spectrometer. The autosampler can be removed and loaded while a second batch of sixteen samples is processed. Less than 100 picograms of agent simulant can be detected, with 2-minute turnaround, with this system.

The rapid determination of trace organics in water and soil is very important for many programs and monitoring activities. We have found that volatile compounds at the 1-10 ppb level can be determined in about 2 minutes by purging them directly into a glow discharge ionization source and thence into a quadrupole mass spectrometer. Some semivolatiles can also be detected, but at higher detection limits. Moreover, scoping experiments have shown that volatiles can be sparged directly into an ITMS with helium and determined with reproducibilities of 10-20%, in 2 minutes. The ITMS has many operational advantages over the quadrupole for this exciting application.

Significant advances in the mass spectrometric analysis of geoporphyrins were made this year, using ITMS and sector instruments. Many features of the ITMS were evaluated and used in these studies, including selected-reagent-ion chemical ionization and MS^n experiments.

We have investigated the application of laser ionization Fourier transform mass spectrometry to characterization of DNA adducts. Methyl-substituted guanosines, thymidine, adenosine, and cytidine have been characterized. Deuterium labelling, sequential collisional dissociation (MS^n), and high resolution, accurate mass measurements were used in these studies. Laser ionization FTMS has also been used to analyze methyl guanosine isomers directly off of TLC plates, following separation.

Work on the differentiation of isomeric compounds by negative ion mass spectrometry has continued. We have now shown that under negative ion chemical ionization, electron-molecule, ion-molecule, and wall-assisted reactions occur. These reactions proved to be key factors to structural differentiation in an extensive study of parent PAHs and methyl-substituted PAHs that we completed this year.

Purine dimers are formed as a result of the action of UV radiation upon DNA in skin, and the photoproducts are thought to be important in skin cancer. We are developing procedures for quantitating these photoproducts at physiologically significant levels. We have developed a multi-dimensional chromatographic method that provides detection limits of 1-5 ppb, almost two orders better than needed to study significant UV exposures.

Packed column supercritical fluid chromatography (SFC) has been used to separate and detect high explosives and/or their manufacturing byproducts. Carbon dioxide is the solvent and bonded cyano phase is the column. Determinations have been made at ca. 0.2 ppm. We are evaluating the use of capillary column SFC for this analysis.

A method was developed for collecting and preserving formaldehyde (from water) as the dinitrophenylhydrazine complex, and determining it by HPLC. The sensitivity is ca. 3 ppb. This procedure was selected by the US Navy for a large study of water contamination in naval housing. (The contamination resulted from the use of polyacetal plumbing fittings.)

We continued to work on concepts and instrumentation for deterrence of currency counterfeiting. The feasibility of two concepts was demonstrated. In the first a taggant is encrypted in the currency during production and verification occurs during sorting operations. In the second near infrared reflectance data of currency are examined by principal component analysis and Kth nearest neighbor algorithms so as to "classify" the currency.

We performed a tank calibration experiment on a 20,000 L plutonium holding tank at and in collaboration with the Savannah River Laboratory (SRL). This experiment proved that our lutetium double-spike mass spectrometric technique is competitive with other calibration techniques. Plans have been made to calibrate a 500,000 L tank at SRL and to conduct several calibration experiments with the Japanese.

We are collaborating with researchers in the Solid State Division, using secondary ion mass spectrometry, to study the stoichiometry of thin film superconductors over the film's thickness. These films are prepared by laser ablation and then annealed in oxygen. Our studies have shown that stoichiometry varies over portions of the film thickness, hence that superconductivity is occurring only in part of the crystal. This type of work will be enhanced tremendously when we acquire a new Cameca ion probe (anticipated 1989).

Much research into the mechanisms of organic SIMS has been carried out. It appears that the primary ion beam causes reactions in solution that result in solute-characteristic secondary ions. We are designing a secondary ionization mass spectrometer for imaging organic compounds in various matrices, such as biological tissues.

Construction of a new high-efficiency, high-resolution von Hamos crystal spectrometer was completed this year. This device is used for research into high-resolution heavy ion X-ray stimulated emission (HIXSE) spectrometry. We believe HIXSE is well suited for non-destructive examination of the local chemistry of implanted ions and thin films in the near surface region (ca. 5 to 500 nm).

Construction of a slow positron source--an appendage to the Oak Ridge Electron Linear Accelerator--has been underway for several years. Installation of the beam line components was completed in February and the first positron beam was detected on March 3, 1988. The design of this facility permits delivery of both slow and high energy positrons to an adjacent experiment room. The highest flux measured for the slow (3keV) component of the beam was 1 x 10^8 positrons per second with ORELA operating at 30% of full power. This is one of the best such sources in the world.

The first experiments with this positron source were directed at positron ionization mass spectrometry. The idea is that large, organic species will attach a positron, eject an electron by annihilation, and leave a positively charged molecule. A time-of-flight mass spectrometer was constructed and interfaced to the positron beam line. Positrons at 2 and 3 eV, obtained through use of a W moderator, were used to obtain spectra of dodecane, toluene, isobutane, and methane.

Initial studies into multiphoton ionization spectroscopy have demonstrated that high sensitivity is attainable and that the ionization signals vary linearly with concentration over several orders of magnitude. These experiments were carried out in our low-pressure spectroscopic cell (reported last year) to eliminate collisional line broadening. Plans have been made and equipment ordered to explore the utility of an ion-trap mass spectrometer with multiphoton ionization and the low pressure burner interface for resonance ionization mass spectrometric experiments.

Our study of the resonance ionization mass spectrometry (RIMS) of Pm was completed this year. In the process, we were able to identify many spectral lines of Pm and Sm. At 584.6 nm there should be no isobaric interference of Nd in the determination of Pm.

We have demonstrated the utility of a GeAlAs diode laser in the RIMS experiment. This was in fact a "diode-assisted" RIMS study because the diode laser was used to pump ground state La atoms to an intermediate excited state from which they were ionized with a copper vapor laser-pumped dye laser. This experiment suggests that multiple diode lasers might be used to design inexpensive RIMS instrumentation.

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 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) diode lasers have been used to initiate electronic transitions in Lu for RIMS analysis; this gives the hope of developing relatively inexpensive RIMS apparatus for future studies; b) a VG-9000 glow discharge mass spectrometer, the first of its kind in DOE, has been delivered and installed; initial experiments have demonstrated its unique ability to analyze all components, from major to trace; c) the first version of our explosive detector underwent successful testing at Sandia; a prototype of a new version, a combination of our atmospheric sampling ion source and an ion trap, is now being tested; early experiments are encouraging; d) construction of the slow positron facility at ORELA is complete; we now have a source of positrons that compares favorably to any in the world and initial experiments are under way; e) the ion trap has been demonstrated to provide MS^n capability ($n \leq 13$ so far) which can be very helpful in elucidation of complex structures; e.g., MS^4 was used to good advantage in porphyrin investigations; and f) construction of the Organic Mass Spectrometry Laboratory will be completed in early 1989; occupancy will follow shortly thereafter.

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 among the DOE Offices of Safeguards and Security, Office of Arms Control, and the Office of Health and Environmental Research. The balance of our effort is funded by Laboratory divisions and programs, the Y-12 and K-25 plants, various interagency agreements (FAA, ISPO/State, and IAEA), and other work-for-others contracts.

LASER SPECTROSCOPY AND INSTRUMENTATION

J. M. Ramsey

This group performs both research and development in the area of laser-based measurements. The focus of our work is very high resolution/high sensitivity spectroscopy including degenerate four-wave mixing, multiphoton, and gas jet spectroscopy. The use of photorefractive optical elements is also being investigated. Projects that are more development oriented include advanced counterfeit deterrence and heat pump efficiency studies. These latter projects also involve the use of laser-based measurements.

High-Resolution Laser Spectroscopy

We have been investigating techniques for performing elemental spectrochemical analysis near the natural linewidth limit (≈ 10 MHz). Conventional atomic sources that use continuous sample introduction consist of plasmas that operate at high temperatures and atmospheric pressure. These conditions lead to spectral line broadening from collisional and Doppler effects. Spectral linewidths are typically a few gigahertz in such an environment. The use of methods that eliminate these line broadening effects can greatly improve spectral resolution and increase qualitative information. These techniques could be used for measurement of isotopic abundances by optical means.

We achieve improved resolution in our experiments while maintaining the convenience of conventional atom sources. The collisional broadening is reduced to the natural linewidth limit by performing the spectroscopy at a reduced pressure. The pressure reduction is accomplished using a sampling orifice on an evacuated chamber as was reported in last year's annual report (ORNL 6474). Species produced in the high temperature and pressure environment of a plasma are extracted into the vacuum chamber through the orifice where the pressure is typically less than one torr. Doppler broadening is greatly diminished by employing nonlinear spectroscopic techniques. Nonlinear spectroscopy reduces Doppler broadening of one-photon transitions by "labeling" the zero velocity species and two-photon transitions through cancelation of the frequency shifts of two photons traveling in opposite directions.

Degenerate Four-Wave Mixing

We have been studying degenerate four-wave mixing (DFWM) to determine its applicability to high-resolution spectrochemical analysis. Initial studies of DFWM were performed in an atmospheric pressure flame. More recently we have compared the use of DFWM performed directly in the flame and in the low-pressure cell. Salt solutions are aspirated through an air-acetylene burner for the production of neutral atoms for analysis. An argon-ion pumped, tunable single-frequency cw dye laser is used as the excitation source. DFWM spectra taken in the low-pressure cell produce spectral features of less than 100 MHz with resolution of the ground state hyperfine structure of the sodium and lithium D lines. Comparison of the spectra obtained in the flame at atmospheric pressure with those from the low-pressure cell suggests a collision-induced shift as well as broadening of the spectra. This has focussed on lithium which has well-resolved hyperfine structure, allowing study of the two isotopes ^{7}Li and ^{6}Li. Fringes from a confocal Fabry-Perot etalon with a free spectral range of 150 MHz were used to aid in the registration of the high- and low-pressure data. Thermal drift of the laser and etalon cavities made registration of spectra to within a sufficient degree of accuracy (≈ 100 MHz) difficult.

To increase the accuracy of comparison of the sharp and broad spectra, a lithium heat-pipe oven was used as a frequency reference. This allows the simultaneous acquisition of calibration spectra with the lithium flame spectra. A portion of the same dye-laser beam that is used to obtain the DFWM spectra is split off and used to obtain saturated absorption spectra in the heat pipe. The heat pipe is operated at low pressure ($\leq$ 1 Torr), and the spectra can be made nearly Doppler-free by the use of counterpropagating beams. Thus, sharp spectra for calibration are obtained with the flame spectra. Comparison of the low-pressure sampled-flame spectra with those from the heat pipe yields excellent agreement of peak positions. The high-pressure spectra show red shifts of up to several hundred MHz, varying for the different hyperfine components. Similar results are observed for ^{7}Li and ^{6}Li. We have also monitored the fluorescence from the flame and found similar red shifts. The concentration of atomic species is important in determining the lineshape in the flame. The shifts are measured at low atomic densities where processes such as self-absorption, which can alter the lineshape, are minimized.

Multiphoton Ionization

We are also investigating the use of multiphoton ionization (resonance ionization) for high-resolution spectrochemical analysis. The photoionization takes place in the low-pressure cell to eliminate the collisional broadening as above. The initial step in the photoionization process is through a two-photon transition to allow the potential for a Doppler-free measurement. We have fitted the cell used in the earlier DFWM studies with a biased wire. The wire collects electrons produced in the cell by resonantly enhanced ionization of the atoms produced in the flame. Initial experiments have employed the pulsed, high-power output of a Nd:YAG-pumped tunable dye laser to drive a two-photon allowed transition. Generally, the excited state produced is photoionized by the absorption of a single additional photon to reach the ionization continuum. In some cases, ionization involving collisions with other atoms or molecules may be important.

The ionization signals are found to depend linearly on concentration over several orders of magnitude for Na, Li and Cu for two-photon transitions to Na 4d ^{2}D, Na 5s ^{2}S, Li 4s ^{2}S and Cu 5s ^{2}S. Excellent sensitivity of detection is demonstrated for these species with limits as follow: Na-20, Li-600, and Cu-25 parts per trillion by weight. Calcium was detected via the two-photon transition to Ca 5s ^{1}S at a limit of 7 parts per billion. Detection of potassium via higher s and d states shows a different nonlinear concentration dependence. Two-color ionization of lithium has been demonstrated. When the dye laser is tuned to the one-photon transition at the D lines, the third-harmonic of the YAG laser ionizes the prepared 2p state with a single photon. This ionization process is efficient and gives signal from aspirated solutions of 100 parts-per-trillion concentration. This is comparable in sensitivity to the single-color ionization via two-photon resonance to the 4s state.

Resonant, one-color ionization spectra of iron, which has many low-lying excited states, gives information on the temperature inside the cell. The iron "thermometer" shows the interesting result that the expansion into the cell cools the electronic temperature well below the flame temperature. Modest vibrational and rotational temperatures are also seen in the spectra of nitric oxide, which is produced in all air flames and which is easily detected by [2+2] multiphoton ionization (MPI) in the sampling cell.

Further studies are aimed at detection of other molecular species of interest, notably carbon monoxide. We are currently investigating the spectroscopy of CO in a clean, static cell using the tunable dye laser and its frequency doubled output.

These initial studies using the pulsed Nd:YAG pumped dye laser have demonstrated that high sensitivity is obtainable with the photoionization approach. The bandwidth of this laser is not small enough to demonstrate the Doppler-free aspect of these experiments. Progress is under way to perform these experiments at high resolution using single frequency lasers.

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

Application of the Ion Trap Mass Spectrometer to Resonance Ionization Mass Spectrometry

An extension of our multiphoton ionization work is to do mass spectroscopy on the resultant photoproduced ions. This experiment is normally referred to as resonance ionization mass spectroscopy (RIMS). Our choice for making the mass to charge ratio measurement is the three dimensional quadrapole or ion trap mass detector. This mass spectrometer has created wide-spread interest in the organic mass spectrometry community. Many potential applications, detailed elsewhere in this report, are being explored within ACD. We believe that the ion trap can also be used advantageously in RIMS. Two such areas of investigation are outlined below.

The theoretical elemental selectivity in resonance ionization often is not realized in single color pulsed RIMS due to power-broadening effects; this results from the need for high laser power in the final ionization step. Alternatively, cw-RIMS offers improved selectivity but suffers from low ionization efficiencies. We plan to implement the ion trap to study the feasibility of collisional ionization of atoms promoted to Rydberg states via laser excitation of resonance transitions. Sample atoms, introduced into the trap by thermal atomization, will be excited to high Rydberg states by laser radiation tuned to specific resonant transitions. These excited atoms will then be ionized inside the ion trap by collisions with inert atoms (He, Ne, Ar, etc.); these gases are present at $\approx 10^{-3}$ torr in a normally operating ion trap mass spectrometer. This technique would obviate the need for high laser power in the ionization step while still maintaining the advantages of highly selective resonant excitation and efficient collisional ionization. In addition, because the resonant transitions are easily saturated, a low-power, cw laser can be used; consequently, the duty factor of excitation will be well matched to continuous thermal atomization from a filament. Ion collection efficiency should also be improved due to the spatial overlap of the ionizing and trapping volumes.

The other area we plan to investigate is the coupling of an analytical flame to the ion trap. The low-pressure interface to the analytical burner developed previously will be used to introduce neutral species into the ion trap. Ions will then be produced by laser excitation and collisions as described above. The addition of mass selectivity should appreciably reduce the background presently observed in multiphoton ionization measurements in the low-pressure cell. The combination of the ion trap mass

---

*ORAU Post-Doctoral Research Associate

**GLCA/ACM Student, Earlham College

***ORAU Post-Doctoral Research Associate spectrometer with the low-pressure burner interface will provide a very sensitive analytical technique for solution samples.

The necessary equipment for these experiments is currently being assembled. The ion trap itself has been constructed from parts obtained from a commercial vendor and the vacuum system has been assembled and tested.Work on the switching circuit serving as an rf generator is now in progress.

(*D. E. Goeringer, W. B. Whitten, J. M. Ramsey*)

Coherent Trapping Effects in Resonance Ionization Mass Spectroscopy

It has been known for almost 20 years that atoms with two or more ground states coupled to a common excited state by resonant laser fields can undergo two-photon optical pumping, called coherent trapping of atomic populations. This phenomenon produces a minimum excited state population when the frequency difference between the two laser fields matches the ground state splitting, and a large excited state population when the fields are slightly detuned. The experimental conditions of many RIMS experiments, namely high intensity multimode-laser excitation of a Doppler-broadened atomic vapor, are similar to those necessary to observe these coherent trapping effects. The resonances in this case would occur if, for a particular isotope, a mode beat frequency of the dye laser is close to the ground state splitting of the analyte. To see how important the trapping might be in causing isotope ratio biases, we have made three- and four-level density matrix calculations of the excited state population for various combinations of laser frequencies and intensities. Among the interesting results of these calculations is the prediction of an unusual saturation behavior at high intensity. Fluorescence excitation spectra as a function of laser beat frequency have been calculated for various experimental conditions.

We have experimentally tested some of the theoretical results by measuring the fluorescence of a beam of sodium atoms excited by two single-frequency ring dye lasers. One laser is tuned to a sodium D_1 hyperfine transition and the other laser scanned in frequency. Far from resonance, the fluorescence is weak because of one-photon optical pumping. When the laser frequency difference approaches the ground-state splitting, the fluorescence increases markedly. There is a central maximum with two sidebands, each displaced by the upper level splitting. There is a very sharp dip in the center of the main peak, corresponding to a 10% decrease in fluorescence from the peak value when the two-photon resonance is established. This observed behavior is predicted by the four-level density matrix calculations.

(*W. B. Whitten, J. M. Ramsey*)

Chemical Measurements using Photorefractive Optical Elements

Degenerate four-wave mixing (DFWM) uses three input beams to generate a fourth signal beam in a nonlinear medium. This technique produces a phase conjugate signal beam only if the two counterpropagating pump beams are phase conjugates. Spatial overlap of these beams can be difficult if refractive index variations serve to distort or to defocus the beams in the sample region. We have investigated the use of a $BaTiO_3$ self-pumped phase conjugate mirror to provide the counterpropagating pump beam. The advantages of using this optical scheme include better discrimination against scattered light at the detector, and higher signals and lower detection limits when significant refractive index perturbations are present in the beam paths. In addition, the most critical alignment in the DFWM experiment is performed by the phase conjugate mirror automatically.

This new approach to DFWM was first demonstrated by introducing phase distortion in the pump beam using the bottom of a cola bottle. The phase conjugate mirror corrects for these distortions, and no change in detection limit is observed, while the detection limit increased (degraded) by two orders of magnitude using a conventional mirror.

This pump beam formation technique is also important when attempting to determine spatial variations of species using DFWM. Refractive index variations can misalign the pump beams leading to incorrect concentration or density measurements. Sodium atom density measurements were made in a slot burner flame as a function of burner position using conventional and phase conjugate mirrors. At the edges of the flame, where the temperature gradient is highest, no signal is observed using a conventional mirror due to beam steering effects, while signal integrity is preserved using the phase conjugate mirror. The conventional approach to DFWM greatly distorts such density measurements.

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

---

*ORAU Post-Doctoral Research Associate

Two-Wave Mixing Interferometer

In the previous annual report (ORNL 6474), we described a novel interferometer based upon two-wave mixing in $BaTiO_3$ that can be used as a self-zeroing refractive index detector. During the past year we studied the characteristics of such an interferometer to optimize the performance as a chromatographic detector. We have set up a system where the pathlength changes in our interferometer are provided by a piezoelectric translation device which translates a mirror under computer control. In this way we separate the interferometer performance from flow cell performance, in order to better determine the properties of the detector. The detection limit of the detector in this configuration is the same as previous experiments, 10^{-6} ΔRI (ΔL = 10 nm). The major advantage of this optical arrangement is the ease with which different time-dependent pathlength changes can be introduced to the detector for the purpose of evaluating system performance and verifying theory. We use step and Gaussian inputs to compare theoretical and experimental waveform shapes, and we are using ramp inputs to simulate the response of the detector to gradient changes in refractive index. We are also investigating the effect of different barium titanate crystal orientations on detector response times and waveform shapes.

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

---

*ORAU Post-Doctoral Research Associate

Continuous Wave Dye Lasers Using Photorefractive Tuning Elements

We have previously shown that the output spectrum from a cw dye laser is reduced to two longitudinal modes when a crystal of photorefractive barium titanate is placed in a dye laser cavity. This bandwidth reduction is the result of a real-time holographic grating written in the crystal by the standing wave intensity patterns in the laser cavity. The grating in the crystal provides bandwidth reduction and wavelength stability. The frequency separation between the two modes oscillating in the cavity can be predicted from the position of the crystal and gain medium within the cavity. The difference frequency must be appropriate to provide complete spatial depletion of the gain in the dye jet and a high contrast interference pattern within the crystal.

We have constructed a modular dye laser cavity to allow for easy modification of the resonator geometry. This dye laser was constructed from Burleigh optical erector set components. The Spectra Physics 375 dye laser used in earlier experiments did not allow such modifications. Three new linear cavity configurations were studied. The first is a three mirror linear cavity using an end mirror with 12 mm radius of curvature, i.e., a dye-jet to end-mirror spacing of 12 mm. The difference frequencies measured at various crystal positions are consistent with the theory developed based on experiments with the Spectra Physics laser. The second configuration investigated is a four mirror symmetrical cavity with a gain medium at the center. With this cavity design the spatial hole burning mode spacing is equal to the longitudinal mode spacing. Experiments show that the position of the crystal in the cavity and the bandwidth of the grating in the crystal must be considered to determine the difference frequency. For crystal spacings between 7.5 and 4.5 cm, the difference frequency is that which provides the highest contrast interference pattern at the crystal. When the crystal is 3.7 cm from the end mirror, the difference is one spatial hole burning mode spacing less than that predicted. At this point the bandwidth of the grating is forcing the two oscillating modes to be closer together despite the lower contrast interference pattern produced in the crystal. At smaller spacings such as 2.5 cm, adjacent longitudinal modes are found to oscillate. The frequency spacing predicted by the crystal position in this case is too large for efficient reflection from the grating while the contrast of the interference pattern from adjacent longitudinal modes is high.

A third configuration involved placing the crystal in the cavity with the c axis pointing away from the gain medium. In this configuration, the laser output spectrum was broadened rather than narrowed. The time average spectrum (τ = 30 ms) covered ≈ 6 nm with a fairly uniform distribution. The spectrum was also found to be much different than that of a free-running cw dye laser. The free-running laser had a spectrum consisting of approximately 3 peaks covering a range of 3 nm. The mode beat spectrum of this laser contained discreet frequencies, but more than just those due to longitudinal modes. We speculate that when the crystal is placed inside the cavity with this orientation, the optically produced grating increases the threshold for frequencies that can utilize this grating. Thus, the high gain mode that initially start oscillating is depressed in favor of lower gain modes. We suspect that the laser systematically scans over this 6 nm range.

(*W. B. Whitten, J. M. Ramsey*)

Counterfeit Deterrence

A program to develop concepts and demonstrate instrumentation for the deterrence of currency counterfeiting continued at a modest level during this year. This work was funded as a subcontract from Sandia National Laboratory, which is providing management oversight of the counterfeit deterrence research programs for the Bureau of Engraving & Printing (BEP), US Department of Treasury.

The technical feasibility of two deterrence concepts was demonstrated. One concept involves encrypting taggant information in the document when it is produced and then using this information to verify the document's authenticity. The encryption will occur at BEP during the final stage of currency production.

Verification will occur during currency sorting operations at the Federal Reserve Banks. These are high-speed high-volume operations. The second concept involves the use of pattern recognition techniques to determine whether a document is authentic. This approach could have application as a "point-of-sale" device to verify the authenticity of a currency note; this application is a relatively low-speed and low-volume operation.

A necessary requirement for the taggant concept is that the taggant must provide multidimensional information that cannot be duplicated by any printing or copying process. Spatial coordinates are two of the random variables. We have shown, through laboratory measurements, that the taggant we proposed, once embedded in the paper matrix, yields four random variables suitable for encryption. Experimental data also indicate that the necessary measurements can be made at the speeds required by the sorting operations. Also, this taggant will not change the visual or tactile characteristics of a note, and should have no effect upon the printing process.

The pattern recognition concept uses near-infrared diffuse reflectance data. The concept was tested with a sample population of uncirculated currency, circulated currency, and high quality bond paper stock. Principal Component Analysis (PCA) and Kth Nearest Neighbor algorithms were applied in this study. PCA involves the transformation of a multidimensional data set so that maximum variance of the data set is retained in only a few principle components or dimensions. By selecting reflectance values at wavelengths which showed the greatest influence on the first principal component of the entire data set, we were able to significantly reduce the dimensionality of the problem. Using the first two principal components of the reduced data set, an excellent separation of the paper stock from the currency specimens was obtained. This reduction in dimensionality is important because it represents a corresponding reduction in the amount of raw data that needs to be gathered and in the computations during data analysis. With five nearest neighbors and an absolute voting rule, 100% correct classification of all members in the data set was achieved. A manuscript describing the pattern recognition work is being prepared for publication.

(J. M. Dale, L. N. Klatt, J. M. Ramsey)

Heat Pump Fluid Measurement Systems

The development of instrumentation for the in-situ measurement of absorbent concentration in operating absorption heat pump systems has continued. Work this past year has focused on the development of a probe for the ammonia-water fluid; the concentration range of interest is 2-50% [w/w] ammonia. The refractive index of this fluid ranges from about 1.33 to 1.37, considerably lower than that of the LiBr-H_2O absorbent.

The sensor being evaluated is fabricated by taking a 600 micron diameter quartz fiber optic and bending it into a arc of about 220°. The polymeric buffer coat and cladding are removed before the arc is formed. The radius of the arc is about 2 mm, and the separation between the straight fiber segments leading to and from the arc is about 2 mm.

The curved segment of the fiber optic allows only a subset of the propagation modes to be transmitted to the exit leg of the sensor. Changes in the refractive index of a fluid surrounding the arc alter the efficiency with which this subset is transmitted. This change in transmission efficiency is the basis of operation of the sensor. T. Takeo and H. Hattori (*Jap. J. Appl. Phys.*, 21, 1509-1512 [1982]) presented a partial theoretical treatment of light propagation around a single bead. The refractive index range of the sensor can be varied by changing the radius of the arc.

Because the total light loss in the arc is very large, a 2 mW near-infrared diode laser is used as the source; a silicon photodiode is the detector. The diode laser contains a photodiode, which is used to provide a source intensity reference signal.

The sensor's response decreases monotonically with increasing refractive index; a second degree polynomial is used to model its response. For diode laser drive currents between 40 and 50 mA the response curve is independent of drive current. Precision of a single measurement corresponds to about 0.2% [w/w] ammonia. Relative errors in ammonia concentration, based upon the statistical repeatability of the response curve, is about 4%. Most of this error is caused by a long term drift in source intensity associated with heat dissipation from the diode laser to the diode mounting and fiber optic coupling hardware. When the response of different probes is normalized with respect to their response for water, the calibration curves differ by about 6%.

A brief compatibility study was completed. At 7% [w/w] ammonia no changes in the sensor were detected after three weeks of continuous exposure to the ammonia-water fluid; temperature was cycled from room temperature to 85°C several times during this study.

(*D. T. Bostick and L. N. Klatt*)

Consolidated Fuel Reprocessing Program

Reports summarizing work in the areas of in-line and hot-cell instrumentation were completed as part of the joint development agreement between the USDOE and the Power Reactor and Nuclear Fuel Development Corporation of Japan (PNC). These reports described development activities sponsored by the Consolidated Fuel Reprocessing Program (CFRP). A third report, not part of the CFRP agreement with PNC, describing in detail the hot-cell titration system was also completed. This instrument has been in service at the TPP facility for about four years. All of these reports were published as ORNL/TMs.

Interaction with the technical staff of the United Kingdom Atomic Energy Authority (UKAEA) and review of progress reports on the in-line photometer technical exchange agreement between the USDOE and the UKAEA has continued.

(*L. N. Klatt*)

ACTINIDES AND ISOTOPIC MASS SPECTROMETRY

R. L. Walker

This group performs research and development in mass spectrometry for improved isotopic analysis of actinides and other elements. We design and improve equipment and analytical methodology for special applications in areas of national and international concern such as the nuclear fuel cycle, safeguards and security, and the environment. Other analytical support is given to stable isotope enrichment programs as well as to a variety of Laboratory research programs.

Safeguards-Related Projects

The lutetium double-spike technique (ORNL-6357, 6474) has been further tested as a nuclear safeguards measurement method. Together with Savannah River Laboratory (SRL) personnel, we performed a volume calibration experiment on a 20,000-liter Pu holding tank located in one of their hot canyon facilities. Our primary goal was to determine the precision of our technique for measuring total tank solution quantity/volume at several different liquid levels. Our results verified the tank's current volume calibration (and mixing/sampling parameters) and showed that we are competitive with the currently used (National Institute of Standards) NIST calibration method. We then discussed with SRL personnel how the technique could be used both for "spot calibration checks" and for volume/level calibration of new tanks as they are put on-line. Plans are being made to assist them in calibrating a new 500,000-liter tank. We have also recently received funding and begun collaborative work with the Japanese on several tank calibration experiments at Tokai Reprocessing Plant.

Collaboration with the International Atomic Energy Agency (IAEA) continued this year. The work consisted of characterization, both isotopic and concentration, of mixed internal calibration spikes to be used for safeguards analyses of U and Pu at the Safeguards Analytical Laboratory (SAL) at Seibersdorf, Austria. The application of internal standards to mass spectrometric analyses has shown great promise for improving bias correction in isotopic measurements of fissile material for accounting purposes. We made uranium measurements to a precision of 0.07% relative standard deviation (RSD) using the VG354E and multicollectors; this compared to a precision of 0.28% that was obtained using the 2-stage instrument. Plutonium isotopic measurements were made to a precision of 0.15% RSD using the 2-stage, resin-bead method. Concentration measurements by isotope dilution gave precisions for Pu or U of 0.1% RSD.

(M. P. May, D. H. Smith, E. G. Miller, R. J. Hydzik, E. H. McBay)

Glow Discharge Mass Spectrometry

In July of this year a VG 9000 glow discharge mass spectrometer (GDMS) was obtained, installed, and made operational. This is the first commercial version of a mass spectrometer using a glow discharge ion source designed for the analysis of solids. This instrumental concept is unique in that it offers for the first time the possibility of measuring major, minor, trace, and ultra-trace sample components with a single instrument. Quantitative analysis requires that a set of elemental relative sensitivity factors (RSFs) be derived from known standards of similar matrix type as the unknown sample. This must be done under carefully controlled conditions such as sample geometry and plasma discharge conditions. Thus, it will be of interest to characterize sensitivity, precision, and accuracy as a function of glow discharge operating parameters. In this context we will study isotope fractionation processes that occur in a glow discharge. We feel that this study will provide basic information on how ions are formed in the plasma and how they are transmitted to the exit slit of the cell and subsequently escape into the electrostatic lens system for mass analysis.

To date preliminary studies have been carried out with four matrix types: low alloy iron, stainless steel, platinum/iridium, and aluminum alloys. These studies show that quantitative work can be done if standards are available to derive a suite of elemental sensitivity factors for each matrix type. An example of the analysis of stainless steel is presented in the following table. In this study, sensitivity factors were derived from the analysis of NBS standards 447 and 449. NBS standard 446A was then treated as an unknown to produce the tabulated results. These results clearly demonstrate the quantitative potential of this technique when it is properly calibrated.

Table 1.1. Quantitative Analysis of NBS 446a

Element	Certified Wt. %	GDMS Value	% Error	Sensitivity Factor
Si	1.19	1.22	2.5	2.16
V	(0.03)*	0.035	-	0.59
Cr	18.35	18.32	0.2	2.45
Mn	0.53	0.54	2.0	1.53
Fe	70.6	70.01	0.8	1.00
Ni	9.11	9.62	5.6	1.54
Cu	0.19	0.20	5.3	6.14

*Tentative value, uncertified

Our instrument is equipped with an Ar purification system that will allow us to measure H, C, O, and N in alloys, a capability that we have not had in a mass spectrometer because of high background in previous instruments.

(W. H. Christie)

Isotope Ratio Mass Spectrometry

The initial concerns of the xenon/krypton project on the VG354E have been addressed and essentially answered. The focus of this project was to measure the ratios of the major isotopes of these gases to errors < 10 ppm standard error of the mean. On the average, isotopes with abundances of 10% and greater could be measured meeting the < 10 ppm requirements with volumes of 1 std. cc of pure xenon or krypton admitted to the ion source via the gas inlet system. A 2 std. cc volume, however, was required to meet the < 10 ppm requirement for the mass 80/84 ratio of krypton, owing to the 2.27% abundance of mass 80. Great concern arose when the instrument began to give different ratio measurements for the same masses measured when only the Faraday collectors to which they were admitted were changed. It was found necessary to apply an empirically determined correction factor to ion beam intensities measured in the axial collector. This is due to the fact that this collector is recessed behind the focal plane of the instrument. Divergence of the ion beam caused this pocket to collect a smaller fraction of its isotope than the other four pockets, which lie on the focal plane.

(E. H. McBay, J. A. Carter, D. H. Smith)

Mass Spectrometric Projects and Support

We continued our support to the stable isotopes and radioactive isotopes sales programs. Analytical support for transuranium production has been severely curtailed this past year due to shut-down of the HFIR. Gas analysis continues as an important activity of the group. The majority of our gas analyses are in support of the Y-12 Plant. However, important contributions are made to research programs at the other Oak Ridge plants as well. The most notable of these was the isotopic and impurity analysis of Kr and Xe enriched in the centrifuge pilot plant at K-25. Additionally, many samples of a more routine nature were analyzed for gas and isotopic composition of the primary element of interest (e.g., hydrogen, carbon, sulfur, sulfur hexafluoride, krypton, and xenon).

We have continued working with members of the Physics Division on the US/UK actinides research program to study the behavior of these nuclides in a fast breeder reactor such as the one at Dounreay, Scotland. This year's effort has been limited to measurement of actinide dosimeters and rechecks of physics specimens from fuel pin #2. Work is expected to start soon on fuel pin #4, the last of the experiments. Fuel pin #4 has been removed from the reactor and is awaiting an approved shipping cask at this time.

Special analytical support has been given to the low-level determination of actinides in waste water streams using our sensitive mass spectrometry techniques. We have continued using the resin bead method and isotope dilution mass spectrometry to enhance the analytical support of waste isolation programs and DOE-supported programs to clean up the local environment as well as at former sites handling radioactive materials.

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

SPECTROSCOPIC R & D PROGRAMS

D. H. Smith

This group performs research, applied and fundamental, in widely disparate areas. The past year has been noteworthy for the completion of two construction projects crucial to further developments in their respective areas of research. An intense beam of positrons has been obtained from ORELA. It has been characterized and found to compare favorably with any such beam in the world. Analytical applications are being explored. A spectrometer for HIXSE has been completed and initial experiments completed. This instrument should pave the way for a series of benchmark experiments. Other areas of concentration for this group are concerned with mass spectrometry. In the inorganic field, studies of resonance ionization as applied to analytical mass spectrometric problems are featured. A major advance this year has been the first application of a solid state diode laser to RIMS. In the organic area, MS/MS, both fundamental and applied, is the major focus. Noteworthy progress has been made this year in application of ion trap mass spectrometry to detection of various ambient species and to fundamental studies.

Resonance Ionization Mass Spectrometry

The study of Pm by RIMS that was discussed in the last annual report (ORNL 6474) has been concluded. By making use of the ETRANS computer program reported two years ago, the first transitions of approximately 50% of the Pm ionization wavelengths were identified and some 95% of the Sm wavelengths identified in the 530 to 560 and 580 to 614 nm spectral ranges. Since the ionization process is believed to involve 3 photons in either a 1+1+1 or 2+1 process, new level information is available for both Pm and Sm within the precision of our wavelength measurements. Based on Nd experimental work reported in the literature, there should be no isobaric interference of Nd on the RIMS determination of Pm at the wavelength of 584.6 nm.

A disposable graphite furnace assembly is being developed and should yield some advantages as an atom and/or ion source for mass spectrometry. The low work function of carbon leads to the generation of atoms as opposed to ions. The presence of carbon should also reduce the yield of gaseous oxide species. With proper design of the furnace, atom effusion from the furnace should be more directional than the effusion from heated metal filaments. We have developed two graphite furnace assemblies that use simple mechanical pressure or gravity to maintain electrical contact. With such designs, assembly of the source is simple, and the sample furnace is disposable. Prototypes of both our designs have been successfully heated in a vacuum. With the former concept, we have fabricated a small furnace consisting of a source, 1.3 cm long graphite tube that can be attached to the sample chamber of existing mass spectrometers. The furnace has been heated to approximately 1200° C with a current 10 amps at 2 volts. No spectral data has been obtained due to excessive outgassing that led to problems with the instrument.

We have undertaken comparative studies of the usefulness of various lasers for the RIMS determination of neodymium. Three lasers have been used in this study: a Nd:YAG pumped dye laser (NDYAG), a copper vapor laser pumped dye laser (CVL), and a flashlamp pumped dye laser (FL). All lasers were operated in the 580 to 620 nm region. Spectral differences were seen with the different lasers. Analytical determinations were previously carried out using the strong Nd RIMS line seen with the FL at 600 nm; this line is very weak with either the NDYAG or CVL systems. With all laser systems, a strong RIMS transition is seen near 586-587 nm; characteristics of the transition are laser dependent, however. The transition is very narrow (FWHM of 0.3 cm^{-1}) when studied with the 0.3 cm^{-1} bandwidth CVL system and consists of one main line. The transition is much broader and double-peaked with the FL; both peaks have a FWHM that corresponds to the bandwidth of the laser, 3 cm^{-1}. With the NDYAG, two lines are also seen with bandwidths of 2-3 cm^{-1}. In the last case, power broadening would seem to be responsible for the broad RIMS peaks.

We are collecting isotope ratio data for the neodymium system at the present time. We have preliminary data at several wavelengths that suggest that the CVL system exhibits large isotope bias effects (> 20%), for odd/even isotope ratios. At a recent RIS meeting, other investigators reported large bias effects for odd/even isotope ratio measurements for various elements and with various laser techniques.

We have demonstrated the utility of a 750 nm GaAlAs diode laser in a three-photon (overall) resonance ionization mass spectroscopy scheme. To our knowledge this represents the first time that such a convenient and inexpensive ($70) laser has been used for this purpose. Tunable dye lasers previously used for RIMS range in cost from $30,000 to over $100,000. The single mode diode laser was temperature tuned to 753.9 nm to pump ground state lanthanum atoms to their $4F_{3/2}$ level at 13260.4 cm^{-1}; from that level the atoms were subsequently excited to an intermediate level and then ionized with two photons from a 6 kHz copper vapor laser-pumped dye laser. Excited state transitions to six higher, intermediate levels have been observed. In the R6G and mixed R6G-Kiton Red dye regions, the CVL-dye wavelengths for these resonant ionization routes from the 13260.4 cm level of La are 5639.4, 566.46, 584.86, 586.54, 600.68, 6017.2, and 601.73 nm. The first experiment was performed at a CVL-dye wavelength of 584.86 nm, and it was expected that photo-ions would be produced based on published emission spectral data for La. The other wavelengths represent allowed transitions to known intermediate states, but these bound-bound transitions have not been seen in emission. There does not always appear to be exact wavelength correlation between the known transition involved and our experimental wavelength. These are small (<< 0.1 nm) but real shifts from the wavelength expected for maximum rate of ion formation.

This demonstration of diode-laser-assisted RIMS used a diode laser as the first step in the resonance ionization process. Multiple diode laser schemes are feasible, and a RIMS instrument can be envisioned where all the bound-bound transitions are accomplished with these inexpensive devices. Depending on the final design of the proposed instrument, it is expected that isotopic selectivity can be obtained through the optical process above.
(R. W. Shaw, J. P. Young, D. H. Smith)

Organic Mass Spectrometry

The overall objectives of this effort are to expand the applicability and improve the performance of analytical organic mass spectrometry. Efforts are directed both to applications and to fundamental studies; the results of the latter may ultimately be analytically useful. The most heavily emphasized application has been the detection of trace quantities of organic explosives in ambient air. We have recently initiated investigation of the possibility of detecting drugs of abuse and other polyatomic molecules of interest. Other applications projects, which have also involved some fundamental studies, include the study of geoporphyrins and cardiac glycosides. A number of studies have been performed to determine and then to demonstrate the analytical utility of the Ion Trap Mass Spectrometer (ITMS). These include the study of ion injection into the ion trap, self-chemical ionization in the ion trap, negative ion studies, laser desorption experiments, multiple stages of mass spectrometry (MS*2) studies, and selected reagent ion chemical ionization. Other types of instrumentation are being developed and used to study dissociative charge exchange and the ionization of polyatomic molecules with positrons.

The explosives detection effort has proceeded along two paths. An instrument based on our original system, consisting of our Atmospheric Sampling Glow Discharge Ionization (ASGDI) source coupled to a quadrupole/time-of-flight (QT) tandem mass spectrometer, was completed for the Y-12 Plant in cooperation with Plant Laboratory personnel. This instrument has been used to screen mail at the Y-12 Plant. Recently it was moved to Albuquerque, NM, and successfully underwent testing and evaluation at Sandia National Laboratories. The results of the tests were received enthusiastically at a program review held at Germantown, MD, in October. The instrument has since been returned to Oak Ridge and is currently being used to evaluate a new operating mode which promises to reduce detection limits and the speed of analysis. This mode of operation is also being investigated on the quadrupole/sector geometry instrument (the QEB) which provides the capability for high resolution daughter ion detection. A second instrument is now being built for Y-12.

The second path in our explosives detection work has been directed along the interfacing of the ASGDI source with an ITMS. The ITMS has several features that make it potentially superior to the QT geometry as the MS/MS instrument in an explosives detector. Among these are greater MS/MS efficiency (possibly resulting in reduced detection limits), better mass resolution in the second stage of mass analysis, and reduced size. The potential for the ITMS in explosives detection with the ASGDI source, however, is contingent on the efficiency with which ions can be injected into the ion trap. Under normal conditions encountered in mass spectrometry, i.e., at low pressures (< 10^{-5} Torr) injection efficiency has been predicted and observed to be very low. However, recently we and other groups have been able to inject ions into the ITMS with efficiencies high enough to be analytically useful. We have been the first to show ion injection from a high pressure ion source and are currently characterizing this experiment. At present, under optimum conditions, we estimate our injection efficiency to be a few percent. With this injection efficiency we find that the ASGDI source/ITMS is comparable in performance to the original QT instrument. However, collision-induced dissociation and electron detachment upon ion injection into the trap are phenomena which must be controlled before the ITMS can be used completely to replace the QT instrument.

We have recently begun to study the possibility of detecting drugs of abuse and certain other organic compounds that might be present in air. These studies are currently at the stage where ions are being formed from the molecules of interest in a variety of ways in order to understand their mass spectrometric behavior. These compounds will then be studied in both the positive- and negative-ion modes on both the QT and ITMS instruments fitted with the ASGDI source.

A number of projects other than ion injection have either been completed or initiated using the ITMS. We have demonstrated the capability for many stages of mass spectrometry. A variety of reactions are possible between stages of mass analysis, including both unimolecular and bimolecular reactions. We have demonstrated a practical application of an MS^4 experiment in the structural characterization of a geoporphyrin. Five or six stages of mass spectrometry should be routinely feasible, and we have done experiments with up to 12 stage.

We have also completed a study demonstrating the usefulness of chemical ionization in the ITMS using mass-selected reagent ions. These studies have highlighted the differences between the conditions for conventional chemical ionization in the ion trap (i.e., without mass-selecting a particular reagent ion), for chemical ionization in a high pressure ion source, and for mass-selected chemical ionization. The latter provides much better control over the energetics and mechanism of ionization. We have been the first to perform laser desorption ionization in an ITMS, demonstrating the approach with quaternary ammonium compounds, porphyrins and sucrose. We have studied the kinetics of a number of ion/molecule reactions involving both positive and negative ions. We have delineated the conditions under which self-chemical ionization in an ITMS (and in the closely related ion trap detector) are likely to occur and have shown that these conditions can be anticipated in a straightforward manner.

We have made significant advances in the mass spectrometric analysis of geoporphyrins using both the ITMS and a reverse geometry sector instrument. We have applied many of the tools available with the ITMS to the geoporphyrins, including selected-reagent-ion chemical ionization and MS^n. We have shown the "true" behavior of ions formed by the gas-phase interaction of a selected reagent ion with a porphyrin molecule and with its corresponding porphyrinogen and contrasted this behavior with that observed in high pressure chemical ionization. Ionization by proton transfer or by electron transfer is readily promoted in the ITMS with precise control over the reaction energetics. Structural information regarding a porphyrin is available from control of fragmentation upon ionization and from MS^n studies. Even more structural information is available from MS^n studies of the in vitro prepared porphyrinogen (i.e., the reduced form of the porphyrin). We are also well along in the study of some charge permutation reactions of porphyrin ions using the reverse-geometry sector mass spectrometer. Porphyrins show intense doubly charged ions in their electron impact mass spectra. We are currently studying the partial neutralization reaction in which the doubly charged ion is converted to a singly charged ion in between stages of mass analysis. We are interested both in optimizing the cross-section for the reaction and in being able to vary the degree of fragmentation that sometimes accompanies it. These reactions of the doubly charged porphyrin ions have not been investigated previously, but they may prove to be particularly useful for mixture analysis.

During the last year we have modified the source to the QEB and designed and built a solids probe. Several experiments are under way to demonstrate features of this instrument. These include: high resolution parent ion scans; a comparison of resolution/sensitivity between low energy and high energy CID; and the performance of a new mode of operation which promises to increase sensitivity without the loss of some selectivity.

The construction of instrumentation for the study of dissociative charge exchange and for the study of ionization by positrons has progressed this year. A system coupling a magnet with a differential time-of-flight system is near completion. Completion of this instrument has been delayed by the inability to get a satisfactorily fabricated anode. Initial studies will concentrate on the dissociation of energetic neutral molecules formed from the neutralization of the corresponding cation. A time-of-flight mass spectrometer has been constructed and attached to a beam line at the ORELA facility. This work, done in collaboration with D.L. Donohue and L.D. Hulett, will focus on the interaction of slow positrons with polyatomic ions. Data have already been obtained with this system for fast positrons (kinetic energies of 3 keV). We are interested in the energetics of the mechanisms leading to ionization and their cross-sections.

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

High-Resolution HIXSE Studies of the Chemical Environment of Ions Implanted in Insulator Matrices

We have initiated a program to study the chemical environment of ions implanted in insulator matrices, e. g., SiO_2 (quartz), as a function of the initial charge state, concentration, energy (depth), deposition rate and substrate matrix using heavy-ion-induced X-ray satellite emission (HIXSE) spectroscopy. Because HIXSE probes the near-surface region (≈ 5- ≈ 500 nm) and yields chemical information, it is ideally suited for the relatively non-destructive examination of the local chemistry of implanted ions and thin film interfaces.

The ability of HIXSE to probe the local chemical environment of implanted moieties depends upon the use of the newly constructed high-efficiency, high-resolution von Hamos crystal spectrometer. This spectrometer combines vertical focusing optics with a multichannel position-sensitive proportional counter detector to allow the observation of relatively weak satellite signals without compromising the resolution of the chemically sensitive satellite lines. The spectrometer is also equipped with an energy-dispersive Si(Li) detector which enables us to obtain cross-sections, branching ratios and fluorescence yields.

In our initial experiments, a series of quartz glass targets was implanted with 70 keV S ions at the Solid State Division's Surface Modification and Characterization (SMAC) facility. The dosage ranged from 5×10^{15} to 1×10^{17}/cm^2. Rutherford backscattering (RBS) measurements were used to verify the dose, the depth (90 nm) and the uniformity of the implantation. A 30 MeV Cl ion beam from the EN tandem accelerator was used to excite the sub-surface ions, and high-resolution S $K_\alpha L^n$ X-ray measurements were obtained with the new spectrometer. The HIXSE spectra of the different targets exhibit marked variations in the shape and intensity of the sulfur KL^5 ($2s^x2p^y$, where $x + y = 3$ and $x \leq = 2$) satellite line. Integrated over HIXSE's sampling range, this translates into an ability to detect changes in the local chemistry of sulfur at a concentration of less than 0.1 at.%. The observed effect appears to be due to changes in the local valence electron density caused by a change in the local sulfur concentration. If this is verified, then HIXSE should be capable of probing the concentration profile of an implanted ion with fewer restrictions on the Z of the implant and substrate than with RBS. Future studies of this model system will explore the effects of such parameters as deposition rate, depth of implantation, charge state, substrate temperature, and substrate on the local chemistry of the implanted ion.

(T. M. Rosseel, J. P. Young, R. A. Zuhr, C. R. Vane,** and R. Peterson***)*

---

*Solid State Division
**Physics Division
***University of Tennessee-Chattanooga

The ORNL Slow Positron Source

The construction of a slow positron source, which makes use of waste gamma energy from the Oak Ridge Electron Linear Accelerator (ORELA) has been completed. The facility is being used for spectroscopic measurements. Installation of the beam line components was accomplished without major difficulty at the end of February, and the first positron beam was detected on March 3.

The ORELA is a pulsed accelerator operated at 150 MeV; pulse widths vary in the range of 10-20 nanoseconds; repetition rates are usually 800-1000 Hz. Maximum power available is about 60 kW. As the electron pulses strike the tantalum target, showers of gamma bremsstrahlung are produced generating photoejected neutrons that are used for nuclear research. About 15% of the gamma energy is forward-scattered beyond the tantalum target. This is intercepted by the tungsten moderator array of the positron facility. The high atomic number of the tungsten induces the formation of positrons, some of which are thermalized and ejected in a monoenergetic state. The 3 eV positrons issue from the moderator in pulses coincident with the ORELA electron pulses striking the tantalum target. The open end of an extraction tube is charged negative (3 kV) as the slow positrons are emitted. As the positrons enter the extraction tube they are focused into spiral trajectories by the magnetic field of a coaxial solenoid. The solenoidal field is continuous, leading to the experiment room; the positrons follow it and are delivered in pulses having shapes that are essentially replicas of the electron pulses of the ORELA. The delivery solenoid has a gentle s-bend that offsets the path of the positrons, allowing the insertion of radiation shielding to block line-of-sight exposure to the ORELA target. The degree of beam offset for the ORELA facility is much lower than that of other facilities. This has allowed the delivery of high energy positron beams, as well as the slow beam, which is an unexpected bonus. In addition to slow positrons, fast positrons are also scattered into the extraction tube and are transported by the magnetic solenoid. If the bends in the solenoid had been sharp, as is the case for other facilities, the fast positrons would not have been delivered.

Various diagnostic techniques have been applied to characterize the beam composition, flux and energy. A NaI(Tl) scintillation detector was mounted 20 m away from the chamber where the positrons emerge into the Electron Room. This detector with its associated data system was used to detect the 0.511 MeV gamma radiation produced by the annihilation of the positrons as a function of their time-of-flight (which then allowed a calculation of their energy). Two main positron-containing components were detected: a high-energy peak at 80 to 100 keV, and a 3000 eV peak that corresponds to low-energy positrons produced in the W moderator assembly and extracted by the pulser tube. The highest flux measured for the 3000 eV component was 1 x 10^8

positrons per second with ORELA operating at 60% of full power. The high-energy positrons represent an additional source of particles with even greater flux. Other components in the beam such as electrons and ions were detected by a channelplate detector but are present in lower abundance.

The first major use for the slow positron facility has been the positron induced mass spectrometry experiment, described below. Other applications in preparation are positron microscopy, radiation mutation studies of DNA, and defect measurements in oxide superconductors.

(L. D. Hulett, D. L. Donohue, T. A. Lewis, S. Pendyala**)*

---

*Instrumentation & Controls Division

**Consultant, State University of New York

Positron Ionization Mass Spectrometry

A time-of-flight mass spectrometer has been constructed and interfaced to the positron beam line at the ORELA. This instrument is being used to study positron interactions with large organic molecules in the gas phase as a function of energy. It is believed that certain organic species will show the effect of positron attachment followed by annihilation of an electron which will leave the molecule as a positive ion. The energetics of this process will be revealed by the fragmentation pattern of the ion, as measured by the mass spectrometer. The instrument consists of a pulsed-grid ion source connected to a 1 m flight tube and a channelplate electron multiplier ion detector. A personal computer is connected to a CAMAC crate for data accumulation and processing. Positrons at 3000 eV are deflected and focussed into the ion source by electrostatic lenses. They strike a W moderator and are re-emitted at very low energy (2 to 3 eV) and directed back into the ion source where they can interact with the target molecular species at a pressure of 1 x 10^{-5} Torr. Spectra have been obtained resulting from positron impact ionization of selected organic molecules such as dodecane, toluene, isobutane, and methane. Future studies will reduce the energy range down to less than 1.0 eV where positron attachment is expected to occur.

(D. L. Donohue, L. D. Hulett, G. L. Glish, S. C. McLuckey)

Transuranium Element Research

Cooperative spectral studies at the Transuranium Research Laboratory (TRL) continues. The work involves spectral studies of actinide and lanthanide compounds and is carried out with R. G. Haire of the Chemistry Division and J. R. Peterson of the Chemistry Department of the University of Tennessee, Knoxville, along with the latter's students. The study of the spectra of trivalent californium doped into various lanthanide halide hosts of differing crystallographic structure has been completed and a paper has been published. The absorption spectrum of californium at low concentrations demonstrates that this cation conforms to the structure of the host. As the concentration of californium is increased to certain critical levels, both host-related and normal californium spectra can be observed. This critical concentration varies from 5 to more than 15% and appears to be related to the crystalline dissimilarity of the host halide and the californium halide. These structural features are very difficult or impossible to observe by X-ray diffraction studies, but they were verified in some related Raman scattering work. The results of this work have suggested several other studies that would involve doping of various lanthanides into other lanthanide hosts. It also points out the spectral similarity of trivalent californium in the monoclinic and rhombohedral form.

We have long been interested in solid-state ion implantation brought about by radioactive transmutation, e.g., Es(III) Bk(III), and Cf(III). In such cases we have found that oxidation state seems to be controlled by the parent, and progeny coordination is controlled by the host. Does this result apply in the general case? One could consider that there are 3 forms of ion implantation: (1) natural, brought about by transmutation; (2) induced, brought about, for example, by neutron irradiation of a target ion in a solid to promote transmutation; or (3) external, brought about by direct ion implantation. Of the three, the last may be the most complex since there is a charge imbalance to correct. We plan to carry out studies in these three areas. A student carried out preliminary work to see if natural transformation studies could be extended to other parts of the periodic table. This work involved developing analytical procedures to determine the stability of the sulfides of iron and to determine changes in the oxidation state of these iron sulfides in the absence of radiation. This work can now be extended to include ^{35}S, which transmutes to ^{35}Cl by β-decay, with a half-life of 87 days. By preparing solid sulfides or polysulfides of Fe, the oxidation state of the daughter Cl can be assessed through its reactivity with Fe(II) or Fe(II) in the system. Preliminary work on the external ion implantation studies are now under way in a HIXSE study of sulfur implanted in SiO_2 (see section on HIXSE).

Other miscellaneous projects were carried out this year. Spectral studies of CfF_3 and CfF_4 were carried out as part of a broader study to characterize the structures of these fluorides. In a cooperative study, high pressure absorption spectral studies (to ≈ 30 GPa) of $NdCl_3$ and $PrCl_3$ were made. Crystal structure of these compounds transform to orthorhombic in the range of 20 GPa. The orthorhombic structure seems to change again at the high pressures as evidenced by both absorption and Raman spectra. It is not clear whether this is indeed a new form or a distorted orthorhombic structure.

(*J. P. Young*)

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 collaboration with workers in the Solid State Division, we are using SIMS to study the stoichiometry of thin film superconductors over the films' thickness. The superconducting thin films (0.5 - 5.0 μm thickness) were prepared by the Solid State Division using a laser ablation technique. The two kinds of films made were $HoBa_2Cu_3Ox$ and YBa_2Cu_3Ox. The film is deposited on a substrate of $SrTiO_3$ or silicon by repeated pulsed laser ablation from a pellet of the superconducting material and is then annealed in oxygen to form the superconducting phase. The stoichiometry of the pellet should be maintained throughout the thickness of the film in order to obtain maximum superconducting properties. However, the laser ablation process of making superconducting materials is not well understood. SIMS measured depth profile distributions of the elements over the thickness of the film are helping to understand parameters of laser sputtering, such as laser fluency, and indicate that there may be diffusion of these elements during annealing.

These films become superconducting between 70 to 80°K and have a maximum current density of 1×10^3 amps/cm^2. The maximum current density of a single crystal of these superconducting materials is in excess of 1×10^6 amps/cm^2, so the maximum current density in the films could be improved. Measurements by SIMS indicate that the stoichiometry of these films after annealing varies over their thickness, which indicates that these films are only superconducting over a portion of their thickness. Further research on this method of making superconducting thin films is needed. Sample charging resulting from the insulating property of $SrTiO_3$ caused difficulties in depth profile reproducibility. An electron flood gun is being developed to neutralize sample charging. Also, further calibration using improved standards of the superconducting materials is needed to better measure the stoichiometry of the films.

Zinc oxide ceramics have found widespread use as surge protection devices (varistors) because of their extremely non-linear current voltage characteristics. Particular impurities, such as bismuth, barium, or rare earths, are necessary for creating the electronic barriers that give rise to the non-linear characteristics. Another class of impurities, such as chromium, manganese, or cobalt, usually used in combination enhances the non-linearity. The details of how these particular impurities act in barrier formation or in enhancement of the non-linear properties are not yet well understood. In an effort to further the understanding of varistors, workers in the Solid State Division implanted bismuth, chromium, and manganese in zinc oxide crystals. Rutherford Back Scattering (RBS) and SIMS analyses were performed to measure the depth profile distribution of these impurities in the zinc oxide after each of a series of annealing treatments. SIMS was used in particular to analyze chromium and manganese, since they could not be detected with RBS, and to verify the bismuth results obtained via RBS.

The object of this investigation was to seek a correlation between the large changes in electrical properties and the annealing behavior of the implanted impurities. To observe changes in the distribution of the implanted Mn and Cr, SIMS was performed after anneals at 500, 700, 900, and 1000°C. Sample nonuniformities and surface charging effects made it difficult to compare the intensities of different profiles to extract quantitative results. Normalization of intensities to a specific reference signal (e.g., ^{64}Zn) as is usually done did not work. Therefore, the SIMS profiles were normalized on the basis of area. Mn and Cr have low vapor pressures at temperatures near 1000°C and for this reason the normalization to total area is probably valid.

SIMS data clearly show that there is very little change in the Mn distribution after the 700°C anneal, but that after annealing at 900°C much of the Mn has diffused both to the surface and to greater depths. SIMS data also show that Cr is not mobile at temperatures up to 1000°C. An additional anneal performed at 1100°C produced a narrowing of the Cr distribution and a greater depth for its peak. These results indicate that at 1100°C Cr becomes mobile, but instead of spreading out and dissolving in the ZnO, as does the Mn, it concentrates. Such concentration would occur if a stable new phase, such as a CrZn spinel was being formed.

A number of SIMS depth profiles were measured on Bi-implanted ZnO. Results for these samples were in qualitative agreement with RBS data. Little change in profile shape could be discerned after a

700°C anneal, but significant broadening was apparent after 900°C annealing.

In a final study, SIMS was used to measure isotopic values in an electromagnetically separated Ge sample and in a Cd glass sample. In the past, the best method for certifying the isotopic values in Ge samples required that the sample first be converted into $BaGeF_6$. This compound was then subjected to electron impact mass spectrometry. Experience has shown that the conversion to $BaGeF_6$ is less than straightforward. The usual analytical ion, GeF_3^+, may or may not be produced in good yield, depending on the quality of the $BaGeF_6$ preparation. SIMS offers a direct and dependable method for certifying the isotopic values in Ge samples. No chemical conversion of the sample is required and samples of the as-received GeO are simply deposited as a thin film on a conducting substrate. Copious Ge^+ ions are produced under O_2^+ bombardment. SIMS is a viable method for these separated samples because of their high purity. In low resolution SIMS, one is always concerned about polyatomic species being formed that would have the same nominal mass number as an isotope of the element being measured.

The Savannah River Plant asked our assistance in certifying the Cd isotopic values in a glass-like material. This material is used to coat components used in the processing of fissionable materials and as such improves safety by reducing the nuclear reactivity of the solutions being processed. Again, SIMS was 2the ideal technique for this particular matrix as no sample chemistry was required and no interfering polyatomic isobars were encountered. We were able to accurately certify that the sample contained natural Cd.

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

Sputter Atomization
Resonance Ionization Mass Spectrometry

Although sputter atomization/resonance ionization mass spectrometry (SA/RIMS) is a useful technique for analysis of targeted elements and for isotope ratio measurements, nonresonant multiphoton ionization (MPI) is better suited for rapid survey analysis of elemental composition. That is, sputtered atomic neutrals can be ionized in a sensitive, nonspecific manner via high intensity, ultraviolet laser radiation. Our current efforts have focused on modifying the instrumentation initially developed for SA/RIMS, which utilized a double-focusing secondary mass spectrometer, to investigate the utility of SA/MPI. Because multiple atomic species can be ionized by each ultraviolet laser shot, it promises to be advantageous to simultaneously detect all corresponding masses via time-of-flight mass spectrometry. Therefore, we have used the ion optical simulation program, SIMION, to design such a mass analyzer for SA/MPI. Because the large energy spread of the laser-generated ions would degrade the resolution in a normal linear time-of-flight instrument, the reflectron geometry, which has been demonstrated to largely compensate for this energy spread, was modeled. Simulation results indicate that with correct adjustment of sample and reflectron voltages, ions can be generated via laser, accelerated, reflected, and detected in such an instrument. The calculations also indicate that secondary ions ejected from the sample surface can either be detected or effectively rejected, depending on voltage settings, thereby reducing background signal.

Preliminary tests on the initial reflectron design, which required a 20 degree vertical deflection for ions exiting the reflectron to reach the detector, were unsuccessful. The design was modified so that no vertical deflection was necessary. After these changes were made, crude, low intensity time-of-flight spectra were observed for secondary ions generated either by ion beam sputtering or by resonance ionization of sputtered neutrals. Although the initial mass resolution was poor, this was attributed to the short flight tube (25 cm) and the fact that the instrument was not operating in the reflectron mode. Our future efforts will concentrate on improvement of the mass resolution by lengthening the flight tube and implementing the reflectron mode of operation.

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

Organic SIMS

Bombarding a solid or liquid sample of thermally fragile molecules with primary ions of some kiloelectron-volts kinetic energy is an unlikely way to generate gas-phase ions characteristic of the sample. By characteristic is meant that the ions are identical in structure to the parent molecules except for the addition/loss of a proton or electron. Nonetheless, by some mechanism that is not yet clear, organic secondary ion emission does occur, and is the basis for organic secondary ion mass spectrometry. By far the most common use of organic SIMS is for determination of molecular weights of involatile organic compounds and, in conjunction with MS/MS, structure determination. However, in principle, SIMS could be used for determination of the spatial distribution of organic compounds in various matrices, i. e., generation of organic ion micrographs.

In our work, we make quantitative measurements of various parameters involved in secondary ion emission, and then correlate these parameters with measured secondary ion intensity. Parameters we have studied and correlated with secondary ion intensity include analyte concentration, solubility and surface activity, primary ion current density, dose and kinetic energy. Such correlations are useful for elucidating the mechanism by which organic secondary ions are formed (a subject of some dispute), and for the projection of sensitivity of the technique when applied to various analyses. For example, the maximum spatial resolution that can be obtained in an organic secondary ion micrograph is determined by the number of characteristic secondary ions that can be detected from a resolution element on the sample.

By using this quantitative approach, we have been able to demonstrate that it is not the surface concentration that determines secondary ion current, but the concentration at some depth below the surface. This was accomplished by obtaining SIMS spectra of amine/glycerol solutions. Amines are surface active in glycerol; that is, they tend to concentrate as a monolayer on the surface, a tendency which depends on a property known as surface activity. Characteristic secondary ion emission would be greater for compounds that were most surface active if secondary ion emission originated from the surface monolayer. In fact, we were able to demonstrate that regardless of surface activity, the intensity of amine-characteristic secondary ions is a function only of the amine concentration. This result indicates that organic secondary ion emission does not occur from the surface monolayer. Because primary ions penetrate to a depth of less that 100 Angstroms, we conclude that the reactions of secondary ion emission occur from a range of between the second monolayer and the twentieth. Simply put, we have found out where secondary ions come from.

We have further found that for a given concentration, secondary ion emission characteristic of solutes decreases as primary ion current density increases. Because we know that the surface monolayer is not involved in organic secondary ion emission, vide supra, the origin of this effect cannot be primary ion-induced surface erosion. More likely, the primary ion beam induces chemical changes in the region from which sputtering occurs. For example, a reasonable hypothesis is that the primary ion beam creates ions and radicals in solution upon impacting a sample. These ions and radicals react with the solute molecules by a number of reactions, such as protonation, deprotonation, and radical addition. It is known that species which can exist as ions in solution prior to bombardment yield more intense secondary ions than species which are not stable as ions in solution. The effects of primary ion current density on secondary ion emission could be explained if dissolved ions were generated in solution by previous sputtering events. Testing this hypothesis is a problem in chemical kinetics.

One way to investigate the kinetics is by pulsing the primary ion beam. Some organic compounds, such as glycerol, show very high yields of secondary ions, even though they are not ionic. By a series of pulsed experiments using pure glycerol, we were able to show that its secondary ion emissivity is not due to the existence of ions formed by prior bombardment. Pulsed experiments on amine/glycerol solutions, however, yield results different from those of pure glycerol samples and consistent with the hypothesis that the primary ion beam is causing reactions in solution that result in emission of solute-characteristic ions.

From a practical point of view, these results indicate that there is, for any given matrix, an optimum primary ion current density that will yield the most intense characteristic secondary ions. This is because while the relative intensity of analyte-characteristic secondary ions increases as primary ion intensity is decreased, the total absolute intensity of all secondary ions decreases linearly as primary ion current density is decreased. Such practical findings are important for the use of secondary ionization as the source of ions for mass spectrometry. In particular, we are designing a mass spectrometer for determining the spatial distribution of organic compounds in various matrices, such as biological tissue. The transmission of the instrument can be optimized by ion optical methods, so that the sensitivity of the instrument itself will be high. However, unless the emissivity of targeted compounds can be enhanced, the utility of the instrument will be limited.

(Peter J. Todd, Casey C. Grimm)*

*Post-doctoral

Polymer Scintillators

Last year we initiated a project to develop a new class of scintillating compound, the polymer scintillator, that can be used for high-efficiency measurements of alpha and beta emitting nuclides in aqueous systems. Such polymers would differ from all other scintillation materials in two important characteristics. First, the polymer will be designed to be considerably hydrophilic. This property will permit significant infusion of an aqueous phase into the polymer matrix. The effect of this intimate contact (essentially on a molecular level) is that radiation emitted in the aqueous phase will undergo virtually no energy degradation before reaching a scintillating nucleus in the polymer matrix. A second difference is that the scintillating structure will be bonded to the polymer substrate rather than be in the form of a solid solution as seen in conventional plastic scintillators. This bonding will permit efficient energy transfer within the polymer matrix and will enhance both the light output and the overall stability of the system.

The successful development of polymer scintillators that combine these two characteristics will have several major applications that include radiation detectors for liquid chromatography, process control flow-cell monitors for nuclear materials, and in vivo detectors for biomedical radiotracer measurements. In addition, the expected unique physical, chemical, and fluorescence characteristics of these compounds suggest some novel applications in the area of analytical fluorescence spectrometry.

Low-energy emitters exhibit special problems when attempts are made to assay them in flowing, aqueous streams. A scintillation flow cell must fulfill certain requirements for efficient operation. Cell volume must be as large as possible. The cell must be designed so that there is no mixing and no dead space where hold-up could occur. Also, there may be requirements for the shape of the cell and the surface-to-volume ratio. Most importantly, there must be molecular contact of fluorescent centers in the scintillator with the energy deposited by the decay event.

We reported last year our work on a scintillation flow cell that used aligned scintillating-glass fibers rather than the conventional scintillator powder. This work revealed that two important effects were responsible for the low efficiency of powdered cell detectors: the already-described geometry effect and an internal scattering effect caused by the finely powdered scintillator. While smaller particles lead to improved geometry, they also generate increased scatter of the generated photons that results in increased or total internal absorption.

To determine if our idea of developing polymer scintillators was feasible, it was essential that we evaluate the following parameters in a well defined solid-scintillator flow-cell detector.

1. Determine the individual contributions of geometry and scattering and demonstrate that both are major factors in detection efficiency.

2. Experimentally show that if scattering can be reduced to a very small value, geometry becomes the efficiency-determining parameter.

Several studies have been carried out to test these ideas. A new technique has been developed to build polystyrene beads into efficient scintillator components. The polystyrene bead starting material (supplied by S. Alexandratos, University of Tennessee) has some important physical properties that are important to this study. These include a transparent matrix, a smooth, almost polished surface, and a relatively uniform size. We have taken this starting material and converted it to a scintillator using a liquid phase diffusion technique of fluors and solvents. The most successful tests have yielded scintillator beads that match the conversion efficiency of PPO-POPOP liquid systems. Scintillating polystyrene beads in different size ranges have been prepared.

The response parameters of the scintillating beads were evaluated using both alpha and beta excitation. Experiments carried out with ^{14}C beta radiation have confirmed the suggestion that scintillator surface scatter plays a major role in detection efficiency and the shape of the pulse-height spectrum. For beads with smooth, almost polished surfaces, spectral shapes are virtually identical with those predicted by computer simulation. Beads with roughened surfaces exhibit greatly reduced counting efficiencies and significant distortion of the pulse-height distribution. These experiments suggest that optical coupling parameters between the scintillator and the PMT play a limiting role in spectral resolution and detection efficiency.

The alpha response from ^{141}Am of the scintillating beads was compared to computer simulations that considered the geometry of beads to be ideal hexagonal close packed. Three different bead sizes were used. The very interesting result of this work was that although the experimental detection efficiencies were all less than those predicted by the computer model, the pulse-height distributions were about 20% greater than those observed in a high-efficiency liquid scintillator! This observation suggests that there is some localization of the scintillation centers within the individual beads and these centers exhibit enhanced photon emission. It further indicates that polymer scintillators of the type we propose to synthesize should exhibit major detection improvements over conventional heterogeneous scintillator systems.

(H. H. Ross)

International Conference on Modern Trends in Liquid Scintillation Counting

The Analytical Chemistry Division and the Center for Applied Isotope Studies, University of Georgia, will host an International Conference on Liquid Scintillation Counting and Organic Scintillators. The meeting will be held in Gatlinburg, TN, and is scheduled for October 2-5, 1989. H. H. Ross, ORNL, and J. Noakes and J. Spaulding, the University of Georgia, are the conference organizers.

The conclave will feature four days of technical presentations along with a large technical exhibit. The three major manufacturers of liquid scintillation equipment will display their most recent products. Technical sessions are planned for new fundamental developments in fluors, instrumentation, sample preparation, instrumentation, data analysis, and special computer techniques. Applied papers are also being solicited in the areas of environmental measurements, process measurement and control, and new techniques applied to biomedical assay. The proceedings of the meeting will be published in a hard-bound, type-set volume.

It is estimated that 200-300 participants will attend, many coming from foreign countries. The meeting will be dedicated to the memory of Donald Horrocks.

(H. H. Ross)

2. RADIOACTIVE MATERIALS ANALYSIS

J. R. Stokely

The Radioactive Materials Analysis Section performs radiochemical and neutron activation analysis (NAA), chemical and instrumental analysis of radioactive materials, and special development work to support a large number of Laboratory and DOE programs. Research and development is done to investigate new techniques for environmental surveillance of radionuclides.

Over the past two years, activities of two laboratories within the section have been curtailed by the shutdown of ORNL's reactors. Start up of the High Flux Isotope Reactor (HFIR) is anticipated in early 1989 which will result in full operation of these laboratories. A new facility at the HFIR for NAA will be put into operation. Much progress has been made this year in development and utilization of analysis methodology for mixed radioactive/hazardous waste. Extensive characterization has been made of numerous waste tanks containing high-level mixed waste. The section has continued to upgrade capabilities and facilities by acquisition of nuclear measurement instrumentation and renovation of radiochemical laboratories.

RADIOCHEMICAL AND ACTIVATION ANALYSIS

J. F. Emery

The Radiochemical and Activation Analysis Group provides general neutron activation analysis (NAA) services, radioisotopic assays, gamma-ray spectrometry services, oxygen analyses by 14-MeV neutrons, and technical assistance for a number of Laboratory divisions and programs. The HFIR was shut down November 1986 and remained so through the end of 1988. All of our operations at the Oak Ridge Research Reactor have been moved to the new General Purpose Neutron Activation Analysis Facility (GPNAAF) at the HFIR. The new pneumatic tube at GPNAAF is operational and ready to be used when the HFIR starts. Operating, training, and test procedures have been written. The test procedures are for periodic tests of the water sensors and check valves in the air supply and rabbit tubes of both the original and new system.

Because of the HFIR shutdown our NAA service work has been reduced to a very low level. Our work for others (WFO) activities are non-existent with the exception of some radon emanation rate analyses for the U. S. Navy which do not require the use of the reactors. We provided gamma-ray spectrometry, isotope product assays (^{153}Gd), and oxygen analysis of lithium metal by 14-MeV NAA for some ORNL programs and divisions.

Use of A Robot for Automated Sample Irradiation in PT-1

The original pneumatic tube at the GPNAAF (PT1) has a number of desirable features including counter current air flow and exhausts, which allow graphite rabbits to terminate their flight on a column of air. The system contains flow elements which indicate the location of a rabbit in the system and safety features such as a decay station, hot cell, and moisture detectors. To complement these features, we are upgrading PT-1 to allow samples to be irradiated automatically.

In the irradiation procedure for PT-1, specimens are placed into high purity graphite rabbits, which are loaded into an air operated cylinder in the hot cell. When the appropriate irradiation time is selected, the rabbit is inserted pneumatically to the in-core irradiation position. At the end of an irradiation, rabbits are returned either to the hot cell or to a decay station in the reactor pool. PT-1 has been used most often for long irradiations which are typically about four hours or greater and require that the sample remain at the decay station an additional thirty minutes to an hour. In the manual operating mode, only two to three specimens can be irradiated per day. In an automated mode this output likely can be doubled. Furthermore, long irradiations can be initiated on weekends and off-hours, making the system available for other uses during weekdays.

(*L. Robinson*)

Update of the Californium Cold Neutron Source

A seed money proposal to perform a feasibility study of the californium cold neutron source (CCNS) was approved for funding in 1988 and 1989. A group of fifteen scientists and engineers from ORNL are involved in this project. One-dimensional neutronics calculations indicate that if neutrons from ten 50 mg californium sources are first moderated by heavy water and then by liquid deuterium, a cold neutron flux of about 1.4 x 10^9 neutrons cm^{-2} s^{-1} could be obtained. If this flux is not greatly diminished in the beam tubes, then capture gamma-ray analysis and neutron depth profiling are certainly possible. Additionally, medical scientists have expressed interest in the CCNS as a means of providing localized neutron irradiations in tumor therapy.

At present the feasibility study is based upon the CCNS being located in "D" and "E" cells at TURF, but due to lofty cost estimates, alternative locations such as the Bulk Shielding Reactor and the Oak Ridge Research Reactor are being considered. A report of our findings is in the final stages and will be published as an ORNL/TM.

(*L. Robinson*)

Field Measurement of Toxic Inorganic Elements-RMA Project

Assistance has continued in the project to help the Army select and test field analytical methods for measurement of toxic chemicals in soil and water at the Rocky Mountain Arsenal (RMA). We purchased a portable X-ray fluorescence (XRF) system and conducted the necessary testing and calibration prior to field evaluation. An X-MET 840 XRF system was purchased from Columbia Scientific Instruments and received at the beginning of August 1988. The X-Met is being evaluated for its capability to determine the toxic elements copper, arsenic, mercury, and lead. The XRF system consists of a computer-based, multichannel analyzer with 256 channels and enough memory to support eight elemental assay models or 100 identification models for the identification of metals and metal alloys. The system has a probe that contains a radioactive source to excite X-rays in a sample and a gas-filled proportional detector to measure the fluorescent X-rays and provide pulses for spectrum acquisition. The probe we have uses ^{244}Cm as an excitation source and has a proportional detector filled with xenon and carbon dioxide. Energy resolution of the detector is about 15 percent.

Each assay model is built by acquiring spectra of up to ten pure elements and up to 30 standard assay samples. The spectra of the pure elements are kept in the instrument with each model. The elements selected are those that one wishes to measure as well as others present in relatively high and variable amounts in the sample matrix. From the pure element spectra, software of the X-MET determines the channel boundaries (FWHM) of full energy peaks and regions where peaks of different elements overlap. Standard assay samples contain known amounts of one or more of the pure elements in the matrix whose analysis is desired. Following spectrum acquisition of each assay standard and input of the known concentration values, a mathematical relation can be established between the concentrations and the net count rates of the elements. Peak overlap is accounted for in the measurement of net count rates. The model is a multivariate one that takes into account interelement matrix effects, such as the fluorescence of one element in the matrix by the fluorescent X-rays of another and nonlinear effects, such as the absorption of an element's fluorescence by the element itself.

Efforts required at ORNL before the instrument is taken to RMA include determinations of detection limits, Class II certifications of ORNL ICP and AA systems, ruggedness testing of the X-MET, equivalency testing of the X-MET against the ORNL ICP and AA systems and Class I certification of the X-Met. The Class I and II certifications refer to United States Army Toxic and Hazardous Materials Agency procedures and protocols for calibration and data analysis. Ruggedness testing refers to an evaluation of the effects of various factors such as temperature, matrix interferences, etc. on analytical results obtained with the X-MET. Field testing of the X-MET will be conducted at RMA in January 1989.

(*F. F. Dyer*)

The Determination of ^{60}Co in Steel from Hiroshima

We are cooperating in an effort organized by George Kerr of the Health and Safety Research Division and funded by the Defense Nuclear Agency to reevaluate the use of trace amounts of cobalt in steel as neutron fluence monitors in areas surrounding the nuclear explosion that occurred in Hiroshima, Japan, August 6, 1945. The samples supplied to us consisted of a section of an iron railing from a tower of the Chugoku Electric Building, a piece of concrete containing a reinforcing rod from the same building, and a section of a steel beam from the Yokogawa Bridge. The railing was comprised of a bottom plate, two upright rods and a top horizontal rod. The railing, concrete, and bridge specimen were located at

687, 676, and 1295 meters, respectively, from the hypocenter of the explosion.

It was possible by the use of a large low-background germanium detector to easily detect ^{60}Co in the rail specimen, and with some difficulty, to detect ^{60}Co in the bridge specimen. However, the counting efficiency of the Ge detector was too low and its background too high to allow quantitative measurement of ^{60}Co in the bridge specimen; thus, an alternate method for this task was required.

It was found that the ^{60}Co could be determined with good counting statistics by non-destructive counting with pairs of large NaI detectors that were operated in coincidence and shielded with very large plastic scintillators that were operated in anticoincidence with respect to the NaI detectors. Two such detectors located at Battelle Pacific Northwest Laboratory (PNL) and operated under the direction of Dr. R. L. Brodzinski were used for the measurements. Two plates from the bridge and a section from the bottom plate of the railing each weighing about 4 kg and having the dimensions 1.27 cm x 20.3 cm x 20 cm (1/2 in x 8 in x 8 in) were counted on the detectors. Two plates made from a steel specimen produced before World War II were counted to serve as background for the measurements. To standardize the measurements, a traditional radiochemical analysis was performed. A 1-kg piece of the rail plate was dissolved, cobalt chemically separated from a portion of the solution that contained about 670 grams of the iron, elemental cobalt determined, and ^{60}Co assayed relative to a known standard of ^{60}Co. The data from all of these measurements are currently being analyzed.

(F. F. Dyer, J. F. Emery)

Radioactive Contamination in Canada Geese from the Oak Ridge Reservation

Previous studies (ORNL 6474) of Canada Geese residing near contaminated ponds within ORNL have shown that there is a possibility for off-site transport of radioactive materials by these birds. Cesium-137 and ^{90}Sr were found to be the major radionuclides accumulated from pond sediments.

Prior to a planned "round-up" of Canada Geese on the Oak Ridge Reservation for tagging for population studies, a flock of 11 geese were observed to utilize the White Oak Creek basin and the 3500 area settling ponds within ORNL for part of their range. This flock had left excrement on the streets and sidewalks at the rear of Building 4508. To check for possible radioactivity transport by the geese, we collected random samples of scat at several locations in the affected area. All scat samples contained measurable ^{137}Cs with a maximum concentration of 17 Bq/g (dry weight).

Geese from four locations (Y-12, ORGDP, Clark Center Park, and ORNL) were trapped in the "round-up" and tagged. Twenty-six birds from the first three locations were measured in a lead-shielded whole-body counter containing a 15x15-cm NaI(Tl) principal detector with a minimum-detectable concentration limit of 0.04 Bq/g for ^{137}Cs. None of the 26 geese from these locations showed any detectable ^{137}Cs. Following the whole-body count, the geese were returned to their capture location and released.

The flock of 11 geese at ORNL were captured and measured on the same day as the other birds. Whole-body screening measurements showed measurable contamination in the three adult and eight juvenile geese. Subsequent sacrifice followed by laboratory measurement of autopsy samples yielded

^{137}Cs concentrations in muscle of 0.7 to 4.0 Bq/g (wet weight).

The removal of the 11 contaminated geese temporarily solves the problem of radioactive transport from the settling basins. Future plans include some mechanism for preventing access to the ponds by migratory waterfowl. Suggested mechanisms include supported covers for the ponds or the use of streamers to frighten the birds.

As a further check of possible offsite transport, plans have been completed for providing a radiological survey of the goose harvest during the current TWRA managed hunts. This survey will be conducted with the same equipment that is used to monitor the deer harvest. The Canada Geese will be counted in the lead-shielded whole-body counter that will be set up at the TWRA checking station on Bethel Valley Road east of ORNL. It is anticipated that there will be no detectable contamination observed in the goose harvest since none was detected in the "round-up" sampling from four locations on the reservation.

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

Radiological Survey for 1987 Deer Hunts

The radiological surveys for the 1987 DOE-TWRA managed deer hunts were performed by Analytical Chemistry Division personnel assisted by students from both Knoxville College and the University of Tennessee. The hunts were broken down into four separate weekend hunts, with the first two restricted to archers (Oct. 17-18 and 24-25). The archery hunts had 1300 permits for each weekend. The permits were issued in two groups; one group of 300 limited to the "Tower Shielding and Park City Road" areas and a second group of 1000 permits for the other archery areas on the Oak Ridge Reservation.

The first hunt yielded a total harvest of 89 deer and the second hunt yielded 59. The remaining two hunts were held on weekends of November 14-15 and December 12-13. The latter hunts were for shotgun or muzzleloader hunters, and 900 hunter permits were issued for each one. The gun hunts yielded harvests of 252 and 130 deer for the two respective dates. The total harvest for the 1987 season was 530 deer with a 53% male distribution. There was a slight increase in the proportion of bucks in the 1987 harvest compared to that of 1986 (58 vs 56.4%). The age distribution of the harvested deer was similar to that of the 1985 hunts. Deer of 2 1/2 years of age and older had a similar percentage distribution irrespective of sex. One and one-half year old bucks exceeded does by about two to one; whereas one-half year old does were harvested about two to one over the bucks.

Soft-tissue radionuclide concentrations continued to be low and acceptable for the entire harvest. Cesium-137 concentrations were determined in all 530 animals by analysis of a liver or muscle sample using a sensitive gamma-ray spectrometer system. Ninety percent of the harvest contained ^{137}Cs at concentrations less than 20 mBq/g, and only 12 animals contained this nuclide at concentrations exceeding 40 mBq/g (maximum value of any animal was 108 mBq/g). With the bone-checking procedure developed for the 1986 hunts, thirty deer were found to contain elevated levels of ^{90}Sr. These animals were retained from the hunters; however, those hunters were allowed to return to that hunt or to a subsequent hunt. The contamination in the harvest (5.7%) is up slightly from that of 1986 (4.4%).

Following the checking station measurements, subsequent quantitative analyses for ^{90}Sr were performed on bone samples from all of the confiscated animals. Data were collected on deer thyroids from the confiscated animals as well as from other selected animals killed during 1987. Iodine-125, ^{129}I and ^{131}I were detected in many of these thyroid glands. The ^{125}I is known to be released from ORNL operations by an airborne pathway. The 1987 collection confirmed the trend noted from the previous hunts: ^{129}I concentrations are generally higher in the thyroids of those deer containing elevated ^{90}Sr concentrations in their bones than for deer without the ^{90}Sr burden. This observation lends support to the idea that the ^{129}I reaches the environment by a different pathway than does the ^{125}I.

(*J. S. Eldridge, B. D. Keele*)

Applications of Autoradiography in Environmental Monitoring

Our recently established procedure for localization of radioactivity via autoradiography has been applied to leg sections taken from confiscated deer from the recent deer hunts. The purpose of the study was to determine the variations in ^{90}Sr concentrations at various positions in the deer femur in order to verify whether our technique for sampling is optimized.

Comparison of results from two animals showed widely different concentrations of ^{90}Sr in small bones between the femurs and the hooves of the two animals. In one of the animals, the small bones between the femur and hoof had considerably more activity than the femur. In the other animal, the activity concentrations were similar for both the femur and small bones. We can speculate that the deer with the higher concentration in the small bones (an area of bone growth) had a recent acute exposure to the contamination, and the one with uniform distribution was from a chronic ingestion.

A second study involved an autoradiogram of bee pollen collected from hives in Yugoslavia. The pollen is sold in health stores for consumption by individuals who feel that there is special nutrition from the pollen. Chernobyl fallout of radioactive cesium was quantitatively determined on a 30-g sample by low-level gamma-ray spectrometry. It was suspected that there might be non-homogeneity in the sample due to the physical characteristics of the material. (Small individual granules with some color variations). A single layer sample of the 1-2 mm granules was placed in apposition to the autoradiographic plates and exposed for a few weeks. Distinct activity differences were noted in some granules compared to the averages. The differences are probably related to the difference in plant species from which the individual bees collected their nectar. Since it is possible to determine the plant from which individual pollen granules originate, this technique could be applied to determine the most contaminated plant species in case of wide-spread airborne contamination. The bees would therefore serve as environmental sample collectors providing material that would be quite labor intensive for people.

(*J. S. Eldridge, B. D. Keele*)

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. We continued to provide analytical support for the DOE Environmental Survey Program during this past year. We completed analytical work on Brookhaven and Argonne samples and are nearing completion on samples from Idaho National Engineering Laboratory

A large effort was required not only to provide analysis of samples but also to prepare documentation that will help those responsible better to interpret the data. A large part of our efforts was expended toward environmental surveillance programs here at ORNL. The Department of Environmental Compliance continued to increase the number of samples submitted to our group during the past year. Approximately 100 new reservation monitoring wells were expected to be in operation by the end of the calendar year. We have been asked to measure gamma emitters, gross alpha, gross beta, isotopic radium, tritium, and total radioactive strontium in these wells. They will be sampled quarterly and then annually thereafter if no appreciable amount of radioactivity is found in them. About thirty new wells near a waste burial area (SWSA6) were sampled during the third quarter of 1988. These wells will be sampled using the same sampling and analysis design as the reservation monitoring wells. We were part of a program to study the temporary above ground storage of waste at the SWSA6 site before it is covered with dirt and capped as a temporary remedial action. We analyzed samples of rainwater runoff for gamma emitters and gross alpha and gross beta to ensure that no radioactivity leaches from the waste containers before they are covered and capped.

We provided analytical support for the Paducah facility on an emergency basis when ^{99}Tc was found in drinking water from wells that were near the plant. Our results compared very favorably with those obtained by the Paducah Plant Laboratory personnel.

We began a program to better utilize AnaLIS within the group during this period. When a group of samples is logged into AnaLIS, a "HOLD" analysis is requested for the first sample. When the sample analysis is complete, the analyst enters the information into the data management system. A preliminary report is generated when all results have been entered but no data are transferred electronically to customers. The group leader or an alternate then reviews the data and deletes "HOLD" which in turn cues the final report to be printed and the data to be electronically transferred, if appropriate. We installed terminals or PC's in each of our four laboratories to make AnaLIS readily accessible to everyone. We began to better utilize the internal control programs of AnaLIS during the past year. All of our internal standards used for method checks and spiking are now entered into the internal data base where they can be analyzed and plotted. Duplicate and blank information is also being stored in the program. Analysts receive immediate feedback when data are entered that do not fall within the confines of the method. A report of the data is generated weekly and is reviewed by the group leader. New forms were designed using IPRINT to further document the QC information.

Our sample receiving/chain of custody area was renovated during this past year to give us more room and allow us to provide the proper security that many customers now demand for their samples. A doorway was cut between F48 and F50 and a door installed with a push button combination lock. Shelving was installed in F48 to accommodate chain of custody samples as well as routine samples. An area in F48 was allocated for completed samples ready for disposal or return to our customers. Sample login and records were moved to F50. This arrangement has worked well and alleviated very cramped conditions that existed before the renovation.

Several changes were made in our counting room operations during the past year. A quality assurance software package was procured from Nuclear Data that is capable of keeping track of QC data associated with the daily standards that are processed to verify instrument calibration. The software has been implemented to automatically store information regarding gamma detector resolution and efficiency whenever a calibration standard is counted. Calibration data acquired from other instruments are manually entered into the data base. The software gives immediate feedback when a result is entered that is out of control. Plots of the data bases are generated monthly and reviewed by the group leader. An automated sample changer for gamma emitters was purchased and put into daily operation during the past year. The system is controlled remotely by the Nuclear Data 9900 MicroVAX based analyzer system and can accept either water samples in a 500-ml bottle or soil samples in a petri dish. Samples can be counted on a 24-hour basis and the system has functioned very well. A direct drive pump was installed on our alpha counting system; it is very quiet when operating and should increase system reliability.

(L. D. Bible, M. T. Davis, D. L. Dihel, P. S. Gouge, C. C. Granger, R. D. Johnson, S. H. Prestwood, N. A. Teasley, J. W. Wade)

AnaLIS: Status Report

Programs were written to allow ACD to electronically transfer data from instruments into AnaLIS by using file transfer technology. This frees ACD from being "ACD/VAX dependent" and allows operation of instrumentation regardless of the operating condition of the ACD/VAX.

A Purchase Requisition database has been installed in AnaLIS and is now in use.

A 'Holding Time' program has been written that searches the backlog for key analyses and calculates the latest date the analysis may be completed or reported based on Contract Laboratory Protocol, National Pollution and Discharge Emission Standards, or Resource Conservation and Recovery Act guidelines.

The report writing programs have been modified to help eliminate the electronic transfer of data to a customer before the laboratory is satisfied with the results. Now, if the only analysis left on a request after the final results have been added is 'HOLD', the laboratory will receive a 'preliminary' report. Once the laboratory is satisfied with the results and removes the 'HOLD' analysis, the laboratory will receive a final report and the data will be electronically transferred to the customer.

Programs have been written for the Organic Analysis Group to allow the preparatory laboratory personnel to enter sample preparation data directly into the organic database. All personnel in the Organic Analysis Group have access to the data base, but only a few of them are authorized to write data to the data base.

(N. A. Teasley, J. W. Wade)

TRANSURANIUM ANALYTICAL LABORATORY

J. L. Botts

This laboratory continued to supply analytical support to the Radiochemical Engineering Development Center (formerly the Transuranium Processing Plant and TURF facility) and to the monitoring of the radioactive waste effluents for the Environmental and Health Protection Division (EHPD). The group has also provided support to both the Emergency Avoidance Solidification campaign and the Inactive Waste Tank Characterization Program within the EHPD. A total of approximately 40,000 analytical determinations were made during 1988, 30,000 of which were in support of the programs from the EHPD.

Laboratory Improvement and Procedure Revision

A high pressure liquid chromatographic method is used to separate the heavy transuranium elements and other products at the Transuranium Analytical Laboratory (TAL). In this method the sample is introduced at the pump intake, then equal fractions are collected from the column and individually analyzed. In the upgraded method, the pump was replaced, a column heater block replaced a heating tape, and an injector was added to inject the sample into the eluent before the column. An alpha detector was built to monitor the column effluent in order to make more efficient and complete separations. Some analyses have been accomplished with the new setup and a clean separation of each element was achieved. Optimization of the procedure and the separation has been performed after the detector was installed. This upgraded procedure should result in a reduction of the analysis time and better separations of the transcurium elements.

The TAL is now sending analytical data directly (by telephone line) to the Waste Operations Center (WOC) using the ND9900 MicroVax Computer and the Data Management Program (SAM). The SAM program allows the user to edit a file which stores the parameters for each counting detector used at the TAL. These detector parameters are then used by SAM to calculate the activity and experimental error from the counting data entered from the various detectors. Since the data generated from the ND9900 are calculated independently, the data are retrieved and stored with all other data for a given sample. All data for a given sample can be retrieved and edited by the user at any time. When all the data for a given sample are processed and stored, a report is then printed by the system printer and validated by the technician. Only after proper validation are the data sent to WOC. The data are sent from one of the MicroVax printers via a direct line to a printer located in WOC. This data transfer eliminates reporting the data to WOC by phone as was done previously and makes the data input into ACD AnaLIS easier and quicker using the AnaLIS file transfer.

A QA plan for the TAL Group was formulated and issued in November 1988. The purpose of the plan is to provide a disciplined and systematic approach for control of the work within TAL. When implemented, it will become an integral part of the QA program for the Radioactive Materials Analysis Section which in turn will become a part of the overall QA program for ACD. The program as formulated is based on the requirements of ANSI/ASME Nuclear Quality Assurance Standard (NQA-1). The plan will provide for QA within TAL that is in compliance with NQA-1. It is based on nine of the NQA-1 requirements which represents a combination of 14 of the 18 elements listed in NQA-1. Development of the plan required that 12 standard operating procedures either be written or revised. These SOP's include: (1) the use and handling of chemicals, (2) documentation of standards and carriers, (3) sample tracking, (4) sample chain of custody, (5) chain of custody sample storage, (6) chain of custody sample security, (7) sample receipt and inspection, (8) training program, (9) use and care of analytical balance, (10) housekeeping responsibilities, (11) personnel signature and initial records and (12) safety and operating requirements for TAL.

The Emergency Avoidance Solidification Campaign is a program in which approximately 50,000 gallons of liquid low-level radioactive waste stored in the two Melton Valley storage tanks will be removed and solidified. The solidification is being done using a commercial solidification cement designed to prepare the waste for storage. Leach testing was conducted to measure the leachability of ^{90}Sr and ^{137}Cs from the solidified waste. The test specimens were prepared on a laboratory scale to test different technologies that could be used in the actual solidification. Specimens were doped with ^{90}Sr and ^{137}Cs.

Concrete specimens were cylindrical in shape with a diameter of approximately 1.6 cm and length of approximately 4.5 cm. They were leached with a specific volume of demineralized water over a period of 90 days. During that period, a total of eleven leach volumes for each specimen were analyzed. The cumulative leaching times were 2 hr, 7 hr, 1 day, 2 day, 3 day, 4 day, 5 day, 19 day, 47 day, and 90 day. At the end of each leaching interval the specimens were removed from the leachate and immediately immersed in fresh leachant. These leachates were then analyzed for ^{90}Sr and ^{137}Cs. A total of eleven specimens were prepared and tested. The analytical data were used to assist in arriving at a final technique to be used for the solidification.

To meet NQA-1 criteria and better meet the needs of TAL customers, we have continued to upgrade our counting room. A new germanium detector has been installed in the counting room. The detector replaces one which was no longer operable or repairable. The detector has been calibrated for frequently used sample geometries. The ND9900 program for controlling sample counting has been modified to allow saving of daily spectra on magnetic tape without interfering with normal counting routines. We are adding a ND9900 20 megabyte removable hard disk drive to the system to improve data storage. A continuing programing update for the system is being evaluated to improve data reduction and system operation. The system will have a QA package installed to monitor all counting room equipment for normal operation.

Other counting room equipment is being evaluated for possible purchase. All equipment will be PC compatible to allow interfacing with the IBM AT, thus providing a data management system for reporting data to AnaLIS and the Waste Operations Center.

(J. M. Peele, D. L. Dihel, R. F. Peacher, C. Cook, C. R. Cooper, R. A. Jones, B. R. Larkins, R. T. Pack, I. deLeon)

INORGANIC AND PHYSICAL ANALYSIS GROUP

D. A. Costanzo

The Inorganic and Physical Analysis Group provides analyses of intermediate and high-level radioactive materials that require glove box or hot cell procedures in the High Radiation Level Analytical Laboratory (HRLAL). In addition, the group provides irradiation, decontamination, and design basis accident tests on coatings and related materials for nuclear applications for outside clients. During this reporting period, HRLAL processed approximately 2100 samples and performed 5740 analyses, essentially the same sample load as for the previous reporting period. However, the major service support efforts of the group shifted in midyear from predominantly uranium process control type measurements, to work related to characterization of liquid low-level radioactive waste and other measurements for environmental and waste management.

In late April 1988, the Consolidated Edison Uranium Solidification Program (CEUSP) completed its mission with final cleanup operations for the processing of over two metric tons of uranium-233 contained in acidic nitrate solution and conversion of the uranium to a solid oxide form for long term safe storage. This program accounted for approximately two-thirds of all samples processed by HRLAL for the past five years. Chemical and physical measurements employed for process control and characterization of the oxide product were free acid, uranium, nitrate, formaldehyde, density, surface area, particle size distribution, oxygen and metal ratio, and carbon and nitrogen content. At the completion of CEUSP, a concerted effort was undertaken at HRLAL to clean up the glove box and hot cell facilities and dispose of uranium-233 contaminated equipment and waste in preparation of new service support activities.

During the period March-June 1988, several programs were initiated within the Chemical Technology and Environmental and Health Protection Divisions that required the characterization of liquid low-level radioactive (LLLW) waste stored in numerous underground tanks, up to 50,000 gal. capacity, that are distributed throughout the Laboratory. These programs now account for greater than 80% of the sample load for HRLAL. Chemical, physical, and radiochemical measurements have been provided to determine and classify the waste as RCRA, TRU, or mixed and to evaluate options for treatment and disposal of the waste.

The LLLW tanks are classified as active or inactive tanks. The active tanks contain LLLW that has been generated since the early 1970's and are still in use. This waste is typically a high pH (10-13) sodium nitrate solution (3-5$\underline{\text{M}}$) with lesser concentrations of potassium, magnesium, calcium, barium, and aluminum as cations and chloride, sulfate, and phosphate as anions. The major radionuclides contained in the waste are ^{90}Sr and ^{137}Cs at MBq/mL levels and lesser amounts of ^{60}Co and europium isotopes. Greater than 95% of the alpha activity due to uranium, plutonium, americium, and curium has been precipitated and is present in the sludge phase of the waste. The inactive tanks contain LLLW that was collected over the period including the late 1940's to early 1970's. These tanks contain wastes that vary from contaminated rainwater to complex multiphase systems of aqueous and organic liquids and sludge. Where appropriate, EPA methods for solid waste and Contract Laboratory Program (CLP) procedures are being employed for the analysis of the radioactive waste. Over the past few months, the HRLAL has provided assistance to the Chemical Technology Division and DOE to identify leaking irradiation sources from a commercial irradiation facility in Decatur, Georgia. Each of the 250 sources at the irradiation facility contains approximately 50,000 curies of cesium-137 encapsulated in cylindrical stainless steel containers. The sources are stored in a large pool of water which is circulated through ion exchange resins for cleanup and a cooling system to remove heat. In the spring of 1988, ^{137}Cs radioactivity was found in the pool water indicating that one or more of the capsules were leaking. Extensive work was done by ORNL to try to identify the leaking sources. Mass spectrometry, ion chromatography, and radiochemical measurements were made on wipes from the surfaces of suspect capsules to try to identify components leaking from the interior of the capsules. Physical examination and leak testing were done by others at ORNL. One leaking capsule was finally identified by observation of an enlargement (or bulge) of the capsule in tests made in Decatur. This capsule has been brought to ORNL for further tests and for opening for detailed analysis of the contents. Attempts are being made to determine the reason(s) for the capsule's expansion and leakage. The HRLAL will be involved in analysis of the ^{137}Cs salt removed from the capsule as part of this effort.

Several new capabilities have been acquired at HRLAL and facilities upgraded to meet the analytical needs of the various LLLW related programs. An inductively coupled plasma atomic emission spectrometer and an atomic absorption spectrometer have been installed in gloved enclosures to permit measurement on radioactive samples, and HRLAL radiochemical counting instrumentation has been upgraded to state-of-the-art .

(C. J. Baggett, R. N. Ceo, H. J. Hall, L. R. Hall, R. E. Jones, J. M. Keller, M. G. McChung, P. W. Pair, M. E. Peters)

Operation of ICP and AA Instrumentation

A Perkin Elmer Model 6500 Inductive Coupled Plasma Spectrometer (ICP) and a Model 5100 Atomic Absorption System (AA) were put into full operation for metal determinations in radioactive samples. Both systems are contained within stainless steel enclosures that prevent radioactive contamination of the laboratory. The ICP has the torch box within the enclosure with the scanning monochromator located outside the contamination zone. The entire AA system is located within a separate custom enclosure. Both systems have been in operation for several months for use with radioactive samples with minimal contamination of the enclosures.

The ICP system has been used primarily to support projects related to mixed radioactive/hazardous waste in active and inactive waste storage tanks and in various other projects that require metal determinations in radioactive materials.

The AA system includes the Perkin Elmer Model 5100 AA spectrometer, a Zeeman graphite furnace, an autosampler, and a MHS-20 mercury hydride system. Operational standards have been established for the system, and personnel have been trained in the use of the instrumentation. The system is currently in use for high sensitivity determinations of metals in radioactive liquid and solid samples.

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

Upgrade of Radiochemical Analysis Capabilities

A Nuclear Data, Inc., Micro MCA was purchased for use in the alpha spectroscopy system in conjunction with a microcomputer (IBM PC/AT). This second microcomputer/MCA setup also provides backup for the gamma ray spectrometry hardware. A constant-current electroplater has recently been delivered which is capable of plating four samples at a time. The electroplated samples will yield much cleaner spectra than were possible using flamed plates.

Gross beta counting has been improved by replacement of obsolete electronic modules. Tennelec NIM modules have been purchased, including a TC174 preamplifier, a TC246 amplifier/SCA, and a TC952 0-3kV power supply. An ORTEC 974 quad counter/timer completes the package, and allows microcomputer-controlled data acquisition and analysis. The new hardware will initially be used for ^{90}Sr samples and for gross gamma counting.

Quality control/quality assurance techniques have been developed for gamma ray spectrometry in cooperation with the other counting facilities in ACD. These procedures are facilitated by the use of computerized data acquisition and analysis.

(R. N. Ceo, J. M. Keller)

Nuclear Coatings Test Program

We were unable to do any radiation tolerance testing during this period because of the High Flux Isotope Reactor (HFIR) shutdown. Two of our customers (RPM Carboline Co. and Valspar Corp.) had Radiation Sterilizers, Inc., do the radiation tolerance testing. They then shipped the panels and blocks to us for evaluation and reporting.

We did not do any design basis accident (DBA) testing during this period because of upgrading and computerization of the autoclave. This work is almost complete and checkout of the system is underway. This system is state-of-the-art and the only one in this country. Testing for customers has been scheduled for January 1989.

We continued to do decontamination testing during this period for several customers.

(*R. E. Jones*)

Fuel Recycle Division Analytical Support Laboratory

The satellite laboratory located in Building 7602 continued to provide routine analytical services to the Fuel Recycle Division in support of the Consolidated Fuel Recycle Program (CFRP) and the Atomic Vaporization Laser Isotope Separation (AVLIS) Product Purification Program. The primary objectives of these programs are to develop technology for reprocessing of breeder fuel and to demonstrate prototypical CFRP equipment for the separation and purification of uranium from AVLIS produced enriched material, respectively. On numerous occasions the laboratory provided 16-24 hours per day coverage for periods of a week in support of continuous processing campaigns, two of which were successful MMES award-fee milestones. Sample turnaround of 1-2 hours was required during these campaigns to verify on-line instrumentation and for process control. During this reporting period, the laboratory processed over 1000 samples and performed about 3000 analyses, which included free acid, uranium, tributyl phosphate, iodine, oxides of nitrogen, density, and conductivity.

(*P. W. Pair*)

Radiation Safety

During the year, Building 2026 and the 14 MeV neutron generator in Building 2011 were 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. The Transuranium Analytical Laboratory and the Inorganic and Physical Analysis Group within the Radioactive Materials Analysis Section had an average radiation exposure of 105 mR/my compared to DOE maximum allowable exposure of 5000 mR/my. This represents a decrease from 1987, which was 182 mR/yr. Exposures have continued to decrease for the last seven years; however, 1988 can be attributed in part to the shutdown of the High Flux Isotope Reactor. No transuranium production campaigns were conducted at Building 7920 during the year. Personnel working with radioactive materials wear visual dosimeters, and their exposure is recorded daily. They also wear the Los Alamos rad alarm audible chirper and the Panasonic supplemental dosimeter, which detects neutrons as well as beta/gamma radiation.

(*R. E. Jones*)

3. INORGANIC CHEMISTRY

W. R. Laing

The Inorganic Chemistry Section provides a wide range of classical chemical, exotic, and sophisticated X-ray and electron optical analytical services to a variety of ORNL, DOE, NRC, DOD, and outside contractor programs. Environmental analytical support was, by far, the largest segment of work performed during the year. Every staff member in the section was involved in the analysis and monitoring of surface and well waters, soils, sediments, and environmental materials for toxic metals, cyanides, sulfides, asbestos, and other potentially hazardous materials. The DOE Environmental Survey Program provided the largest single segment of support to the section, followed by the ORNL effluent monitoring programs, which ensure compliance with DOE, EPA, and state discharge regulations.

Other important programs to which the section contributed included EPRI Acid Rain, several Remedial Action Studies, the Nuclear Regulatory Commission in the decommissioning of former uranium mines and mills, the US Air Force surveys, the Strategic Petroleum Reserve, and Resource Conservation and Recovery Act and National Pollution and Discharge Elimination System monitoring.

Increased emphasis on quality assurance documentation in response to EPA, Martin Marietta Central Staff, DOE, and internal quality audits resulted in very favorable ratings. The audits did recommend detailed written standard operating procedures (SOP). This resulted in 14 new SOP documents to meet the audit recommendations. The EPA accepted the rapid PCB extraction procedure developed by the section; other improved procedures included microwave dissolution of uranium ore concentrates and fish tissue, and increased use of computers for quality assurance and instrumental data evaluation.

New equipment included a mercury analyzer, upgrade of the ICP scanning monochromator and of the X-ray fluorescence analyzer, and a replacement vacuum evaporator for preparation of electron microscopy specimens.

ENVIRONMENTAL ANALYTICAL LABORATORY

N. M. Ferguson

The Environmental Analytical 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 65,000 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) supply 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 performance evaluation samples from EPA for the Contract Laboratory Program Performance Evaluation were analyzed. During this period we completed all analyses from five DOE sites (Livermore, Sandia, Argonne, Brookhaven, and Idaho). We received aqueous samples (water, wastewater) and solid samples (sediment, soil) from each site. Eleven elements (Hg, As, Pb, Cr, Ni, Sb, Cs, Be, Ag, Se, Tl) were determined in the solid and aqueous samples. Mercury was measured using a cold vapor atomic absorption method. All other metals were measured using a Perkin-Elmer 500 graphite furnace with Zeeman background correction. Five final data packages were also completed during this period. Case narratives, method descriptions, data evaluation, EPA packages, etc. were completed for Pantex, Sandia, Livermore, Argonne, and Brookhaven. All program commitments were met.

Work for the Environmental and Health Protection Division 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.

There was a considerable increase in technical support for the Industrial Hygiene Group. Numerous blood, urine, and air filters were analyzed. We placed into operation a new safety program for the analysis of urine and blood to protect personnel from potential infections. We consider all body fluid samples as potentially infectious.

We received a new mercury analyzer (LDC/Milton Roy/235) this year. It is a high performance recording UV photometer for measuring the absorbance of 253.7 nm radiation by mercury vapor in a gaseous stream referenced to a mercury-free stream. The instrument will detect 0.001 ug of mercury, and full scale deflection of the recorder is attained at 0.005 ug. The capacity of the bubbler tube (reaction vessel) is 250 ml. The analyzer uses the Hatch and Ott procedure to determine mercury. This is the same procedure (oxidized mercury reacts with stannous chloride which reduces mercury to elemental form) we have used for years.

During this year we expanded the usage of the Lachat ion analyzer. The ion analyzer has replaced three Technicon auto analyzers. We have determined ammonia-nitrogen, nitrate-nitrogen, and phosphorus in a variety of complex matrices (KCl leachates, HCl leachates, $HCl\text{-}NH_4F$ leachates). Total phosphorus and total Kjeldahl nitrogen are now measured simultaneously in a single sample aliquot. Ammonia-nitrogen and phosphorus are also measured simultaneously.

The Analytical Chemistry Division (ACD) is attempting to improve the quality of data taken from instruments and stored in its data base. One avenue for realizing this goal is to reduce the manipulation of data by hand operations. During this period a program (FIS_PARSE) was written and implemented to extract desired information from the output of the Lachat flow injection optical spectrometer (FIS) and prepare it for storage in the Analytical Chemistry Division's data base (AnaLIS). It is necessary to review the data before transmitting them to the data base; therefore, direct transfer of results from the computer used to operate the spectrometer was not selected as the method of choice. The data were written in ASCII format to a floppy disc. The data can then be listed and inspected at any work station equipped to read the disc. If the work station is, or can be, connected to the ACD computer system, data may be transferred from the floppy disc to it electronically.

During this time the workload for the graphite furnace has been extremely heavy. Even though two people were involved full time with graphite furnace work, many overtime hours were required to meet program commitments. We use a Perkin-Elmer 5000 instrument equipped with a model 500 graphite furnace (approximately 5 years old) and a Perkin-Elmer 603 instrument with a 2200 graphite furnace (approximately 15 years old) to perform the analyses. The updated features (Zeeman background correction, computer controlled furnace, etc.) of the 500 furnace allows us to analyze samples with complex matrices directly from a calibration curve. This instrument is used to analyze all compliance samples. The 2200 furnace (deuterium background correction) is only capable of analyzing samples with simple matrices and the standard addition method is required. We have now obtained funds to purchase a new computer controlled atomic absorption (AA) system (flame and flameless mode) to replace the older system.

We have investigated, modified, and placed in use several new procedures this year. These include: an improved method to determine Pb in blood; a new method to determine Cd in blood; improved microwave techniques for the dissolution of fish tissue (freeze dried and fresh); several improved flow injection methods.

(*L. S. Barringer, T. L. Baxter, J. D. Mayton, S. A. MacIntyre, M. L. Moore, S. J. Morton, T. R. Mueller, W. R. Musick, T. B. Shope, J. Summers, C. L. Watson*)

CHEMICAL AND PHYSICAL ANALYSIS

J. H. Stewart

The Chemical and Physical Analysis (CAPA) group provides extensive technical support to a multitude of ORNL programs, and to a variety of external organizations including the US Air Force, the Nuclear Regulatory Commission, Strategic Petroleum Reserve, Savannah River Laboratory, and the massive DOE Environmental Survey Program. New methods and modifications of existing procedures were required to meet requirements of these programs, as well as significant efforts to meet the stringent time limitations for completion of the specimens. During this year the CAPA Group received, allocated and distributed, analyzed, and reported 130,000 analyses within the holding time requirements of the Site Survey and the effluent monitoring requirements for the DOE and to the State of Tennessee. The quality level was maintained at an exemplary level of 97.5 percent as measured by the analysis of Performance Evaluation mandatory anonymous soil and water specimens, our own internal standards programs, and by the external quality assurance specimens from the Applied Research Group. The laboratory quality was also measured by numerous formal audits of the CAPA Group by the EPA Region IV, Martin Marietta Energy Systems staff, EMSL/Las Vegas, PEER Corp. (special audit group for DOE), as well as our own internal audits. The audit findings were generally favorable; however, several recommendations were received and acted upon. A significant recommendation required the preparation of 14 separate, formal, standard operating procedures (SOP) to include very detailed daily procedures for responsibility and duties of sample custodian, specimen receiving, distribution, chain-of-custody, data review, data logging, specimen analysis verification, and completed specimen disposal. The required Environmental Survey documentation requirements far exceeded our initial expectations, and additional staff was assigned to meet the contract-required delivery schedules.

The SWSA-6 (solid waste storage area) site evaluation was similar to the larger Environmental Survey program, but was more complex since careful coordination between ORGDP, Bechtel, EDGe Corp.and IT Corp. was necessary. Within ORNL we coordinated work with Engineering, Operations, Waste Management staff and the Environmental Analysis, Low Level Radiochemistry, and Chemical and Physical Analysis groups. The electronic data transfers between IT, ORNL, and Bechtel worked smoothly and the entire operation was completed within budget and also met the stringent Bechtel completion date.

The CAPA Group also provided Dyess Air Force Base with 4000 ICP analytical results as part of their site evaluation. Other external support included analytical methods development for the Strategic Petroleum Reserve (toxic metals measurements by ICP), and NRC analyses for potentially toxic metals in groundwater and aquifers near mining sites being decommissioned.

New instrumentation and procedural development included a method for selectively measuring uranium as UO_2 and as UF_4 in fused salts of Li-Ca-U fluorides; the demonstration of a rapid and complete dissolution of uranium ore concentrate by a microwave technique prior to Davies-Gray uranium measurement; and successful validation and acceptance of the ORNL petition for use of the Soxtec rapid extraction of PCB from soil and sediment in meeting EPA and state regulations. In conjunction with Y-12 laboratory staff we evaluated the ORNL kinetic laser phosphorimeter and demonstrated 50 part-per-trillion measurement capability for uranium in well and surface waters. We placed in operation a low cost X-ray fluorescence quantitative analysis system, and have demonstrated a quantitative compound measurement capability for the rutile-anatase TiO_2 system using the new Scintag X-ray diffractometer. The 1-meter scanning ICP monochromator was used to measure gold, lanthanum, ytterbium, uranium, and yttrium in special samples. The very high precision and accuracy required for our support of the ORNL superconductivity program demanded classical chemical separations followed by gravimetry and electrochemistry to permit compound metal-oxide stoichiometry calculations in the copper-barium-yttrium oxide systems. The value of the hexamethylenedisiloxane (HMDS) vapor transport of fluoride was again demonstrated in the measurement of 10 microgram levels of fluoride in very concentrated LiNaK nitrate solutions. The HMDS technique was also used to measure these levels of fluoride in ceramic Al_2O_3.

We participated in educational and training programs by hosting advanced science students from area high schools, university science students, the ORNL Family Day tours, and with a cooperative 3-month working assignment of two IAEA students. One student was from Venezuela and one from Sri-Lanka; both worked in the areas of X-ray fluorescence and X-ray diffraction.

(*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 SECTION

M. R. Guerin

This section is responsible for a diversified program of research, methods development and analytical services for organic compounds. Research is directed toward spectroscopic and separations techniques that can be used for identification and quantitation of trace organics in natural media. Gas-phase ion molecule chemistry using Fourier transform mass spectrometry and ion trap mass spectrometry are two techniques being studied. New and improved separations procedures have been developed and a cooperative modeling effort to improve fraction collection cutpoints in HPLC separations is under way. A large number of different atmospheric, smoke obscurant, motor exhaust, and military chemical agent studies have continued; these involve both laboratory and field investigations. Closely related are new programs in field-portable instrumentation and mixed wastes characterization. Service capabilities have been upgraded so as to provide better analytical services in compliance with regulatory requirements for characterization of soils, waters, and wastes. Data quality and data usability assessments are now possible under these improved procedures. Determinations of organic constituents in a great variety of sample matrices, including mixed wastes, have been successfully completed and reported using these upgraded methods and facilities.

ORGANIC SPECTROSCOPY

M. V. Buchanan

The focus of this group is the development of spectroscopic techniques for the unambiguous identification of trace organics. Progress has been made in a number of areas this year, including 1) the elucidation of wall-catalyzed oxidation reaction mechanisms under negative ion conditions and analytical applications of these reactions to differentiate parent and substituted polycyclic aromatic hydrocarbons using mass spectrometry, 2) differentiation of isomeric modified nucleotides and nucleosides by laser desorption Fourier transform mass spectrometry (FTMS), 3) development of FTMS-based techniques for the characterization of environmental tobacco smoke particulates, 4) development of techniques for the rapid detection of trace organics in air using thermal desorption processes with an ion trap mass spectrometer (ITMS), 5) development of mass spectrometry-based techniques for the rapid detection of volatile organics in water and soil, and 6) development of a multiplexed version of the multimode ionization chromatographic detector. The group also provides specialized spectroscopic support, including GC/MS, FTMS, FTIR, and FTNMR, to other programs in the Organic Chemistry Section, as well as to other groups within the Laboratory.

Negative Ion Processes for Identification of Polycyclic Aromatics

Work has continued on the investigation of negative ion processes for the differentiation of isomeric compounds by mass spectrometry. We have established that, under negative ion chemical ionization conditions, ions are produced by three different processes, including electron-molecule, ion-molecule, and wall-assisted reactions. Our initial work in this area had shown that electron capture reactions may be used to differentiate isomeric polycyclic aromatic hydrocarbons (PAH) on the basis of relative electron affinity values. More recently, it has been determined that compounds can undergo wall-catalyzed oxidation reactions in the mass spectrometer source to form species which readily capture electrons. These reactions are enhanced by the addition of oxygen and elevated source temperatures.

In a study of over thirty parent PAHs, it was found that depending upon the parent PAH structure, products such as ketones, quinones, and anhydrides were formed by wall-catalyzed oxidation reactions. These different oxidation products allow structural isomers such as naphthacene, benz(a)anthracene, chrysene, and triphenylene, all of which have a molecular weight of 228 to be readily distinguished. Methyl-substituted PAHs also undergo surface-catalyzed reactions with oxygen to form aldehydes, quinones, and anhydrides. Forty-six methyl- and dimethyl-substituted PAHs were studied, and it was found that information regarding the position of methyl groups on the aromatic ring system can be determined using these reactions.

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

---

*Postdoctoral Research Associate

Examination of Modified Nucleic Acid Constituents by Laser Ionization Fourier Transform Mass Spectrometry

The structural characterization of DNA adducts (DNA which has been modified by the addition of an alkyl group, for example) is vital for understanding the nature of carcinogenesis. Modern biochemical techniques can detect the presence of adducts and, when standard DNA adducts are available, chromatographic coelution can be used to identify adducts in a biological mixture. By contrast, the structural identification of unknown adducts is quite difficult, since they are usually present in low concentrations in a complex biological matrix. The sensitivity of mass spectrometry, coupled with the potential of obtaining structural information at the isomeric level, is well suited for the investigation of these adducts. Using the 1064 nm fundamental line from a Nd:YAG laser, we find that methyl-substituted guanosines may be ionized to form anions corresponding to $(M-H)^-$ and to the free nucleic base (elimination of the sugar ring). We have determined that collisional dissociations of these ions yield sufficient information to differentiate all six methyl-substituted isomers, such as O^6-methyl guanosine from 7-methyl guanosine. The fragmentation mechanisms of these negative ions were investigated by accurate mass and high resolution measurements of both the parent ions and daughter ions, by sequential collisional dissociation experiments (MS^n), and by deuterium labelling. Methyl isomers of the other three nucleosides, thymidine, adenosine, and cytidine, have also been characterized. As part of a collaborative project, researchers at the Proctor and Gamble Company have provided standards of ethyl adducts of thymidine and guanosine for study using laser desorption FTMS.

We find that modified DNA oligomers may also be characterized using laser ionization FTMS. For example, the identity and position of the methyl group in the modified hexamer d(N^6mA-T-G-C-A-T) can be determined readily using this technique. The sequence of the oligomer is difficult to obtain, however, due to fragmentation to the individual bases.

Laser ionization has also been used to analyze methyl guanosine isomers directly off of thin layer chromatographic plates. High performance silica gel plates were used with a chloroform/methanol mobile phase for the separation of the isomers. The combination of separation techniques with FTMS will be very important for the analysis of trace DNA adducts in complex mixtures. Research in this area is continuing.

FTMS has also been used to study ultraviolet photo-induced pyrimidine dimers in a collaboration with C.-h. Ho of the Separations and Synthesis Group. Laser ionization yields negative ions corresponding to $(M-H)^-$, which can be used to determine the identity of the dimer. Collisional dissociation of these ions provides several fragmentation products which can be used to determine ring orientations and structure of the pyrimidine dimers.

(*R. L. Hettich*)

Development of FTMS-based Techniques for the Analysis of Tobacco Smoke Constituents

The nonvolatile, high molecular weight components of tobacco smoke have been studied by laser ionization FTMS. The goal of this work was to develop a method to characterize these high molecular weight compounds and compare the compositions of environmental tobacco smoke (ETS), mainstream smoke, and sidestream smoke. The tar samples were collected by J. H. Moneyhun (Special Projects Group) on aluminum disks using either a cascade impactor or an electrostatic precipitator. The latter method was found to coat the disk more uniformly, which is more desirable for laser ionization studies.

Laser ionization of the tobacco smoke tar generates an abundance of positive and negative ions at virtually every mass in the 100 to 600 dalton range. The positive and negative ion profiles for mainstream and sidestream tobacco tar appear to be quite similar, although the sidestream smoke does appear to have a higher concentration of the volatile components. Intense ions for nicotine and alpha-tocopherol are especially prominent in the positive ion spectra. The ETS samples collected so far have been too dilute to characterize.

It has been suggested that solanesol is an important component of ETS. Laser desorption of a solanesol standard yielded an ion at m/z 630 corresponding to $(M-H)^-$, as well as fragment ions. Work is in progress to determine the detection limits for this compound. Future work will also focus the characterization and quantitative determination of high mass compounds in ETS particulate matter.

As part of our efforts to increase the analytical utility of FTMS, software was written to perform neutral loss scans. This software enables real-time display of five neutral losses and five constant losses, with disk storage of the complete daughter spectrum if desired. This software was tested with a home heating oil, which was applied to the tip of the direct sample probe. Alkyl-substituted aromatic compounds were easily identified without chromatographic separation.

(*R. L. Hettich, M. B. Wise, and M. R. Guerin*)

Thermal Desorption MS/MS of Military Chemical Agents

The development of an instrument for the positive identification and quantitation of trace levels of chemical agents in air surrounding demilitarization facilities is nearing completion. This instrument consists of a direct thermal desorption unit which has been designed and built in-house and interfaced to a tandem mass spectrometer (MS/MS). For this project, a Finnigan ion trap mass spectrometer (ITMS) was purchased; a decision based on its superior detection limits over conventional triple quadrupole tandem mass spectrometers. The ITMS was installed during April 1988. Initial tests were performed by interfacing a single thermal desorber (designed at ORNL) to the ITMS via the open split transfer line normally used for GC/MS. The two main requirements of this project, detection limit of 100 picograms of agent simulant and two-minute sample turnaround time, were easily met with this arrangement. Compound identification is based on the ions present in full scan daughter ion MS/MS spectra. Quantitation is performed by integrating the desorption profile using one or more characteristic daughter ion masses.

The final version of the thermal desorption MS/MS incorporates an automated thermal desorber unit with a 16-port autosampler, which can be removed from the unit and reloaded while a second batch of sixteen samples is being run. The automated thermal desorption unit employs an electronically multiplexed design to minimize the number of moving parts and improve reliability. This unit can be operated either from the computer that controls the ITMS or as a stand alone unit which might be used with a gas chromatograph.

The thermal desorption ITMS is currently undergoing final testing with agent simulants. It will be shipped to an Army facility located in Utah early in 1989 where it will be used on a routine basis.

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

New Mass Spectrometric Techniques for the Rapid Determination of Organic Compounds in Water and Soils

The development of a rapid method for the direct determination of trace organics in water and soil has continued. The Finnigan 3200 quadrupole mass spectrometer fitted with a concentric cylindrical glow discharge ionization source was interfaced with an IBM AT computer, and complete software was written for data acquisition, storage, display, and quantitation. Earlier work with trace levels of volatile organics, including benzene and trichloroethylene, in water was repeated, and additional volatile compounds were examined, including perchloroethylene. Some semivolatile compounds, such as phenol and nitrobenzene, were also tested. Detection limits for volatiles in water were found to be between 1 and 10 ppb, while the semivolatiles were substantially higher. In addition to excellent detection limits, this method has the advantage of rapid sample analysis (2 minutes per sample) and good reproducibility (± 10%). A new mass spectrometer system is being purchased to upgrade the present instrument and improve its reliability. In addition, this instrument will employ an off-axis multiplier (which should reduce present noise levels) and will be able to detect both positive and negative ions.

Some scoping experiments were also performed using an ion trap mass spectrometer (ITMS). Volatile organics in water were purged directly into the open-split interface of the ITMS using helium at a flow rate of 100 ml/min. Using this approach, detection limits for benzene, toluene, trichloroethylene, and perchloroethylene were in the range of less than 1 ppb to 10 ppb. Calibration curves were linear from one to 100 ppb, with correlation coefficients of 0.98 or better. The reproducibility of the measurements was determined to be typically ± 10% (at 100 ppb) to 20% (at 1 ppb) at the 95% confidence interval. In addition to having detection limits for volatiles in water similar to that of the glow discharge mass spectrometer, the ITMS has added capabilities of collisional dissociation (MS/MS), selective chemical ionization, single ion storage, and rapid clear-down time. Sample turnaround time was typically 2 minutes.

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

Multiplexed Multimode Ionization Detector

New multiplexing electronics for the multimode ionization detector (MMID) were designed and built; these allow chromatograms from all three detection modes to be obtained in one chromatographic run. The MMID is operated at a reduced pressure, and the three different modes (electron capture, argon ionization, and selective) are determined by the intervals between detection pulses. Three electrometers, one for each detection mode, are sampled for a specified interval (for example, 0.3 seconds), and a sampling trigger generated by the multiplexer flags the computer interface to collect a sample.

Menu-driven software has been written for the acquisition of data from the MMID. The capabilities of this software include real-time color display of all three detector responses (updated every five seconds), individual gain and range controls for all channels, color hard copies, storage of data files and foreground/background data aquisition and manipulation. At present, the computer does not control the instrument itself. Work is under way to allow computer control.

(C. C. Grimm, M. B. Wise, and M. V. Buchanan)*

Special Spectroscopic Studies

This year, a wide variety of specialized studies were performed for projects within the Organic Chemistry Section and for researchers in other divisions. The majority of these studies employed GC/MS, although an increasing number used FTMS, as well as NMR and GC/FTIR.

A project for the Department of Defense involving the characterization of combustion products from colored smoke grenades was completed. An additional red grenade, composed primarily of α-methoxy-azo-β-naphthol (MBN) and 1,4-diamino-2-methoxyanthraquinone (DMA), was combusted because prior lots of red grenades did not produce typical bright red smokes. Combined GC/MS was used to characterize both the particulate matter and volatile organics from the combusted red grenade smoke. In some cases, additional confirmation of compound identity was obtained using GC/FTIR, ^{13}C NMR, and FTMS. The major combustion products identified included 2-methoxyaniline, 2-naphthol, naphtho(1,2-d)-oxazole and methoxyphenylnaphthol, all of which presumably arise from the decomposition of DMA.

Thermal desorption GC/MS was used to identify volatile organics in air samples in support of several aerosol generation studies being conducted in this

---

*Postdoctoral Research Associate

section, including the combustion of red phosphorus rubber mixtures and emissions from rocket motors. In addition, conventional electron impact and negative ion chemical ionization was used with GC/MS to determine the quantities of nitroaromatics and polycyclic aromatics from rocket engine exhaust trapped on filter pads.

Other GC/MS studies were performed for researchers in Health and Safety Research, Chemistry, Chemical Technology, Environmental Sciences, and Biology Divisions. In addition, the ability of FTMS to analyze high mass and nonvolatile materials and to establish exact elemental composition has been utilized frequently this year by researchers in other divisions.

(*C. Y. Ma, R. L. Hettich, R. H. Ilgner, and M. V. Buchanan*)

SEPARATIONS AND SYNTHESIS

W. H. Griest

The focus of this group is the development and application of organic chemical separations and synthesis methods to environmental and health issues. Programmatic support is provided by the U. S. Department of Energy Office of Health and Environmental Research; additional support comes from services to other ORNL programs and investigators. Facility management also has been a part of the group's activities.

Programmatic Methods Development

A major part of the group's work in the past year involved the separation and determination of modified nucleic acid constituents, particularly DNA photoproducts. Purine dimers are formed as a result of the action of UV irradiation upon skin DNA, and some of these photoproducts are thought to be involved in the development of skin cancer. We are developing sensitive procedures for quantitating photoproducts at physiologically significant UV exposure levels. Cyclobutane dimers of thymine, uracil, and a mixed uracil-thymine dimer have been photochemically synthesized and isolated, and methods for hydrolytic liberation of the dimers from DNA have been adopted. In the development of a multidimensional chromatographic method for determining these compounds as their hydrolyzed nucleic bases, an off-line HPLC-GC/ECD method has been devised. Reverse phase HPLC has been used to separate the dimers from their monomers in DNA hydrolyzates for collection and electrophore labeling. GC/ECD of the pentafluorobenzyl derivatives allows detection limits in the 1 to 5 ppb range (1 to 5 fmol injected), which is almost two orders of magnitude better than that considered necessary for study of physiologically significant UV exposures. The HPLC separation of the photoproduct dimers from the monomers is being modeled in a collaborative study with George Guiochon's group. From a practical standpoint, the model will allow the prediction of the optimum dimer fraction collection cutpoints under severely overloaded monomer concentrations. Work on the development of online HPLC/MS detection with the Analytical Spectroscopy Section and the structural identification of modified nucleic acid components has been completed. An atmospheric sampling glow discharge ionization source was designed and evaluated using nucleosides and nucleic bases. MS/MS spectra were demonstrated. Flow rates up to 0.3 ml/min can be accommodated, which is well within the range of microbore LC.

The separation and detection of high explosives compounds by packed column supercritical fluid chromatography has been investigated for the first time. Seven high explosives or manufacturing byproducts have been efficiently separated in 20 min using carbon dioxide and a bonded cyano phase packed column. Determinations have been made at ca. 0.2 ppm with UV absorbance detection. Because the surface activity of the silica particles apparently limits the application of packed column SFC, capillary separations are being investigated to extend the elution range.

(*R. S. Ramsey, C-h. Ho, K. H. Row,* B. M. Dittmar,** C. Guzman,*** and W. H. Griest*)

Methods Development and Application for Organic Analytical Services

A large part of this group was involved in methods development and applications in support of other ORNL investigators and programs. Methods for the determination of high explosives, petroleum hydrocarbons, and soil gases were improved and adapted to conform to EPA Contract Laboratory Program (CLP) requirements for the DOE Environmental Survey program. A particularly important part of this adaptation was the inclusion of rigorous QC measures such as careful documentation and extensive reporting in the EPA CLP format and the incorporation of spikes, blanks, replicates, and control samples in the protocols. Eight high explosives are determined in water to ca. 0.2 ppm and in soil to ca. 1 ppm with generally very good spike recoveries (≥ 80%) using reverse phase HPLC with UV detection. Several hundred samples were analyzed for both ORNL and other DOE laboratories participating in the program. Fuel contamination of water and soil are determined with limits of detection of 10 ppm and 100 ppm, respectively, by solvent extraction and capillary GC with flame ionization detection. The spike recoveries are generally ≥ 90%. The procedure also identifies the type of fuel (e.g., gasoline, jet, or diesel fuel), a capability not offered by the EPA infrared analytical method. Soil gases collected in solid sorbent tubes are tentatively identified and measured by thermal-desorption capillary column GC with flame ionization detection. The limit of detection is 7 to 58 ng of compound per trap. Considerable difficulties were overcome in achieving retention time and peak area reproducibilities of 0.2-0.8% RSD and 12-37% RSD, respectively.

Modification of EPA SW-846 methodology was made to allow the determination of regulated organic compounds in mixed waste samples from nuclear storage tanks. Procedures for purging volatiles from waste liquids and extracting semivolatiles from liquids and sludges in a hot cell, glove box, or contamination zone hood, and for direct aqueous injection GC analysis of common solvents in radioactive liquids, have been adapted from SW-846 and applied to nuclear waste tank programs. The extracts from highly radioactive sludges and liquids can be analyzed by EPA methods in conventional chemical laboratories with a minimum of precautions. Surrogate and matrix spike recoveries generally have been good.

A method for the determination of formaldehyde in water at ppb levels has been developed, evaluated and applied to numerous samples in response to a U.S. Navy program's needs to determine water contamination resulting from polyacetal plumbing fittings. A method was developed for collecting and preserving formaldehyde as the dinitrophenylhydrazone

---

*Postdoctoral Research Associate

**ORAU student research participant, Duke University

***HBCU student research participant, Catholic University of Puerto Rico derivative and for solvent extracting it for measurement by HPLC. The sensitivity is ca. 3 ppb and is limited mainly by the blank level in reagent water. Matrix spike recoveries over the range of 60 to 180 ppb are ≥ 90%, and the stability of spiked samples is very good. More recent work suggests that a solid sorbent resin may be able to replace the much more laborious derivatization and solvent extraction method.

The leaching of uranium from Y-12 waste streams was studied to provide an input for the environmental impact statement for the low-level waste disposal program. We subjected four batches each of four Y-12 wastes to the EPA Toxicity Characteristic Leaching Procedure and the concentrations of uranium and several RCRA metals in the leachates were determined by the Inorganic Chemistry Section. Differences in the leaching behavior of uranium among the wastes were observed, although inhomogeniety in the waste composition (even for a given waste stream) complicated the interpretations.

(B. A. Tomkins, R. Merriweather, J. M. McMahon, W. M. Caldwell, V. S. Ong,[*] *M. E. Garcia,*[**] *and W. H. Griest)*

Facility Management

The Synthetic Fuels Repository is being decommissioned upon instructions from DOE because of concerns that aging of the fuels will render them nonrepresentative of their production processes and because of the current lack of interest in synthetic fuels. In the past year, the sample handling laboratory was decommissioned, and a study of disposal options for the synfuels collection was initiated. The capabilities of the Hazardous Materials Analytical Laboratory at Building 2026B were improved by additional staff and instrumentation. One laboratory room was contamination-zoned, and two GCs were installed for mixed waste analyses. The air conditioning system to the large instrument lab was supplemented with an auxiliary cooling unit, and the compressed gas tank farm was improved by the construction of an empty tank rack and concrete pad.

(W. H. Griest and B. A. Tomkins)

SPECIAL PROJECTS

R. A. Jenkins

The generation, sampling, instrumental monitoring, and chemical characterization of controlled and/or specialty atmospheres continues to be a major focus of this group. There is also a field environmental analytical component of its efforts. In general, funding for the group continues to be issue-oriented, originating predominantly with the Department of Defense and the National Institutes of Health.

Tobacco Smoke Studies

The characterization of environmental tobacco smoke (ETS) has continued as a major activity this year. A small interior office, with modified ventilation to simulate that achieved in more conventional office space, is being used to contain test atmospheres of ETS. Simulated ETS is generated through the use of one or more smoking machines, puffing at controlled

---

[*] GLCA/ACM student, Coe College

[**] Environmental Safety and Health Protection Division.

rates. The room can be monitored with several real-time or near real-time instruments, in order to document the composition of the atmosphere being tested. The focus of much of the experimental work being conducted in this test room has been to determine the organic vapor phase composition of ETS under conditions where no other combustion or noncombustion sources can confound interpretation of the results. The results can then be compared with indoor air compositions encountered in more natural environments. In this way the contribution which ETS makes to indoor air pollution can be more accurately estimated. For example, during the past year, the concentrations of a number of volatile organics, including nicotine, benzene, toluene, isoprene, butyronitrile, pryrrole, propionitrile, and vinyl pyridine were measured in test atmospheres contaminated only with the tobacco smoke. Typically, the concentrations ranged from 1 to 10's of $\mu g/m^3$ for atmospheres heavily contaminated with ETS. The relative ratios of selected marker compounds to other organic volatiles in the test environments were calculated. The ratios obtained in test atmospheres were compared with those obtained in real environments, which had been contaminated with ETS and other indoor air pollutants. For compounds which arise nearly exclusively from tobacco related combustion, the ratios obtained in real indoor air samples were similar to those obtained in test environments. In contrast, for those constituents of indoor air likely to have multiple sources (such as benzene, styrene, and toluene), the ratios determined in real environments were considerably different from those obtained in test environments contaminated only with ETS. Determination of such ratios can act to establish the relative contribution of ETS to indoor air pollution. The effects of ETS aging and concentration on the marker/constituent ratios are now being examined.

The physical characteristics of ETS are also being examined. The size distribution of the ETS particulates is an important characteristic, since it ultimately determines the site in the lung where the material will be deposited and the fraction of the inhaled particles which is likely to be retained. In order to determine the size distribution of such particles, we have been re-examining the use of electron microscopic (EM) techniques. Previous studies in our laboratory showed that methyl cyanoacrylate (MCA) vapor fixation of mainstream cigarette smoke droplets was required to prevent volatilization of the collected material during EM processing and viewing procedures. However, ETS droplets, given their long residence time in the air, would be expected to have most of the volatile material already evaporated out of the droplets, making the remaining droplets much less susceptible to further evaporation during EM procedures. Indeed, experiments performed with a dotriacontane aerosol (thought to mimic the "waxy" ETS droplets) demonstrated that small particles could be observed following EM processing without prior MCA fixation. Therefore, we have been examining the use of filtration and electrostatic precipitation as a means of concentrating ETS particles within a viewable field. Results to date have been mixed. A few particles much smaller than 0.1 μm have been observed. However, we are unable to prove that these are in fact ETS droplets. The fabrication of a continuous concentrator to acquire much larger samples is now being considered, because the relative fraction of viewable surface area that a small ETS particle would cover is exceedingly small.

Our initial development and validation of a personal sampler for ETS related nicotine was completed early in the year. The monitor consists of a small personal sampling pump drawing ambient air through a Tenax cartridge. The cartridge is subjected to triethylamine-assisted thermal desorption, followed by gas chromatographic analysis. The system, which has a detection limit in its current sampling configuration of 0.07 $\mu g/m^3$, was used to determine ambient nicotine levels in selected area restaurants, as described in last year's Annual Report (ORNL-6474). Discussions are under way with project sponsors to consider investigating the extension of this personal monitor to important indoor air vapor contaminants other than nicotine.

(C. E. Higgins, J. H. Moneyhun, C. V. Thompson, M. R. Guerin, and R. A. Jenkins)

Military Atmosphere Studies

Our aerosol technology program has also included projects related to the generation and characterization of test atmospheres with military purposes. During the past year, our work has had both an experimental and technology assessment component. We have completed development and characterization of a laboratory scale generator for a military biphasic obscurant aerosol. One phase is generated from a petroleum-based liquid (PBL) 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 inhalation exposure. Experiments indicated that the system can deliver the PBL with about 90% efficiency and the solids with about 50% efficiency over a range of 0.1 to 10 g/m^3. UV spectrophotometry of the PBL indicated an increase in absorption near 255 nm with aerosolization, but detailed chromatographic characterization suggested little change in the relative composition of the major fractions of the liquid aerosol. This suggests that any chemical transformation occurring as a result of aerosolization probably occurs in those components that are not chromatographable. We have constructed five aerosol generation systems and are preparing to install them in a bioassay laboratory off-site.

Characterization of exhaust from test scale rocket motors from hand-held anti-tank weapons systems has been proceeding. The purpose of the effort is both to provide a data base for health risk assessment and to input into improved computer model projections of exhaust products. For this study, the concentrations of both major and minor vapor and particle phase constituents are being determined. Because many of the potential species are reactive, real-time monitoring systems to determine levels of particulates, ammonia, oxides of nitrogen, hydrogen cyanide, carbon monoxide and carbon dioxide, and hydrogen chloride are used. Analytical methods validation experiments are being performed using free burns of small quantities of the candidate propellants in a small chamber at Building 5507. Samples for the actual characterization studies are collected from firings of scaled down test motors in a 19.6 m^3 chamber at the Army's Signature Characterization Facility (ASCF) at Redstone Arsenal in Huntsville, Alabama. One sampling trip was made to the ASCF in late June. Two propellant systems were fired and subsequently sampled. A new double-stage filtering system was fabricated at ORNL and employed successfully to collect a much larger fraction of the particulates generated from the firings. These concentrations ranged up to 175 mg/m^3. A perchlorate-based propellant produced exhaust which, when diluted with clean air in the chamber, was less than 1 ppm HCl and about 300 ppm CO. A nitroglycerine/nitrocellulose propellant produced comparable quantities of

CO, but five times as much CO_2. We are moving into a new phase of the project, in which we will be interacting with staff from the Computing and Telecommunications Division. Data generated from experimental studies will be used to refine existing predictive models, so that more accurate estimates of the concentrations of hazardous substances in the exhausts can be made.

A major technology assessment and strategy development effort was recently completed for the Army's Biomedical R&D Laboratory, in which the criteria for a facility to generate, characterize, and expose animals to the exhaust products of large guns and rifles were defined. A number of approaches to the controlled generation of gun exhaust atmospheres were considered, including gun simulators, closed bombs, and mixtures of toxic constituents. The most practical approach and the one that will likely generate the most relevant atmosphere was determined to be the use of real guns firing actual or scaled-down ammunition. The exhaust, breech- or muzzle-only, or a combination of the two, would be handled by a collection/distribution system (CDS). The CDS is designed to provide for a continuous concentration atmosphere for inhalation exposure and chemical characterization studies, despite the fact that the gun firing is an inherently intermittent phenomenon. It would accomplish this by using a continuously variable, programmable gas proportioning system. Important aspects of the facility were also addressed, including the limiting toxic constituents, noise, spent projectile handling, safeguards and security, and staff requirements. Recommendations have been made to the Army in the form of a draft final report.

Sampling and analysis methodology for the determination of chemical warfare agents in incineration stacks, workplace and ambient atmospheres has been reviewed and assessed for the Office of the Program Manager for Chemical Demilitarization. Such an assessment has wide ranging implications for the determination of many airborne semivolatile organic species, since many of the problems associated with agent sampling are generic to other organic substances. The assessment indicated that the agents, in the case of incineration failure, would be present in the stack dissolved in a light aerosol of phosphoric acid. The current sampling systems (coarse filters followed by resin filled vapor adsorption traps) would thus be challenged by small droplets, which they were never designed to collect. This situation could result in a substantial underestimation of the amount of the chemical agent released into the environment. A relatively straightforward solution to this problem has been proposed to the Army: installing a filter which has a high collection efficiency for submicron droplets upstream of the existing sampling train. A series of validation experiments designed to determine the efficacy of the approach has also been proposed.

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

---

*Instrumentation and Controls Division

Environmental Studies

The other major component of the group's activities is directed toward field analysis, the goal being to reduce reliance on (or replacing) laboratory analyses of environmental samples. We have moved into the experimental phase of a project in support of the environmental restoration of the Army's Rocky Mountain Arsenal (RMA). Essentially, the mission is to evaluate the utility of selected commercially available analytical instrumentation for use under actual field conditions. A detailed experimental plan was developed to evaluate a portable gas chromatograph (GC) for the determination of volatile organics in soil and water, and a portable X-ray fluorescence (XRF) unit for the determination of selected toxic metals. The portable GC selected has a built-in air sampling pump and resin cartridge, so it was relatively simple to modify the system for purge and trap analysis of soil and water. Unfortunately, although a number of evaluation studies have indicated that the constituents of interest could be quantified with relatively low limits of detection using a photoionization detector, the instrument appears to be insufficiently reliable from a mechanical or electronic standpoint to be of utility in this particular application. The portable XRF system evaluation has been performed jointly with the Radioactive Materials Analysis Section. The goal has been to determine if the system can be used effectively on the wide variety of sample matrices likely to be encountered at RMA. Early results from our laboratory studies appear promising. Limits of detection for copper, arsenic, lead, and mercury have been determined to be 25, 12, 60, and 29 ppm, respectively, in wet RMA soil. We expect to test the performance of the system under actual field conditions at RMA early in 1989.

This work for the Army has led to the initiation of a small project for DOE. The purpose of the work is to develop rapid sample processing techniques, which can be utilized in the field for the more timely determination of semivolatile organics in soil and water, and the use of colorimetric indicator tubes for the semiquantitative screening of volatile organics in environmental samples. The experimental phase is scheduled for the beginning of 1989.

Another small project involving aerosol technology and environmental analysis consists of the development of an aerosol concentration inlet for an atmospheric pressure ionization source mass spectrometer. This would be used for the determination of semivolatile organics in water samples. The device functions by directing an aerosolized stream of the sample against a countercurrent air stream. The water has a higher volatility than the semivolatile, components, therefore, the droplets tend to become enriched in the organic species. At the maximum enrichment point, the droplets can be skimmed into the ionization source. Early evaluation experiments using the device appear promising.

(*R. A. Jenkins, F. F. Dyer, R. L. Moody, C. V. Thompson, and J. H. Moneyhun*)

Laboratory Upgrading

We have successfully relocated our rocket exhaust characterization chamber and associated instrumentation to Building 5507. Our ultimate goal is to have the laboratory functioning as an aerosol technology facility. We have acquired a new, near-real-time particle mass concentration monitoring system, a Tapered Element Oscillating Microbalance (TEOM), and have integrated it into our rocket exhaust characterization studies. One of the prime advantages of the TEOM is that it can be directly mass calibrated with the material of interest, and a sample of the ambient particulates measured can be collected on a filter and subjected to chemical analysis. We have also acquired a new portable GC with photoionization and electron capture detectors. We have added an electrochemical HCl monitor and a nondispersive infrared CO_2 analyzer to our suite of real-time monitoring instrumentation. Also, we have upgraded our data handling capabilities through the recent acquisition of a multichannel data logging software package.

(R. A. Jenkins, C. V. Thompson, and J. H. Moneyhun)

ORGANIC ANALYSIS

J. E. Caton
M. P. Maskarinec

The Organic Analysis Group works on the qualitative and quantitative determination of organic compounds in a wide variety of sample matrices. The great majority of the samples are either environmental in nature or are associated with waste disposal. Multiple compounds are usually determined at trace levels, (parts-per-million to parts-per-billion and even lower for pesticides and polychlorinated biphenyls), in any given sample. 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 almost entirely from the services provided.

The primary organic analysis effort can be characterized as consisting of four subefforts: (1) receiving, logging, and distributing samples; quality controlling and reporting results; and administration; (2) preparing samples for analysis; (3) analysis utilizing gas chromatography/mass spectrometry; and (4) analysis by gas chromatography. In all these areas, personnel and/or equipment capabilities have been expanded significantly during the past year. We have established the capability to determine herbicides in environmental samples. In addition, we are in the process of establishing Appendix IX methodology (as required by 40CFR part 264, July 1986) for volatile and semivolatile organic compounds. Much effort has gone into data quality reviews and data reporting in support of earlier work to assist in preparing major environmental survey reports. The increased capacity and efficiency now available will be demonstrated as personnel complete their assignments to data review activities.

Progress in Receiving, Reporting, and Quality Control

The organic analysis effort is highly dependent on sample handling, data processing, and quality control. This activity has been expanded and personnel have been committed to the effort on a full-time basis. In addition to increasing the person-power in this area, both space and equipment have also been increased. Formal procedures for tracking samples have been upgraded so that a paper track is now updated on a daily basis to indicate the exact sample status. Ultimately our goal is to have the current status of each sample immediately available on the division data management system. However, such a tracking system requires some refinement and the participation of all segments of the Organic Analysis Group because a single sample may involve the determination of more than 100 specific components with all the subefforts in the Organic Analysis Group participating. With the increased commitment of personnel such a complete tracking system for organic sample status should be established in 1989.

During the past year specific office space and a full time staff member has been committed to this function. It is anticipated that a second staff member will be added as an assistant. With this expanded staff increased emphasis will be placed on quality control and enhanced customer interactions. In addition to these increased staff capabilities, improvements in sample distribution have been instituted. All samples are now formally grouped so that they can readily be associated with the proper quality control samples. The samples in these groups along with the controls, (blanks, spikes, etc.) are prepared and analyzed at the same time thereby facilitating assessment of data quality. Reporting capabilities have also been enhanced by having one or more personal computers available for each of the separate laboratory efforts, (preparation, gas chromatography, and gas chromatography/mass spectrometry) to communicate directly with the division's data management system. Thus, personnel in each of these laboratories can directly acquire sample information and add results as they are generated.

Much extra effort was devoted to reporting results during this year because of the extensive reporting requirements for the large consignment of samples associated with the Department of Energy Environmental Survey. The reporting activity required more person-hours of effort than did the analyses! Not only were complete data packages, as specified by the Environmental Protection Agency Contract Laboratory Program, assembled for nearly 1500 samples, but the same data were also prepared in corrected ASCII files for use in report preparation. After these reports were prepared, both data quality assessments and data usability evaluations were made on all results. Although this reporting task required approximately six person-years effort, the Organic Analysis Group derived noteworthy benefits which should enhance future efforts. The capability of assembling data packages, assessing data quality and usability, writing narratives concerning analytical problems and quality control for a given set of samples, and proofing results critically is now quite widespread within this group.

(*J. E. Caton, L. J. Wachter, M. P. Maskarinec, S. F. Glover, S. H. Harmon, and L. H. Johnson*)

Developments in Sample Preparation

The sample preparation effort is currently staffed by three technicians who are continually improving their skills. Hardware additions to this function have been minimal during this year; however, several significant improvements have been instituted. One small but very important improvement has been to move the final clean-up and concentration of pesticide samples to a separate ("clean") laboratory. This step has aided greatly in reducing interferences often encountered by the high sensitivity electron capture detectors employed in the analysis of pesticides and polychlorinated biphenyls. Another step that has improved the preparation of pesticide samples is the addition of mercury to prepared pesticide samples before analysis to remove any elemental sulfur present in the prepared extract. Electron capture detectors are quite sensitive to sulfur and many environmental samples contain elemental sulfur, which tends to follow the pesticides and polychlorinated biphenyls through the preparation process.

The preparation of herbicide extracts from either aqueous or solid samples, (soil, sludge, etc.) has been added to our preparation capabilities. Although the extraction of herbicides is not very different from the procedures employed for semivolatiles or for pesticides and polychlorinated biphenyls, the final preparation is much more complicated and represents a substantial increase in our routine capabilities. Because many of the common herbicides are fairly low molecular weight acids they must be derivatized before analysis by gas chromatography. Following concentration of the initial acid extract this process consists of a chemical reaction to prepare the derivative, extractive cleanup, drying, and concentration to a final known volume.

With the help of the data management system coordinator, the capability now exists on the division data management system to directly record all preparation data as it is generated in the laboratory. Although a written record of the preparation must still be maintained, this capability negates the necessity of passing multiple copies of such records to the laboratories where the preparation information is incorporated into final analytical calculations. As a consequence such records are not misplaced and transcription errors are minimized.

The preparation effort is located in 4500S where good facilities including continuous liquid-liquid extraction, separatory funnel liquid-liquid extraction, ultrasonic extraction, preparative chromatography, tissue extraction, soxhlet extraction and various methods for solvent removal, extract drying, and concentration are available. We can now process in excess of 50 samples per week for short periods with the capability being approximately 30 samples per week on a continuing basis.

(J. E. Caton, G. M. Henderson, D. D. Pair, and C. D. Vamadore)

Developments in Gas Chromatography/Mass Spectrometry

The routine gas chromatography/mass spectrometry effort is presently staffed by three persons. Four instruments, which are maintained on vendor service contracts, are available. The vendor services these contracts quite well and downtime has been minimal. Two instruments are equipped with packed columns and are devoted to volatiles analysis; the remaining two instruments are equipped with capillary columns and are used for the determination of semivolatile organic compounds. Peak capacity for this laboratory approaches 50 samples per week for semivolatiles and 70 per week for volatiles; the continuing capacity is 30 samples for the analysis of semivolatile compounds and 45 samples for the analysis of volatile compounds.

Hardware improvements in gas chromatography/mass spectrometry have been significant during this year. A central computer that can receive files from all instruments is now functioning. This greatly facilitates the processing of data and the formatting of results. In addition, improved equipment interfacing the instruments devoted to semivolatiles with this new computer has been installed. This hardware allows a sequence of samples to be analyzed without operator intervention with the data being directly reported to the central computer as it is generated. As a result, initial data manipulation and calculations can be completed in the operator's absence. The full capabilities of this system have not yet been tested, but it should contribute significantly to an increase in efficiency.

During this year there were significant personnel changes in the gas chromatography/mass spectrometry effort. Our most experienced operator accepted employment elsewhere leaving the present staff with only one individual with more than two years experience. Thus, training has become very important. Accordingly, two staff members attended different training sessions offered by the instrument vendor. Two other staff members attended mass spectral interpretation classes. Although such training will continue as it becomes conveniently available, staff inexperience has been a minor problem because there is a wealth of expertise in other groups within the Organic Chemistry Section upon which the organic analysis staff may call.

In addition to the somewhat routine analysis for volatile and semivolatile organic compounds, the gas chromatography/mass spectrometry staff also carries out some more specialized analyses, especially for volatile compounds. The special nature of these procedures generally evolves from the method of sample introduction. The staff is quite proficient at the determination of volatiles in a sample collected elsewhere on a sorbent trap by a third party. Gas samples collected in valved-traps have also been analyzed.

(*G. S. Fleming, S. H. Harmon, and M. D. Edwards*)

Improvements in Gas Chromatography

The gas chromatography effort presently has a staff of two. Seven instruments are available with four of these instruments equipped with autosamplers. The laboratory also has a Nelson Chromatography System and adequate personal computing equipment to support it. Although the amount of equipment in this laboratory is certainly adequate, the reliability of at least two of the more critical instruments (with autosamplers) has caused problems. At present the peak capacity of this laboratory is 70 pesticide samples per week with the ongoing capacity being 35 to 40 samples per week. Although the majority of routine samples processed are pesticides and/or polychlorinated biphenyls, there are many other miscellaneous or specialty samples. Among these are aqueous samples for total chromatographable organic content, aqueous samples for petroleum hydrocarbons, aqueous samples for solvents, waste oils for solvent content, and waste oils for polychlorinated biphenyl content.

This laboratory has had several hardware improvements during the past year. A fully equipped gas chromatograph was added. This addition was vital in view of the fact that only one of the other instruments is less than ten years old. Also, a very fast personal computer has been associated with the Nelson Chromatography System resulting in much faster chromatographic data processing. Software improvements have also been instituted. LOTUS programs have been developed to process results from the chromatography system and incorporate them into report forms.

The staff of the gas chromatography laboratory is relatively inexperienced. Therefore, there is, and will continue to be, an emphasis on training. During this year one staff member attended an ACS gas chromatography training course and three staff members attended a vendor-sponsored seminar. While the efficiency and capabilities of the gas chromatography function will increase as the staff gains experience, significant expertise in gas chromatography is available within the Organic Analysis Group and within the Organic Chemistry Section. Thus, consultation is immediately available and training should be expedited.

(*M. P. Maskarinec, R. M. Edwards, and L. H. Johnson*)

ORNL/UTK DISTINGUISHED SCIENTIST PROGRAM

G. A. Guiochon

The research work conducted by the group at ORNL deals with two main topics, both related to nonlinear chromatography: the behavior of large concentration bands in chromatographic columns and the surface energy distribution of ceramic powders.

Nonlinear Chromatography

The main thrust of this program is the theory and applications of preparative high performance liquid chromatography. The theoretical work deals with the simulation of the migration of large concentration bands through chromatographic columns, using a general model based on basic principles of physical chemistry. The model is a set of partial differential equations obtained by writing a mass balance equation and a kinetic equation for each compound involved (including the components of the mobile phase). The solution of this system predicts, depending on the boundary conditions, elution profiles of large bands (with or without volume overload), displacement profiles or system peaks.

The system of partial differential equations is hyperbolic and nonlinear. We have generated computer programs which permit the numerical calculations of its solution under all experimental conditions. We are presently studying the properties of the column response to changes in the input conditions, when the concentration of the input band is large and/or the kinetics of mass transfer between phases is fast. This approach does not work when the kinetics of mass transfer between phases is slow and the column efficiency is below ca. 100 theoretical plates. We are currently investigating this difficult theoretical problem which is of great practical interest for certain applications such as in affinity chromatography.

We are also studying the degree of agreement between experimental results and the prediction of the model. This requires that we carry out important studies on the thermodynamics of liquid-solid (adsorption) equilibria and on the kinetics of adsorption. For example, we have to determine competitive equilibrium isotherms of several compounds between phases and the cross coefficients of their kinetic equations of transfer. These parameters are difficult to measure and have been little studied in the past. We have an important research program beginning in this area, since progress there is necessary to achieve complete understanding of preparative liquid chromatography. We study also the relationship between isotherm and mass transfer properties on the one hand, and the nature of the surface of the packing material involved on the other hand.

This experimental study in turn generates theoretical work on the thermodynamics and kinetics of adsorption and desorption.

(*G. A. Guiochon, J. V. H. Schudel,* and A. M. Katti**)

---

*Predoctoral student, University of Tennessee

Surface Properties of Ceramic Powders

During the past year, we have begun an investigation of the adsorption energy distribution of polar vapors on the surface of solid particles such as those used in the preparation of high temperature resistant ceramics, using gas chromatography. This work is closely related to the one described in the previous section, as it involves another aspect of adsorption.

The production of advanced ceramic material for energy research (high structural strength material above 1400°C) or for sophisticated electronic devices is plagued by the lack of methods permitting the timely elimination of lots which will result in the production of faulty parts. We are developing a gas chromatographic method to determine rapidly and accurately the energy distribution function of the surface of ceramic powders for a series of probe molecules, prior to their use in the manufacturing process. The energy distribution function is related to the chemical composition of the external layer of the solid material and controls the wettability of the particles by their adhesion to any organic material, such as binder, dispersant, or solvent.

Recent results in the theory of large concentration chromatography permit the rapid determination of the thermodynamic and kinetic parameters of the adsorption of probe compounds on an adsorbent contained in a chromatographic column. The energy distribution function is determined directly from the adsorption isotherm. It can be used to calculate other data, such as the monolayer formation energy. Combining the energies determined for a set of probe solutes permits a classification of the surfaces with respect to similar ones. This could permit us to sort satisfactory materials from unsatisfactory ones.

Experimentally, porous layer open tubular columns are prepared from ceramic powder samples and measurements are carried out on these columns, using dry or wet carrier gases.

(G. A. Guiochon, E. V. Dose, and J. L. Roles*)*

---

*Postdoctoral Research Associate

5. ORNL ENVIRONMENTAL PROGRAMS

W. R. Laing

Environmental analyses continued to be a growth area for the division. Work from the Environmental and Health Protection and the Environmental Sciences Divisions and the DOE Environmental Survey Program totaled 235,000 measurements; this accounted for 58% of ACD analytical support work. The number of groundwater samples from monitoring wells increased as did the number from remedial action activities related to the waste area groups and inactive storage tanks. Details of these activities are found in the individual section reports.

DOE ENVIRONMENTAL SURVEY

In 1988 the DOE Environmental Survey sampling team visited Brookhaven National Laboratory and Idaho National Engineering Laboratory. Samples from these sites were analyzed along with work in process from three sites sampled the previous year. A total of 93,000 measurements, 23% of the total reported by ACD, were completed. In January an audit of the organic work raised questions about its quality. In July, after more of the data packages had been prepared, an EPA team began a data quality and usability evaluation. This study was completed by ACD personnel over the remainder of the year. It was found that 16% of the data was quality level 1 (highest), 32% was quality level 2 and 52% was quality level 3. All of the level 3 data was further evaluated by a usability test. This showed that 90% of the level 3 data was useable. In practice, however, all the data were used in the preparation of the site reports.

At year-end all analytical measurements were completed except for a few radiochemical samples. Results, along with blanks, standards, spikes, duplicates and QC samples, were organized into sample delivery groups (SDGs). Case narratives were prepared for each SDG which described any deviation from protocols. Data tables showing the analytical results grouped by environmental problem were prepared from the survey data base. Quality control programs were described and results were tabulated for the site reports. Site reports have been issued for Rocky Flats and Pantex. Four other reports are in process. We have also provided assistance to Argonne National Laboratoary and Battelle-Columbus in the preparation of these reports.

Quarterly performance evaluation samples from EPA were analyzed for inorganic and organic constituents. The average score was 91.6 for inorganics and 71.3 for organics.

REMEDIAL ACTION PROGRAMS

Last year's decision to subcontract the ORNL remedial investigations and feasibility studies (RI/FS) was implemented this year. The first contract was awarded for work in Waste Area Group 6. ACD participated through contract input and review.

The inactive tank sampling program began this year and provided some unusual analytical problems. Because of EPA responsibility for waste materials, the use of EPA procedures were required. Application of methods for groundwater and municipal waste to highly salted, radioactive solutions provided many challenges. The final agreement with EPA was to follow established procedures as closely as possible and to document all deviations. This work is discussed in detail in the reports from the Organic Chemistry and the Radioactive Materials sections.

Three hundred groundwater monitoring wells were drilled in the area surrounding ORNL. These wells will be qualified over the next few years and brought on-line as routine sampling points. They will provide the data to define off-site environmental problems.

6. QUALITY ASSURANCE, SAFETY AND TRAINING

QUALITY ASSURANCE/QUALITY CONTROL

P. L. Howell*

The Quality Assurance (QA) Program continued in an effort of upgrading and revitalization to requirements, under the direction of the newly-organized ORNL Quality organization. DOE Order 5700.6A invoked the use of Quality Assurance Program Requirements for Nuclear Facilities (NQA-1) in preparing QA Programs. The development of an 18-element NQA-1 QA Program began and progressed with procedures written for six of the 18 NQA-1 elements. To develop the procedures, small group meetings were held to discuss the requirements of each NQA-1 element followed by discussion of how the requirements were applicable within ACD. Procedures were then drafted and sent out for review and comment. Manager training to the NQA-1 requirements and the applicable ACD procedure is ongoing and takes place in monthly supervisor meetings. Division-wide training will take place upon completion of the QA Program manual. The remainder of the procedures are to be written and the manual completed by July, 1989.

The use of Quality Circles to identify and resolve problems was reinstated in April 1988. The quality circle concept, coupled with the Performance Improvement Process (PIP), operates under the ACD Quality Council, the purpose of which is the "study of any issue concerning quality of work, efficiency of work, or methods of improvement." PIP projects on "Centralized Personal Computer (PC) Resource Facility", "Standardization of Secretarial PCs", and "Investigation of Provisions for Greater Flexibility in Changing Job Assignments" are currently in progress. Council members were chosen from each section in the division.

Quality Assurance and Quality Control staff members participated in eight training courses and served as instructors in several training sessions. All group leaders, section heads, the division director, and the Quality Assurance Specialist participated in the ORNL "Managing For Quality" seminar presented by the Quality Department to introduce management to NQA-1.

Three ACD sections drafted in excess of 60 standard operating procedures (SOPs). Associated with the development of SOPs has been updating and revising the division document control system to promote a more systematic and disciplined approach to distribution and control. The enhanced system will be refined and put to use in 1989. A number of forms have been developed, using a commercially available software package, to aid in standardizing formats used across all sections.

Database tracking systems were implemented for tracking audit and surveillance findings, corrective action status and plans, and other programmatic QA activities. This was "The Year of the Audit" with a total of 28 quality assurance audits, surveillances, and reviews taking place in ACD laboratories and facilities.

Of the 28, eight were customer-sponsored audits or reviews, three were ORNL audits, one was an internal audit, and four were segments of a program-wide review for both ORNL and K-25. There were 12 surveillances: one customer sponsored, four internal, and seven associated with the ORNL Isotope Generation and Depletion (ORIGEN) Code Verification Program activities.

One program review by DOE resulted in removal of the Organic Analysis Group from DOE Environmental Survey because of failure to follow programmatic quality and evidentiary protocols. This led to an in-depth review of "data quality" and "data usability". This review found that most of the ORNL data was "useful" according to data review protocals.

Quarterly and monthly quality control solutions were provided to the ACD inorganic, organic and radiochemical analysis laboratories. Monthly control solutions are purchased from Analytical Products Group (APG), Marietta, Ohio, and submitted as "blinds". Results are reported to APG and statistically evaluated. Thirty laboratories throughout the country participate in this evaluation, including the analytical chemistry laboratories from all Martin Marietta Energy Systems, Inc., sites.

ACD laboratories performing analyses for the DOE Environmental Survey participated in the EPA performance evaluation (PE) program this year. Both organic and inorganic PE samples were received, analyzed, and scored. The average score for four inorganic PE sample sets was 91.6 (out of a possible 100). The average score for three organic PE sample sets for the same period was 71.3. Additional "water pollution" QC samples were submitted quarterly and annually to the laboratories to monitor inorganic measurements not covered by the PE sets. A score of "acceptable" was received for all such sets.

Quality Control summaries for the ACD QC program were not available because of the changeover to the Analytical Laboratory Information System (AnaLIS) data management system. Software to summarize and generate summary reports has not been completed.

(W. R. Laing, S. K. Holladay, J. S. Eldridge)

---

*ORNL Quality Department

SAFETY PROGRAM

A. L. Harrod

R. E. Jones

A Safety Program and Safety Action Plan was implemented as a guide toward better safety in the Analytical Chemistry Division during 1988. The division experienced seven first aid cases against four during last year. Increased awareness of safety has been emphasized in all our Safety Meetings as a result of these incidents.

The division has not experienced a lost workday case since June 1972.

Notable achievements during this reporting period:

1. Safety meetings held - 51, total attendance - 774.
2. Training guide updates for our facilities at Building 2026 and 7920 continue.
3. Spring Clean-Up Week activities in May included fire training at Building 2026 and 4500N.
4. Orientation meeting for new employees was conducted in March 1988.
5. Appointment of a Division Quality Circle Council; PIP Projects for Safety and Job Performance Improvements.

6. Training Sessions - 1988
    a. Transuranium Waste - 2
    b. Low Level Waste - 2
    c. RCRA - 1 (Resource Conservation & Recovery Act)

This division continues to maintain a high level of safety awareness and accident prevention among our personnel as evidenced by our superior performance rating this year.

NON-REACTOR NUCLEAR TRAINING

M. P. May

Development of safety-related training programs continues for ORNL employees working at, visiting, or using our non-reactor nuclear facilities.

Staff at 2026 have completed a "training milestone." They conducted a training program for all facility workers on the Building 2026 Operational Rules, Safety Guidelines and Emergency Evacuation & Alarm Procedures, complete with a review, written exam and necessary documentation. They also worked with craft supervision (I&C,P&E) to hold a separate training session designed specifically for the maintenance and craft personnel assigned to the building.

ACD and ChemTech personnel at 7920 have worked to consolidate training records and provide documentation for our division office files.

A "Contact-Handled TRU Waste Disposal" program is being developed at 9735 for the Actinides & Isotopic Mass Spectrometry Lab. We are currently working to dispose of three drums of waste generated prior to the program initiation.

New training material and equipment include videotapes on "Radioisotopes" and "ORNL Isotopes Production;" and a division-wide portable video player.

The DSO has created a file for materials related to training, located in 4500S.

Internal plant training audits were conducted at 7920, 2026 and 2011 (the neutron generator facility). The results show progress being made in training program development Some remaining problems include insufficient staff, incomplete documentation, programs not designed to the worker's needs, and out of date and vague division and facility training guides. We still have much work to do, both at the division and facility levels. The ACD training coordinator has met with and will be working with our Quality Assurance Specialist on standardizing division/section/group procedure writing. Together with the Training Design and Development Staff of ORAU, we are also in the process of submitting a proposal to the Office of Operational Safety for developing and maintaining an Interactive Video Disc Computer-Based Training Program.

Many changes have occurred at the laboratory level. Over the past year, the plant Training Coordinator has met monthly with Division Training Coordinators, to keep them up to date on DOE and Energy Systems developments, to review programs and to recieve input from other Training Coordinators. The Technical Training and Resources Group of the Environmental and Health Protection Division has assumed training responsibility for many plant-wide programs: Radiation Protection, Low Level Waste and Hazardous Waste Generator Certification, Industrial Hygiene, and soon, CH-TRU Waste Training. This is a step toward centralizing plant training responsibilities.

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 1988 Advisory Committee was composed of:

D. D. Bly, Central Research Department, DuPont Experimental Station, P.O. Box 80328, Wilmington, DE.

R. G. Cooks, Chemistry Department, Purdue University, West Lafayette, IN.

J. C. Giddings, Department of Chemistry, University of Utah, Salt Lake City, UT.

E. D. Pellizzari, Research Triangle Institute, Research Triangle Park, NC.

CONSULTANTS

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

L. M. Diana, University of Texas, Arlington, TX.

S. Perone, Lawrence Livermore National Laboratory, Livermore, CA.

N. M. Spyrou, University of Surrey, Surrey, England.

J. Warren, EPA, Washington, DC.

F. Strobel, University of Delaware, Newark, DE.

P. Jurs, Pennsylvania State University, University Park, PA.

G. Christian, University of Washington, Seattle, WA.

R. Yost, University of Florida, Gainesville, FL.

N. Winograd, Pennsylvania State University, University Park, PA.

M. Comisarow, University of British Columbia, Vancouver, B.C., Canada.

M. Bonner Denton, University of Arizona, Tucson, AZ.

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.

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 1988.

OAK RIDGE ASSOCIATED UNIVERSITIES PROGRAMS

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

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

Two post-docs working with Michelle Buchanan in the Organic Chemistry Section completed their research: John W. Haas (University of Massachusetts) and Elizabeth A. Stemmler (Indiana University). Both were 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 completed his predoctoral studies with P. J. Todd.

Donald B. Coyle, The American University, worked with Mike Ramsey on the development of Doppler-free laser spectroscopy techniques for atoms in supersonic beams.

Becky Dittmar, Angelo State University, worked with Rose Ramsey on the separation of nucleotides and nucleosides using reverse-phase high pressure liquid chromatography.

ORAU Faculty Research Program. Subrahmanyam Pendyala (SUNY, Fredonia) returned for the summer to continue his work with Dave Smith and Lester Hulett on positron spectroscopy.

J. A. Howell (Earlham College) completed his sabbatical assignment with Mike Ramsey on laser spectroscopy.

ORAU Faculty Research Program for Historically Black Colleges and Universities. Wedad Hussein, an associate professor at Jackson State University, worked with Ted Mueller in the Chemical and Physical Analysis Group on R&D projects in the field of electroanalytical chemistry.

GLCA Program. Robert B. Clark worked with Mike Ramsey using photorefractive optical elements to control the bandwidth and wavelength of CW dye lasers using real-time holographic concepts.

SUMMER INTERN PROGRAM

Summer Intern Program. The division was host to C. Wayne Lyle, Southern Arkansas University, who participated in the summer intern program. He worked with Michelle Buchanan in the Organic Chemistry Section on DNA separations using high performance liquid chromatography.

CO-OP PROGRAM

<u>Co-Op Program.</u> G. C. Keyes completed her co-op assignment with Jeff Wade in the low-level radiochemical analysis group, participating in various group projects; she was replaced by Jamie McKinney, who continued working in this same area with Jeff Wade.

UNIVERSITY OF TENNESSEE PROGRAMS

<u>Distinguished Scientist Program.</u> Dr. Georges Guiochon is continuing his work through the University of Tennessee/ORNL Distinguished Scientist Program. Mark L. Aubel, a post-doc student from the University of Georgia, and two predoctoral students, Jennifer Schudel and Anita Katti, are continuing their work at ORNL. In addition, two post-docs, Eric Dose, Rice University, and Martin Czok, a German citizen from the University of Tennessee, and a predoctoral student, Jeffry Roles, also from the University of Tennessee, have been added to Dr. Guiochon's staff. Both Samir Ghodbane, an Algerian citizen from Georgetown University, and Michel F. Righezza, a French citizen from Georgetown University, completed their assignments on this program.

<u>Science Alliance Research Program.</u> Luis E. Cuellar from El Salvador is a student at the University of Tennessee; he completed his work with Lester Hulett measuring positron lifetime spectra of composite materials and other solids. Lisa Rosevink, a student at the University of Tennessee, worked with Harley Ross on radiochemical research.

<u>Postgraduate Research Program.</u> Gary J. VanBerkel (Washington State University) is continuing his work 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) completed 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.

Casey Grimm (Florida State University) is working with Peter Todd doing research in the study of organic secondary ion emission.

INTERNATIONAL ATOMIC ENERGY AGENCY PROGRAM

The division was host to two IAEA Fellows, Anil R. Hikkaduwa Liyanage from Sri Lanka, and Jose Alvarado Duran from Caracas, Venezuela, for a four-month period. They were assigned to Sue MacDougall to receive on-the-job training in X-ray fluorescence and X-ray diffraction via assisting experts in these techniques.

OAK RIDGE SCIENCE RESEARCH SEMESTER PROGRAM

Melanie Mock, Mars Hills College, worked with Leon Klatt on the development of fiber optic probe for the determination of ammonia concentration in heat absorption equipment and with Jack Young doing a spectrophotometric study of air oxidation of cuprous sulfide.

Carmen Guzman, Catholic University of Puerto Rico, worked with Wayne Griest on high pressure liquid chromatography.

SERVICE ACADEMY RESEARCH ASSOCIATES (SARA) PROGRAM

Stephen C. Formella, a student in the United States Military Academy, completed a five-week internship with the Laser Spectroscopy and Instrumentation Group. He worked with Mike Ramsey using lasers in advancing spectrochemical analysis techniques.

SCIENCE TEACHERS RESEARCH INVOLVEMENT FOR VITAL EDUCATION (STRIVE)

Bobbi J. Grogan, a science teacher at Farragut High School, Concord, TN, worked with Wayne Griest on the separation and determination of surfactants and other polar organics by liquid and supercritical fluid chromatography.

ORNL/DOE CONFERENCE ON ANALYTICAL CHEMISTRY IN ENERGY TECHNOLOGY

Due to a question of charging registration fees for conferences at which the majority of participants are employees of DOE or DOE contractors, a high quality program could not be organized within the required time frame for the Annual ORNL/DOE Conference on Analytical Chemistry. The Conference will be resumed in October 1989.

SEMINAR PROGRAM

Speaker	Title	Date
Diana, L. M. Ctr. for Positron Studies, Univ. of Texas, Arlington, Arlington, TX	"The Positron Spectroscopy of Gases"	March 21, 1988
Perone, Sam Lawrence Livermore Natl. Lab., Livermore, CA	"Information Theory and the Design of Chemical Sensors"	June 3, 1988
Spyrou, N. M. Dept. of Physics, Univ. of Surrey, Guildford, Surrey, England	"Nuclear Analysis and Imaging in Biological Systems"	June 22, 1988
Warren, John EPA, Washington, DC	"The Integration of Sampling and Quality Assurance"	August 2, 1988

Strobel, Fred Univ. of Delaware, Newark, DE	"Effects of Electronic Excitation on the Reactivity of Transition Metal Ions"	August 17, 1988
Jurs, Peter Dept. of Chemistry, Pennsylvania State Univ., University Park, PA	"Computer-Assisted Studies of Molecular Structure and Physiochemical Properties"	Sept. 15, 1988
Christian, Gary Dept. of Chemistry, Univ. of Washington, Seattle, WA	"Flow Injection Analysis in Process Analysis"	Sept. 19, 1988
Yost, Richard Dept. of Chemistry, Univ. of Florida, Gainesville, FL	"Tandem Mass Spectrometry: Present Capabilities and Future Prospects"	Oct. 14, 1988
Winograd, Nicholas Dept. of Chemistry, Pennsylvania State Univ., University Park, PA	"Surface Studies Using Ion Beam and MPRI"	Nov. 4, 1988
Comisarow, Mel Dept. of Chemistry, Univ. of British Columbia, Vancouver, B.C., Canada	"Fourier Transform Ion Cyclotron Resonance Mass Spectroscopy"	Nov. 15, 1988
Denton, M. Bonner Dept. of Chemistry, Univ. of Arizona, Tucson, AZ	"New Technologies for Spectroscopic Analysis"	Dec. 5, 1988

AWARDS AND HONORS

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

Bill Laing was presented a Quality Recognition Award by the ORNL Quality Department. This award recognizes Bill's many contributions to quality programs in the division and elsewhere over many years.

Beth Stemmler, a post-doc student, received an ASTM Travel Stipend Award; she was one of only a dozen to receive this award.

Michelle Buchanan won an award for "Distinguished Scientific Achievement" from the Association of Women in Science (AWIS).

Mike Guerin received a Technical Achievement Award at the Martin Marietta Awards Night for pioneering the development and application of modern organic analytical chemistry within the health and environmental programs of DOE and many other agencies and institutions.

Michelle Buchanan was elected Treasurer for the American Society of Mass Spectrometry.

Three more people have received the Distinguished Service Award established by the division to recognize deserving permanent staff members of ACD. Arnold Harrod received the award for his long-term efforts in assisting everyone and his contributions to the people of ACD. Leon Klatt received the award for his contributions in instrumentation and computer technology, in addition to his normal duties. Cecil Higgins received the award for the long-term dedication to research, his willingness to offer assistance to others, and his kindness and courteous attitude to everyone.

Lester Hulett, T. A. Lewis, and Dave Donohue received a Significant Event Award for putting into operation a high-intensity source of low-energy positrons.

Gary Glish and Scott McLuckey also received a Significant Event Award for development of an explosive vapor detector.

ADDITIONAL PROFESSIONAL ACTIVITIES

Bostick, D. A.

Member: Association of Women in Science
ACD Representative: University Relations Coordinator

Buchanan, M. V.

Editorial Board: *Biomedical and Environmental Mass Spectrometry*
Organic Mass Spectrometry
Chairman: Environmental Applications Group of the American Society for Mass Spectrometry
Treasurer: American Society for Mass Spectrometry
Advisory Board: National Science Foundation, Biological Centers Program
Chairman Elect: 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

Eldridge, J. S.

Chairman: Membership Committee, Environmental Radiation Section of the Health Physics Society
ACD Quality Circle

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.

ACD Representative: Laboratory Emergency Sample Coordinator

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.

ACD Representative: Energy Systems Environmental Analysis Committee

Glish, G. L.

Consultant: Finnigan MAT, San Jose, CA

Councilor: East Tennessee Mass Spectrometry Discussion Group

Member: Board of Directors, Asilomar Conference on Mass Spectrometry

ORAU Travelling Lecture Program

Organizer: Symposium at 15th Annual FACSS Meeting

Vice President: American Society for Mass Spectrometry

Griest, W. H.

Consultant: Department of Energy - SBIR Review

Department of Energy - BES

Electric Power Research Institute

Environmental Protection Agency - IERL/TSO

Member: ORAU Travelling Lecture Program

Guerin, M. R.

Member: National Institute of Environmental Health Sciences Program Project Review of MIT Combustion Products Carcinogenesis Program

University of Kentucky Tobacco and Health Research Institute Advisory Board

Consultant: SBIR Reviews, DOE, NCI

Organizer: Workshop on Analytical Problems Unique to DOE

Hettich, R. L.

Chairman: ACD Seminar Committee

Member: ORAU Travelling Lecture Program

Treasurer: East Tennessee ACS Section

Ilgner, R. H.

ACD Representative: ACD Environmental Protection Officer

Consultant: Fourth Judicial District Court, Monroe, LA

Jenkins, R. A.

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

Klatt, L. N.

Member: Energy Systems, Ph.D. Recruiting Team
ACD Seminar Committee

Laing, W. R.

Chairman: ASTM Subcommittee 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 Chairman: Analytical Chemistry in Energy Technology Conference

May, M. P.

Instructor and Coordinator: "Beginning Radiochemistry" in-house course

McLuckey, S. A.

Chairman: Ion Physics and Instrumentation Interest Group
American Society for Mass Spectrometry

Organizer: Ion Trapping Symposium for 1988 ASMS Annual Meeting, San Francisco
Member: ACD Seminar Committee

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
Consultant: SBIR Reviews, NCI

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
Editorial Board: *Journal of Radioanalytical and Nuclear Chemistry, Letters*
Conference Organizer: International Conference on New Trends in Liquid Scintillation Counting and Organic Scintillators, Gatlinburg, TN, October 2-5, 1989.
Judge: 39th Int. Science & Engineering Fair, Knoxville, tN

Rosseel, T. M.
Judge: 39th Int. Science & Engineering Fair, Knoxville, TN
Treasurer: American Vacuum Society (AVS) Tennessee Valley Chapter
Member: AVS Tennessee Valley Chapter, Scholarship Committee
AVS, 35th National Symposium, Local Organizing Committee
Asst. Treasurer: AVS, 35th National Symposium
Treasurer: 8th Annual TVC-AVS Symposium & Equipment Exhibit

Smith, D. H.
Member: ORNL PIP Committee on Expert Systems
ASMS Isotope Ratio Interest Group

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
	Martin Marietta Energy Systems PIP Committee on Optimizing Analytical Services
	Martin Marietta Energy Systems Management Advisory Committee for Nonexempt Reevaluation Program
	Martin Marietta Energy Systems PIP Committee on Travel Expenses
	Martin Marietta Energy Systems Pilot Program on Mentoring
Chairman:	ORNL Division/Program Directors' Caucus
	ORNL PIP Committee on Expert Systems
	Education Committee, ACS Division of Analytical Chemistry
	Martin Marietta Energy Systems United Way Campaign 1988

Stewart, J. H., Jr.

Member:	ASTM Committee D-34 Waste Disposal
	International Working Group, "Analytical Standards of Minerals, Ores, and Rocks"
	Manuscript Review Board for *Analytical Chemistry*
ACD Coordinator:	Technician Advisory Group
	ASTM C-26 Nuclear Fuel Cycle Committee
	ACD Nonradiological Emergency Sample Coordinator

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

Wise, M. B.

Member:	ACD Safety Committee
	ORAU Travelling Lecture Program
Judge:	39th Int. Science & Engineering Fair, Knoxville, TN

Young, J. P.

ACD Representative:	Graduate Fellow Selection Panel, ORNL
Fellow:	American Association for the Advancement of Science
Judge:	39th Int. Science & Engineering Fair, Knoxville, TN
Member:	ACS Program Advisory Committee, Division of Nuclear Chemistry & Technology

8. PRESENTATION OF RESEARCH RESULTS

As in past years, the division has actively responded to the evolvig 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
Asano, K. G.	Grant, B. C.; Asano, K. G.; Glish, G. L.; McLuckey, S. A., "Trace Analysis for Organics in Air Using an Atmospheric Sampling Glow Discharge Ionization Source," *Proceedings of the 36th ASMS Conference on Mass Spectrometry and Allied Topics*, 1988, p. 47.
	Glish, G. L.; Van Berkel, G. J.; Asano, K. G.; McLuckey, S. A., "'MS^n' Experiments with an Ion Trap Mass Spectrometer," *Proceedings of the 36th ASMS Conference on Mass Spectrometry and Allied Topics*, 1988, p. 1112.
	McLuckey, S. A.; Asano, K. G.; and Glish, G. L., "Negative Chemical Ionization and MS/MS of Negative Ions in an Ion Trap Mass Spectrometer," *Proceedings of the 36th ASMS Conference on Mass Spectrometry and Allied Topics*, 1988, p. 1108.
	Asano, K. G.; Glish, G. L.; McLuckey, S. A., "Coupling of an Atmospheric Sampling Ion Source with an Ion Trap Mass Spectrometer," *Proceedings of 36th ASMS Conference on Mass Spectrometry and Allied Topics*, 1988, p. 636.
Bertram, L. K.	Goeringer, D. E.; McLuckey, S. A.; Bertram, L. K., "Instrumentation for Mass Spectrometry/Coincident Fragment Detection," *Proceedings of the 36th ASMS Conference on Mass Spectrometry and Allied Topics*, 1988, p. 835.

Buchanan, M. V.	Guerin, M. R.; Buchanan, M. V., "Environmental Exposure to N-Aryl Compounds," *Carcinogenic and Mutagenic Responses to Aromatic Amines and Nitroarenes*, C. M. King, L. J. Romano, and D. Schuetzle, eds., Elsevier Publishers, 1988, p. 37.
	Hettich, R. L.; Buchanan, M. V.; Wise, M. B., "Characterization of Substituted Glycerols by Thermal Desoprtion Fourier Transform Mass Spectrometry," *Ohio State University FT/ICR Newsletter*, Alan Marshall, ed., Spring, 1988.
	Buchanan, M. V.; Stemmler, E. A., "Gas Phase Ion-Molecule and Surface-Catalyzed Oxidation Reactions of PAH Under Negative Ion Chemical Ionization Conditions," *Proceedings, 36th Annual Conference on Mass Spectrometry and Allied Topics*, 1988, p. 767.
	Stemmler, E. A.; Buchanan, M. V., "Isomeric Differentiation of PAH and Methyl-PAH Using Surface Catalyzed Oxidation Reactions," *Proceedings, 36th Annual Conference on Mass Spectrometry and Allied Topics*, 1988, p. 769.
Christie, W. H.	James, R. B.;* Bolton, P. R.;* Alvarez, R. A.;* Christie, W. H.; Valiga, R. E., "Melting of Silicon Surfaces by High-Power Pulsed Microwave Radiation," *SAND* 87-8987, January 1988.
	James, R. B.;* Bolton, P. R.;* Alvarez, R. A.;* Christie, W. H., "Rapid Heating of Ion-Implanted Silicon by High-Power Pulsed Microwave Radiation," *SAND* 88-8759, May 1988.
	Christie, W. H.; Goeringer, D. E., "Investigation of Matrix Effects on the Neutral Fractions Ejected from Ion-Bombarded, Uranium-Containing Solids Using Resonance Ionization Mass Spectrometry," *Secondary Ion Mass Spectrometry - SIMS VI*, A. Benninghoven, A. Huber, G. Slodzian and H. Werner, eds., John Wiley & Sons, 1988, p. 805.
Glish, G. L.	Busch, K. L.* Glish, G. L.; McLuckey, S. A., *Mass Spectrometry/Mass Spectrometry: Techniques and Applications of Tandem Mass Spectrometry*, VCH Publishers, Inc., New York, 1988.
	Asano, K. G.; Glish, G. L.; McLuckey, S. A., "Coupling of an Atmospheric Sampling Ion Source with an Ion Trap Mass Spectrometer," *Proceedings of the 36th ASMS Conference on Mass Spectrometrometry and Allied Topics*, 1988, p. 636.

Glish, G. L.

Van Berkel, G. J.; Glish, G. L.; McLuckey, S. A., "Analysis of Porphyrins Using an Ion Trap Mass Spectrometer," *Proceedings of the 36th ASMS Conference on Mass Spectrometry and Allied Topics*, 1988, p. 639.

Grant, B. C.; Asano, K. G.; Glish, G. L.; McLuckey, S. A., "Trace Analysis for Organics in Air Using an Atmospheric Sampling Glow Discharge Ionization Source," *Proceedings of the 36th ASMS Conference on Mass Spectrometry and Allied Topics*, 1988, p. 47.

McLuckey, S. A.; Asano, K. G.; and Glish, G. L., "Negative Chemical Ionization and MS/MS of Negative Ions in an Ion Trap Mass Spectrometer," *Proceedings of the 36th ASMS Conference on Mass Spectrometry and Allied Topics*, 1988, p. 1108.

Glish, G. L.; Van Berkel, G. J.; Asano, K. G.; McLuckey, S. A., "MS^n Experiments with an Ion Trap Mass Spectrometer," *Proceedings of the 36th ASMS Conference on Mass Spectrometry and Allied Topics*, 1988, p. 1112.

Van Berkel, G. J.; Glish, G. L.; McLuckey, S. A., "Structural Analysis of Porphyrins: Analysis of In Vitro Prepared Porphyrinogens Using an Ion Trap Mass Spectrometer," *Proceedings of the 36th ASMS Conference on Mass Spectrometry and Allied Topics*, 1988, p. 300.

Glish, G. L.; McLuckey, S. A., "Scan Modes for Hybrid Mass Spectrometers," *Proceedings of the 36th ASMS Conference on Mass Spectrometry and Allied Topics*, 1988, p. 823.

Glish, G. L.; McLuckey, S. A., "Kinetic Energy Release Effects on Resolution in MS/MS," *Proceedings of the 36th ASMS Conference on Mass Spectrometry and Allied Topics*, 1988, p. 825.

Van Berkel, G. S.; Glish, G. L.; McLuckey, S. A., "Mechanism of Prophyrin Hydrogenation and Decomposition in a Chemical Ionization Plasma," *Proceedings of 36th ASMS Conference on Mass Spectrometry and Allied Topics*, 1988, p. 302.

Goeringer, D. E.

Christie, W. H.; Goeringer, D. E., "Investigation of Matrix Effects on the Neutral Fractions Ejected from Ion-Bombarded, Uranium-Containing Solids Using Resonance Ionization Mass Spectrometry," *Secondary Ion Mass Spectrometry - SIMS VI*, A. Benninghoven, A. Huber, G. Slodzian and H. Werner, eds., John Wiley & Sons, 1988, p. 805.

Goeringer, D. E.

Goeringer, D. E.; McLuckey, S. A.; Bertram, L. K., "Instrumentation for Mass Spectrometry/Coincident Fragment Detection," *Proceedings of the 36th ASMS Conference on Mass Spectrometry and Allied Topics*, 1988, p. 835.

Young, J. P.; Shaw, R. W.; Goeringer, D. E.; Smith, D. H., "Influence of Laser Characteristics on Resonance Ionization Mass Spectrometry," *Resonance Ionization Spectroscopy 1988* (Institute of Physics Conf. Series No. 94), T. B. Lucarto and J. E. Parks, eds., Inst. of Physics, Bristol, p. 251.

Grant, B. C.

Grant, B. C.; Asano, K. G.; Glish, G. L.; McLuckey, S. A., "Trace Analysis for Organics in Air Using an Atmospheric Sampling Glow Discharge Ionization Source," *Proceedings of the 36th ASMS Conference on Mass Spectrometry and Allied Topics*, 1988, p. 47.

Griest, W. H.

Jenkins, R. A.; Griest, W. H.; Moneyhun, J. H.; Tomkins, B. A.; Ilgner, R. H.; Higgins, C. E.; Gayle, T. M.,* "Sampling and Chemical Characterization of Workplace Atmospheres Contaminated with Airborne Diesel Exhaust," *Proceedings of the 1988 EPA/APCA International Synposium on Measurements of Toxic and Related Air Pollutants*, U.S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory, and the Association Dedicated to Air Pollution Control and Waste Management, Research Triangle Park, NC, May, 1988, p. 119.

Griest, W. H.; Higgins, C. E.; Guerin, M. R., "Comparative Chemical Characterization of Shale Oil- and Petroleum-Derived Diesel Fuels," *Health and Environmental Research on Complex Organic Mixtures*, R. H. Gray, et al., eds., Pacific Northwest Laboratory, Richland, WA, 1987, p. 63.

Ho, C.-h.; Jones, A. R.; Smith, L. H.;* Epler, J. L.;* Griest, W. H.; Guerin, M. R., "Upgrading Coal-Derived Liquids to Reduce Toxicity. I. Evaluation of Nonhydrotreatment Processes as Alternatives or Supplements to Hydrotreatment," *Health and Environmental Research on Complex Organic Mixtures*, R. H. Gray, et al., eds., Pacific Northwest Laboratory, Richland, WA, 1987, p. 211.

Guerin, M. R.

Guerin, M. R.; Buchanan, M. V., "Environmental Exposure to N-Aryl Compounds," in *Carcinogenic and Mutagenic Responses to Aromatic Amines and Nitroarenes*, C. M. King, L. J. Romano, and D. Schuetzle, Eds., Elsevier Publishers, 1988, p. 37.

Guerin, M. R.

Wise, M. B.; Guerin, M. R., "Direct Determination of Part-per-Billion Levels of Volatile Organics in Water and Soil Samples Using a Glow Discharge Mass Spectrometer," *Proceedings, Fourth Annual Waste Testing and Quality Assurance Symposium, Volume II*, Washington, DC, July 11-15, 1988, p. G-53.

Griest, W. H.; Higgins, C. E.; Guerin, M. R., "Comparative Chemical Characterization of Shale Oil- and Petroleum-Derived Diesel Fuels," *Health and Environmental Research on Complex Organic Mixtures*, R. H. Gray, et al., eds., Pacific Northwest Laboratory, Richland, WA, 1987, p. 63.

Ho, C.-h.; Jones, A. R.; Smith, L. H.;* Epler, J. L.;* Griest, W. H.; Guerin, M. R., "Upgrading Coal-Derived Liquids to Reduce Toxicity. I. Evaluation of Nonhydrotreatment Processes as Alternatives or Supplements to Hydrotreatment," *Health and Environmental Research on Complex Organic Mixtures*, R. H. Gray, et al., eds., Pacific Northwest Laboratory, Richland, WA, 1987, p. 211.

Guiochon, G. A.

Guiochon, G.; Guillemin, C.,* *Quantitative Gas Chromatography, Journal of Chromatography, Volume 42*, Elsevier, Amsterdam, The Netherlands, May 1988.

Hettich, R. L.

Hettich, R. L.; Buchanan, M. V.; Wise, M. B., "Characterization of Substituted Glycerols by Thermal Desoprtion Fourier Transform Mass Spectrometry," *Ohio State University FT/ICR Newsletter*, Alan Marshall, Ed., Spring, 1988.

Hettich, R. L., "Laser Ionization Fourier Transform Mass Spectrometry for the Differentiation of Methyl Guanosine Isomers," *Proceedings, 36th Annual Conference on Mass Spectrometry and Allied Topics*, 1988, p. 1122.

Higgins, C. E.

Jenkins, R. A.; Thompson, C. V.; Higgins, C. E., "Development and Application of a Thermal Desorption-Based Method for the Determination of Nicotine in Indoor Environments," *Indoor and Ambient Air Quality*, R. Perry and P. W Kirk, eds., Selper, Ltd., London, 1988, p. 557.

Jenkins, R. A.; Griest, W. H.; Moneyhun, J. H.; Tomkins, B. A.; Ilgner, R. H.; Higgins, C. E.; Gayle, T. M.,* "Sampling and Chemical Characterization of Workplace Atmospheres Contaminated with Airborne Diesel Exhaust," *Proceedings of the 1988 EPA/APCA International Symposium on Measurements of Toxic and Related Air Pollutants*, U.S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory, and the Association Dedicated to Air Pollution Control and Waste Management, Research Triangle Park, NC, May, 1988, p. 119.

Higgins, C. E.	Griest, W. H.; Higgins, C. E.; Guerin, M. R., "Comparative Chemical Characterization of Shale Oil- and Petroleum-Derived Diesel Fuels," *Health and Environmental Research on Complex Organic Mixtures*, R. H. Gray, et al., eds., Pacific Northwest Laboratory, Richland, WA, 1987, p. 63.
Ho, C.-h.	Ho, C.-h.; Jones, A. R.; Smith, L. H.;* Epler, J. L.;* Griest, W. H.; Guerin, M. R., "Upgrading Coal-Derived Liquids to Reduce Toxicity. I. Evaluation of Nonhydrotreatment Processes as Alternatives or Supplements to Hydrotreatment," *Health and Environmental Research on Complex Organic Mixtures*, R. H. Gray, et al., eds., Pacific Northwest Laboratory, Richland, WA, 1987, p. 211.
Holladay, S. K.	Maskarinec, M. P.; Johnson, L. H.; Holladay, S. K., "Recommendations for Holding Times of Environmental Samples," *Proceedings, Fourth Annual Waste Testing and Quality Assurance Symposium, Volume II*, Washington, DC, July 11-15, 1988, p. H-29.
Holmes, R. L.	Stewart, J. H., Holmes, R. L., Rogers, W. F., Maskarinec, M. P., "Evaluation of a Rapid Quantitative Organic Extraction System for Determining the Concentration of PCB in Soils," *Proceedings of USEPA Symposium on Waste Testing and Quality Assurance*, Vol. II, July 11-15, 1988, Washington, DC, July 11-15, 1988, p. G-37.
Ilgner, R. H.	Jenkins, R. A.; Griest, W. H.; Moneyhun, J. H.; Tomkins, B. A.; Ilgner, R. H.; Higgins, C. E.; Gayle, T. M.,* "Sampling and Chemical Characterization of Workplace Atmospheres Contaminated with Airborne Diesel Exhaust," *Proceedings of the 1988 EPA/APCA International Symposium on Measurements of Toxic and Related Air Pollutants*, U.S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory, and the Association Dedicated to Air Pollution Control and Waste Management, Research Triangle Park, NC, May, 1988, p. 119.
Jenkins, R. A.	Jenkins, R. A.; Thompson, C. V.; Higgins, C. E., "Development and Application of a Thermal Desorption-Based Method for the Determination of Nicotine in Indoor Environments," *Indoor and Ambient Air Quality*, R. Perry and P. W Kirk, eds., Selper, Ltd., London, Publishers, 1988, p. 557.

Jenkins, R. A.	Jenkins, R. A.; Griest, W. H.; Moneyhun, J. H.; Tomkins, B. A.; Ilgner, R. H.; Higgins, C. E.; Gayle, T. M.,* "Sampling and Chemical Characterization of Workplace Atmospheres Contaminated with Airborne Diesel Exhaust," *Proceedings of the 1988 EPA/APCA International Symposium on Measurements of Toxic and Related Air Pollutants*, U.S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory, and the Association Dedicated to Air Pollution Control and Waste Management, Research Triangle Park, NC, May, 1988, p. 119.
Johnson, L. H.	Maskarinec, M. P.; Johnson, L. H.; Holladay, S. K., "Recommendations for Holding Times of Environmental Samples," *Proceedings, Fourth Annual Waste Testing and Quality Assurance Symposium, Volume II*, Washington, DC, July 11-15, 1988, p. H-29.
Maskarinec, M. P.	Maskarinec, M. P.; Moody, R. L., "Storage and Preservation of Environmental Samples," *Principles of Environmental Sampling*, L. H. Keith, ed., American Chemical Society, 1988, p. 145.
	Maskarinec, M. P.; Johnson, L. H.; Holladay, S. K., "Recommendations for Holding Times of Environmental Samples," *Proceedings, Fourth Annual Waste Testing and Quality Assurance Symposium, Volume II*, Washington, DC, July 11-15, 1988, p. H-2.
	Stewart, J. H., Holmes, R. L., Rogers, W. F., Maskarinec, M. P., "Evaluation of a Rapid Quantitative Organic Extraction System for Determining the Concentration of PCB in Soils," *Proceedings of USEPA Symposium on Waste Testing and Quality Assurance*, Vol. II, July 11-15, 1988, Washington, DC, July 11-15, 1988, p. G-37.
McLuckey, S. A.	Busch, K. L.;* Glish, G. L.; McLuckey, S. A., *Mass Spectrometry/Mass Spectrometry: Techniques and Applications of Tandem Mass Spectrometry*," VCH Publishers, Inc., New York, 1988.
	Grant, B. C.; Asano, K. G.; Glish, G. L.; McLuckey, S. A., "Trace Analysis for Organics in Air Using an Atmospheric Sampling Glow Discharge Ionization Source," *Proceedings of the 36th ASMS Conference on Mass Spectrometry and Allied Topics*, 1988, p. 47.
	Goeringer, D. E.; McLuckey, S. A.; Bertram, L. K., "Instrumentation for Mass Spectrometry/Coincident Fragment Detection," *Proceedings of the 36th ASMS Conference on Mass Spectrometry and Allied Topics*, 1988, p. 835.

McLuckey, S. A.

Asano, K. G.; Glish, G. L.; McLuckey, S. A., "Coupling of an Atmospheric Sampling Ion Source with an Ion Trap Mass Spectrometer," *Proceedings of the 36th ASMS Conference on Mass Spectrometry and Allied Topics*, 1988, p. 636.

Van Berkel, G. J.; Glish, G. L.; McLuckey, S. A., "Analysis of Porphyrins Using an Ion Trap Mass Spectrometer," *Proceedings of the 36th ASMS Conference on Mass Spectrometry and Allied Topics*, 1988, p. 639.

McLuckey, S.A.; Asano, K.G.; Glish, G.L., "Negative Chemical Ionization and MS/MS of Negative Ions in an Ion Trap Mass Spectrometer," *Proceedings of the 36th ASMS Conference on Mass Spectrometry and Allied Topics*, 1988, p. 1108.

Glish, G.L.; Van Berkel, G.J.; Asano, K.G.; McLuckey, S.A., "MS^n Experiments with an Ion Trap Mass Spectrometer," *Proceedings of the 36th ASMS Conference on Mass Spectrometry and Allied Topics*, 1988, p.1112.

Van Berkel, G.J.; Glish, G.L.; McLuckey, S.A., "Structural Analysis of Porphyrins: Analysis of In Vitro Prepared Porphyrinogens Using an Ion Trap Mass Spectrometer," *Proceedings of the 36th ASMS Conference on Mass Spectrometry and Allied Topics*, 1988, p. 300.

Glish, G. L.; McLuckey, S. A., "Scan Modes for Hybrid Mass Spectrometers," *Proceedings of the 36th ASMS Conference on Mass Spectrometry and Allied Topics*, 1988, p. 823.

Glish, G. L.; McLuckey, S. A., "Kinetic Energy Release Effects on Resolution in MS/MS," *Proceedings of the 36th ASMS Conference on Mass Spectrometry and Allied Topics*, 1988, p. 825.

Van Berkel, G. S.; Glish, G. L.; McLuckey, S. A., "Mechanism of Porphyrin Hydrogenation and Decomposition in a Chemical Ionization Plasma," *Proceedings of 36th ASMS Conference on Mass Spectrometry and Allied Topics*, 1988, p. 302.

Moneyhun, J. H.

Jenkins, R. A.; Griest, W. H.; Moneyhun, J. H.; Tomkins, B. A.; Ilgner, R. H.; Higgins, C. E.; Gayle, T. M.,* "Sampling and Chemical Characterization of Workplace Atmospheres Contaminated with Airborne Diesel Exhaust," *Proceedings of the 1988 EPA/APCA International Symposium on Measurements of Toxic and Related Air Pollutants*, U.S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory, and the Association Dedicated to Air Pollution Control and Waste Management, Research Triangle Park, NC, May, 1988, p. 119.

Moody, R. L.	Maskarinec, M. P.; Moody, R. L., "Storage and Preservation of Environmental Samples," *Principles of Environmental Sampling*, L. H. Keith, ed., American Chemical Society, 1988, p. 145.
Rogers, W. F.	Stewart, J. H., Holmes, R. L., Rogers, W. F., Maskarinec, M. P., "Evaluation of a Rapid Quantitative Organic Extraction System for Determining the Concentration of PCB in Soils," *Proceedings of USEPA Symposium on Waste Testing and Quality Assurance*, Vol. II, Washington, DC, July 11-15, 1988, p. G-37.
Shaw, R. W.	Smith, D. H.; Shaw, R. W.; Young, J. P., "Resonance Ionization Mass Spectrometry of Promethium," *Proceedings of the 36th ASMS Conference on Mass Spectrometry and Allied Topics*, San Francisco, CA, 1988, p. 210. Shaw, R. W.; Young, J. P.; Smith, D. H., "Resonance Ionization Mass Spectrometric Study of the Promethium-Samarium Isobaric Pair," *Resonance Ionization Spectroscopy 1988* (Institute of Physics Conf. Series No. 94), T. B. Lucarto and J. E. Parks, eds., Inst. of Physics, Bristol, 1988, p. 251. Young, J. P.; Shaw, R. W.; Goeringer, D. E.; Smith, D. H., "Influence of Laser Characteristics on Resonance Ionization Mass Spectrometry," *Resonance Ionization Spectroscopy 1988* (Institute of Physics Conf. Series No. 94), T. B. Lucarto and J. E. Parks, eds., Inst. of Physics, Bristol, 1988, p. 367.
Smith, D. H.	Smith, D. H.; Shaw, R. W.; Young, J. P., "Resonance Ionization Mass Spectrometry of Promethium," *Proceedings of the 36th ASMS Conference on Mass Spectrometry and Allied Topics*, San Francisco, CA, 1988, p. 210. Shaw, R. W.; Young, J. P.; Smith, D. H., "Resonance Ionization Mass Spectrometric Study of the Promethium-Samarium Isobaric Pair," *Resonance Ionization Spectroscopy 1988* (Institute of Physics Conf. Series No. 94), T. B. Lucarto and J. E. Parks, eds., Inst. of Physics, Bristol, 1988, p. 251. Young, J. P.; Shaw, R. W.; Goeringer, D. E.; Smith, D. H., "Influence of Laser Characteristics on Resonance Ionization Mass Spectrometry," *Resonance Ionization Spectroscopy 1988* (Institute of Physics Conf. Series No. 94), T. B. Lucarto and J. E. Parks, eds., Inst. of Physics, Bristol, 1988, p. 367.

Stemmler, E. A.	Buchanan, M. V.; Stemmler, E. A., "Gas Phase Ion-Molecule and Surface-Catalyzed Oxidation Reactions of PAH Under Negative Ion Chemical Ionization Conditions," *Proceedings, 36th Annual Conference on Mass Spectrometry and Allied Topics*, 1988, p. 767. Stemmler, E. A.; Buchanan, M. V., "Isomeric Differentiation of PAH and Methyl-PAH Using Surface Catalyzed Oxidation Reactions," *Proceedings, 36th Annual Conference on Mass Spectrometry and Allied Topics*, 1988, p. 769.
Stewart, J. H.	Stewart, J. H., Holmes, R. L., Rogers, W. F., Maskarinec, M. P., "Evaluation of a Rapid Quantitative Organic Extraction System for Determining the Concentration of PCB in Soils," *Proceedings of USEPA Symposium on Waste Testing and Quality Assurance*, Vol. II, Washington, DC, July 11-15, 1988, p. G-37.
Thompson, C. V.	Jenkins, R. A.; Thompson, C. V.; Higgins, C. E., "Development and Application of a Thermal Desorption-Based Method for the Determination of Nicotine in Indoor Environments," *Indoor and Ambient Air Quality*, R. Perry and P. W Kirk, eds., Selper, Ltd., London, 1988, p. 557.
Tomkins, B. A.	Jenkins, R. A.; Griest, W. H.; Moneyhun, J. H.; Tomkins, B. A.; Ilgner, R. H.; Higgins, C. E.; Gayle, T. M.,* "Sampling and Chemical Characterization of Workplace Atmospheres Contaminated with Airborne Diesel Exhaust," *Proceedings of the 1988 EPA/APCA International Symposium on Measurements of Toxic and Related Air Pollutants*, U.S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory, and the Association Dedicated to Air Pollution Control and Waste Management, Research Triangle Park, NC, May, 1988, p. 119.
Valiga, R. E.	James, R. B.;* Bolton, P. R.;* Alvarez, R. A.;* Christie, W. H.; Valiga, R. E., "Melting of Silicon Surfaces by High-Power Pulsed Microwave Radiation," *SAND* 87-8987, January 1988. Sonder, E.;* Zuhr, R. A.;* Valiga, R. E., "Annealing of Damage and Stability of Implanted Ions in ZnO Crystals," *Bulletin APS*, Vol. 3, No. 3, March 1988.
Van Berkel, G. J.	Van Berkel, G. J.; Glish, G. L.; McLuckey, S. A., "Analysis of Porphyrins Using an Ion Trap Mass Spectrometer," *Proceedings of the 36th ASMS Conference on Mass Spectrometry and Allied Topics*, 1988, p. 639.

Van Berkel, G. S.	Glish, G.L.; Van Berkel, G. L.; Asano, K. G.; McLuckey, S. A., "MS^n Experiments with an Ion Trap Mass Spectrometer," *Proceedings of the 36th MSMS Conference on Mass Spectrometry and Allied Topics*, 1988, p.1112.
	Van Berkel, G. J.; Glish, G. L.; McLuckey, S. A., "Structural Analysis of Porphyrins: Analysis of In Vitro Prepared Porphyrinogens Using an Ion Trap Mass Spectrometer," *Proceedings of the 36th ASMS Conference on Mass Spectrometry and Allied Topics*, 1988, p. 300.
	Van Berkel, G. S.; Glish, G. L.; McLuckey, S. A., "Mechanism of Porphyrin Hydrogenation and Decomposition in a Chemical Ionization Plasma," *Proceedings of 36th ASMS Conference on Mass Spectrometry and Allied Topics*, 1988, p. 302.
Wise, M. B.	Hettich, R. L.; Buchanan, M. V.; Wise, M. B., "Characterization of Substituted Glycerols by Thermal Desoprtion Fourier Transform Mass Spectrometry," *Ohio State University FT/ICR Newsletter*, Alan Marshall, Ed., Spring, 1988.
	Wise, M. B.; Guerin, M. R., "Direct Determination of Part-per-Billion Levels of Volatile Organics in Water and Soil Samples Using a Glow Discharge Mass Spectrometer," *Proceedings, Fourth Annual Waste Testing and Quality Assurance Symposium, Volume II*, Washington, DC, July 11-15, 1988, p. G-53.
Young, J. P.	Smith, D. H.; Shaw, R. W.; Young, J. P., "Resonance Ionization Mass Spectrometry of Promethium," *Proceedings of the 36th ASMS Conference on Mass Spectrometry and Allied Topics*, San Francisco, CA, 1988, p. 210.
	Shaw, R. W.; Young, J. P.; Smith, D. H., "Resonance Ionization Mass Spectrometric Study of the Promethium-Samarium Isobaric Pair," *Resonance Ionization Spectroscopy 1988* (Institute of Physics Conf. Series No. 94), T. B. Lucarto and J. E. Parks, eds., Inst. of Physics, Bristol, p. 251.
	Young, J. P.; Shaw, R. W.; Goeringer, D. E.; Smith, D. H., "Influence of Laser Characteristics on Resonance Ionization Mass Spectrometry," *Resonance Ionization Spectroscopy 1988* (Institute of Physics Conf. Series No. 94), T. B. Lucarto and J. E. Parks, eds., Inst. of Physics, Bristol, p. 367.

Articles

Asano, K. G.	McLuckey, S. A.; Glish, G. L.; Asano, K. G.; Grant, B. C., "Atmospheric Sampling Glow Discharge Ionization Source for the Analysis of Trace Organics in Ambient Air," *Anal. Chem.* 60, 2220 (1988).
	McLuckey, K. G.; Asano, K. G.; Glish, G. L.; Van Berkel, G. J., "Self Chemical Ionization in an Ion Trap Mass Spectrometer," *Anal. Chem.* 60, 2312 (1988).
Blazewicz, P. R.	Nolan, T. G.; Koutny,* L. B.; Blazewicz, P. R.; Whitten, W. B.; Ramsey, J. M., "Low-Pressure Degenerate Four-Wave Mixing Spectroscopy with Flame Atomization," *Appl. Spectrosc.* 42, 1045 (1988).
Buchanan, M. V.	Haas, J. W.;* Buchanan, M. V.; Wise, M. B., "Differentiation of Polycyclic Aromatic Hydrocarbons Using a Multimode Ionization Chromatographic Detector," *J. of Chromatogr. Sci.* 26, 29 (1988).
	Stemmler, E. A.; Buchanan, M. V., "Differentiation of Methyl-Substituted Fluorenes, Anthracenes and Benz(a)anthracenes Using Surface-Catalyzed Oxidation Reactions and Negative Ion Chemical Ionization Mass Spectrometry," *Rapid Commun. in Mass Spectrom.* 2, 184 (1988).
Christie, W. H.	Geohegan, D. B.;* Masburn, D. N.;* Culbertson, R. J.;* Pennycook, S. J.;* Budai, S. J.;* Valiga, R. E.; Sales, B. C.;* Lowndes, D. H.;* Boatner, L. A.;* Sonder, E.;* Eres, D.;* Christen, D. K.*; Christie, W. H., "Pulsed Laser Deposition of Thin Superconducting Films of $Ho_1Ba_2Cu_3O_7$-x and $Y_1Ba_2Cu_3O_7$-x," *J. Mater. Res.* 3, 1170 (1988).
	James, R. B.;* Bolton, P. R.;* Alvarez, R. A.;* Christie, W. H.;* Valiga, R. E., "Melting of Silicon Surfaces by High-Power Pulsed Microwave Radiation," *J. Appl. Phys.* 64, 3243 (1988).
	Goeringer, D. E.; Christie, W. H.; Valiga, R. E., "Investigation of Matrix Effects on the Neutral Fractions Ejected from Ion-Bombarded, Uranium-Containing Solids Using Resonance Ionization Mass Spectrometry," *Anal. Chem.* 60, 345 (1988).

Christie, W. H.	Young, J. P.; Shaw, R. W.; Goeringer, D. E.; Smith, D. H.; Christie, W. H., "Analytical Capabilities of Inorganic Resonance Ionization Mass Spectrometry," *Anal. Instrum.* 17, 41, (1988).
Donohue, D. L.	Fiedler, R.* and Donohue, D. L., "Pocket Sensitivity Calibration of Multicollector Mass Spectrometers," *Fresenius Z. Anal. Chem.* 331, 209 (1988).
	Smith, D. H.; McKown H. S.; Young, J. P.; Shaw, R. W.; Donohue, D. L., "Prediction and Identification of Multiple-Photon Resonant Ionization Processes," *Appl. Spectrosc.* 42, 1057 (1988).
Dyer, F. F.	Dyer, F. F.; Emery, J. F.; Robinson, L.; Teasley, N. A., "Design and Use of the ORNL HFIR Pneumatic Tube Irradiation Systems," *J. Trace and Microprobe Techniques* 6, 147 (1988).
	Dyer, F. F.; Hobbs, R. W.;* Pugh, L. P.;* Snelgrove, J. L.,* "An Automated Fuel Element Scanning System," *J. of Radioanal. Nucl. Chem. Art.* 25, 317, 1988.
Emery, J. F.	Dyer, F. F.; Emery, J. F.; Robinson, L.; Teasley, N. A., "Design and Use of the ORNL HFIR Pneumatic Tube Irradiation Systems," *J. Trace and Microprobe Techniques* 6, 147 (1988).
Ghodbane, S.	Guiochon, G. A.; Ghodbane, S., "La Solution du Probleme a Deux Corps en Chromatographie Semi-Ideale a Forte Concentration," *Comptes Rendus de l'Academie des Sciences* (Paris) 306 (Series II), 575 (1988).
	Guiochon, G. A.; Ghodbane, S., "Computer Simulation of the Separation of a Two-Component Mixture in Preparative Scale Liquid Chromatography," *J. of Phys. Chem.* 92, 3682 (1988).
	Ghodbane, S.; Guiochon, G. A., "Study of the Influence of the Relative Concentration of Two Components of a Binary Mixture on Their Overloaded Elution Profile," *J. of Chromatogr.* 440, 9 (1988).
	Guiochon, G. A.; Ghodbane, S., "La Separation d'un Melange de deux Corps in Chromatographie Semi-ideale a Forte Concentration," *Bulletin de la Societe Chimique de France* 440 (1988).

Ghodbane, S.	Ghodbane, S.; Guiochon, G. A., "Optimization of Concentration Overload in Preparative Liquid Chromatography," *J. of Chromatogr.* 444, 275 (1988).
	Ghodbane, S.; Guiochon, G. A., "Column Loading and Relative Retention in Overloaded Elution Chromatography," *J. of Chromatogr.* 450, 27 (1988).
	Ghodbane, S.; Guiochon, G. A., "Effect of Mobile Phase Velocity on the Recovery Yields and Production Rates in Overloaded Elution Chromatography," *J. of Chromatogr.* 452, 209 (1988).
	Golshan-Shirazi, S.;* Ghodbane, S.; Guiochon, G. A., "Comparison Between Experimental and Theoretical Band Profiles in Non-Linear Liquid Chromatography with a Pure Mobile Phase," *Anal. Chem.* 60, 2630 (1988).
Glish, G. L.	McLuckey, S. A.; Glish, G. L.; Asano, K. G.; Grant, B. C. "Atmospheric Sampling Glow Discharge Ionization Source for the Analysis of Trace Organics in Ambient Air," *Anal. Chem.* 60, 2220 (1988).
	McLuckey, K. G.; Asano, K. G.; Glish, G. L.; Van Berkel, G. J., "Self Chemical Ionization in an Ion Trap Mass Spectrometer," *Anal. Chem.* 60, 2312 (1988).
Grant, B. C.	McLuckey, S. A.; Glish, G. L.; Asano, K. G.; Grant, B. C., "Atmospheric Sampling Glow Discharge Ionization Source for the Analysis of Trace Organics in Ambient Air," *Anal. Chem.* 60, 2220 (1988).
Griest, W. H.	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," *Sep. Sci. and Technol.* 23, 1905 (1988).
	Griest, W. H.; Tomkins, B. A.; Caffrey, J. R.,* "Improved Ultrasonic Extraction Recovery of Benzo(a)pyrene from Stack Ash Using High Power/Mass Ratios," *Anal. Chem.* 60, 2169 (1988).
Goeringer, D. E.	Goeringer, D. E.; Christie, W. H.; Valiga, R. E., "Investigation of Matrix Effects on the Neutral Fractions Ejected from Ion-Bombarded, Uranium-Containing Solids Using Resonance Ionization Mass Spectrometry," *Anal. Chem.* 60, 345 (1988).

Goeringer, D. E.

Young, J. P.; Shaw, R. W.; Goeringer, D. E.; Smith, D. H.; Christie, W. H., "Analytical Capabilities of Inorganic Resonance Ionization Mass Spectrometry," *Anal. Instrum.* 17, 41, (1988).

Guiochon, G. A.

Guiochon, G. A., Ghodbane, S., "La Solution du Probleme a Deux Corps en Chromatographie Semi-Ideale a Forte Concentration," *Comptes Rendus de l'Academie des Sciences* (Paris) 306 (Series II), 575 (1988).

Guiochon, G. A.; Ghodbane, S., "Computer Simulation of the Separation of a Two-Component Mixture in Preparative Scale Liquid Chromatography," *J. of Phys. Chem.* 92, 3682 (1988).

Ghodbane, S.; Guiochon, G. A., "Study of the Influence of the Relative Concentration of Two Components of a Binary Mixture on Their Overloaded Elution Profile," *J. of Chromatogr.* 440, 9 (1988).

Guiochon, G. A.; Ghodbane, S., "La Separation d'un Melange de deux Corps in Chromatographie Semi-ideale a Forte Concentration," *Bulletin de la Societe Chimique de France* (1988).

Ghodbane, S.; Guiochon, G. A., "Optimization of Concentration Overload in Preparative Liquid Chromatography," *J. of Chromatogr.* 444, 275 (1988).

Guiochon, G. A.; Golshan-Shirazi, S.;* Jaulmes, A.,* "Computer Simulation of the Propagation of a Large Concentration Band in Liquid Chromatography," *Anal. Chem.* 60, 1856 (1988).

Tchapla, A.;* Heron, S.;* Colin, H.;* Guiochon, G. A., "The Behavior of Homologous Series in Reverse Phase Liquid Chromatography: Role of the Temperature," *Anal. Chem.* 60, 1443 (1988).

Lin, B.; Guiochon, G. A., "Numerical Simulations of Chromatographic Band Profiles at Large Concentrations. Length of Space Increment and Height Equivalent to a Theoretical Plate," *Sep. Sci. and Technol.* 24, 31 (1988).

Ghodbane, S.; Guiochon, G. A., "Column Loading and Relative Retention in Overloaded Elution Chromatography," *J. of Chromatogr.* 450, 27 (1988).

Guiochon, G. A.

Righezza, M. F.; Guiochon, G. A., "Effects of the Nature of the Solvent and Solutes on the Response of a Light Scattering Detector," *J. of Liq. Chromatogr.* 11, 1967 (1988).

Ghodbane, S.; Guiochon, G. A., "Effect of Mobile Phase Velocity on the Recovery Yields and Production Rates in Overloaded Elution Chromatography," *J. of Chromatogr.* 452, 209 (1988).

Huang, J. X.;* Guiochon, G. A., "Application of Preparative HPLC to Peptides and Proteins," *Biochromatogr.* 3, 140 (1988).

Righezza, M. F.; Guiochon, G. A., "Effect of the Wavelength of the Laser Beam on the Response of an Evaporative Light Scattering Detector," *J. of Liq. Chromatogr.* 10, 234 (1988).

Schudel, J. V. H.; Guiochon, G. A., "The Effect of Random Noise and Peak Asymmetry on the Precision and Accuracy of Measurements of the Column Efficiency in Chromatography," *J. of Chromatogr.* 457, 1 (1988).

Katti, A. M.; Guiochon, G. A., "Prediction of Band Profiles in Displacement Chromatography by Numerical Integration of a Semi-ideal Model," *J. of Chromatogr.* 449, 25 (1988).

Golshan-Shirazi, S.;* Guiochon, G. A., "Study of the Elution Profiles of Large Concentration Chromatographic Bands. Special Properties in the Case of a Langmuir Isotherm," *Anal. Chem.* 60, 2364 (1988).

Golshan-Shirazi, S.;* Ghodbane, S.; Guiochon, G. A., "Comparison Between Experimental and Theoretical Band Profiles in Non-Linear Liquid Chromatography with a Pure Mobile Phase," *Anal. Chem.* 60, 2630 (1988).

Golshan-Shirazi, S.;* Guiochon, G. A., "Comparison Between Experimental and Theoretical Band Profiles in Non-Linear Liquid Chromatography with a Binary Mobile Phase," *Anal. Chem.* 60, 2634 (1988).

Lin, B.;* Golshan-Shirazi, S.;* Ma, Z.;* Guiochon, G. A., "Shock Effects in Non-Linear Chromatography," *Anal. Chem.* 61, 2647 (1988).

Guiochon, G. A.	Guiochon, G. A.; Moysan, A.;* Holley, C.,* "Influence of Various Parameters on the Response Factors of the Evaporative Light Scattering Detector for a Number of Non-Volatile Compounds," *J. of Liq. Chromatogr.* 10, 2547 (1988).
Katti, A. M.	Katti, A. M.; Guiochon, G. A., "Prediction of Band Profiles in Displacement Chromatography by Numerical Integration of a Semi-ideal Model," *J. of Chromatogr.* 449, 25 (1988).
Leibman, C. P.	Leibman, C. P.; Todd, P. J.; Mamantov, G., "Enhanced Positive Secondary Ion Emission from Substituted Polynuclear Aromatic Hydrocarbon/Sulfuric Acid Solution," *Org. Mass Spectrom.* 23, 634 (1988).
Lyon, W. S.	Lyon, W. S., "The Energy Idea Drought," *J. Radioanal. Nucl. Chem. Lett.* 128, 251 (1988).
	Lyon, W. S., Book Review, "Survival Strategies for New Scientists," *Scientometrics* 13, 173 (1988).
Maskarinec, M. P.	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," *Sep. Sci. and Technol.* 23, 1905 (1988).
McKown, H. S.	Smith, D. H.; McKown H. S.; Young, J. P.; Shaw, R. W.; Donohue, D. L., "Prediction and Identification of Multiple-Photon Resonant Ionization Processes," *Appl. Spectrosc.* 42, 1057 (1988).
McLuckey, S. A.	McLuckey, S. A.; Glish, G. L.; Asano, K. G.; Grant, B. C., "Atmospheric Sampling Glow Discharge Ionization Source for the Analysis of Trace Organics in Ambient Air," *Anal. Chem.* 60, 2220 (1988).
	McLuckey, K. G.; Asano, K. G.; Glish, G. L.; Van Berkel, G. J., "Self Chemical Ionization in an Ion Trap Mass Spectrometer," *Anal. Chem.* 60, 2312 (1988).
Moody, R. L.	Vo-Dinh, T.;* Alak, A.;* Moody, R. L., "Recent Advances in Surface-Enhanced Raman Spectrometry for Chemical Analysis," *Spectrochimica Acta* 43B, 605 (1988).

Nolan, T. G.	Nolan, T. G.; Koutny, L. B.;* Blazewicz, P. R.; Whitten, W. B.; Ramsey, J. M., "Low-Pressure Degenerate Four-Wave Mixing Spectroscopy with Flame Atomization," *Appl. Spectrosc.* **42**, 1045 (1988). Koutny, L. B.;* Whitten, W. B.; Nolan, T. G.; Ramsey, J. M., "Supersonic Jet Atomic Spectroscopy with Flame Atomization," *Anal. Chem.* **60**, 958 (1988).
Ramsey, J. M.	Nolan, T. G.; Koutny, L. B.;* Blazewicz, P. R.; Whitten, W. B.; Ramsey, J. M., "Low-Pressure Degenerate Four-Wave Mixing Spectroscopy with Flame Atomization," *Appl. Spectrosc.* **42**, 1045 (1988). Koutny, L. B.;* Whitten, W. B.; Nolan, T. G.; Ramsey, J. M., "Supersonic Jet Atomic Spectroscopy with Flame Atomization," *Anal. Chem.* 60, 958 (1988).
Righezza, M. F.	Righezza, M. F.; Guiochon, G. A., "Effects of the Nature of the Solvent and Solutes on the Response of a Light Scattering Detector," *J. of Liq. Chromatogr.* **11**, 1967 (1988). Righezza, M. F.; Guiochon, G. A., "Effect of the Wavelength of the Laser Beam on the Response of an Evaporative Light Scattering Detector," *J. of Liq. Chromatogr.* **10**, 234 (1988).
Robinson, L.	Dyer, F. F.; Emery, J. F.; Robinson, L.; Teasley, N. A., "Design and Use of the ORNL HFIR Pneumatic Tube Irradiation Systems," *J. Trace and Microprobe Techniques* **6**, 147-159 (1988).
Ross, H. H.	Rucker, T. L.; Ross, H. H.; Schweitzer, G. K.* "Computer Modeling of Fiber-Scintillator Flow-Cell Radiation Detector Geometry," *Trans. Am. Nucl. Soc.* 55, 52 (1987). Rucker, T. L.; Ross, H. H.; Schweitzer, G. K.,* "Monte Carlo Modeling of Fiber-Scintillator Flow-Cell Radiation Detector Geometry," *Nucl. Instrum. Methods in Phys. Res.* A267, 511 (1988). Rucker, T. L.; Ross, H. H.; Schweitzer, G. K.,* "Scintillator-Fiber Flow-Cell Radioactivity Detector for Liquid Chromatography," *Chromatographia* **25**, 31 (1988).

Row, K. H.	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," *Sep. Sci. and Technol.* 23, 1905 (1988).
	Row, K. H., "Comparison of the Elution Curves of Pure Components and Their Mixtures and Prediction of Elution Curves by a Theoretical Plate Model in Preparative Gas-Liquid Chromatography," *Chromatographia* 25, 961 (1988).
Schudel, J. V. H.	Schudel, J. V. H., Guiochon, G. A., "The Effect of Random Noise and Peak Asymmetry on the Precision and Accuracy of Measurements of the Column Efficiency in Chromatography," *J. of Chromatogr.* 457, 1 (1988).
Shaw, R. W.	Shaw, R. W.; Young, J. P.; Smith, D. H., "Removal of the Samarium Isobaric Interference from Promethium Mass Analysis," *Anal. Chem.* 60, 282 (1988).
	Smith, D. H.; McKown H. S.; Young, J. P.; Shaw, R. W.; Donohue, D. L., "Prediction and Identification of Multiple-Photon Resonant Ionization Processes," *Appl. Spectrosc.* 42, 1057 (1988).
	Young, J. P.; Shaw, R. W.; Goeringer, D. E.; Smith, D. H.; Christie, W. H., "Analytical Capabilities of Inorganic Resonance Ionization Mass Spectrometry," *Anal. Instrum.* 17, 41, 1988.
Smith, D. H.	Shaw, R. W.; Young, J. P.; Smith, D. H., "Removal of the Samarium Isobaric Interference from Promethium Mass Analysis," *Anal. Chem.* 60, 282 (1988).
	Smith, D. H.; McKown H. S.; Young, J. P.; Shaw, R. W.; Donohue, D. L., "Prediction and Identification of Multiple-Photon Resonant Ionization Processes," *Appl. Spectrosc.* 42, 1057 (1988).
	Young, J. P.; Shaw, R. W.; Goeringer, D. E.; Smith, D. H.; Christie, W. H., "Analytical Capabilities of Inorganic Resonance Ionization Mass Spectrometry," *Anal. Instrum.* 17, 41, (1988).
Stemmler, E. A.	Stemmler, E. A.; Buchanan, M. V., "Differentiation of Methyl-Substituted Fluorenes, Anthracenes and Benz(a)anthracencas Using Surface-Catalyzed Oxidation Reactions and Negative Ion Chemical Ionization Mass Spectrometry," *Rapid Commun. in Mass Spectrom.* 2, 184 (1988).

Teasley, N. A.	Dyer, F. F.; Emery, J. F.; Robinson, L.; Teasley, N. A., "Design and Use of the ORNL HFIR Pneumatic Tube Irradiation Systems," *J. Trace and Microprobe Techniques* 6, 147 (1988).
Todd, P. J.	Todd, P. J., "Secondary Ion Emission from Glycerol under Continuous and Pulsed Primary Ion Current," *Org. Mass Spectrom.* 23, 419 (1988). Leibman, C. P.; Todd, P. J.; Mamantov, G., "Enhanced Positive Secondary Ion Emission from Substituted Polynuclear Aromatic Hydrocarbon/Sulfuric Acid Solution," *Org. Mass Spectrom.* 23, 634 (1988).
Tomkins, B. A.	Griest, W. H.; Tomkins, B. A.; Caffrey, J. R.,* "Improved Ultrasonic Extraction Recovery of Benzo(a)pyrene from Stack Ash Using High Power/Mass Ratios," *Anal. Chem.* 60, 2169 (1988).
Valiga, R. E.	Goeringer, D. E.; Christie, W. H.; Valiga, R. E., "Investigation of Matrix Effects on the Neutral Fractions Ejected from Ion-Bombarded, Uranium-Containing Solids Using Resonance Ionization Mass Spectrometry," *Anal. Chem.* 60, 345 (1988). James, R. B.;* Bolton, P. R.;* Alvarez, R. A.;* Christie, W. H.; Valiga, R. E., "Melting of Silicon Surfaces by High-Power Pulsed Microwave Radiation," *J. Appl. Phys.* 64, 3243, (1988). Sonder, E.;* Zuhr, R. A.;* Valiga, R. E., "Annealing of Damage and Stability of Implanted Ions in ZnO Crystals," *J. Appl. Phys.* 64 1140 (1988). Geohegan, D. B.;* Masburn, D. N.;* Culbertson, R. J.;* Pennycook, S. J.;* Budai, S. J.;* Valiga, R. E.; Sales, B. C.;* Lowndes, D. H.;* Boatner, L. A.;* Sonder, E.;* Eres, D.;* Christen, D. K.; Christie, W. H., "Pulsed Laser Deposition of Thin Superconducting Films of $Ho_1Ba_2Cu_3O_7$-x and $Y_1Ba_2Cu_3O_7$-x," *J. Mater. Res.* 3, 1170 (1988).
Van Berkel, G. J.	McLuckey, K. G.; Asano, K. G.; Glish, G. L.; Van Berkel, G. J., "Self Chemical Ionization in an Ion Trap Mass Spectrometer," *Anal. Chem.* 60, 2312 (1988).

Whitten, W. B.	Koutny, L. B.;* Whitten, W. B.; Nolan, T. G.; Ramsey, J. M., "Supersonic Jet Atomic Spectroscopy with Flame Atomization," *Anal. Chem.* 60, 958 (1988). Nolan, T. G.; Koutny, L. B.;* Blazewicz, P. R.; Whitten, W. B.; Ramsey, J. M., "Low-Pressure Degenerate Four-Wave Mixing Spectroscopy with Flame Atomization," *Appl. Spectrosc.* 42, 1045 (1988).
Wise, M. B.	Haas, J. W.;* Buchanan, M. V.; Wise, M. B., "Differentiation of Polycyclic Aromatic Hydrocarbons Using a Multimode Ionization Chromatographic Detector," *J. of Chromatogr. Sci.* 26, 29 (1988).
Young, J. P.	Young, J. P.; Shaw, R. W.; Goeringer, D. E.; Smith, D. H.; Christie, W. H., "Analytical Capabilities of Inorganic Resonance Ionization Mass Spectrometry," *Anal. Instrum.* 17, 41 (1988). Young, J. P., "Analytical Applications of Resonance Ionization Spectroscopy (RIS) are Expanding," *Trends in Analytical Chemistry* 7, 237 (1988). Wilmarth, W. R.;* Haire, R. G.;* Young, J. P.; Ramey, D. W.;* Peterson, J. R.,* "Absorption Spectrophotometric and X-ray Diffraction Studies of the Trihalides of Promethium in the Solid State," *J. Less-Common Met.* 141, 275 (1988). Smith, D. H.; McKown H. S.; Young, J. P.; Shaw, R. W.; Donohue, D. L., "Prediction and Identification of Multiple-Photon Resonant Ionization Processes," *Appl. Spectrosc.* 42, 1057 (1988). Shaw, R. W.; Young, J. P.; Smith, D. H., "Removal of the Samarium Isobaric Interference from Promethium Mass Analysis," *Anal. Chem.* 60, 282 (1988).

Reports

Buchanan, M. V.	Jenkins, R. A.; Maskarinec, M. P.; Griest, W. H.; Dyer, F. F.; Moody, R. L.; Ho, C.-h.; Buchanan, M. V., *Technology Assessment of Field Portable Instrumentation for Use at Rocky Mountain Arsenal*, ORNL/TM-10542, June 1988. Gammage, R. B.;* Buchanan, M. V.; Higgins, C. E.; White, D. A.;* Dreibelbis, W. G.;* Olerich, G.; Guerin, M. R.; Hawthorne, A. R.,* *Measurement of Volatile Organic Compounds (VOC) in Eight East Tennessee Homes*, ORNL-6286, September 1988.
Carter, J. A.	Carter, J. A., *Analytical Chemistry Division Research and Development Summary: Analytical Spectroscopy Section*, ORNL/CF-88/50, February 1, 1988. Carter, J. A., *Analytical Chemistry Division Research and Development Summary: Analytical Spectroscopy Section*, ORNL/CF-88/205, June 1, 1988. Carter, J. A., *Analytical Chemistry Division Research and Development Summary: Analytical Spectroscopy Section*, ORNL/CF-88/335, November 10, 1988 Carter, J. A.; Smith, D. H.; Walker, R. L.; May, M. P., *An Independent Technique for Fissile Inventory Verification of Holding Tanks in the Nuclear Fuel Cycle: Status Report*, ORNL/TM-10814, May 1988.
Christie, W. H.	Christie, W. H., *Report of Foreign Travel of W. H. Christie*, ORNL/FTR-3044, September 1988.
Dale, J. M.	Dale, J. M.; Keller, J. M.; Sparks, C. J, Jr.,* *Part II: Evaluation of a Wavelength Dispersive X-Ray System for the Determination of Uranium and Plutonium in Highly Radioactive Solutions*, ORNL/6407, November 1987.
Dyer, F. F.	Jenkins, R. A.; Maskarinec, M. P.; Griest, W. H.; Dyer, F. F.; Moody, R. L.; Ho, C.-h.; Buchanan, M. V., *Technology Assessment of Field Portable Instrumentation for Use at Rocky Mountain Arsenal*, ORNL/TM-10542, June 1988.
Glish, G. L.	Glish, G. L.; McLuckey, S. A., *Evaluation of the Ion Trap Mass Spectrometer for Potential Application in the Space Station*, ORNL/TM-10751, April 1988.

Goeringer, D. E.	Smith, D. H.; Goeringer, D. E., *An Expeditious Technique for Keyboard Input of Information to FORTRAN Computer Programs*, ORNL/TM-10770, May 1988.
Griest, W. H.	Ross, R. H.;* Pal, B. C.;* Ramsey, R. S.; Jenkins, R. A.; Lock, S.;* Griest, W. H.; Guerin, M. R., *Problem Definition Study on Techniques and Methodologies for Evaluating the Chemical and Toxicological Properties of Combustion Products of Gun Systems, Volume 1*, Department of Defense FINAL REPORT, ORNL-634/V1, March 1988.
	Jenkins, R. A.; Maskarinec, M. P.; Griest, W. H.; Dyer, F. F.; Moody, R. L.; Ho, C.-h.; Buchanan, M. V., *Technology Assessment of Field Portable Instrumentation for Use at Rocky Mountain Arsenal*, ORNL/TM-10542, June 1988.
	Griest, W. H.; Jenkins, R. A.; Tomkins, B. A.; Moneyhun, J. H.; Ilgner, R. H.; Gayle, T. M.;* Higgins, C. E.; Guerin, M. R., *Sampling and Analysis of Diesel Exhaust and the Motor Pool Workplace Atmosphere*, ORNL/TM-10689, June 1988.
	Griest, W. H.; Guerin, M. R.; Smith, L. H.;* Witschi, H. P.;* Higgins, C. E.; Tomkins, B. A.; Ho, C.-h.; Ilgner, R. H., *Synthetic and Alternate Fuels Characterization*, ORNL/TM-10706, August 1988.
	McMahon, J. M.; Tomkins, B. A.; Griest, W. H., *Leaching Studies of Uranium-Bearing Waste Solids from Manufacturing Operations, Chapter 7 in Data Package for the Low-Level Waste Disposal Development and Demonstration Program Environmental Impact Statement*, ORNL/TM-10939, Volume 1, September 1988.
Guerin, M. R.	Ross, R. H.;* Pal, B. C.;* Ramsey, R. S.; Jenkins, R. A.; Lock, S.;* Griest, W. H.; Guerin, M. R., *Problem Definition Study on Techniques and Methodologies for Evaluating the Chemical and Toxicological Properties of Combustion Products of Gun Systems, Volume 1*, Department of Defense FINAL REPORT, ORNL-634/V1, March 1988.
	Griest, W. H.; Jenkins, R. A.; Tomkins, B. A.; Moneyhun, J. H.; Ilgner, R. H.; Gayle, T. M.;* Higgins, C. E.; Guerin, M. R., *Sampling and Analysis of Diesel Exhaust and the Motor Pool Workplace Atmosphere*, ORNL/TM-10689, June 1988.
	Griest, W. H.; Guerin, M. R.; Smith, L. H.;* Witschi, H. P.;* Higgins, C. E.; Tomkins, B. A.; Ho, C.-h.; Ilgner, R. H., *Synthetic and Alternate Fuels Characterization*, ORNL/TM-10706, August 1988.

Guerin, M. R.	Gammage, R. B.;* Buchanan, M. V.; Higgins, C. E.; White, D. A.;* Dreibelbis, W. G.;* Olerich, G.;* Guerin, M. R.; Hawthorne, A. R.,* *Measurement of Volatile Organic Compounds (VOC) in Eight East Tennessee Homes*, ORNL-6286, September 1988. Guerin, M. R., *Analytical Chemistry Division Research and Development Summary; February, 1988, Organic Chemistry Section*, ORNL/CF-88/93, March 31, 1988. Guerin, M. R., *Analytical Chemistry Division Research and Development Summary; June, 1988, Organic Chemistry Section*, ORNL/CF-88/266, July 31, 1988. Guerin, M. R., *Analytical Chemistry Division Research and Development Summary; October, 1988, Organic Chemistry Section*, ORNL/CF-88/364, November 30, 1988.
Higgins, C. E.	Griest, W. H.; Jenkins, R. A.; Tomkins, B. A.; Moneyhun, J. H.; Ilgner, R. H.; Gayle, T. M.;* Higgins, C. E.; Guerin, M. R., *Sampling and Analysis of Diesel Exhaust and the Motor Pool Workplace Atmosphere*, ORNL/TM-10689, June 1988. Griest, W. H.; Guerin, M. R.; Smith, L. H.;* Witschi, H. P.;* Higgins, C. E.; Tomkins, B. A.; Ho, C.-h.; Ilgner, R. H., *Synthetic and Alternate Fuels Characterization*, ORNL/TM-10706, August 1988. Gammage, R. B.;* Buchanan, M. V.; Higgins, C. E.; White, D. A.;* Dreibelbis, W. G.;* Olerich, G.;* Guerin, M. R.; Hawthorne, A. R.,* *Measurement of Volatile Organic Compounds (VOC) in Eight East Tennessee Homes*, ORNL-6286, September 1988.
Ho, C.-h.	Jenkins, R. A.; Maskarinec, M. P.; Griest, W. H.; Dyer, F. F.; Moody, R. L.; Ho, C.-h.; Buchanan, M. V., *Technology Assessment of Field Portable Instrumentation for Use at Rocky Mountain Arsenal*, ORNL/TM-10542, June 1988. Griest, W. H.; Guerin, M. R.; Smith, L. H.;* Witschi, H. P.;* Higgins, C. E.; Tomkins, B. A.; Ho, C.-h.; Ilgner, R. H., *Synthetic and Alternate Fuels Characterization*, ORNL/TM-10706, August 1988.
Hulett, L. D., Jr.	Hulett, L. D., Jr., *Report of Foreign Travel*, ORNL/FTR-3048, September 26, 1988
Ilgner, R. H.	Griest, W. H.; Jenkins, R. A.; Tomkins, B. A.; Moneyhun, J. H.; Ilgner, R. H.; Gayle, T. M.;* Higgins, C. E.; Guerin, M. R., *Sampling and Analysis of Diesel Exhaust and the Motor Pool Workplace Atmosphere*, ORNL/TM-10689, June 1988.

Ilgner, R. H.	Griest, W. H.; Guerin, M. R.; Smith, L. H.;* Witschi, H. P.;* Higgins, C. E.; Tomkins, B. A.; Ho, C.-h.; Ilgner, R. H., *Synthetic and Alternate Fuels Characterization*, ORNL/TM-10706, August 1988.
Jenkins, R. A.	Ross, R. H.;* Pal, B. C.;* Ramsey, R. S.; Jenkins, R. A.; Lock, S.;* Griest, W. H.; Guerin, M. R., *Problem Definition Study on Techniques and Methodologies for Evaluating the Chemical and Toxicological Properties of Combustion Products of Gun Systems, Volume 1*, Department of Defense FINAL REPORT, ORNL-634/V1, March 1988. Jenkins, R. A.; Maskarinec, M. P.; Griest, W. H.; Dyer, F. F.; Moody, R. L.; Ho, C.-h.; Buchanan, M. V., *Technology Assessment of Field Portable Instrumentation for Use at Rocky Mountain Arsenal*, ORNL/TM-10542, June 1988. Griest, W. H.; Jenkins, R. A.; Tomkins, B. A.; Moneyhun, J. H.; Ilgner, R. H.; Gayle, T. M.;* Higgins, C. E.; Guerin, M. R., *Sampling and Analysis of Diesel Exhaust and the Motor Pool Workplace Atmosphere*, ORNL/TM-10689, June 1988.
Klatt, L. N.	Klatt, L. N., *A Summary of In-Line Monitor Developments*, ORNL/TM-10713, April 1988. Klatt, L. N., *A Summary of Hot-Cell Laboratory Instrument Developments*, ORNL/TM-10774, July 1988. Klatt, L. N., *A Hot-Cell Titration System*, ORNL/TM-10780, July 1988.
Keller, J. M.	Dale, J. M.; Keller, J. M.; Sparks, C. J, Jr.,* *Part II: Evaluation of a Wavelength Dispersive X-Ray System for the Determination of Uranium and Plutonium in Highly Radioactive Solutions*, ORNL/6407, November 1987.
Laing, W. R.	Laing, W. R., *Analytical Chemistry Division Summary Report: Inorganic Chemistry Section*, ORNL/CF-88/41, February 1, 1988. Laing, W. R., *Analytical Chemistry Division Summary Report: Inorganic Chemistry Section*, ORNL/CF-88/159, June 1, 1988. Laing, W. R., *Analytical Chemistry Division Summary Report: Inorganic Chemistry Section*, ORNL/CF-88/309, October 1, 1988.

Maskarinec, M. P.	Jenkins, R. A.; Maskarinec, M. P.; Griest, W. H.; Dyer, F. F.; Moody, R. L.; Ho, C.-h.; Buchanan, M. V., *Technology Assessment of Field Portable Instrumentation for Use at Rocky Mountain Arsenal*, ORNL/TM-10542, June 1988.
May, M. P.	Carter, J. A.; Smith, D. H.; Walker, R. L.; May, M. P., *An Independent Technique for Fissile Inventory Verification of Holding Tanks in the Nuclear Fuel Cycle: Status Report*, ORNL/TM-10814, May 1988.
McLuckey, S. A.	Glish, G. L.; McLuckey, S. A., *Evaluation of the Ion Trap Mass Spectrometer for Potential Application in the Space Station*, ORNL/TM-10751, April 1988.
McMahon, J. M.	McMahon, J. M.; Tomkins, B. A.; Griest, W. H., *Leaching Studies of Uranium-Bearing Waste Solids from Manufacturing Operations, Chapter 7 in Data Package for the Low-Level Waste Disposal Development and Demonstration Program Environmental Impact Statement*, ORNL/TM-10939, Volume 1, September 1988.
Moneyhun, J. H.	Griest, W. H.; Jenkins, R. A.; Tomkins, B. A.; Moneyhun, J. H.; Ilgner, R. H.; Gayle, T. M.;* Higgins, C. E.; Guerin, M. R., *Sampling and Analysis of Diesel Exhaust and the Motor Pool Workplace Atmosphere*, ORNL/TM-10689, June 1988.
Moody, R. L.	Jenkins, R. A.; Maskarinec, M. P.; Griest, W. H.; Dyer, F. F.; Moody, R. L.; Ho, C.-h.; Buchanan, M. V., *Technology Assessment of Field Portable Instrumentation for Use at Rocky Mountain Arsenal*, ORNL/TM-10542, June 1988.
Ramsey, J. M.	Ramsey, J. M., *Report of Foreign Travel*, ORNL/FTR-2867, May 13, 1988.
Ramsey, R. S.	Ross, R. H.;* Pal, B. C.;* Ramsey, R. S.; Jenkins, R. A.; Lock, S.;* Griest, W. H.; Guerin, M. R., *Problem Definition Study on Techniques and Methodologies for Evaluating the Chemical and Toxicological Properties of Combustion Products of Gun Systems, Volume 1*, Department of Defense FINAL REPORT, ORNL-634/V1, March 1988.
Smith, D. H.	Smith, D. H.; Goeringer, D. E., *An Expeditious Technique for Keyboard Input of Information to FORTRAN Computer Programs*, ORNL/TM-10770, May 1988.
	Carter, J. A.; Smith, D. H.; Walker, R. L.; May, M. P., *An Independent Technique for Fissile Inventory Verification of Holding Tanks in the Nuclear Fuel Cycle: Status Report*, ORNL/TM-10814, May 1988.

Stokely, J. R.	Stokely, J. R., *Analytical Chemistry Division Summary Report: Radioactive Materials Analysis Section*, ORNL/CF-88/33, February 1, 1988.
	Stokely, J. R., *Analytical Chemistry Division Summary Report: Radioactive Materials Analysis Section*, ORNL/CF-88/238, June 1, 1988.
	Stokely, J. R., *Analytical Chemistry Division Summary Report: Radioactive Materials Analysis Section*, ORNL/CF-88/331, November 4, 1988.
Tomkins, B. A.	Griest, W. H.; Jenkins, R. A.; Tomkins, B. A.; Moneyhun, J. H.; Ilgner, R. H.; Gayle, T. M.;* Higgins, C. E.; Guerin, M. R., *Sampling and Analysis of Diesel Exhaust and the Motor Pool Workplace Atmosphere*, ORNL/TM-10689, June 1988.
	Griest, W. H.; Guerin, M. R.; Smith, L. H.;* Witschi, H. P.;* Higgins, C. E.; Tomkins, B. A.; Ho, C.-h.; Ilgner, R. H., *Synthetic and Alternate Fuels Characterization*, ORNL/TM-10706, August 1988.
	McMahon, J. M.; Tomkins, B. A.; Griest, W. H., *Leaching Studies of Uranium-Bearing Waste Solids from Manufacturing Operations, Chapter 7 in Data Package for the Low-Level Waste Disposal Development and Demonstration Program Environmental Impact Statement*, ORNL/TM-10939, Volume 1, September 1988.
Walker, R. L.	Carter, J. A.; Smith, D. H.; Walker, R. L.; May, M. P., *An Independent Technique for Fissile Inventory Verification of Holding Tanks in the Nuclear Fuel Cycle: Status Report*, ORNL/TM-10814, May 1988.

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.	Grant, B. C.; Asano, K. G.; Glish, G. L.; McLuckey, S. A., "Trace Analysis for Organics in Air Using an Atmospheric Sampling Glow Discharge Ionization Source," 36th Annual Conference on Mass Spectrometry and Allied Topics, San Francisco, CA, June 5-10, 1988 (invited).
	Asano, K. G.; Glish, G. L.; McLuckey, S. A., "Coupling of an Atmosphere Sampling Ion Source with an Ion Trap Mass Spectrometer," 36th Annual Conference on Mass Spectrometry and Allied Topics, San Francisco, CA, June 5-10, 1988.
Blazewicz, P. R.	Blazewicz, P. R.; Whitten, W. B.; Ramsey, J. M., "Resonance Ionization Spectroscopy of Flame Species in a Low-Pressure Cell," Fourth International Laser Science Conference, Atlanta, GA, October 2-6, 1988.
Buchanan, M. V.	Buchanan, M. V., "FTMS: Evolution, Innovation, and Applications," Chemical Physics Seminar, Oak Ridge National Laboratory, Oak Ridge, TN, March 14, 1988 (invited).
	Buchanan, M. V.; Ma, C. Y.; Moneyhun, J. H.; Guerin, M. R., "Chemical Characterization of Combustion Products from Four Colored Signal Smoke Grenades," Smoke/Obscurants Symposium XII, Kossiakoff Conference and Education Center, The Johns Hopkins University, Laurel, MD, April 19-21, 1988 (invited).
	Buchanan, M. V.; Stemmler, E. A., "Negative Ion Processes for Isomer Differentiation by Mass Spectrometry," Southeast Association of Analytical Chemists (SEACC) Meeting, Knoxville, TN, April 22, 1988 (invited).
	Buchanan, M. V.; Stemmler, E. A., "Gas-Phase Ion-Molecule and Surface-Catalyzed Oxidation Reactions of PAH Under Negative Ion Chemical Ionization Conditions," 36th American Society for Mass Spectrometry Conference, San Francisco, CA, June 5-10, 1988.

Buchanan, M. V.	Buchanan, M. V., "Negative Ion Processes for Structure Information," University of Wisconsin, Department of Chemistry, McElvain Lecture, October 1, 1988 (invited).
	Buchanan, M. V., "Gas Phase Processes for the Isomeric Identification of Trace Organics Using Mass Spectrometry," DOE OHER Site Review, Oak Ridge National Laboratory, Oak Ridge, TN, October 15-16, 1988.
Ceo, R. N.	Ceo, R. N., "Gas Jet Fission Products: Delivery Rates," American Chemical Society 20th Central Regional Meeting, Morgantown, WV, June 2, 1988.
	Ceo, R. N., "Principles of Gamma Ray Spectrometry," GMI Engineering and Management Institute, Flint, MI, November 28, 1988.
Donohue, D. L.	Donohue, D. L.; Hulett, L. D., "Experimental Applications of the Positron Facility at the ORELA," Engineering Physics and Mathematics Division Seminar, Oak Ridge National Laboratory, Oak Ridge, TN, October 20, 1988.
	Donohue, D. L.; Glish, G. L.; McLuckey, S. A.; Hulett, L. D., Jr.; McKown, H. S.; Pendyala, S.,* "Positron Ionization Mass Spectrometry," 8th International Conference on Positron Annihilation, Gent, Belgium, August 29-September 3, 1988.
Costanzo, D. A	Costanzo, D. A.; Griest, W. H., "ORNL Approach to the Characterization of Highly Radioactive Waste Using EPA-Related Procedures," Workshop on Environmemtal Analytical Problems Unique to DOE, Oak Ridge, TN, December 12-13, 1988.
	Costanzo, D. A., "Building 2026 Facility Orientation and Emergency Preparedness Training," Training Session at Building 2026, Oak Ridge National Laboratory, Oak Ridge, TN, March 4, 1988.
Eldridge, J. S.	Eldridge, J. S., "An Historical Review of ORNL Emergency Response: The Role of Radiochemistry in Several Real and Mock Exercises," Emergency Preparedness Brown Bag Seminar, Oak Ridge National Laboratory, May 23, 1988.
Ghodbane, S.	Ghodbane, S., "Separation of Two Overlapping Compounds in Preparative Liquid Chromatography," 1988 Pittsburgh Conference and Exposition, New Orleans, LA, February 22-26, 1988.
Glish, G. L.	Glish, G. L., "New Advances in Mass Spectrometry: Ion Traps and Glow Discharge Ionization," Savannah River Laboratory, Aiken, SC, January 27, 1988 (invited).

Glish, G. L.

Glish, G. L., "A New Dimension in Mass Spectrometry: Mass Spectrometry/Mass Spectrometry," Chemistry Department, University of West Florida, Pensacola, FL, February 3, 1988 (invited).

Glish, G. L.; McLuckey, S. A., "Performance of an Atmospheric Sampling Ionization Source," Finnigan MAT, San Jose, CA, April 25, 1988 (invited).

Glish, G. L.; McLuckey, S. A., "MS/MS Instruments: Is Bigger (and More Expensive) Really Better?" Bay Area Mass Spectrometry Discussion Group, Berkeley, CA, April 27, 1988 (invited).

Glish, G. L., "A New Dimension in Mass Spectrometry: Mass Spectrometry/Mass Spectrometry," Chemistry Department, University of Missouri, Columbia, MO, April 29, 1988 (invited).

Glish, G. L.; McLuckey, S. A.; Asano, K. G.; Grant, B. C., "Application of Mass Spectrometry to the Detection of Vapors from Explosive Compounds," The Analytical Environment Symposium, Chicago, IL, May 10, 1988.

Glish, G. L., "Studies of Gas Phase Reaction Mechanisms," Lawrence Livermore National Laboratory, Livermore, CA, June 2, 1988 (invited).

Glish, G. L.; Van Berkel, G. J.; Asano, K. G.; McLuckey, S. A., "MS^n" Experiments with an Ion Trap Mass Spectrometer," 36th Annual Conference on Mass Spectrometry and Allied Topics, San Francisco, CA, June 5-10, 1988.

Glish, G. L.; McLuckey, S. A., "Scan Modes for Hybrid Mass Spectrometers," 36th Annual Conference on Mass Spectrometry and Allied Topics, San Francisco, CA, June 5-10, 1988.

Glish, G. L.; McLuckey, S. A., "Kinetic Energy Release Effects on Resolution in MS/MS," 36th Annual Conference on Mass Spectrometry and Allied Topics, San Francisco, CA, June 5-10, 1988.

Glish, G. L.; McLuckey, S. A.; Van Berkel, G. J.; Asano, K. G.; McKown, H. S.; Goeringer, D. E., "Developments in Ion Trap Mass Spectrometry," Analytical Chemistry Division Information Meeting, Oak Ridge National Laboratory, Oak Ridge, TN, July 18-19, 1988.

Glish, G. L.

Glish, G. L.; McLuckey, S. A., "Coupling an Atmospheric Sampling Ion Source to an ITMS," Finnigan MAT International Users' Forum, Bordeaux, France, August 28, 1988 (invited).

Glish, G. L.; Van Berkel, G. J.; McLuckey, S. A., "Selected Reagent Ion Chemical Ionization Using an Ion Trap," 11th International Mass Spectrometry Conference, Bordeaux, France, August 29-September 2, 1988.

Glish, G. L.; Van Berkel, G. J.; McLuckey, S. A., "Analysis of Porphyrins by Mass Spectrometry," 11th International Mass Spectrometry Conference, Bordeaux, France, August 29-September 2, 1988.

Glish, G. L.; Van Berkel, G. J.; Asano, K. G.; McLuckey, S. A., Multiple Stages of Mass Spectrometry with an Ion Trap Mass Spectrometer," 11th International Mass Spectrometry Conference, Bordeaux, France, August 29-September 2, 1988.

Glish, G. L.; Van Berkel, G. J.; McLuckey, S. A., "Multiple Stages of Mass Spectrometry with an Ion Trap Mass Spectrometer," 15th Annual FACSS Meeting, Boston, MA, October 30-November 4, 1988.

Glish, G. L.; McLuckey, S. A., "Quadrupole/Sector Geometry Hybrid Instruments," Workshop on Hybrid Mass Spectrometers, Lake Louise, Canada, November 17-18, 1988.

Goeringer, D. E.

Goeringer, D. E.; McLuckey, S. A.; Bertram, L. K., "Instrumentation for Mass Spectrometry/Coincident Fragment Detection," 36th Annual Conference on Mass Spectrometry and Allied Topics, San Francisco, CA, June 5-10, 1988 (invited).

Goeringer, D. E., "Practical Aspects of Lasers in Resonance Ionization Mass Spectrometry," 36th Annual Conference on Mass Spectrometry and Allied Topics, San Francisco, CA, June 5-10, 1988 (invited).

Glish, G. L.; Goeringer, D. E.; McLuckey, S. A., "Laser Desorption in an Ion Trap Mass Spectrometer," 15th Annual Meeting of the Federation of Analytical Chemistry and Spectroscopy Societies, Boston, MA, October 30-November 4, 1988.

Griest, W. H.	Griest, W. H.; Row, K. H.; Maskarinec, M. P., "Comparison of Mobile Phases for the Micellar Electrokinetic Capillary Chromatography Separation of Normal and Modified Nucleic Acid Constituents," 1988 Pittsburgh Conference and Exposition, New Orleans, LA, February 22-26, 1988. Griest, W. H., "How to Deal with Complex Mixtures - The Chemists' Perspective," Biological Evaluation of Complex Mixtures Workshop, Carmel-by-the-Sea, CA, May 11-13, 1988 (invited). Griest, W. H., "Analytical Applications of Supercritical Fluid Chromatography," Y-12 Analytical Laboratory, Oak Ridge, TN, June 29, 1988 (invited).
Guerin, M. R.	Guerin, M. R., "Formation and General Characteristics of Environmental Tobacco Smoke," APCA Specialty Conference on Combustion Processes and the Quality of Indoor Environment, Niagara Falls, NY, September 27-29, 1988 (invited).
Guiochon, G. A.	Guiochon, G. A., "Prediction of Large Concentration Band Profiles in Non-Linear Chromatography," Department of Chemistry, Louisiana State University, Baton Rouge, LA, January 22, 1988 (invited). Guiochon, G. A.; Ghodbane, S.; Golshan-Shirazi;* Bing-Cheng, L.,* "A Numerical Solution of the General Problem of Chromatography," 2nd International Symposium on Preparative Liquid Chromatography, Baden, FRG, February 2, 1988 (invited). Guiochon, G. A., "Preparative Liquid Chromatography. Guidelines Suggested by the Theory," 1988 Pittsburgh Conference and Exposition, New Orleans, LA, February 22-26, 1988 (invited). Guiochon, G. A., "Optimization Criteria in Liquid Chromatography," 1988 Pittsburgh Conference and Exposition, New Orleans, LA, February 22-26, 1988 (invited). Guiochon, G. A., "Theory of Preparative-Liquid Chromatography," Washington Chromatography Discussion Group, Rockville, MD, March 17, 1988 (invited). Guiochon, G. A., "Computer Simulation of Preparative Chromatographic Separations," E. I. DuPont de Nemours, Central Research Station, Wilmington, DE, March 16, 1988 (invited).

Guiochon, G. A.

Guiochon, G. A.; Ghodbane, S.; Golshan-Shirazi;* Huang, J. X.;* Katti, A. M.; Lin, B. C.;* Ma, Z.,* "Non-Linear Chromatography," Analytical Chemistry Division Information Meeting, Oak Ridge National Laboratory, Oak Ridge, TN, July 18-20, 1988 (invited).

Guiochon, G. A., "Non-Linear Chromatography," Southeast Association of Analytical Chemists (SEACC) Meeting, Knoxville, TN, April 22, 1988.

Guiochon, G. A., "Theory of Band Movement Through Chromatographic Columns with Emphasis on Preparative Chromatography," West Carolina Chromatography Discussion Group Annual Conference, Greenville, SC, July 20, 1988 (invited).

Guiochon, G. A., "Theory of Band Separation in Chromatography," Southern Illinois University Seminar, Carbondale, IL, October 21, 1988 (invited).

Guiochon, G. A., "Non-Linear Chromatography," Department of Chemistry, University of Maryland, Baltimore Campus, November 29, 1988 (invited).

Guiochon, G. A., "These Strange Chromatographic Effects," HPLC '88, 12th International Symposium on Column Liquid Chromatography, Washington, DC, June 24, 1988 (invited).

Guiochon, G. A., "Preparative Liquid Chromatography: Theory of Band Separation," 17th International Symposium on Chromatography, Vienna, Austria, September 26, 1988 (invited).

Guiochon, G. A., "Theory of Non-Linear Chromatography," Eastern Anaytical Symposium, New York, NY, October 4, 1988 (invited).

Guiochon, G. A., "The Theory of Non-Linear Chromatography," International Symposium on Chromatography, Weismann Institute, Rehovoth Israel, November 16, 1988 (invited).

Hettich, R. L.

Hettich, R. L., "Combining Laser Technology with Fourier Transform Mass Spectrometry for the Characterization of Non-Volatile Compounds," Oak Ridge Associated Universities Travelling Lecture, Hampton University, Hampton, VA, February 10, 1988 (invited).

Hettich, R. L.	Hettich, R. L., "Combining Laser Technology with Fourier Transform Mass Spectrometry for the Characterization of Non-Volatile Compounds,' Oak Ridge Associated Universities Travelling Lecture, University of Puerto Rico - Mayaguez Campus, Mayaguez, Puerto Rico, May 12, 1988 (invited).
	Hettich, R. L., "Laser Ionization Fourier Transform Mass Spectrometry for the Differentiation of Methyl Guanosine Isomers," 36th American Society for Mass Spectrometry Conference, San Francisco, CA, June 5-10, 1988.
	Hettich, R. L., "Laser Ionization and Laser Photodissociation with Fourier Transform Mass Spectrometry," 36th American Society for Mass Spectrometry Conference, San Francisco, CA, June 5-10, 1988.
	Hettich, R. L., "Examination of Alkylated Nucleosides and Oligonucleotides by Laser Ionization Fourier Transform Mass Spectrometry," Analytical Chemistry Division Information Meeting, Oak Ridge National Laboratory, Oak Ridge, TN, July 18-20, 1988.
Hulett, L. D., Jr.	Hulett, L. D., Jr.; Donohue, D. L.; Pendyala, S.,* "Surface Imaging by Positron Emission," 35th International Field Emission Symposium, Oak Ridge, TN, July 18-22, 1988.
	Hulett, L. D., Jr.; Donohue, D. L.; Lewis, T. A.; * Pendyala, S.,* "A High Intensity Positron Facility for Analytical Research," Analytical Chemistry Division Information Meeting, Oak Ridge National Laboratory, Oak Ridge, TN, July 16-20, 1988.
	Hulett, L. D., Jr.; Lewis, T. A.;* Donohue, D. L.; Pendyala, S.,* "The Oak Ridge National Laboratory Slow Positron Source," 8th International Conference on Positron Annihilation, Gent, Belgium, August 29-September 3, 1988.
	Hsu, F. H.;* Shao, M. J.;* Hulett, L. D., Jr.; Rosseel, T. M.; Dale, J. M., "Surface Effects on Positron Annihilation Characteristics in Cab-O-Sil (SiO2)," 8th Int. Conf. on Positron Annihilation, Gent Belgium, August 29-September 3, 1988.
	Lewis, T. A.;* Hulett, L. D., Jr.; Donohue, D. L., "The Extraction of Linac-Generated Slow Positrons Using the Single-Gap Accelerator Technique," 8th International Conference on Positron Annihilation, Gent, Belgium, August 29-September 3, 1988.

Hulett, L. D., Jr.

Hulett, L. D., Jr.; Pendyala, S.;* "A Theorem for the Determination of Resolution Functions of Positron Lifetime Spectra," 8th International Conference on Positron Annihilation, Gent, Belgium, August 29-September 3, 1988.

Hulett, L. D., Jr.; Pendyala, S.;* "The Practicality of High Magnification Imaging by Positron Emission," 8th International Conference on Positron Annihilation, Gent, Belgium, August 29-September 3, 1988.

Hulett, L. D., Jr., "Positron Spectroscopy as a Tool for Materials Analysis and Characterization," Polymer Engineering Seminar Series, University of Tennessee, Knoxville, TN, October 6, 1988.

Jenkins, R. A.

Jenkins, R. A.; Maskarinec, M. P., "An Assessment of Commercially Available Instrumentation for Field Analysis and Evaluation at Hazardous Waste Sites," 9th Rocky Mountain American Chemical Society Regional Meeting, Las Vegas, NV, March 28-30, 1988 (invited).

Moneyhun, J. H.; Gayle, T. M.;* Jenkins, R. A., "A Laboratory Scale Liquid/Solid Smoke Generator to Simulate the XM55 Field Systems," Smoke/Obscurants Symposium XII, Kossiakoff Conference and Education Center, The Johns Hopkins University, Laurel, MD, April 19-21, 1988.

Jenkins, R. A.; Griest, W. H.; Moneyhun, J. H.; Tomkins, B. A.; Ilgner, R. H.; Higgins, C. E.; Gayle, T. M.,* "Sampling and Chemical Characterization of Workplace Atmospheres Contaminated with Airborne Diesel Exhaust," 1988 EPA Symposium on Measurement of Toxic and Related Air Pollutants, Raleigh, NC, May 2-4, 1988.

Jenkins, R. A.; Thompson, C. V.; Higgins, C. E., "Development and Application of a Thermal Desorption-Based Analytical Method for the Determination of Nicotine in Indoor Environments," Indoor and Ambient Air Quality Conference, London, England, June 13-15, 1988.

Jenkins, R. A.; Thompson, C. V.; Higgins, C. E., "Development and Application of a Personal Sampler and Detection Method for Monitoring Nicotine in Indoor Environments," Analytical Chemistry Division Information Meeting, Oak Ridge National Laboratory, Oak Ridge, TN, July 18-20, 1988.

Jenkins, R. A.	Jenkins, R. A.; Moneyhun, J. H.; Gayle, T. M.;* Ross, R. H., "Chemical and Toxicological Properties of the Combustion Products of Rifle and Gun Systems," Pulmonary Research Review and Analysis Meeting, Fitzsimons Army Medical Center, Auroro, CO, September 20-22, 1988 (invited).
Jones, R. E.	Jones, R. E., "Radiation Safety," Orientation Meeting for New Employees in ACD, Oak Ridge National Laboratory, Oak Ridge, TN, March 18, 1988.
Katti, A. M.	Katti, A. M.; Guiochon, G. A., "Numerical Simulation of Band Profiles for Two-Component Separations by Displacement Chromatography," 1988 Pittsburgh Conference and Exposition, New Orleans, LA, February 22-26, 1988.
Keller, J. M.	Keller, J. M., "Installation of a Hot ICP," Martin Marietta Energy Systems, Inc. Analytical Committee Meeting, Oak Ridge National Laboratory, Oak Ridge, TN, October 25, 1988.
Klatt, L. N.	Dale, J. M.; Klatt, L. N., "Concepts for the Deterrence of Currency Counterfeiting," Analytical Chemistry Division Information Meeting, Oak Ridge National Laboratory, Oak Ridge, TN, July 18-20, 1988. Klatt, L. N.; Bostick, D. T.; Perez Blanco, H.,* "Fiber Optic Sensors for the Study of Falling Liquid Films," 196th Meeting of the American Chemical Society, Los Angeles, CA, September 25-30, 1988 (invited). Klatt, L. N.; Bostick, D. T.; Perez Blanco, H.,* "Sensors for Determining the Composition of Falling Liquid Films," 174th Meeting of the Electrochemical Society, Chicago, IL, October 9-14, 1988 (invited). Klatt, L. N., "Recent Instrumentation Developments," Department of Chemistry, University of Alabama - Birmingham, Birmingham, AL, November 11, 1988.
Laing, W. R.	Laing, W. R., "Measurement Control for a Non-Nuclear Facility," Symposium on Measurement and Control for MC&A, Savannah River Laboratory, Aiken, SC, October 25-27, 1988.
Maskarinec, M. P.	Maskarinec, M. P., "Evaluation of Analytical Problems Associated with the EPA Toxicity Characteristic Leaching Procedure (TCLP) Test," Solid Waste Forms: Characteristics and Evaluations Workshop on Waste Forms, Oak Ridge National Laboratory, Oak Ridge, TN, April 8, 1988 (invited).

Maskarinec, M. P.

Maskarinec, M. P.; Holladay, S. K.; Ryan, M. A.,* "Technical and Economic Issues Related to Consistency of Quality Assurance Programs," 12th Aquatic Toxicology and Hazard Assessment Symposium, Sparks, NV, April 24-26, 1988 (invited).

Maskarinec, M. P., "Technical and Economic Benefits of Harmonization of Quality Assurance Programs," 12th Aquatic Toxicology and Hazard Assessment Symposium, Sparks, NV, April 24-26, 1988 (invited).

Maskarinec, M. P.; Johnson, L. H.; Holladay, S. K., "Determination of Stability of Environmental Samples," Analytical Chemistry Division Information Meeting, Oak Ridge National Laboratory, Oak Ridge, TN, July 18-20, 1988.

May, M. P.

May, M. P.; Shepherd, M.;* Wetzel, M.;* McCauley, B.,* "The Use of Interactive Computer Based Training at ORNL as a Tool for Training Workers at Our Non-Reactor Nuclear Facilities," Training Coordinator's Meeting, Oak Ridge National Laboratory, Oak Ridge, TN, May 5, 1988.

May, M. P., Walker, R. L.; Carter, J. A.; Wolf, D.,* "Level and Volume Calibration of a Large Hot Canyon Tank using the Lutetium Double Spike Method," Savannah River Laboratory, Aiken, SC, September 21-22, 1988.

May, M. P.; Wetzel, M.,* "Interactive Video Disc Computer Based Training for ORNL," Office of Operational Safety, Oak Ridge National Laboratory, Oak Ridge, TN, October 11, 1988.

McLuckey, S. A.

McLuckey, S. A.; Glish, G. L.; Asano, K. G., "Glow Discharge Ionization for Trace Analysis of Organics in Ambient Air," Pittsburgh Conference, New Orleans, LA, February 25, 1988.

McLuckey, S. A., "Organic Mass Spectrometry: Highlights," Basic Energy Sciences Site Review, Oak Ridge, TN, April 11, 1988.

McLuckey, S.A.; Asano, K.G.; Glish, G.L., "Negative Chemical Ionization and MS/MS of Negative Ions in an Ion Trap Mass Spectrometer," 36th ASMS Conference on Mass Spectrometry and Allied Topics, San Francisco, CA, June 9, 1988.

McLuckey, S.A.; Glish, G. L.; McKown, H. S.; Grant, B.C., "Explosives Detection at ORNL: Status Report," Analytical Chemistry Division Information Meeting, Oak Ridge, TN, July 18, 1988.

McLuckey, S. A.	McLuckey, S. A.; Glish, G.L.; Asano, K.G., "The Coupling of an Atmospheric Sampling Ion Source with an Ion Trap Mass Spectrometer," 196th ACS National Meeting, Los Angeles, CA, Sept. 28, 1988 (invited).
	McLuckey, S.A.; Glish, G. L.; Grant, B.C.; Asano, K. G., "The Status of Explosives Detection at Oak Ridge National Laboratory," 4th Annual Conference on Explosives Detection, FBI Academy, Quantico, VA, Oct. 12, 1988 (invited).
	McLuckey, S. A.; Glish, G. L.; Carter, J. A., "Progress in Explosives Detection at Oak Ridge National Laboratory," DOE Office of Safeguards and Security Program Review, Germantown, MD, Oct. 14, 1988.
	McLuckey, S. A.; Glish, G.L., "MS/MS Instrumentation for Rapid Trace Level Analysis," FACSS XV, Boston, MA, Nov. 2, 1988 (invited).
Nolan, T. G.	Nolan, T. G.; Whitten, W. B.; Ramsey, J. M., "Refractive Index Detection Using Two-Wave Mixing in Photorefractive Barium Titanate," Fourth International Laser Science Conference, Atlanta, GA, October 2-6, 1988.
Ramsey, J. M.	Ramsey, J. M., "Applications of Nonlinear Optics to Chemical Analysis," North Carolina State University, Department of Chemistry, Raleigh, NC, February 29, 1988 (invited).
	Ramsey, J. M., "High Resolution Analytical Laser Spectroscopy," Universidad de Los Andes, Merida, Venezuela, April 25, 1988 (invited).
	Ramsey, J. M., "Nonlinear Optical Experiments Using Photorefractive Materials," Universidad de Los Andes, Merida, Venezuela, April 26, 1988 (invited).
	Ramsey, J. M., "Analytical Spectroscopy Using Time-Domain Measurements," Universidad de Los Andes, Merida, Venezuela, April 27, 1988 (invited).
	Ramsey, J. M., "Nonlinear Optics in Chemical Measurement Systems," Universidad Central de Venezuela, Caracas, Venezuela, April 29, 1988 (invited).
	Ramsey, J. M., "Laser-Based Chemical Measurements," DOE Chemical Sciences Site Review, Oak Ridge, TN, April 11, 1988.

Ramsey, J. M.	Ramsey, J. M.; Whitten, W. B., "Analytical Laser Spectroscopy at High Resolution: New Techniques," ACS Summer Symposium, "Lasers in Analytical Chemistry," Stanford University, Stanford, CA, June 26-29, 1988. Ramsey, J. M.; Whitten, W. B.; Nolan, T. G.; Blazewicz, P. R., "Very High Resolution Laser-Based Spectrochemical Analysis," Analytical Chemistry Division Information Meeting, Oak Ridge National Laboratory, Oak Ridge, TN, July 18-20, 1988. Ramsey, J. M.; Whitten, W. B., "Frequency and Bandwidth Control of CW Dye Lasers Using Intracavity Photorefractive Elements," Fourth International Laser Science Conference, Atlanta, GA, October 3, 1988. Ramsey, J. M.; Whitten, W. B.; Blazewicz, P. R., "Multi-Ionization in a Low Pressure Cell," Federation of Analytical Chemistry and Spectroscopy Societies (FACSS), Boston, MA, October 30-November 4, 1988.
Ramsey, R. S.	Ramsey, R. S.; Ho, C.-h.; Row, K. H., "Determination of UV-Induced Photosensitive Lesions in DNA by HPLC/GC," Analytical Chemistry Division Information Meeting, Oak Ridge National Laboratory, Oak Ridge, TN, July 18-20, 1988.
Robinson, L.	Dyer, F. F.; Robinson, L.; Emery, J. F., "The Use of Automation with the New Pneumatic Irradiation Facility of the ORNL HFIR," American Nuclear Society, 1988 Annual Meeting, San Diego, CA, June 12-16, 1988. Robinson, L.; Hobbs, R. W.; Dyer, F. F.; Pugh, L. P.;* Snelgrove, J. L.;* Teasley, N. A., Jr., "An Automated Fuel Scanning System," ANS Topical Meeting on Industrial Radiation and Radioisotope Measurement Applications, Pinehurst, NC, September 6-9, 1988.
Ross, H. H.	Ross, H. H., "Radioactivity in the Environment: A Major Analytical Challenge," Chemistry Department, North Georgia College, Dahlonega, GA, November 17, 1988 (invited). Ross, H. H., "Radioactivity in the Environment: A Major Analytical Challenge," Chemistry Department, University of West Florida, Pensacola, FL, November 10, 1988 (invited).

Ross, H. H.	Ross, H. H., "Radioactivity in the Environment: A Major Analytical Challenge," Chemistry Department, Florida Southern College, Lakeland, FL, November 9, 1988 (invited).
	Ross, H. H., "Fiber-Scintillator Flow-Cell Radiation Detector," Analytical Chemistry Division Information Meeting, Oak Ridge National Laboratory, Oak Ridge, TN, July 18-20, 1988.
Rosseel, T. M.	Rosseel, T. M., "High Resolution Studies of the Chemical Effects in Heavy Ion Induced X-Ray Satellite Emission (HIXSE) Spectra," DOE Basic Energy Sciences Site Review, Oak Ridge, TN, April 11, 1988.
Shaw, R. W.	Shaw, R. W.; Young, J. P.; Smith, D. H., "Resonance Ionization Mass Spectrometric Study of the Promethium/Samarium Isobaric Pair," 4th International Symposium on Resonance Ionization Spectroscopy & Its Applications, Gaithersburg, MD, April 10-15, 1988.
	Shaw, R. W.; Young, J. P.; Smith, D. H., "RIMS Removal of Isobaric Interference for the Pm/Sm Pair," DOE Basic Energy Sciences Site Review, Oak Ridge, TN, April 11, 1988.
	Shaw, R. W.; Young, J. P.; Smith, D. H., "Diode Laser-Initiated, Two-Color Resonance Ionization Mass Spectrometry of Lanthanum," 4th International Laser Science Conference, Atlanta, GA, October 3-6, 1988.
	Shaw, R. W.; Young, J. P.; Smith, D. H., "Uranium Isotope Ratio Determinations by Resonance Ionization Mass Spectrometry," 4th International Laser Science Conference, Atlanta, GA, October 2-6, 1988.
Shults, W. D.	Shults, W. D., "Analytical Chemistry at a Multipurpose Laboratory," Southeastern Association of Analytical Chemists-88, Knoxville, TN, April 22-23, 1988.
Schudel, J. V. H.	Schudel, J. V. H.; Guiochon, G. A., "Column Efficiency Calculations: A Study of the Precision and Accuracy of Eight Measurement Methods," 1988 Pittsburgh Conference and Exposition, New Orleans, LA, February 22-26, 1988.
Smith, D. H.	Smith, D. H.; Shaw, R. W.; Young, J. P., "Resonance Ionization Mass Spectrometry of Promethium," 36th Annual Conference on Mass Spectrometry and Allied Topics, San Francisco, CA, June 5-10, 1988.

Smith, D. H.	Smith, D. H., "Feasibility Study of Isotope Ratio Measurements of Xe and Kr Using a Multi-Collector Mass Spectrometer," DOE Advisory Group for Effluent Research, Washington, DC, October 7, 1987.
Stemmler, E. A.	Stemmler, E. A.; Buchanan, M. V., "Isomeric Differentiation of PAH and Methyl-PAH Using Surface-Catalyzed Oxidation Reactions," 36th American Society for Mass Spectrometry Conference, San Francisco, CA, June 5-10, 1988. Stemmler, E. A.; Buchanan, M. V., "Isomeric Differentiation Using Negative Ion Chemical Ionization and Surface-Catalyzed Oxidation Reactions," Analytical Chemistry Division Information Meeting, Oak Ridge National Laboratory, Oak Ridge, TN, July 18-20, 1988.
Stewart, J. H.	Stewart, J. H., "Approval Petition for Use of XOXTEC Rapid PCB Extraction," Martin Marietta Energy Systems, Inc., Analytical Communication Meeting, Oak Ridge National Laboratory, Oak Ridge, TN, October 25, 1988. Stewart, J. H., "Analysis of Crude Oils for Strategic Petroleum Reserves," Fossil Energy Program Review, Oak Ridge National Laboratory, Oak Ridge, TN, August 22, 1988.
Todd, P. J.	Todd, P. J., "Mass Transport in Liquid SIMS," 36th Annual Conference on Mass Spectrometry and Allied Topics, San Francisco, CA, June 5-10, 1988 (invited). Todd, P. J., "Organic Ion Imaging," Analytical Chemistry Division Information Meeting, Oak Ridge National Laboratory, Oak Ridge, TN, July 18-20, 1988.
Van Berkel, G. J.	Van Berkel, G. J., "The Effect of Operating Parameters on Chemical Ionization Mass Spectra Obtained with an Ion Trap Mass Spectrometer," 1988 Pittsburgh Conference, New Orleans, LA, February 22-26, 1988. Van Berkel, G. J., "Mass Spectrometric Analysis of Porphyrins," Oak Ridge National Laboratory, DOE Basic Energy Sciences Site Review, Oak Ridge, TN, April 11-12, 1988. Van Berkel, G. J., "New Mass Spectrometric Approaches to Geoporphyrin Analysis," V. M. Goldschmidt Conference, Baltimore, MD, May 1988 (invited).

Van Berkel, G. J.	Van Berkel, G. J.; Glish, G. L.; McLuckey, S. A., "Structural Analysis of Porphyrins: Analysis of In Vitro Prepared Porphyrinogens Using an Ion Trap Mass Spectrometer," 36th Annual Conference on Mass Spectrometry and Allied Topics, San Francisco, CA, June 5-10, 1988.
	Van Berkel, G. J.; Glish, G. L.; McLuckey, S. A., "Mechanism of Porphyrin Hydrogenation and Decomposition in a Chemical Ionization Source," 36th Annual Conference on Mass Spectrometry and Allied Topics, San Francisco, CA, June 5-10, 1988.
	Van Berkel, G. J.; Glish, G. L.; McLuckey, S. A., "Analysis of Porphyrins Using an Ion Trap Spectrometer," 36th ASMS Conference on Mass Spectrometry and Allied Topics, San Francisco, CA, June 5-10, 1988.
	Van Berkel, G. J., "Selected Reagent Ion Chemical Ionization and Ms^n Analysis," Finnigan-MAT Ion Trap Mass Spectrometer Users Meeting, San Francisco, CA, June 5-10, 1988.
	Van Berkel, G. J.; Glish, G. L.; McLuckey, S. A., "New Mass Spectrometric Approaches to Porphyrin Analysis," Analytical Chemistry Division Information Meeting, Oak Ridge National Laboratory, Oak Ridge, TN, July 18-20, 1988.
	Van Berkel, G. J., "Ion Trap Mass Spectrometry," Chemistry Department, Lawrence University, Appleton, WI, October 21, 1988 (invited).
Vane, R.*	Raman, S.;* Kahane, S.;* Rosseel, T.; Stewart, S.;* Walkiewicz, T.* "Chemical Sensitivities of Heavy-Ion Induced X-rays from Al Compounds and Alloys," APS Division of Atomic Molecular and Optical Physics Meeting, Baltimore, MD, April 18-22, 1988.
Wade, J. W.	Wade, J. W., "Our Experience with the DOE Environmental Survey and Lessons Learned," 34th Annual Conference on Bioassay, Analytical, and Environmental Radiochemistry, Las Vegas, NV, October 17-21, 1988.
Whitten, W. B.	Whitten, W. B.; Ramsey, J. M., "Isotopic Biases in Resonance Ionization Mass Spectrometry Due to Ground State Coherences," 1988 International Laser Science Conference, Atlanta, GA, October 2-6, 1988.

Whitten, W. B.	Whitten, W. B.; Ramsey, J. M.; Blazewicz, P. R.; Nolan, T. G., "Low Pressure Spectrometry for High Pressure Combustion Diaganostics," 4th International Laser Science Conference, Atlanta, GA, October 2-6, 1988.
Wise, M. B.	Wise, M. B., "FTMS - A Powerful Tool for Gas Phase Ion Chemistry," University of Georgia, Oak Ridge Associated Universities Travelling Lecture, Athens, GA, April 5, 1988 (invited).
	Wise, M. B.; Buchanan, M. V., "Fourier Transform Tandem Mass Spectrometry for the Qualitative Analysis of Complex Mixtures," 36th American Society for Mass Spectrometry Conference, San Francisco, CA, June 5-10, 1988.
	Wise, M. B.; Guerin, M. R., "Glow Discharge Ionization Mass Spectrometry for Environmental Analysis Applications," 36th American Society for Mass Spectrometry Conference, San Francisco, CA, June 5-10, 1988.
	Wise, M. B.; Buchanan, M. V.; Ilgner, R. H., "Detection of Trace Organics in the Environment Using New Mass Spectrometry - Based Techniques," Analytical Chemistry Division Information Meeting, Oak Ridge National Laboratory, Oak Ridge, TN, July 18-20, 1988.
	Wise, M. B., "Tandem Mass Spectrometry in Trapped Ion Instruments: A Direct Comparison of FTMS and ITMS," American Chemical Society National Meeting, Symposium on Trapped Ion Techniques, Los Angeles, CA, September 25, 1988 (invited).
	Wise, M. B.; Guerin, M. R., "Detection of Part-per-billion Levels of Organics in Water and Soil Samples Using a Glow Discharge Mass Spectrometer," EPA Meeting on Waste Testing and Quality Assurance, Washington, DC, July 11-15, 1988.
	Wise, M. B., "Analysis of Volatile Organics in Water and Soil Samples Using Glow Discharge Mass Spectrometry," 36th American Society for Mass Spectrometry Conference, San Francisco, CA, June 5-10, 1988 (invited).
Young, J. P.	Young, J. P.; Shaw, R. W.; Goeringer, D. E.; Smith, D. H., "Influence of Laser Characteristics on Resonance Ionization Mass Spectrometry," Fourth International Symposium on Resonance Ionization Spectroscopy & Its Applications," Gaithersburg, MD, April 10-15, 1988.

Young, J. P.	Young, J. P.; Shaw, R. W.; Goeringer, D. E.; Smith, D. H.; Christie, W. H., "Resonance Ionization Mass Spectrometry: Current Status and Future Potential," Eastern Analytical Symposium, New York, NY, October 2-7, 1988.

ARTICLES REVIEWED OR REFEREED FOR PERIODICALS

	Anal. Chem.	Biomed & Environ. Mass Spect.	Environ. Sci. & Technol.	Health Phys.	J. of Chromatogr.	J. of Phys. Chem.	Proposals	Other	Total
Buchanan, M. V.	1	3					13	2	19
Carter, J. A.	1						2		3
Dyer, F. F.							1		1
Eldridge, J. S.				3					3
Emery, J. F.							1		1
Feldman, C.	1							2	3
Glish, G. L.	1							2	3
Griest, W. H.	4		1				5	1	11
Guerin, M. R.							5	3	8
Guiochon, G. A.					3	2	3		8
Jenkins, R. A.							1	3	4
Ramsey, J. M.	1							3	4
Ramsey, R. S.	3						3		6
Ross, H. H.	1							1	2
Shaw, R. W.							3		3
Stewart, J. H.								2	2
Stokely, J. R.							1		1
Todd, P. J.	1						3		4
Tomkins, B. A.	5		2		1		1	1	10
Whitten, W. B.							2		2
Wise, M. B.							1		1
Young, J. P.	2	—	—	—	—	—	3	1	6
Total	21	3	3	3	4	2	48	20	104

SUMMARY OF ANALYTICAL SERVICE WORK

Organization	Number of results reported by Analytical Spectroscopy	Radioactive Materials Analysis	Organic Chemistry	Inorganic Chemistry	Total
ORNL Unit					
Analytical Chemistry	316	4523	8309	7239	20387
Central Management		434		124	558
Chemical Technology	3991	6158	2202	11881	24232
Chemistry	213	19		207	439
Energy		481		7128	7609
Engineering Physics and Mathematics	155	4		6	165
Engineering Technology	736	3		694	1433
Environmental and Health Protection	9241	41024	37100	13115	100480
Environmental Sciences		966	24793	80149	105908
Fuel Recycle	4217	259		132	4608
Fusion Energy	198	68		107	373
Health			111		111
Health and Safety Research	107	1232	8120	5309	14768
Metals and Ceramics	2851	98		6980	9929
Physics	56	31		30	117
Plant and Equipment	329	194	82	823	1428
Research Reactor		400		214	614
Solid State	157	19		151	327
Others					
DOE Environmental Survey Program			67068	26294	93362
K-25	1781	292	2575		4648
Miscellaneous	1372	219	339	1854	3784
Nuclear & Engineering Tech.				4937	4937
Paducah Gaseous Diffusion Plant			527		527
Savannah River Laboratory	243				243
Work for Others	1349				1349
Y-12	1782	399	1025		3206
TOTAL	29094	56823	152251	167374	405542

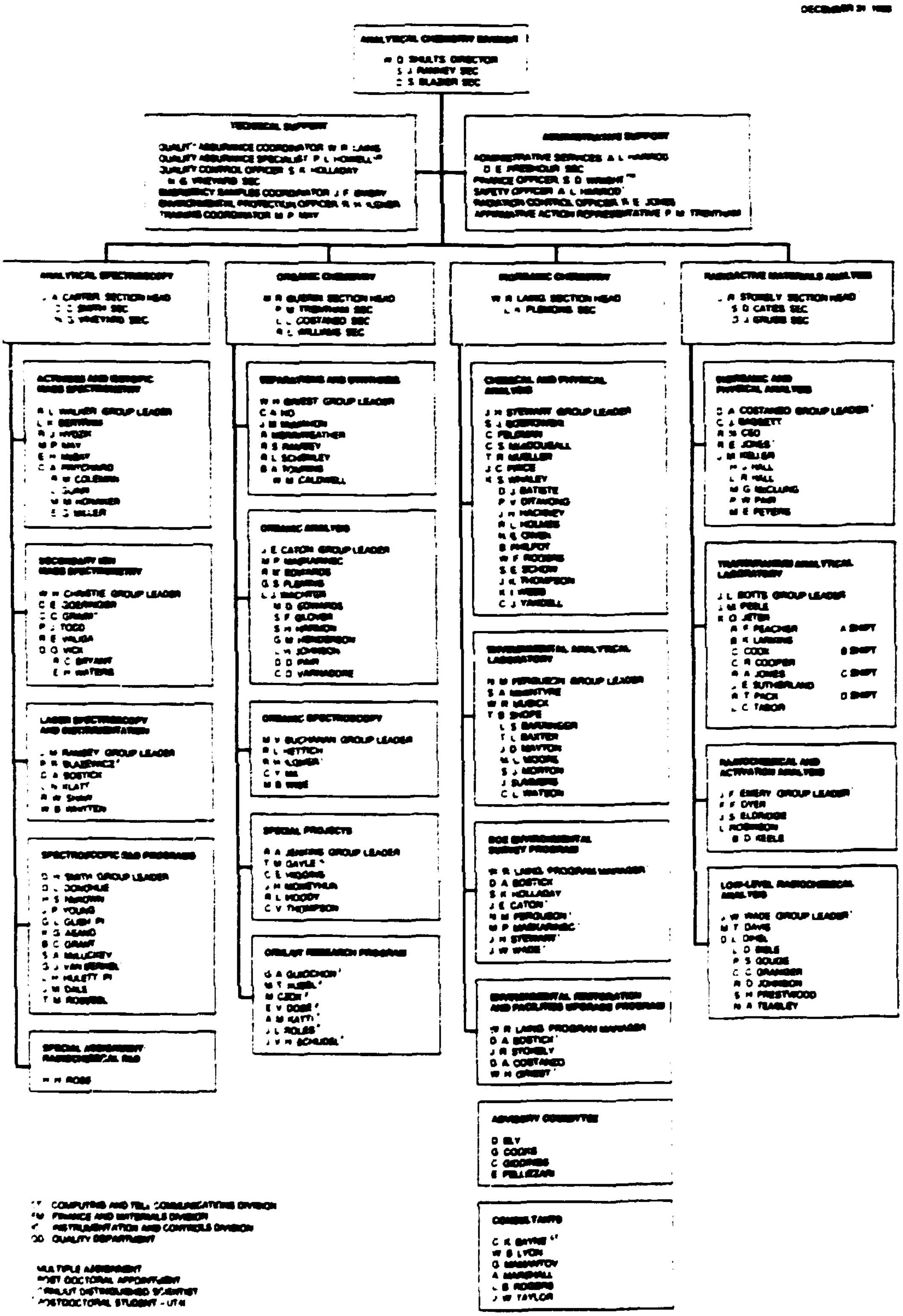
DIVISIONAL MANPOWER AND FINANCIAL SUMMARY
FY 1988

Source	$K	PY[a]
DOE programs		
Energy Research		
Basic Energy Sciences	1,685	11.1
Health and Environmental Research	573	3.3
Environmental Site Survey	1,955	13.6
Safeguards and Security	573	4.1
Nuclear Energy	272	1.8
Multi-program Facilities Support	268	1.6
Fossil Energy	12	0.1
Miscellaneous	429	2.7
Total DOE Programs	5,767	38.3
Work for others - federal agencies		
Department of Defense	1,561	7.3
National Cancer Institute	287	2.0
Environmental Protection Agency	309	1.9
Bureau of Engraving and Printing	45	0.2
Federal Aviation Administration	81	0.7
National Institute on Drug Abuse	42	0.3
Miscellaneous	140	0.6
Total WFO - federal Agencies	2,465	13.0
Work for others - nonfederal agencies		
Council for Tobacco Research	81	.6
Protective Coating Companies	5	0.0
Uranium Ore Testing Companies	45	0.0
Miscellaneous	46	0.3
Total WFO - nonfederal agencies	177	0.9
Support/Services		
ORNL Divisions/Programs	8,646	84.7
Other Clients	1,555	15.2
Total Support/Services	10,201[b]	99.9
TOTAL FINANCIAL PLANS	18,610	152.1

---

[a] Person Years

[b] Does not reflect Laboratory general and administrative expense or general plant services.


DECEMBER 31 1988
ANALYTICAL CHEMISTRY DIVISION
W D SHULTS DIRECTOR
S J RAMSEY SEC
TECHNICAL SUPPORT
QUALITY ASSURANCE COORDINATOR W R LAING
QUALITY ASSURANCE SPECIALIST P L HOWELL
QUALITY CONTROL OFFICER S K HOLLADAY
EMERGENCY SAMPLES COORDINATOR J F EMERY
TRAINING COORDINATOR M P MAY
ADMINISTRATIVE SUPPORT
ADMINISTRATIVE SERVICES A L HARROD
FINANCE OFFICER S D WRIGHT
SAFETY OFFICER A L HARROD
RADIATION CONTROL OFFICER R E JONES
AFFIRMATIVE ACTION REPRESENTATIVE P M TRENTHAM
ANALYTICAL SPECTROSCOPY
J A CARTER SECTION HEAD
ORGANIC CHEMISTRY
INORGANIC CHEMISTRY
W R LAING SECTION HEAD
RADIOACTIVE MATERIALS ANALYSIS
J R STOKELY SECTION HEAD
S D CATES SEC
ADVISORY COMMITTEE
CONSULTANTS

ORNL/6474
Distribution Category UC-4

INTERNAL DISTRIBUTION

1. H. Adair
2. T. D. Anderson
3. B. R. Appleton
4. K. G. Asano
5-15. J. T. Atherton
16. J. B. Ball
17. C. K. Bayne
18. L. K. Bertram
19. S. Bobrowski
20. J. T. Bradbury
21. J. L. Botts
22. M. V. Buchanan
23-26. J. A. Carter
27. J. A. Caton
28. R. N. Ceo
29. W. H. Christie
30. D. A. Costanzo
31. J. M. Dale
32. R. M. Davis
33. D. C. Dihel
34. D. L. Donohue
35. J. G. Dorsey
36. J. S. Eldridge
37. C. Feldman
38. M. Ferguson
39. G. S. Fleming
40. T. M. Gayle
41. R. Genung
42. G. L. Glish
43. D. E. Goeringer
44. W. H. Griest
45-48. M. R. Guerin
49. G. Guiochon
50. A. L. Harrod
51. R. L. Hettich
52. C-h. Ho
53. S. K. Holladay
54. P. L. Howell
55. L. D. Hulett
56. R. H. Ilgner
57. R. A. Jenkins
58. S. V. Kaye
59. J. M. Keller
60. L. N. Klatt
61. E. H. Krieg
62. W. R. Laing
63. W. S. Lyon
64. C. A. Ma
65. S. A. McIntyre
66. F. Maienschien
67. S. A. McLuckey
68. J. M. McMahon
69. J. H. Moneyhun
70. R. W. Morrow
71. T. R. Mueller
72. R. T. Pack
73. J. M. Peele
74. M. L. Poutsma
75. C. A. Pritchard
76. J. M. Ramsey
77. D. E. Reichle
78. C. R. Richmond
79. L. Robinson
80. P. S. Rohwer
81. T. M. Rosseel
82. T. M. Row
83. D. R. Rupert
84-134. W. D. Shults
135. D. H. Smith
136. J. H. Stewart
137. J. R. Stokely
138. N. A. Teasley
139. P. J. Todd
140. B. A. Tomkins
141. R. E. Valiga
142. D. O. Vick
143. J. W. Wade
144. R. L. Walker
145. C. W. Weber
146. W. B. Whitten
147. S. D. Wright
148. F. W. Young
149. J. P. Young
150. Biology Library
151. Laboratory Shift Supv.
152. ORNL Y-12 Library, Doc. Reference
153. Lab Records, ORNL-RC
154. ORAU Technical Library (MERT)
155. ORAU/UPD Beth Jinkerson
156. Patent Section
157. PGDP Library (B. Barbre)
158-159. Central Res. Lib.
160-161. Lab Records
162-163. Central Publ.

EXTERNAL DISTRIBUTION

162. D. D. Bly, E. I. du Pont de Nemours, E328/120, Wilmington, DE 19898
163. J. Burnett, Division of Chemical Sciences, Office of Energy Research, USDOE, ER-142, GTN,
Washington, DC 20545
163. D. A. Coffin, Health Effects Research Laboratory, MD-52, EPA, Research
Triangle Park, NC 27711
164. G. Cooks, Department of Chemistry, Purdue University, Lafayette, IN
165. P. Duhamel, Office of Health & Environmental Research, USDOE, GTN,
Washington, DC 20545
165. J. E. Garneir, Sohio Engineered Materials Company, P.O. Box 832, Niagara
Falls, NY 14302
166. C. Giddings, 3978 Emigration Canyon, Salt Lake City, UT 84108
169. G. Goldstein, Office of Health & Environmental Research, USDOE, GTN,
Washington, DC 20545
170. R. Gordon, Division of Chemical Sciences, Office of Energy Research, USDOE, ER-142, GTN,
Washington, DC 20545
170. David W. Green, Chemical Technology Division, Argonne National
Laboratory, 9700 South Cass Avenue, Argonne, IL 60439
171. R. Hirsch, Division of Chemical Sciences, Office of Energy Research,
USDOE, ER-162, GTN, Washington, DC 20545
172. Institute for Energy Analysis, P.O. Box 117, Oak Ridge, TN 37830
173. G. Mamantov, Department of Chemistry, University of Tennessee,
Knoxville, TN 37916
174. R. S. Marianelli, Division of Chemical Sciences, Office of Energy
Research, USDOE, ER-14, GTN, Washington, DC 20545
175. T. B. Owen, National Cancer Institute, Bethesda, MD 20014
176. E. Pellizzari, Director for Analytical Sciences, Research Triangle Park,
NC 27709
177. John Phillips, Los Alamos National Laboratory, ADWT, MS-F-628, Los
Alamos, NM 87545
178. D. Stevenson, Division of Chemical Sciences, Office of Basic Energy
Sciences, USDOE, ER-142, GTN, Washington, DC 20545
179. U.S. Department of Agriculture, National Agriculture Library, Gift
Program, Room 002 D88/063, Beltsville, MD 20705
180. R. W. Wood, Deputy Director, Office of Health & Environmental Research,
USDOE, ER-74, GTN, Washington, DC 20545
181. D. J. Wynn, USATHAMA, DRX TH-TE-A, Bldg. E4585, Aberdeen Proving Ground,
MD 21010
182. Office of Assistant Manager, Energy Research and Development, DOE, Oak
Ridge Operations Office, Oak Ridge, TN 37830
183-283. Distribution as shown in DOE/TIC-4500 under category UC-4 (25 copies--NTIS)