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**Analytical Chemistry Division
Annual Progress Report
For Period Ending
December 31, 1981**

W. D. Shults

MASTER

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For Period Ending December 31, 1981

W. D. Shults, Director

W. S. Lyon, Editor

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INTRODUCTION AND SUMMARY

W. D. Shults

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

1. Analytical Research, Development, and Implementation. The division maintains a program to conceptualize, investigate, develop, assess, improve, and implement advanced technology for chemical and physicochemical measurements. Emphasis is on problems and needs identified with ORNL and Department of Energy (DOE) programs, but attention is also given to needs in the analytical sciences themselves. This program is composed of medium- to long-term projects and is supported primarily by the DOE. The program constituted approximately 20% of the FY 1981 budget.

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 results or special analytical capabilities to expedite programmatic interests. The effort in this category comes from ORNL and DOE programs and from "Work-for-Others" agreements. Emphasis here is on "applied" chemistry. This type of activity accounted for approximately 30% of the division's budget in FY 1981.

3. Technical Support. The division performs chemical and physicochemical analyses and tests of virtually all types. 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. In general, this work is short-term in nature and comes from other divisions and programs within ORNL. A significant fraction originates outside of ORNL and involves the use of talent and/or facilities in which the division is particularly strong or unique. This type of work accounted for approximately 50% of the budget during FY 1981.

The Analytical Chemistry Division is organized into five major sections, each of which may carry out any type of work falling in the three categories mentioned above. Chapters 1 through 5 of this report highlight progress within the five sections during the period January 1, 1981, to December 31, 1981. A short summary introduces each chapter to indicate work scope. Information about quality assurance and safety programs is presented in Chap. 6, along with a tabulation of analyses rendered. Publications, oral presentations, professional activities, educational programs, and seminars are cited in Chaps. 7 and 8. Approximately 56 articles, 31 proceedings publications and 33 reports have been published, and 119 oral presentations given during this reporting period.

1. ANALYTICAL METHODOLOGY

W. S. Lyon

Analytical Methodology comprises two groups: Analytical Instrumentation and Physicochemical Analyses. Both perform basic research and both have an applied aspect to their program.

The DOE Office of Basic Energy Sciences sponsors most of the research in this section. Because the section is small we have been forced to select specialty areas for exploitation. Lasers and their applications is the primary interest in the instrumentation group, whereas positron spectroscopy and heavy ion excitation and spectroscopy provide the main thrust for the physicochemical group. Research results flow within the section to the applied programs; thus, on-line instrumentation and remote equipment logically follow in one group and surface analysis and special electron and x-ray analyses are performed in the other. Work here is supported by a number of ORNL programs. Our research projects are dynamic: ideas generated during research usually result in new projects. At least two of our current efforts are the direct result of ORNL seed money studies. Our primary product, we believe, is an idea; our ultimate goal, an analytical utilization of the same.

ANALYTICAL INSTRUMENTATION

H. H. Ross

Spectroscopy Research

Our program in analytical spectroscopy continues to embrace a broad range of research areas. Specific topics for the past year include time-resolved fluorescence, time-of-flight spectroscopy, delayed lasing, Fourier transform microwave spectroscopy, optoacoustic spectroscopy, resonance ionization spectroscopy, laser excited fluorescence, heavy ion x-ray fluorescence, and the development of the glow discharge detector for the analysis of oxygen or fluorine. The status of these projects ranges from almost complete (glow discharge detector) to barely started (Fourier transform microwave spectroscopy). In virtually each case, the research approach to the project is the same: (1) development of theory and experimental verification and (2) demonstration of the concept in a "real life" analytical problem situation. Although these projects are carried out in a basic research mode--that is, untargeted to a specific application--our interest in them is high because of their potential analytical utility, the high degree of creativity needed and the challenge of the experimental sophistication that is required.

Our work in time-resolved fluorescence has led to the interesting observation that an argon-ion laser can be operated in a mode such that the output is nearly 100% self modulated at five frequencies between 408 and 737 MHz. This type of operation can be obtained when the laser gain is adjusted slightly above threshold where the homogeneous line width of the Ar^+ transitions are broader than the longitudinal mode spacing of the laser cavity. The practical consequences of this observation are that the experimental setup for time-resolved measurements can be simplified considerably and also made cheaper.

We have already tested such a system for the measurement of short fluorescence lifetimes by combining a self-modulated laser with a phase null detection scheme. The phase of the laser modulation frequency is compared to that of the

laser induced fluorescence modulation by using a microwave double-balanced mixer. The mixer output is minimum when inputs are 90° out of phase. A delay line (optical) in one leg of the mixer is used to develop the null for both a sample and standard; delay differences are then related to sample fluorescent lifetime.

A significant milestone in our time-of-flight spectroscopy program was reached this year when we demonstrated the collection of an optical absorption spectrum in 10 psec. The measurement was accomplished by combining a high-speed continuum light source with the dual path fiber optic dispersion system. The light source consists of a high-power Nd glass laser, operating at about 1 micron, that self mixes in a carbon tetrachloride medium to produce the continuum. Also, we have recently acquired a laser amplifier which will yield a 10-fold increase in peak power at 1 micron. These intense laser pulses should push the frequency range of the continuum further toward the ultraviolet. Using the lower power continuum and the dual beam fiber optic time-of-flight spectrometer, absorption spectra of test samples were obtained using the single pulse mode. Prominent absorption peaks in didymium glass and oxazine 170 laser dye were readily observed and were in roughly quantitative agreement with spectra measured on a conventional photometer. Future studies with this system will be directed toward response optimization and practical testing--hopefully at the Brookhaven National Laboratory Light Source Facility.

We have already shown that the time delay between the impulsive pumping of a laser and the resulting laser emission depends in general upon losses within the laser cavity. Such a "delayed lasing" system can be used to measure very small absorbances by means of a time delay rather than conventional intensity measurements. Our first demonstration of this concept was rather unpretentious experimentally; several modifications of the original equipment have been investigated in an attempt to improve performance.

Time jitter has proved to be a major limiting factor in the system performance. Time uncertainty also degrades lower detection limits. The jitter was found to be correlated to pulse-to-pulse energy fluctuations ($\pm 10\%$) of the pump laser. We attempted to overcome this problem by

making measurements using single laser pulses whose energy was individually monitored. Unfortunately, only small improvements were realized. A second approach involving energy discrimination was only moderately successful also. Further system testing has led us to acknowledge that time jitter has several possible originating sources, and our future work will be directed toward a better understanding of this as well as making a workable, tunable system.

A new program in the group this year is Fourier transform microwave spectroscopy--a novel scheme for measuring microwave absorption spectra. A \$48,000 ORNL seed money grant was awarded to the project for the design and evaluation of such a microwave spectrometer. The device will measure the electric field cross-correlation function in a way similar to that used in FTIR spectroscopy. The novel feature of the proposal is that the temporal delay required for the correlation procedure is produced digitally rather than by a physical path-length change. Our first objective is to prove that such an instrument can be built and that it can provide the theoretically predicted S/N advantage. Most of the necessary electronic components are in hand. The digital circuit has been designed, fabricated, and tested; some optimization is still needed. The fabrication of the microwave components is also in process.

Another exciting event this year was the demonstration of our ability to obtain an optoacoustic spectrum at temperatures as low as 1.5K. At these temperatures electronic absorption spectra are both sharper and less complex, resulting in enhanced analytical selectivity in mixtures. The acoustic detector used was a lead titanate piezoelectric transducer which was coupled to a sample of neodymium hydroxide. Measurements were made of the range 1.5 to 77K.

We have also used a quartz transducer to record the optoacoustic spectrum of 3-bromoquinoline, isolated in an argon matrix at 10K. At present, energy trapping in metastable states of organic molecules along with elevated background signals limits our detection sensitivity. Experiments are currently underway aimed at reducing the importance of these problems. We are also pushing techniques that employ frozen solutions at cryogenic temperatures

for the analysis of weakly and non-fluorescent polycyclic organic compounds.

One application of resonance ionization spectroscopy is to detect recoil species of radioactive decay. We are presently studying the requirements for the detection of ^{218}Po , a 100 keV recoil daughter of ^{222}Rn . We found that the daughter is completely neutralized in a gas mixture of helium with 1-3% of added isobutane but only partially neutralized in helium, argon, or mixtures of argon with methane or isobutane.

Work was also carried out on the detection of the lithium daughter from the decay of ^7Be -acetylacetonate (application to solar neutrino detection). We have assayed our ^7Be compound and are ready to load it. The proportional counter was also found to operate satisfactorily at 150C. Unfortunately, in carrying out the temperature response tests, vacuum leaks developed in the electrical connectors.

Our development of the glow discharge detector has reached a point where a number of practical and valuable analytical measurements can be made with it. A good example is the determination of organic and inorganic fluorine. We have shown that an absolute detection limit of 0.07 pg of fluorine can be attained from diphenyldifluorosilane when introduced into a glow discharge (operating with helium). The diphenylsilane compound suggests itself as a possible general, volatile form of fluorine for use with the detector. The use of the compound is not essential. Inorganic fluorine is detected by liberating HF from the sample. A few (1-3) microliters of sample is placed on a platinum heating block that can be located in the detector chamber upstream but close to the glow discharge. After a weak acidulant is added (KH_2PO_4), the mixture is dried in air. The heater is replaced into the glow chamber and the glow is turned on. A fast heating of the sample generates a pulse of HF which generates the fluorine signal. Fifty to 100 ng F appears to be a reasonable sample load.

The glow discharge detector is also being used to detect oxygenated compounds in the eluate of a capillary gas chromatograph. A major problem in this application is the reduction of the background oxygen signal in the carrier gas (He). Several different traps and

combinations of traps were evaluated with little success. However, recently a helical trap containing a BASF proprietary catalyst has reduced the level of oxygen bearing species in helium to <0.1% of its original level for about 2 hours. A real advantage of this approach is that the catalyst is quickly and easily regenerated in situ. We are just starting initial tests of the detection scheme using simple mixtures of organic oxygenated compounds.

Finally, in spectroscopy, two small efforts are being made in the areas of laser excited fluorescence and heavy-ion x-ray fluorescence. The established technique of selective laser excited fluorescence of precipitated fluorides has been modified and tested for the determination of ultratrace quantities of lanthanides and actinides. This modified technique involves the coprecipitation of the sample with lanthanum fluoride rather than calcium fluoride. The result is a product that is at least, if not more, sensitive to the F-element ion precipitate and is also less influenced by some common interferences in the CaF_2 method. We have shown that curium at concentrations lower than 10^{-10}g can be detected by the revised technique. Unfortunately, preliminary tests with plutonium were negative; this may be due to a problem in the sample preparation.

(C. Feldman, J. M. Ramsey, R. W. Shaw, W. B. Whitten, J. P. Young, H. E. Howell,* G. Mamantov*)

Applied Instrumentation

Essentially all of our applied instrumentation activities are related to specific projects or general analytical needs located within the Consolidated Fuel Recycle Program. These efforts have been particularly rewarding this past year. After more than two years of planning, research, and development, several finished instrument designs—hardware and software—have been installed or are being bench tested. Although all test data are not in, initial evaluations indicate that our conservative

approach to the design concepts will result in instrument systems that require only minimal "fine tuning". Our specific activities fall into two major categories: (1) hot-cell instrumentation, and (2) in-line process instrumentation.

Development work has been completed on the modification of the prototype microcomputer-controlled remote pipetter to a hot-cell version of the instrument. This modified unit was installed in the mock-up hot-cell located in Building 2026 and operating personnel were trained and also asked to evaluate the system. On the basis of suggestions and operating experience, significant improvements were added to the control software and to the remote mechanical hardware. Also, several mechanical fixtures were re-designed for easier operation and greater reliability. Overall, the changes resulted in a factor of two improvement in the overall accuracy and precision of aliquot delivery. The completed instrument has been installed in hot cell No. 4 of Building 2026 and is in routine use.

We also initiated the development work on a prototype instrument for the titration of highly radioactive samples in a remote environment. The microcomputer controller for the unit is very similar to that used in the remote pipetter. The electromechanical design for the titrant delivery unit features interchangeable variably sized burets with a stepping motor-driven plunger and solenoid-actuated delivery valves. Electrochemical transducers having potentiometric or amperometric responses will be available for titration monitoring. The Control Program automatically varies the rate of titrant addition and determines the end point for a titration. A plot of the titration curve is provided and analytical results will be printed directly on the work control sheet.

Detailed evaluation of the Hewlett-Packard Model 8450 multiwavelength spectrophotometer has shown that it is not feasible to modify this system for hot-cell applications. The optical and detector units can be shielded adequately; however, due to cable driver capability and signal propagation requirements, the maximum allowable cable length between the remote detector electronics and the operator's console is one meter, only one-tenth our required

*University of Tennessee, Knoxville

length. Optical communications cables were considered as an alternative. This approach was discarded due to radiation shielding problems and the unwillingness of the vendor to guarantee performance specifications.

Work for the in-line process instrumentation has been broad based in concept but restricted in scope. A large majority of work has been directed toward the measurement of uranium and plutonium under a host of different conditions. The project has been very challenging because of the severe environmental demands placed upon the instrumentation and the long-term reliability that is needed. Nevertheless we have managed to complete the research and development for the free acid monitor and the multiwavelength photometer (uranium systems) this year. Spectral studies of plutonium systems were also completed but much of this work must be converted to working algorithms that can be used with "real" process streams.

The free acid monitor has undergone extensive bench testing to delineate any potential design weaknesses. The equipment has operated without failure using sample solutions containing nitrate, $\text{Al}(\text{NO}_3)_3$, 10-300 μm particles, and entrained organic solvent. Final design changes are being incorporated into the construction of a field version of the acid monitor to reduce construction costs and improve performance.

The multiwavelength filter photometer has also undergone bench testing. Photometric analyses of blind organic and aqueous uranium samples were compared with results obtained by standard colorimetric, titrimetric and coulometric procedures, as well as by x-ray fluorescence. The average absolute error between the known and analyzed U concentration was less than 2.5 g/l U over a 10-200 g/l U concentration range for all methods. We are particularly pleased that several other facilities are interested in using our device in their process areas. General Electric, Y-12, K-25, and the Savannah River Plant and Laboratory have sent representatives to visit and obtain additional information.

We have recently completed a report describing the in-line photometric analysis of Pu in aqueous nitric acid streams. The most accurate and precise measurement of total Pu concentrations is obtained by summing the calculated

Pu(III) and Pu(IV) concentrations, determined simultaneously using 602 nm and 660 nm absorbance data. Future studies in this area will include an evaluation of the photometric determination of Pu in organic PUREX solutions, as well as bench testing the prototype photometer with organic and aqueous Pu standards.

Fluorescent studies indicate that nitrate quenching of uranyl fluorescence cannot be circumvented by temporal resolution of the emission signal as we had hoped. Electrochemical procedures to remove nitrate ion from uranium solutions prior to fluorescent analysis also were not successful. Several alternate measuring techniques are being pursued for the measurement of trace U in organic and aqueous solutions.

(D. A. Bostick, C. Feldman, D. E. Goeringer, L. N. Klatt, J. E. Strain)

PHYSICOCHEMICAL ANALYSES

L. D. Hulet, Jr.,

Problem Solving Using Electron and X-ray Methods

Scanning electron microscopy (SEM), transmission electron microscopy (TEM), x-ray fluorescence (XRF) and x-ray diffraction (XRD), when used in concert, are very powerful methods for solving a large variety of problems. The SEM is equipped with an energy dispersive x-ray fluorescence detector (EDX). As the electron beam scans the specimen it excites x-rays characteristic of the elements present, thus enabling a chemical analysis to be performed at the same time images are recorded. X-ray fluorescence is also induced by bombardment with other x-rays such as monochromatic $\text{AgK}\alpha$ or with ^{241}Am gamma rays. This technique reveals trace elements in the ppm concentration range. Thus with the SEM-EDX and x-ray induced XRF techniques used in combination we can determine shapes and sizes of specimens, element distributions and both major and minor element components. By also recording x-ray diffraction patterns of

specimens their crystal structures can be determined to reveal compound forms present. The SEM has a resolution of about $0.01\text{ }\mu\text{m}$. To examine particles smaller than this the TEM is used. Selected area electron diffraction can also be done with the TEM, allowing crystal structures to be identified in particles as small as $1\text{ }\mu\text{m}$.

The above techniques are especially useful in materials analysis. A very interesting problem involved the strengthening of alumina ceramics by the addition of zirconia. It is believed that the zirconia absorbs stress energies undergoing a crystallographic change from a tetragonal to a monoclinic structure. SEM-EDX examination showed that for a high-strength material to be achieved it is necessary for the zirconia to be of particle sizes less than one μm and to be thoroughly dispersed in the alumina. The above methods are also very useful for trouble shooting. For example, $\text{Ni}_{20}\text{Ti}_{27}\text{B}_6$ is being evaluated as a substitute for diamond in drill bits. In one of the preparations the melt reacted violently with the crucible, which fractured. SEM-EDX studies of the melt-crucible interface showed that aluminum from the crucible had reacted with titanium of the melt, but that Ni of the melt was not involved. Secondary ion mass spectroscopy showed that boron also was not involved.

Several problems related to nuclear energy have been studied. Silver zeolite is used for removing methyl iodide from fuel reprocessing gas streams. Reduction of the silver zeolite in hydrogen increases its adsorption capacity. SEM-EDX showed that small silver nodules ($0.2\text{ }\mu\text{m}$) were possibly responsible for the increased iodide adsorption. Particulate matter composed of U_3O_8 , Na_2O_2 , and iron oxide that is likely to result from core melt in light water reactor accidents has been studied with TEM. Particle sizes ranging from $0.05\text{--}0.10\text{ }\mu\text{m}$ were found. Na_2O_2 tended to form large aggregates. X-ray diffraction is being used to study the fixing of wastes as crystalline phosphates. When CePO_4 was reacted with P_2O_5 a new compound was apparently produced. The lattice spacings of the diffraction pattern did not match any known compounds, but its symmetry was very close to that of NdP_3O_9 . Also the cerium and phosphorus concentrations were 1:3; thus we tentatively conclude that CeP_3O_9 (cerium trimetaphosphate) has been formed.

The confirmation that x-ray diffraction can be used to quantify asbestos in building material has been completed. The method uses aspirin, which has a diffraction line close to that of asbestos, as an internal standard. Levels of chrysotile asbestos as low as 0.5% can be detected. Three sets of standards using different matrices were measured, MgO to simulate firebrick, dimethyl oxalate to simulate organic material, and gypsum which is a major component in wallboard. Asbestos concentrations were varied between 0.5% and 60% in each matrix. It is necessary to thoroughly mix the asbestos in the matrix and to rotate the sample during measurement to avoid preferred orientation errors in diffraction intensities. Using aspirin as an internal standard, relative errors of 10% or lower were maintained.

To detect crystalline materials in low concentrations signal-to-noise must be as high as possible in the diffractogram. The GE XRD-5 used in the asbestos work was modified by adding a post monochromator to suppress noise from fluorescence and Compton scatter; details have been described in previous reports. This year the XRD-5 has been automated by interfacing an Apple computer package with special software for sample change and slewing to major diffraction peaks. Diffractograms are recorded with a multichannel analyzer instead of a strip chart; this will allow low intensity peaks to be deconvoluted from neighboring interferences, thus further increasing detection sensitivity.

(F. L. Ball, H. W. Durr, R. L. Sherman, J. H. Stewart)

Positron and Heavy Ion Spectroscopy

In previous annual reports and in open literature our work on the generation and use of monoenergetic positrons has been discussed. Positrons from radioactive sources have Fermi distributions in their energies, extending over several hundred keV. If a properly prepared moderator surface, such as annealed tungsten, is placed so as to intercept the fast positrons they will be thermalized and a certain fraction will be re-emitted with an energy equal to the

negative work function (about 2 eV for tungsten) of the moderator. These are then extracted from the moderator cavity by an electrostatic field and accelerated to higher energies. Inelastic scattering spectra of the monoenergetic positrons show promise that chemical information from solid surfaces can be obtained; low energy positron diffraction spectra have been recorded.

The ability to use our tungsten moderator under poor vacuum conditions (10^{-5} - 10^{-6} torr) is of highly practical advantage. For this reason workers at several other institutions, Brandeis, Texas A&M, and Lawrence Livermore, have adopted its use. Moderating techniques by other workers require ultra high vacuum conditions. There is a joint effort between our division and the Physics Division to provide a high intensity positron beam by proton bombardment of B_4C . We expect fluxes in excess of 1 million counts $\text{sec}^{-1} \text{cm}^{-2}$.

A stainless steel high vacuum bell jar has been fabricated and attached to the positron spectrometer. Surface analyses will be performed with this system. More measurements will be made of energy loss spectra to look for peaks characteristic of elements and compounds. Low energy diffraction patterns will be compared for electrons and positrons. We have shown that microchannel plates can be used to intensify and record positron spectra as well as electrons. We were fearful that annihilation gamma rays would saturate the plate, but this doesn't seem to be a problem. The microchannel plate will greatly improve statistics of the diffraction patterns.

Heavy ion induced x-ray fluorescence (HIIXRF) is being examined as a technique for the direct determination of the bulk chemical environment of an element. Recent results obtained from the collision of 36.0 MeV Ar^{9+} ions with a series of sulfur compounds at ORIC have been analyzed. The analysis, which agrees with and extends the results of another group at Texas A&M for third row elements, demonstrates that variations in the $K\alpha$ satellite intensity distribution can be correlated with the effective charge on the element and with the bulk valence electron density of the target.

To develop this method into a viable analytical tool, elements beyond the third row must be examined. Towards this goal, a more limited

series of molybdenum compounds has been examined. Although the intensity distribution appears to be more complex than that of sulfur, the results clearly demonstrate for the first time that the chemical environment also affects the L x-ray satellite intensity distribution. It may be possible from these results to obtain information on the valence state of metal alloys. Future experiments will attempt to explore the possibility for a series of molybdenum alloys. In addition, we plan to make a systematic study of molybdenum and selenium compounds so as to open a larger section of the periodic table to analysis by the HIIXRF technique.

(R. Auble,* L. D. Hulett, H. J. Kim,* W. Milner,* G. D. O'Kelly,** S. Raman,* T. M. Rosseel, G. Slaughter,* C. R. Vane,* J. P. Young)

*Physics Division

**Chemistry Division

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2. MASS AND EMISSION SPECTROMETRY

J. A. Carter

In this section, research, development, and support analyses are carried out in three topical groups: Elemental and Ion Probe Surface Spectroscopy, Inorganic Mass Spectrometry, and Organic Mass Spectrometry. Each group has many opportunities to apply newly developed techniques to support laboratory energy programs more efficiently or to offer new knowledge altogether. The section's work is quite varied and involves the study of techniques for: surface characterization, both inorganic and organic; multi-element trace metal analysis in a variety of alloys and ceramics; special nuclear materials analysis for various safeguarding applications, domestic and international; organic MS/MS and GC/MS analysis; actinides isotopic abundance measurements and burnup analysis; and stable isotopes mass spectrometry.

About 30% of our support comes from the Division of Chemical Sciences of the Office of Basic Energy Sciences. This effort, which is generic in nature, provides the technical base for addressing current and future analytical mass spectrometric needs as related to DOE energy programs. Another 20% of our support comes through the Office of Safeguards and Security for domestic and international efforts. The remaining support comes from several other laboratory programs and from various Work-for-Others programs, as, for example, the analytical effort for the Three-Mile Island reactor cleanup.

ELEMENTAL AND ION PROBE SURFACE SPECTROSCOPY

W. H. Christie

Our secondary ion mass spectrometer (SIMS) was relocated into a new facility that was especially renovated for this purpose. The move has contributed to an enhanced utilization of the SIMS technique. We have used SIMS to determine the composition of tritium permeation-barrier oxide films grown on SANICRO-32 surfaces. This alloy was of interest because unlike a similar alloy, INCOLOY-800, a good permeation barrier oxide was not formed under similar experimental conditions. The concentration depth profiles produced in this work are being studied by Chemistry Division personnel in an attempt to relate observed film composition to permeation behavior.

As part of a study to improve efficiency of a thermal ion source, we have used SIMS to study the distribution of uranium on ion source lens elements. A surprising observation was that we were able to differentiate between uranium delivered to the target as ions vs. as neutral species. For uranium ions at 7 keV impact energy, a Gaussian peak shape for the implanted uranium ions was observed, whereas the neutrals were easily differentiated as being on the surface. The implanted ion distribution was in excellent agreement with Lindehard, Scharff, and Schiott (LSS) theory calculations giving a projected range of 2.95 nm. The neutral uranium surface film was less than 0.8 nm thick. Surface distribution maps produced by SIMS can be correlated with changes in ion optics in an attempt to improve the ion transmission of these devices. In conjunction with this work, our SIMS study of the resin bead as a uranium thermal ion source has continued.

A significant portion of our SIMS effort this year was devoted to studying the fate of a variety of dopants in silicon and GaAs substrates as a consequence of laser and thermal annealing. The development of quantitative depth profiling methods based on calibration via ion implantation standards combined with instrument computer control has reduced the determination of dopant redistribution profiles and dopant losses to a more or less routine task.

Preliminary studies have been done that demonstrate the applicability of SIMS to the direct analysis of fuel element dissolver solutions. Isotopics for Cs, U, and Pu were determined from a very radioactive solution by simply making a 1000:1 dilution and then using only 0.3 μ L of solution for the determination. This small sample presented little radiation hazard, allowing conventional handling, and the SIMS measured isotopics were in excellent agreement with other reference measurements.

A new facility was completed for the analysis of highly radioactive, gamma-emitting samples that cannot be handled by conventional methods. Information on hazardous sample types has been badly needed in nuclear fuel burnup studies, in fuel reprocessing chemistry, and in various nuclear experiments. Analyses must provide isotopic ratios and quantitative elemental data at trace concentrations.

To satisfy the above analytical requirements, we have adapted a commercial spark-source mass spectrometer (SSMS) to handle radioactive materials directly in a shielded environment. The completed facility was designed to handle irradiated nuclear fuel solids in which the most significant gamma radiation after aging is that due to ^{137}Cs (0.66 MeV). Other features of the facility are: a relocation of all control and monitor functions to a remote panel, a leaded-glass view window, an external video camera for remote observation of the ion-producing rf spark, a 20-cm diameter shielded glove port for entry of sample electrodes and ion source parts, and a continuous, filtered air flow toward the hot cell which ensures negative pressure containment of radioactive debris.

Photoplates, used to record sample ion intensities, are processed on site via a computerized microdensitometer. A number of processing options were designed into the software. These have been very helpful with unusual sample types, such as highly irradiated material. In an early study with this instrument undissolved solids, present after acid dissolution of a fuel element, were analyzed. A knowledge of the composition of this solid material is of importance to the nuclear fuel reprocessing effort. These highly radioactive solids produced mass spectra containing Mo, Te, Ru, Rh, Pd, I, and Ag. It should be pointed out that this is the first

time that samples with a high gamma intensity have been analyzed directly for a very broad range of elements with some elements at matrix and others at trace concentration levels.

(R. E. Eby, L. Landau, J. R. Sites, R. J. Warmack)

INORGANIC MASS SPECTROMETRY

R. L. Walker and D. H. Smith

We have improved the resin bead technique for sequential plutonium and uranium analysis in spent reactor dissolver solutions by developing a bulk resin bead method. This approach increases the amount of sample exposed to the beads, thus reducing the effect of contamination in the preparations. We have developed resin bead methods for the isotope dilution determination of thorium in Ir-W alloys for the Space Power Program, the determination of fission neodymium for estimation of burn-up of reactor fuels, and low-level thorium and uranium analysis in zircaloy cladding material. We are assisting with characterization of the actinides for the US/UK Actinides Experiment, and continue to support the TMI clean-up effort by providing low-level plutonium and uranium analyses. Finally, uranium concentrations and isotopic ratios are determined for Rockwell for their Waste Isolation Program.

A technique for overcoating resin-bead-loaded samples with a layer of carbon dissolved in rhenium led to dramatic improvements in beam stability and ionization efficiency such that, with a new source design, an improvement in precision of about a factor of two has been realized.

We have continued to provide actinide analyses for the TRU and CFRP programs, stable isotopes analyses for the Chemical Technology Division's separations and isotope sales efforts, and gas analyses on a variety of samples. Three developments have been undertaken that provide more reliable gas analyses and broader

capability. First, the Micromass MS-1201 gas instrument has been interfaced to an Apple-II microcomputer, which serves as a data reduction device. This system eliminates most of the hand work and has improved reliability and efficiency of routine gas analyses. Secondly, a Micromass MS-622 gas instrument has been installed which has the capability for accurate carbon, nitrogen, and sulfur ratio measurements. Finally, a data network involving intercommunication of four datataking computers interfaced to mass spectrometers and a central host computer has become fully operational. This has resulted in improved efficiency.

Work using a double isotopic spike as an internal standard has been initiated; early results are promising, and we are hopeful of improving precision to the 0.1% level.

A modified quadrupole mass spectrometer and sample preparation equipment have been installed in a van. The primary function of this mobile laboratory is to provide on-site isotopic analyses of uranium and plutonium for both safeguards and environmental monitoring purposes. This unique facility has thus far been very successful. It has made three field trips: to the Paducah and Portsmouth Gaseous Diffusion Plants and to the National Lead Operation in Cincinnati. Samples ranging in concentration from <0.5 ppb to pure uranium have been analyzed. In each case, good agreement was obtained with results acquired by other means. We have devised the techniques necessary to analyze natural waters for uranium down to the 1-2 ppb level without requiring chemical separation.

An area of continuing interest for this section is that of isotopic ratio measurements. Safeguards and other applications of this type of mass spectrometry demand the highest possible precision, and we have devoted considerable time in the past year to improving our methodology in this regard. A new ion source has been developed whose optics are of circular rather than rectangular symmetry. This source provides radial focusing and leads to improved ion transmission and lower operating temperatures.

(L. K. Bertram, H. S. McKown, T. R. Mueller, C. A. Pritchard, J. R. Sites, J. R. Walton)

ORGANIC MASS SPECTROMETRY

W. H. Christie and D. H. Smith

A simple, novel collision region has been incorporated into the third field-free region of the triple sector (MS/MS) mass spectrometer. It consists of a hypodermic needle connected to a collision gas supply that is perpendicular to the ion beam and pointing into a diffusion pump. This design reduces the amount of gas diffusing into other portions of the instrument as well as the size of the interaction region of the ion beam and the gas, when compared to conventional collision cells. The overall efficiency of this new design is better than any collision chamber reported in the literature for high energy collisional activation. This new cell gives us the capability for collision-induced dissociation, which is an integral part of most MS/MS experiments.

The major objective in the design and construction of an organic secondary ion mass spectrometry (SIMS) source was to create an inexpensive secondary ion source suitable for involatile organic samples. The completed source proved to be amenable to inorganic SIMS and, by design, is compatible with other type (isotope ratio, MS/MS) mass spectrometers. Initial tests showed it produced approximately 1.5 mA secondary ions under bombardment by 50 nA of 5 keV Ar^+ ions, which represents an improvement over commercial sources.

In conjunction with efforts in MS/MS, the SIMS source was attached to the ORNL triple sector MS/MS instrument. In these preliminary experiments, the collision spectrum of ions formed from involatile organic salts was determined to be identical to those results obtained from ions formed from other ionization techniques. Furthermore, the collision spectra of some inorganic ions studied proved easily interpretable and point the way for further studies of polyatomic inorganic ions using SIMS and MS/MS. Following these initial experiments, the ion source was returned to single magnetic sector mass analyzer where efforts are currently underway to place the system under computer control for the study of chemical enhancement of organic secondary ion production.

A project to elucidate the fragmentation patterns of chalcogen-containing fatty acids and their methyl esters is almost finished. This study has used the high resolution capability of the AEI MS-50 mass spectrometer in addition to its metastable scanning modes. Besides the conventional accelerating voltage (HV) scanning mode from the manufacturer, we have incorporated a linked B/E scanning circuit so that we can obtain both parent ion spectra (HV) and daughter ion spectra (B/E). The sectors have also been electrically uncoupled to perform experiments where consecutive metastable transitions in the first and second field-free regions are observed.

Work on the Kratos MS-25 has focused primarily on a joint project with Exxon and the University of Tennessee involving the pyrolysis of coal. The objective of this study is to determine hydrogen migration routes during pyrolysis. To achieve this goal, coals in which specific sites have been deuterated are pyrolyzed, with the products being analyzed by GCMS. A standard procedure has been developed and numerous runs at various temperatures have been done on normal coal and O-perdeuteromethylated coal.

(G. L. Gligh, E. H. McBay, W. T. Rainey, P. J. Todd)

3. ANALYTICAL TECHNICAL SUPPORT

L. T. Corbin

The Analytical Technical Support Section is composed of four groups: General Analytical Laboratory, Radioactive Materials Analytical Laboratory, Transuranium Laboratory, and Analytical Development Group. The section is supported primarily by programs in the Chemical Technology, Fuel Recycle, Operations, Metals and Ceramics, and Environmental Sciences divisions. The section's work includes testing of oil (primarily for polychlorinated biphenyl content), coal, hazardous wastes, nuclear coatings, organics, and high burnup, highly radioactive fuels. Plans for future work include preparation of site-specific material standards and measurement for the Nuclear Regulatory Commission.

New capabilities of the section include analyses by x-ray fluorescence and inductively coupled plasma emission spectrometry. A catalog of analytical services was added to the Data Management System this year so that customers have ready access to ACD's capabilities and costs.

RADIOACTIVE MATERIALS ANALYTICAL LABORATORY

W. R. Laing

Routine measurements for metals and nonmetals are now being made in aqueous and organic samples by x-ray fluorescence (XRF). When this technique was applied to the solution of chemical analysis problems, it became evident that the technique itself was not free of problems. Changes in matrix were found to be extremely important, and interference from unsuspected elements was another hazard. The results were erratic and often unreliable. As experience has been gained, a philosophy of operation that will minimize these interference problems has evolved. The sample matrix is matched as closely as possible in the preparation of standard solutions. An internal standard is added to all samples and standards. Where a possibility exists for a direct spectral interference, a check is made using a different excitation energy or by looking at other lines of the elements. Background on radioactive samples is measured and subtracted.

A new program, RECAL, obtained from United Scientific Company, is being used to recalibrate for spectral line shift before making x-ray fluorescence measurements. The need for this adjustment was discovered during a matrix effect study, in which uranium, acidity, and nitrate contents were varied. When RECAL was used each day, an improvement of 5% in accuracy was found. Using this approach, over 3000 determinations have been made for U, Pu, Zr, Tc, Th, Mo, and Re in aqueous and organic simulated reprocessing solutions.

A Claigge fluxer has been used to prepare disks for XRF measurements of fly ash, uranium ores, and synfuel catalysts. The preparation of these disks by fusion with lithium tetraborate is relatively easy, but sample disks must be compared with standard disks for a quantitative measurement. The usual ratio of 300 mg sample to 6 g flux permits easy weighing of the standard matrix material, but makes it difficult to weigh the additions necessary for low percentage and microgram/gram levels of minor constituents. Tests which confirm that minor additions can be made using small aliquots (<500 μ L) of standard

solutions were completed this year. Tests of the preparation of Al_2O_3 -based standards containing additions of Ni, Co, Mo, Zn, and an internal standard of Cu, were made. Linear calibration curves were obtained for all elements.

With the current emphasis on precious metals accountability, XRF has been a valuable asset. It has been used to identify metals thought to be gold, silver, or platinum. It has also been used for the analysis of gold plating solutions from the research shops, with considerable time savings. A number of pipe components were identified as Inconel or 304L stainless steel for the Fusion Energy Division. Qualitative scans and alloy identification continue to be a major portion of XRF sample load.

The O/M apparatus was used for the first time to measure an Am-Cm oxide sample. A different oxygen potential for the equilibration gas was necessary from that normally used for U-Pu oxides. In this case, 4% H_2 in helium was passed through a liquid nitrogen cold trap before passage over the sample at 800°C. An O/M of 1.56 was obtained for this sample.

A study of chloride release during nuclear coatings tests has been conducted. Our results indicated that >95% of the chloride was leached in the first four days of the test. Identical samples, tested under the same conditions except for the absence of radiation, also showed the rapid release of chloride. The conclusion was that major factors influencing leachability are the presence of water and elevated temperature. Radiation had little or no effect.

Nuclear coatings work has shifted emphasis from protective coatings for an original facility to coatings, filler compounds, and caulking compounds that would be used in facility repair. Both irradiation tests and design basis accident tests have been performed on these materials. Changes have been made on the control circuit of the superheater installed on the design basis accident test facility. These changes will allow the testing of coatings up to 195°C to more closely simulate reactor accident conditions.

(R. F. Apple, J. L. Botts, R. D. Brooksbank, P. L. Howell, R. E. Jones)

GENERAL ANALYTICAL LABORATORY

R. R. Rickard and N. M. Ferguson

The General Analytical Laboratory (GAL) provided a broad base of technical support for Laboratory programs and approved outside agencies. Over 60,000 analytical results were reported even though the staff has been reduced by 20%. The sample receiving laboratory within GAL frequently routed analytical requests to specialty laboratories outside the group. Approximately 4400 consignments of this type were made. Traceability was maintained with the use of our data management and sample transaction system. Some of the analytical projects undertaken are reported here.

A hazardous waste testing laboratory has been established jointly with the Industrial Safety and Applied Health Physics Division to perform tests on solid wastes. Solid wastes, which include gases and liquids by definition, were tested in compliance with regulations promulgated by the Environmental Protection Agency in keeping with the intent of the Resource Conservation and Recovery Act. Regulations required testing to include the following criteria: (1) ignitability, (2) corrosivity, (3) reactivity, and (4) toxicity.

Gas chromatographic profiles of approximately 37 oils and 4 liquid fuels have been made. These profiles were developed by two different teams, each with its own instrument. The redundancy was justified by the need to respond to oil spill emergencies quickly and effectively. The end use of the profiles was for the rapid identification of oils to determine their sources. All known Laboratory reservoirs (such as pumps, transformers, etc.) that contain oils were sampled and analyzed for polychlorinated biphenyl (PCB) content. Concentrations ranged from over 90% PCB to less than 1 μ g/g. The sample load justified the acquisition of another electron capture detector and a Hall detector.

An ambitious liquid-effluent sampling program initiated by the ORNL Department of

Environmental Management in July required a large number of analyses. The Environmental Analysis Laboratory (EAL) determined arsenic, lead, mercury, thallium, ammonia, Kjeldahl nitrogen, nitrate, organic carbon, and total phosphorus in approximately 15 effluent samples per week. Other analyses were assigned to other laboratories. This workload posed a burden on the EAL staff because other sample-intensive programs were underway. Work schedules were changed, and appropriate personnel were borrowed to increase analytical throughput. The effluent sampling program carried an unusually heavy quality assurance burden.

Fossil energy programs required a wide variety of analytical support. This included Btu measurements, ultimate and proximate analyses, ash-fusion temperature measurements, the determination of neutralization numbers, and the concentration of halogens, sulfide, and trace elements. In addition, many simulated distillation curves were obtained by gas chromatography, and many viscosity measurements were made. A new coal analyzer has greatly simplified proximate analyses.

A sizeable effort was expended to determine leachability of certain elements from simulated waste forms. The graphite furnace technique for determining trace elements by atomic absorption spectrophotometry was employed to determine cesium at the 5 to 10 ng-mL⁻¹ level and strontium at the 0.2 to 40 ng-mL⁻¹ level in the leachate solutions.

Air filters, human blood, and urine samples have been analyzed frequently for lead content. The air filters were also analyzed for other trace metals. For human health and safety reasons, the urine and blood samples required rapid turnaround times. This work, which involved analysis of control samples from the National Institute for Occupational Safety and Health and commercial "blind" standards, was performed for the Industrial Hygiene Department.

(J. E. Attrill, H. G. Davis, W. R. Musick, C. K. Talbott)

TRANSURANIUM ANALYSIS LABORATORY

J. H. Cooper

Approximately two-thirds of the samples analyzed this past year were generated by the Chemical Technology Division in support of transuranium element production and studies of power reactor fuel reprocessing. A mini-mixer-settler in the cell bank is used to study the parameters involved in the solvent extraction of spent fuel elements from a commercial power reactor. Although the extraction tests are of short duration, a large number of analyses are requested to follow the paths of the various elements of concern. In addition to the usual analyses requested for uranium, plutonium, and acidity, the capability for fission product analysis by gamma spectrometry was added. An ND-6600 analyzer system is in routine use by our personnel for these determinations.

Determinations of zirconium concentration were required in a study of the behavior of zirconium in solutions of varying acidity. Our usual analysis for zirconium is by colorimetry. After acquisition of the x-ray fluorescence analyzer by the Radioactive Materials Analytical Laboratory, these samples were analyzed by the x-ray method. Some discrepancies were noted between the two methods of analysis in samples of low acidity. Investigation of this problem showed that the zirconium in some samples was colloidal and not being measured by our colorimetric technique. A combination of the two methods was used to measure total and dissolved zirconium in these samples.

We were requested to provide assistance to TRU engineers in determining the best method for stripping radiozirconium from di-2-ethylhexylphosphoric acid. Several strippants were tried, and the one selected was 0.5 M oxalic acid at elevated temperature.

An acid titration system capable of recording pH vs volume of titrant, as well as the first derivative, has been installed in our laboratory. After thorough evaluation of the instrument and training of personnel, it is being routinely used on samples outside the hot cells.

(J. M. Peele, J. C. Price, J. W. Wade)

ANALYTICAL DEVELOPMENT GROUP

D. A. Costanzo

The Reactor Programs Group was renamed the Analytical Development Group in January 1981 to more closely reflect the nature of the group's analytical methods development and technical support activities. We provide technical assistance to other groups in this section; however, major efforts of the group continue to be directed toward support for ongoing Laboratory nuclear technology programs. BET surface area, helium pycnometry, and mercury porosimetry are being used to characterize the structural properties of reactor core graphite for the High-Temperature Gas-Cooled Reactor Program. A computer-controlled BET surface area analyzer, which operates on a constant volume/variable pressure regime, has been developed for these studies. Other techniques that have been used to characterize reactor fuels and materials include thermal analysis; mass spectrometry; particle size analysis; high-temperature chlorination; hot-vacuum extraction of gases in metals; and ion, liquid, and gas chromatography.

We also function in a liaison/task management role for ACD in support of the Consolidated Fuel Reprocessing Program. This is an intradivisional effort and involves members from the Analytical Methodology, Mass and Emission Spectrometry, Nuclear and Radiochemical Analysis, and Technical Support sections. The effort is concerned with the development of analytical methods, equipment, and support facilities for the overall program. Included are the development of chemical, nuclear, radiochemical, x-ray fluorescence, and mass spectrometric methods suitable for high burnup, highly radioactive fuel; the remotization and automation of analytical equipment; and the development of sampling requirements, mechanisms, and laboratory investigations to support design studies for the Hot Experimental Facility, a proposed demonstration pilot plant. An x-ray fluorescence analysis capability was established for the determination of uranium and plutonium in highly alpha-active process solutions, and a proof-of-principle x-ray fluorescence system was designed and assembled for uranium and plutonium

analyses in highly gamma-active dissolver solutions; evaluation of the proof-of-principle analyzer is underway. Also, gas and liquid chromatographic methods have been developed for the determination of hexamethylenetetramine and formaldehyde in internal gelation process solutions and organic degradation products in recycle tributylphosphate-normal paraffin hydrocarbon recycle solutions. In support of engineering design studies, a laboratory study of the decomposition of hydrogen peroxide in nitric acid was performed.

A new organic service analysis laboratory was established in cooperation with the Bio-Organic Analysis Section to meet their ongoing programmatic needs. Initial efforts have been directed at the analysis of volatile and particulate organic matter in air samples. Future plans are to expand this capability to include organic service analysis to support other Laboratory environmental, industrial hygiene, and process development programs.

An inductively coupled plasma (ICP) atomic emission spectrometer was acquired in February 1981 to meet ORNL's needs for cost-effective multielement analysis at the submicrogram-per-milliliter level. The ICP comprises a radio-frequency argon plasma where atomization and excitation of the analytes occur; a polychromator which can simultaneously determine up to 35 elements; a sequential scanning monochromator, which is useful for the determination of other elements; and a computer system for data acquisition and data management. Trace and major elements analyses have been performed on a variety of sample matrices including coal fly ash and soil digests, leachates, brines, ground waters, and organic liquids. The ICP computer (LSI-11/23) is being upgraded with a multitasking, real-time operating system (TSX-Plus) and a Data Systems DSD 880x/30 Winchester disk system. The vendor-supplied software has been modified to operate under TSX-Plus, and software has been developed for simultaneous operation of the polychromator and monochromator. Software has also been developed to provide communication between the ICP computer and the ORNL DEC-10 System, for direct transfer of ICP data to the ACD's Data Management System.

(D. R. Heine, J. M. Keller, F. L. Layton, D. A. Lee, C. S. MacDougall, J. L. Marley, J. H. Stewart)

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4. BIO/ORGANIC ANALYSIS SECTION

M. R. Guerin

This section is responsible for organic analytical chemistry. Chemical and physical methods are developed and used to isolate organic constituents of complex mixtures. Spectroscopic and chromatographic methods are developed and used to identify and quantitate the constituents. Compounds are synthesized to provide calibration standards and to test hypothesized identifications. Aerosol generation, sampling, and monitoring are carried out in support of inhalation toxicology programs. Analytical research emphasizes spectroscopic approaches to distinguish between isomers and high resolution chromatographic methods to quantitate individual compounds. Applied programs emphasize environmental and health issues associated with human exposure to smokes, with synthetic fossil fuels technologies, and with environmental pollution. Approximately 35 people, including 23 professionals, are engaged in work supported by the Environmental Protection Agency, Electric Power Research Institutes, National Cancer Institute, Department of the Army, and the Department of Energy.

QUANTITATIVE METHODS AND APPLICATIONS

W. H. Griest and J. E. Caton

This group develops and applies methods for the quantitative analysis of environmental and fossil fuels-related samples. We are also responsible for managing sample storage and distribution activities in support of ORNL synfuels programs and an EPA air pollution program.

Our work has relied heavily upon chromatographic methods, particularly gas chromatography (GC) and high performance liquid chromatography (HPLC). Many of the problems encountered this year were solved by direct chromatographic analysis. For example, we have found that catechol, resorcinol, phenol, and the cresols can be measured directly in coal liquefaction process waters by direct reversed phase HPLC using fluorescence detection. Extensive sample preparation or fractionation is not required. The HPLC method has also been successfully applied to the determination of phenols in synthetic fossil fuels by analyzing a basic extract of the fuel. Our results for an NBS Shale Oil reference sample are in good agreement with the NBS certification results.

Methods have been developed to determine vapor phase organic compounds in ambient outdoor air, indoor air, headspace vapors of fossil-fuel derived liquids, and cigarette smoke. The volatile species are collected on Tenax® resin and analyzed by thermal desorption GC using high resolution glass capillary columns. Over 100 components are visualized in the cigarette smoke gas phase profiles. The 34 major species can now be determined quantitatively. We find that components ranging in volatility from methyl butene to limonene can be measured for cigarettes delivering tar over the range of <0.01 to 50 mg/cigt.

The adoption of chromatographic methods preceded by chemical derivatization has considerably improved our ability to determine the major gas chromatographable organic compounds in ambient air particulates and coal fly ash. Solvent extracts are concentrated and trimethylsilyl-derivatized to render polar species sufficiently volatile for GC. Mono- and di-carboxylic acids were identified and quantitated in ambient

air particulate samples collected in the Ohio River Valley. Also, for the first time, the presence of a host of polar organic compounds was demonstrated in a coal fly ash sample which contained mutagenic activity. Derivatization increased the mass accounting of solvent-extractable material by a factor of two (up to ca. 30-40%) for some fly ashes.

Sequential HPLC/GC and HPLC/HPLC methods have been developed to bring the specific advantages of both techniques to bear on especially difficult problems. The routine determination (separately) of benzo(a)pyrene and β -naphthylamine in synthetic fuels and air particulate samples is now achieved using normal phase, semi-preparative scale HPLC to obtain fractions enriched in the analyte. Reversed phase analytical HPLC with fluorescence detection is employed for the final determination. These procedures are relatively less laborious than traditional methods, sensitive (to ppb levels) and highly specific.

Sequential chromatography is being studied as a universal approach to the analysis of complex organic mixtures. Using a mobile phase gradient of increasing polarity allows the elution from a normal phase HPLC column of separate fractions enriched in saturated hydrocarbons, aromatic hydrocarbons, nitrogen heterocyclics plus aromatic amines, and phenols plus carboxylic acids. A final "intractable" fraction consisting of an apparent high polar species also can be obtained. Each of these fractions is analyzed by GC, HPLC, or by FTIR (Fourier Transform Infrared) spectrometry. This procedure has been employed to fractionate and analyze synthetic crude oils and air particulate samples in detail, although polycyclic aromatic hydrocarbons were a main focus of the analyses. We are now improving the methodology for the isolation and the determination of the primary aromatic amines because of their importance as the determinant mutagens in some synthetic crude oils.

To complement chromatographic methods, diffuse reflectance FTIR spectrometry (DRIFT) also has been applied to the characterization of many environmental and fossil-fuel related samples. Compared to conventional transmittance spectrometry, the diffuse reflectance technique offers several advantages including an inherently higher signal-to-noise ratio, easier sample preparation, and the capability of

studying a much wider range of samples under identical conditions. Using this technique mid-infrared spectra of more than 40 organic compounds of environmental significance have been carefully documented. These spectra serve as an initial library for future chemical, biological or environmental characterizations. In addition, similarly generated spectra of a wide variety of fossil fuels-derived products have been recorded for correlations of spectral features with bioassay results.

Approximately 250 aliquots of synthetic fossil fuels-related samples have been distributed to approximately 50 laboratories around the country as part of our synfuels repository function. Chemical and physical stability monitoring of the most important samples is continuing. Samples from the ORNL Synthetic Fuels Life Sciences Program resulting from the University of Minnesota Low Btu Gasifier and the Catlettsburg Kentucky H-Coal Liquefaction Pilot Plant are being stored and distributed as specified by the program plans.

(B. D. Barkenbus, S. H. Harmon, C. E. Higgins, C. S. MacDougall,* R. R. Reagan, B. A. Tomkins, L. B. Yeatts)

ISOLATION AND IDENTIFICATION

C.-h. Ho and M. V. Buchanan

This group conducts preparative scale separations and spectroscopic analyses primarily to provide materials for biological testing and to identify bioactive constituents. Analytical services and research involving mass spectrometry and nuclear magnetic resonance spectrometry are also carried out. Syntheses are performed to produce calibration standards and compounds for biological testing.

A method was developed to extract adsorbed organic compounds from large quantities (0.5-1.0 kg) of coal combustion fly ash. The use of stainless steel mesh thimbles and isolating the extracting solvent from room air allows reproducibly high recoveries of adsorbed organics and very low analytical blanks by Soxhlet extraction. Chemical class separations of petroleum-, shale-, and coal-derived oils have been

routinely applied to determine their chemical compositions and to provide materials for biological testing. Direct slurries of the crude oils in dimethylsulfoxide (DMSO) are found to be promising substitutes for chemical class fractions for mutagenicity testing. Bioteasing efforts required to determine the mutagenicity of oils might be reduced by a factor of ten using this procedure. In complementary research, we find that crude oils and fractions dissolved in DMSO for biological testing can be quantitatively recovered for further study by Sephadex LH-20 column chromatography using tetrahydrofuran as eluent. A preparative scale (0.5-5 gms) high performance liquid chromatograph and a bulk (3 liter charge) distillation apparatus have been added to our separations equipment.

Research to identify mutagens and carcinogens in synthetic fossil fuels has focused on the neutral components of the oils. Mutagenicity is found to be due to the moderately polar components of the neutral fraction. Carbonyl-containing compounds, possibly quinones, have been confirmed to be present in the most mutagenic subfractions. Oils which have undergone hydrotreatment and exhibit great reductions in mutagenicity are found to contain much smaller quantities of neutral polar constituents. Subfractions of the neutral fractions of two coal oils were prepared for mutagenicity and skin carcinogenicity testing. Carcinogenicity and mutagenicity were found by our collaborators to be due to constituents of polycyclic aromatic and polar-substituted polycyclic aromatic subfractions, respectively.

Research to identify the impurity in commercial acridine responsible for gross teratogenesis in crickets has continued. Several high performance liquid chromatography subfractions of the acridine impurities composite exhibit teratogenicity, suggesting that more than one compound may be responsible. One especially active (5 ng results in 9% abnormal embryo development) subfraction has been isolated. Chemical characterization is being hampered by the small sample size available. Two unusual acridine isomers, benz(f)isoquinoline and benz(h)isoquinoline, have been successfully synthesized and are currently being bioassayed.

We have continued our investigation of chemical ionization techniques to distinguish isomeric compounds using mass spectrometry. Ammonia and perdeuteroammonia have been successfully used as ionization reagents to distinguish

*Analytical Technical Support Section

primary, secondary, and tertiary amines in complex mixtures. Chemical ionization has also been used to distinguish isomeric oxygen-containing compounds, such as phenols, aromatic ethers and aromatic alcohols, using methanol and perdeuteromethanol as ionization reagents. Ammonia chemical ionization has shown that primary aromatic amines and azaarenes are the major constituents in the mutagenic acetone subfractions of several synthetic fossil fuels. Trace amounts of primary amines and azaarenes were also found in the mutagenic neutral polar-substituted polycyclic aromatic subfractions of the fuels. These subfractions contained substantial amounts of oxygen-containing compounds which were identified as phenols, carbonyls, and aromatic ethers using methanol chemical ionization.

Nuclear magnetic resonance (NMR) has also been used to characterize isomeric amines in complex mixtures. The amines are derivatized using trifluoroacetic anhydride and the derivatives are analyzed using ^{19}F NMR. Primary and secondary amine substituted aromatics and indole-type secondary amines are widely separated over the ^{19}F NMR spectral range. This allows the rapid determination of the relative amounts of these types of compounds in complex mixtures.

Proton NMR has been investigated as a possible technique for the rapid assessment of fossil fuels with respect to mutagenic activity. After the spectra of a large number of aromatic compounds were studied and compared with each compound's mutagenic activity, it was found that, in general, compounds with aromatic protons shifted downfield of 7.25 ppm displayed activity, whereas those upfield were not active. This technique has been applied to a number of fossil fuels and found to be useful in predicting trends in the mutagenic activities of these materials.

We also have been involved in the characterization of phosphorus smokes. Phosphorus-31 NMR has been used to determine the amount of polyphosphates present and to establish the average chain length of these compounds. Colorimetric procedures have been used to determine total inorganic phosphate content. Thin layer chromatography has been found to be useful in the separation of polyphosphates with up to five phosphate units.

Spectroscopic services in support of Laboratory-wide programs were initiated in

mid-summer. We have provided data and technical consultation to a number of divisions within the Laboratory, including Chemical Technology, Chemistry, Metals and Ceramics, Environmental Sciences, Health and Safety Research, Industrial Safety and Applied Health Physics, and Biology.

(A. R. Jones, G. L. Kao, C. Y. Ma, G. Olerich, R. B. Quincy, I. B. Rubin)

PHYSICAL PROJECTS

R. W. Holmberg

Emphasis continues to be placed on aerosol technology research and development in support of investigations into the inhalation toxicology of Army smokes and obscurants. During the past year our responsibilities have increased significantly. In addition to the ongoing investigation of a diesel fuel aerosol in cooperation with the Biology Division, we now are working on three other obscurant systems: fog oil, red phosphorus-butyl rubber, and white phosphorus-felt.

Two incendiary phosphorus formulations are being studied. One contains white phosphorus impregnated into a wool felt matrix and the other is red phosphorus containing butyl rubber as a binder. In the field, these are deployed explosively (in grenades or artillery shells) where the phosphorus burns to form phosphorus pentoxide which then hydrates to form an obscuring cloud consisting primarily of phosphoric acids. In distinct contrast to their field use where they are deployed and burn in minutes, a toxicological investigation requires that the aerosol be generated for times up to four hours at a uniform and reproducible concentration. We have found that the red phosphorus-butyl rubber formulation, softened by the addition of small amounts of hexane, can be extruded through a small orifice. The ribbon burns as it contacts a flowing air stream to form the aerosol which is carried to chambers for study. A hydraulic extrusion system has been developed to carry out this process. A high pressure pump metering hydraulic fluid into a hydraulic ram controls the extrusion rate and hence the rate at which the aerosol is formed by combustion. A laboratory mock-up has been extensively tested.

Prototypes suitable for delivery to the biological team that will do the toxicology have been completed and are being tested. Preliminary results indicate that this system may also be adapted to the generation of the aerosol from the white phosphorus-felt formulation.

Active support continues to be given to the DF-2 diesel fuel aerosol toxicology studies that are being conducted in the Biology Division. Our responsibilities include maintaining the generators and the exposure systems, documenting particle size distributions of the aerosols, and calibrating the exposure monitoring system. Significant improvements have been made to provide for more reliable operation. In particular, a second thermocouple control system has been added to prevent thermal runaway by shutting the exposure system down if the controlling circuitry fails. New diesel oil pumps have been installed to improve the reproducibility of the aerosol concentration.

Another obscurant system in the Army inventory is deployed with a portable "M3A3" generator system. "Fog oil", a petroleum distillate similar to No. 10 lubricating oil, is evaporated in the generator and recondenses when expelled into the cooler air to form a dense and persistent aerosol. As with the diesel fuel aerosol system, the size of the M3A3 generator precluded its use for laboratory scale inhalation toxicology studies. We have found that a generator system very similar to that used for the diesel aerosol can be used. Experiments in our test chambers have been made to demonstrate that uniform aerosols of fog oil at concentrations up to 10 mg/l can readily be generated for periods as long as six hours. Preliminary particle size distributions, measured by cascade impaction, indicate mass median aerodynamic diameters of about one micrometer. The Environmental Protection Agency at Research Triangle Park, N.C., has been chosen to perform the toxicology experiments. Our responsibilities are to provide generators and monitors for the aerosol and to give technical assistance in their use. We have recently delivered equipment necessary to outfit one exposure chamber and have visited the responsible scientists there to discuss the use of this equipment. In the next few months we will fabricate and deliver the necessary equipment for them to outfit the five chambers that will be used for the exposure studies.

A data logger system for the acquisition and management of data from peak producing instruments (especially chromatographs) is being developed. Two major components are involved; (a) an off line data logger which digitizes, preprocesses, and stores on magnetic tape and, (b) a minicomputer system to accept the tape data, process, plot, and archive it. The data logger is based on an AIM-65 single board microcomputer with a 12 bit analog-to-digital converter and a Phillips type minicassette for temporary data storage. FORTH is being used as the programming language. This device is complete except for interfacing the A/D module. An LSI 11/23 computer system with multiuser RT11/TSXPlus operating system to manage and archive the data has been acquired and is operational.

Our studies for the Council for Tobacco Research (CTR) have been completed with the successful transfer of all instrumentation from Microbiological Associates, Bethesda, Md., to the University of Kentucky, Lexington, Ky. Manuals detailing operating and maintenance procedures were completed to assist the transfer.

(P. Berlinski, T. M. Gayle,* J. H. Moneyhun, C. W. Rogers)

SAMPLING AND SPECIAL PROJECTS

R. A. Jenkins and M. P. Maskarinec

This group is responsible for field sampling and for chemical analyses in support of special projects. Military obscurants, cigarette smoke, and solid wastes are being studied. Comparing compositions of laboratory-generated test materials with authentic field samples and determining the dose achieved during biological testing are among the special services provided.

We carried out one major sampling expedition in this reporting period. The Veteran's Administration Hospital Research Laboratory, East Orange, N.J., was visited to carry out instrumental monitoring and to collect samples of exhaled air and of expelled urine from dogs

*Instruments and Controls Division

being chronically exposed to cigarette smoke. Otherwise, sampling activities were limited to studies of the influence of sampling flow rate on the apparent composition of diesel fuel aerosols under study in the Biology Division and to providing guidance and hardware in support of research on indoor air pollution (Health and Safety Research Division) and on the H-Coal Pilot Plant in Catlettsburg, Ky. (Synfuels Life Sciences Program, Chemical Technology Division). Several new analytical methods have been developed as part of our cigarette smoke research. N-nitrosamines can now be determined quantitatively in complex mixtures with great selectivity using our recently acquired "Thermal Energy Analyzer" (proprietary catalytic conversion of organic NO-functionality to a form detected by chemiluminescence) as a gas chromatographic detector. Results for vapor phase nitrosamines in cigarette smokes have been in excellent agreement with those reported by others. A method based on extensive purification and the final nitrogen-selective gas chromatographic measurement of nicotine and cotinine in plasma has been developed. It is being applied to determine the dose accompanying the exposure to dogs to cigarette smoke inhalation. A method has been developed to collect and determine volatile constituents of urines from dogs exposed to cigarette smoke. Statistical pattern recognition methods based on the ten predominant gas chromatographically visualized components allows the correct assignment of animals to smoking versus control groups.

Applied cigarette smoke research has shown that even low "tar" products can deliver quantities of "tar" and nicotine which are comparable to those delivered by the more popular filter and nonfilter cigarettes if the smoking characteristics (puff volume and frequency) are sufficiently altered. Studies of foreign commercial cigarettes have shown that products consumed in the Peoples Republic of China exhibit a much decreased quantity of nicotine relative to tar as compared to Western cigarettes. Applied research currently focuses on determining the constituents of commercial cigarette smokes not measured by the Federal Trade Commission and on changes in smoke delivery with changes in smoking conditions.

Support of research into the health effects of inhalation exposure to diesel fuel military obscuration has continued. We find that the constituents of the starting diesel fuel are

distributed between the vapor and particulate phases of the obscuration at a ratio depending on the particle phase concentration. Vapor phase components are greater at lower smoke concentrations. Thus far, we find the composition of laboratory-generated smoke to be comparable to that of field-generated smoke. We also find that decachlorobiphenyl (DCBP) can serve as an excellent marker compound for diesel fuel aerosol dosimetry. DCBP added to the diesel fuel before aerosolization is distributed into the particle phase independent of particle size and may be quantitatively determined by electron capture gas chromatography in the tissues and organs of exposed rodents.

Several methods have been developed to support research into the characterization of solid wastes. Carboxylic acids, common major constituents of solid waste leachates, can now be quantitatively determined by high performance liquid chromatography using conductivity detection. Volatile components of solid wastes can now be determined by trapping on polymer resins and subsequent thermal desorption gas chromatographic analysis. The sequential equilibration of solid wastes with acidic, alkaline, and neutral extractants has proved to be a superior method for isolating organic constituents of the wastes for subsequent biological and chemical analysis. These methods are being used in collaboration with the staff of the Environmental Sciences Division, to establish the relevance of laboratory leachates vs. authentic landfill leachates.

(R. S. Brazell, R. W. Harvey, D. L. Manning, J. S. Wike)

5. NUCLEAR AND RADIOCHEMICAL ANALYSIS

J. R. Stokely

This section is organized into three groups: Low-Level Radiochemical Analysis, Radiochemical and Activation Analysis, and Special Projects. The groups perform analyses and R&D to support several DOE sponsored programs at ORNL and the other Nuclear Division facilities. A small but growing activity is work for other government agencies and private corporations.

About 75% of the section's work is involved with routine and special analyses for Nuclear Division programs. These analyses range from simple routine measurements of radioactivity to complicated determinations involving extensive radiochemical separations and high-sensitivity counting. Special capabilities are available in neutron activation analysis, gamma-ray spectroscopy, and environmental radiochemistry. This year the section provided over 100,000 analyses in support of Nuclear Division programs.

Research and development projects are conducted to improve routine operations or to provide new techniques for radiochemical analysis. We are especially active in development of computer software for gamma-ray spectroscopy and activation analysis, in application of specialized radiochemical analysis techniques for environmental monitoring and surveillance, and in development of radiochemical separations for low-level analysis.

RADIOCHEMICAL AND ACTIVATION ANALYSIS

J. F. Emery

Over the past seven years, we have been involved in the National Uranium Resource Evaluation (NURE) program. Our involvement has been to determine uranium in sediment samples by neutron activation analysis using delayed neutron counting. Over the last year, approximately 48,000 uranium analyses were made for this program. Because of program deadlines and delays in receipt of samples, this work had to be done over a three-month period at the end of FY 81. To analyze the large number of samples, we operated the ORR activation analysis facility on a 3-shift per day schedule for about eight weeks and made modifications in the analysis procedure to improve efficiency and decrease the analysis time. The program deadlines were met with about a week to spare. Quality control was maintained at a high level even with the high sample throughput. Blind control samples were analyzed routinely and showed coefficients of variation of 7 and 2 percent for 0.6 and 5 ppm uranium, respectively. The work done this year completes our involvement in this program; experimental work on the program was terminated at the end of FY-81.

This group has continued to provide general radiochemical and activation analysis services for a number of Laboratory programs. Multi-element neutron activation analysis was used to establish the elemental content of samples for a site specific study of a gasification plant at the University of Minnesota. These elemental analyses were made to assess the health and environmental effects of the plant and to provide data for a mass balance study. As part of the Laboratory's efforts in decontamination and decommissioning of old nuclear facilities, we provided radioanalytical support for studies related to Building 3505. We continued to provide support for reactor fuel reprocessing development. Reactor flux maps were obtained from assays of cobalt wires irradiated in the Bulk Shielding and Oak Ridge Research Reactors. We have continued routine analysis of radioisotopic

products made by the Laboratory for use by outside organizations.

Work for other government agencies and private organizations has increased somewhat this year. A project with IBM Corporation was successfully completed that involved the ultra low-level determination of uranium and thorium in semiconductor materials. This effort helped IBM identify sources of uranium and thorium contamination in computer memory units. We were able to extend our NAA sensitivities to the sub-ppb level as a result of development activities connected with this project. We have also been involved in work with the U.S. Bureau of Mines and U.S. Geological Survey on analysis of various materials for uranium and its daughters and multielement NAA of soil samples. Radiochemical analyses have been performed for General Public Utilities (TMI) and Florida Power and Light Co. to assist these organizations in monitoring radionuclides at their nuclear reactor facilities.

Development activities are performed to provide new methodology or to improve routine operations. We have made several improvements in computer software for gamma-ray spectral analysis and neutron activation analysis on the Nuclear Data (ND 6620) data acquisition system. Software has been written and put into use for calculation of counting efficiencies at any chosen source-to-detector distance without the need to obtain an efficiency calibration curve for the specific source-to-detector geometry. A program has been written to correct gamma-ray spectral results for gamma-ray absorption by the sample or sample container. New software was developed to allow the ND 6620 to be used for absolute neutron activation analysis calculations. Radiochemical methods were developed to determine iodine valence states in reactor cooling water, and a method is now under development to utilize neutron activation analysis for determination of boron in fission product contaminated reactor primary coolant water.

(L. C. Bate, F. F. Dyer, L. R. Hall, J. W. Hama, M. P. May, K. J. Northcutt, H. A. Parker, J. W. Wade)

LOW-LEVEL RADIOCHEMICAL ANALYSIS

T. G. Scott

The Low-Level Radiochemical Analysis Group performs low-level radioanalyses for both natural and man-made radionuclides in a variety of materials to support programs and projects conducted by UCC-ND and by outside organizations. During the past year, we gave support to various programs in the following proportions: environmental monitoring, 40%; environmental surveillance, 30%; research and development, 25%; and work for others, 5%.

Much of the environmental monitoring work was of a statutory nature. We routinely analyzed charcoal and filter papers from atmospheric monitors and off-gas stacks, water from local streams and area lakes, well water from waste burial grounds, fish collected from the mouth of White Oak Creek, and grass and soil from areas around atmospheric sampling stations. Approximately 9000 determinations were made on these materials. Gamma emitting radionuclides (primarily ^{131}I , ^{137}Cs , and ^{60}Co) were determined by Ge(Li) gamma spectrometry. Radioisotopes of strontium, plutonium, americium, curium, uranium, and thorium were determined by radiochemical methods that incorporate low-background beta and alpha counting techniques. A recently-purchased Tri-CarbTM 460C liquid scintillation counting system was used to screen aqueous samples for ^{90}Sr by Cerenkov radiation measurements as well as to measure tritium.

A large portion of the work concerning environmental surveillance was directed toward the Formerly Used Sites, Remedial Action Program (FUSRAP). The purpose of this program is to survey sites formerly utilized by the Manhattan Engineer District (MED) and the AEC for work involving the handling of radioactive materials. Over the past year, we have analyzed materials (predominantly aqueous solutions) from sites in New Jersey, Pennsylvania, Massachusetts, Utah, and Missouri. The same analyses were usually required on samples from all of the sites, i.e., ^{226}Ra , ^{230}Th , ^{210}Pb , and ^{238}U . The largest suite of samples was from Salt Lake City and contained some of the most intractable materials. Special procedures were developed to

determine ^{226}Ra and ^{210}Pb in materials that did not respond satisfactorily to normal methods. The use of selective sulfate precipitations from EDTA solutions proved to be effective in making the above determinations and is being adapted to methods for determining ^{90}Sr , ^{230}Th and transuranium elements.

Soil samples that were suspected of containing plutonium in a highly refractory form were solubilized by sintering with sodium peroxide. The sintering process was found to be more satisfactory than fusions in that it requires less time and presents fewer interferences when sequential separations are desired. Also, the sintering process was found to be useful in determining ^{90}Sr and ^{99}Tc in intractable materials.

Methods were developed, tested, and used to analyze fuel, products, and wastes related to a coal gasification plant. Emphasis was placed on a method for the determination of ^{210}Po because of the relatively high volatility and questions regarding the fate of Po during plant operations. Coal, bottom ash, cyclone fines, tar, and impinger solutions were analyzed. Wet ashing followed by pyrosulfate fusion was used to solubilize materials prior to chemical separation, which involved liquid-liquid extraction with triauryllamine, acid stripping, and spontaneous deposition on silver discs. Alpha spectrometry was used to determine ^{210}Po by relative measurements with ^{209}Po tracer. Results from the analysis of the above-mentioned materials indicate that ^{210}Po tends to concentrate in the bottom ash.

Operations in the low-level group have been improved this year by the installation and use of the ACD data management system and by use of additional programs written for our TI-59 printing calculators. We communicate with the PDP-10 through a DECwriter III printing terminal for logging and tracking samples, composing reports, and maintaining a backlog. Programs for the TI59 calculators are stored on magnetic cards and, when executed, provide alpha-numeric printed tapes for a check-off list and permanent documentation of variables and parameters used in each calculation.

(G. I. Gleason, P. S. Gouge, C. C. Granger, C. V. Holmes, W. C. Massey, S. H. Prestwood, L. M. Roseberry, N. A. Teasley, A. J. Weinberger)

SPECIAL PROJECTS

J. S. Eldridge

The Special Projects Group provides technical support for environmental surveillance activities in the Department of Environmental Management of the Industrial Safety and Applied Health Physics Division. In addition, nondestructive assay assistance is provided for special nuclear materials and precious metals accountability in the Laboratory Protection Division. Radiological measurements under emergency conditions, for both Laboratory and DOE-ORO operations, is another responsibility of this group.

One of the major efforts in environmental surveillance is the investigation of radionuclide movement via activity of aquatic insects. Extensive field studies are being performed in the storage pond at the old hydrofracture disposal site in Melton Valley. Study parameters have included sediment, water, suspended matter, phytoplankton, zooplankton, collector-gatherer invertebrates, collector-filterer invertebrates, predator invertebrates, and emergent adult insects. Information provided by these studies will be incorporated into the design of environmental surveillance procedures at waste disposal ponds and nuclear fuel storage pools. An ancillary insect experiment involved the preparation of mosquitoes tagged with radioactive tracers (^{125}I and ^{65}Zn) for assessing the magnitude of radionuclide transfer to man by mosquito feeding. Small but measurable transfers were determined.

We have performed environmental surveillance measurements in a variety of other studies: honeybees as bioindicators of radionuclide contamination, radionuclide uptake and transport by wild ducks inhabiting a contaminated lake, radioactivity residuals following decontamination and decommissioning activities, and radionuclide leaching and transport from shallow land disposal sites.

Assistance in performing inventory audits for special nuclear materials is provided for the Laboratory Protection Division in the form of nondestructive assay procedures (NDA). By the use of a portable γ -ray spectrometer system, representative inventory samples are examined by NDA measurements at various "Balance Areas"

within the Laboratory. Such NDA procedures have been in use for approximately two years, and a few examples of mislabeling have been uncovered. These procedures have improved the inventory reconciliations. A new application of NDA is being implemented for inventory verification in precious metal audits. We are acquiring a semi-portable system for performing nondestructive x-ray fluorescence analyses at precious metal storage areas. By the use of source-excited x-rays, we will determine the major element composition of objects carried in precious metal inventories.

Rapid response for assistance in radiological emergencies is an area in which we participate at two levels: off-site for environmental surveillance, and on-site in support of local events involving radioactive materials. Through a cooperative program with the ORNL Department of Environmental Management, we provide analytical assistance and operate the radioactivity measuring equipment in the DOE emergency van. We have calibrated the gamma spectrometer system and verified the performance of the alpha and beta counting equipment for field use. Two off-site drills have been performed recently in which teams from DOE and ORNL have responded to simulated releases of radioactivity to the environment. The first such drill was a local one in which we operated the mobile lab in Oak Ridge. The second response was in conjunction with an annual exercise of the TVA emergency plan at the Sequoyah plant. The latter drill required movement of the lab and survey teams to an emergency response center in Chattanooga. We also participated in an on-site drill involving a simulated criticality incident at a nuclear fuel storage facility. The latter response involved coordination of facility and manpower resources as well as rapid response analyses.

(C. V. Holmes, N. A. Teasley)

6. QUALITY ASSURANCE, SAFETY, AND TABULATION OF ANALYSES

L. T. Corbin, Quality Assurance Officer
F. L. Layton, Quality Control Officer
J. S. Wike, Division Safety Officer

QUALITY ASSURANCE

The quality assurance (QA) program for the Analytical Chemistry Division has continued as in past years. The QA program for the Technical Support Section of the division has been a part of the Data Management System (DMS) for two years. Quality assurance/control program reports are issued internally on a monthly basis with quarterly summaries of the statistical data. Being a part of the DMS has greatly facilitated the preparation of monthly and quarterly control reports.

Table 6.1 shows the quality level for each of the service laboratories within the Technical Support Section and compares the results with

Table 6.1. Distribution of control tests by laboratory (October 1980 through September 1981)

Laboratory	Number of control results		Quality level (%) ^a	
	Total	Outside fixed limits	1980	1981
General Analytical	471	15	96.16	96.82
Environmental Analysis	600	60	93.27	90.00
Radioactive Matls Analytical	324	13	97.32	95.99
Transuranium	100	17	95.39	83.00
Total	1495	105	95.00	92.98

^aQuality level (%) = [(No. determinations inside fixed limits)/(total No. determinations)] x 100.

those from the previous year. Lists of the different control programs and the number of results reported for each program are given in Table 6.2. During the past year, there has been a 38% decrease in the number of control determinations reported and a 20% decrease in the number of control programs.

Several internal QA audits are held each year, and each section of the division has been audited at least once.

SAFETY

Employees of the division have accumulated over two million workday hours since the last lost time accident in 1972. This record is possible because of the enthusiastic support of safety programs within the division. For example, eighty-five percent of employees attended a safety meeting each quarter. Thirty percent attended special training sessions, one of which was an in-house CPR course conducted for members of emergency teams. Most employees participated in a division-wide safety show by Magician Austinini. One safety suggestion of many rendered was incorporated into emergency evacuation procedures for buildings with multiple access corridors. Housekeeping and safety awareness within the division continue to improve.

SUMMARY OF ANALYSES RENDERED

Table 6.3 contains a tabulation of support analyses performed by the various laboratories and/or groups within the division during this reporting period.

Table 6.2. Distribution of control results (by method)
for October 1980 through September 1981

Method	Constituent	Number of programs	Number of results	Total
Atomic absorption spectrometry	Aluminum	1	22	
	Calcium	2	56	
	Chromium	1	14	
	Copper	2	15	
	Iron	1	43	
	Lead	1	10	
	Lithium	2	7	
	Magnesium	2	28	
	Nickel	1	7	
	Potassium	2	35	
	Sodium	2	47	
	Zinc	2	<u>10</u>	294
Colorimetry	Chloride	1	18	
	Molybdenum	1	13	
	Nitrogen	2	107	
	Phosphorus	1	43	
	Sulfate	1	60	
	Thorium	1	1	
	Uranium	2	98	
	Zirconium	1	<u>3</u>	343
Coulometry	Uranium	2	<u>32</u>	32
Fluorometry	Uranium	2	<u>315</u>	315
Gravimetry (Leco)	Carbon	1	<u>61</u>	61
Infrared absorption spectrophotometry	Carbon	1	<u>78</u>	78
Volumetric titrimetry	Acid	3	71	
	Chloride	1	1	
	Nitrate	1	48	
	Sulfur	1	67	
	Thorium	1	1	
	Uranium	2	<u>184</u>	372
Total		44		1495

Table 6.3 Summary of analytical work

Organization	Number of results reported by						Total
	Mass and Emission Spectrometry	General Analytical Laboratory	Transuranium Laboratory	Radioactive Mate- rials Analytical Laboratory	Physico- Chemical Analysis	Nuclear and Radiochemical Analysis	
ORNL Unit							
Analytical Chemistry	905	4,946		33	80	4,513	10,477
Biology	372	49		14		1	436
Central Management		172	51		21		244
Chemical Technology	12,782	10,939	19,558	8,843	1,601	5,409	59,132
Chemistry	1,105	1,076	111	15	1,067	1,019	4,393
Engineering Physics	273	112			20		405
Engineering Technology	321	6,284			137	795	7,537
Environmental Sciences		26,651		18	159	3,757	30,585
Fuel Recycle				81	130		211
Fusion Energy	323				16		339
Health	1,250	461			3		1,714
Health and Safety Research		506	10	191	223	2,831	3,761
Industrial Safety and Applied Health Physics		3,476			5	7,091	10,572
Information						1	1
Instrumentation and Controls	1,205	78	15	78		22	1,398
Metals and Ceramics	14,452	4,028		946	815	2,706	22,947
Neutron Physics						181	181
Operations		735	9,603	292		5,856	16,486
Physics			62	59		29	150
Plant and Equipment	155	369				8	532
Quality Assurance and Inspection	204	16					220
Solid State	2,275	435		70	153	199	3,132
Others							
Protective Coating Testing				1,171			1,171
NURE ^a						75,692	75,692
K-25 and Y-12	2,078					8	2,086
Miscellaneous	14,232	406		85	72	334	15,129
Savannah River Laboratory	982						982
TOTAL	52,914	60,739	29,410	11,896	4,502	110,452	269,913

^aNational Uranium Resource Evaluation.

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7. SUPPLEMENTARY ACTIVITIES

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

ADVISORY COMMITTEE

This year the division Advisory Committee was composed of:

- L. L. Lewis, Head, Analytical Chemistry Department, Research Laboratories, General Motors Corporation, 12 Mile, Mounds Road, Warren, MI.
- H. B. Mark, Jr., Chairman, Chemistry Department, University of Cincinnati, Cincinnati, OH.
- F. W. McLafferty, Chemistry Department, Cornell University, Ithaca, NY.
- M. V. Novotny, Chemistry Department, Indiana University, Bloomington, IN.
- H. L. Pardue, Chemistry Department, Purdue University, West Lafayette, IN.

CONSULTANTS

- A. E. Cameron* advised the Mass and Emission Spectrometry section of the division.
- M. T. Kelley advises the Analytical Methodology section, with particular emphasis on computer applications.

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

- R. E. Dessy, Virginia Polytechnic Institute and State University, Blacksburg, VA.
- C. Poole, Wayne State University, Detroit, MI.
- E. Milne, National Institute of Health, Bethesda, MD.
- R. Hagemann, Centre D'Etudes Nucleaires de Saclay, France.
- R. F. Fleming, National Bureau of Standards, Washington, DC.
- R. Conzemius, Ames Laboratory, Ames, IA.
- J. VanLoon, University of Toronto, Toronto, Canada.
- H. Blount, University of Delaware, Newark, DE.

*Deceased.

PARTICIPATION IN ORNL IN-HOURS PROGRAM

The following divisional people completed courses during the 1981 winter term of the Continuing Education Program for Scientific and Technical Personnel:

- M-310E. Introduction to the DECsystem-10: D. E. Goeringer, C. C. Granger, and N. A. Tensley, Jr..
- M-158. Introduction to System 1022 Data-Base Management System: T. G. Scott and J. W. Wade.
- S-202H. Design and Structure of Technical Writing: B. D. Barkenbus and R. D. Brooksbank.

The following courses were completed during the spring 1981 term:

- M-120. Introduction to FORTH Programming: D. R. Heine.
- M-310F. Introduction to DECsystem-10: W. E. Gilliam and M. P. May.
- M-605A. Practical Statistics II: B. A. Tomkins.
- S-203F. Technical Writing Workshop: J. L. Marley.

S-800. Frontiers in Biological Sciences: P. J. Todd.

Courses completed during the fall 1981 term were:

M-158A. Introduction to System 1022 Data-Base Management System: C. S. MacDougall.

M-600E. Practical Statistics I: B. D. Barkenbus.

IAEA FELLOWSHIP PROGRAM

Francia P. Verceluz, Nuclear Research Assistant, Philippine Atomic Energy Commission, Diliman, Quezon City, Philippines, was assigned to the division as an IAEA fellow June 2, 1981, on a one-year appointment. She is working in the Analytical Technical Support Section in the General Analysis Laboratories including the Environmental Analysis Laboratory and our Analytical Clean Room learning techniques such as atomic absorption spectrometry, cold vapor and graphite furnace techniques; Auto Analyzers, and inductively coupled plasma emission spectrometry.

GUEST ASSIGNMENTS

Geoffrey Gleason, Senior Scientist from ORAU, continued his work as a consultant on the staff of the Nuclear and Radiochemical Analyses section until May, at which time he was placed on our staff.

The University of Tennessee continued to fund Brisco L. Harward and Vincent E. Norvell to work in the Analytical Methodology section on the study of rapid scan spectrometry. Norvell completed the requirements for a Ph.D. and left the division, having accepted a position at Y-12. Also funded is Huston E. Howell, Jr., who is studying matrix isolation photoacoustic spectroscopy.

John Vargo is assigned to the Bio/Organic Analysis section under the subcontract initiated with the University of Tennessee. This contract provides the technical services of advanced graduate students in environmental analytical chemistry to the division. Also working under this contract in the Bio/Organic Analysis section is Donna Taylor Dixon.

W. J. Carter, a University of Tennessee Post-Doc, worked in the Bio/Organic Analysis section for a four-month period.

SCUU PROGRAM

S. L. Severs, Centre College, was selected for the 1981 SCUU Science Semester program. Her assignment involved working with M. P. Maskarinec in the Bio/Organic Analysis section on the hazardous wastes project and the cigarette smoke program.

SUMMER PROGRAM

During the summer, the division was host to representatives of the ORAU Undergraduate Research Program, which has been developed to offer laboratory experience to college students as well as to promising high school graduates. B. A. Tomkins served as "Dean" for these guests during their visits.

J. F. Graham, University of Missouri, worked in the Analytical Methodology section on the development of computerized instrumentation.

M. A. Nussbaum, Taylor University, was assigned to work with the inductively coupled plasma emission spectrometry program in the Analytical Technical Support section.

J. R. O'Lear, University of Colorado, worked in the Analytical Methodology section on laser spectroscopy.

The division also hosted an ORAU Faculty Research Participant during the summer. M. J. Sepaniak worked for a ten-week period in the Bio/Organic Analysis section on R&D in high pressure liquid chromatography.

Two people were assigned to the division as part of the Summer Research Internship Program.

V. S. Adams, University of Georgia, was assigned to the Bio/Organic Analysis section to work with R. S. Brazell on the analysis of volatile components in urine.

R. L. Robinson, Carleton College, also worked in the Bio/Organic Analysis section on the fossil fuels project with M. V. Buchanan.

GLCA PROGRAM

During the fall semester, three students were assigned to the division. E. W. Kristensen from Saint Olaf College in Minnesota was assigned to the Analytical Methodology section writing programs for AIM computer. The Bio/Organic Analysis section hosted E. C. Tews, who worked with M. P. Maskarinec on chromatographic analysis. C. I. Reigel, Wabash College, worked in the Analytical Technical Support section on the ICP with D. A. Costanzo.

ORNL CO-OP PROGRAM

Vanessa H. Ostrum and Gregory B. Hurst, both from the University of Tennessee, alternated quarters working in the Bio/Organic Analysis section with the fossil fuels program.

SPECIAL AWARDS

W. S. Lyon, Jr., section head for the Analytical Methodology section, received the George Hevesy Medal for outstanding contributions to the field of analytical chemistry. The award, sponsored by the *Journal of Radioanalytical Chemistry*, was presented jointly to Lyon and Max Peisach of South Africa, at the Modern Trends in Activation Analysis Conference in Toronto, Canada. Lyon is only the fourth American to receive the award since it was instituted in 1968.

L. T. Corbin and J. H. Stewart, Jr. received the Harlan J. Anderson meritorious award at the July, 1981, meeting of ASTM C-26 Committee in Reno, NV.

25th ANNUAL ORNL CONFERENCE ON ANALYTICAL CHEMISTRY IN ENERGY TECHNOLOGY

This year's conference was held at the Riverside Motor Lodge in Gatlinburg, Tennessee, October 6-8, 1981. The theme of the conference was "Analytical Chemistry in Nuclear Technology." Attendance was 200, including 18 exhibitors and 21 booths. Topics covered during the meeting included heavy metal analyses, general analytical, mass spectrometry, instrumentation, and Three-Mile Island and environmental analysis.

A. L. Harrod was General Chairman, W. S. Lyon was Technical Program Chairman, and M. L. Emery was Treasurer. Linda Plemons and Doris Smith were Conference Secretaries. Other Conference Committee members included L. T. Corbin, D. A. Costanzo, J. F. Emery, H. H. Ross, W. D. Shults, J. R. Stokely and R. L. Walker.

ADDITIONAL PROFESSIONAL ACTIVITIES

APPLE, R. F.

Member: ASTM Committee D-33, Protective Coating and Lining Work for
Power Generation Facilities
Chairman Task Group on Methods for Coatings Analysis, ASTM

ATTRILL, J. E.

Member: ASTM Committee E-38 on Resource Recovery
American Chemical Society
Advisory Committee: Subcommittee E-38.01 on Energy, ASTM

BALL, F. L.

Secretary: Electron Microscopy Society of America
Member: Analytical Chemistry Division Seminar Committee

BRAZELL, R. S.

Lecturer: ORAU Traveling Lecture Program

BUCHANAN, M. V.

Chairman: Analytical Chemistry Division Seminar Committee
Treasurer: East Tennessee Section, American Chemical Society
Invited Lecturer: National Autonomous University of Mexico, Mexico City,
January 12-16, 1981

CARTER, J. A.

Secretary: Subcommittee C-5:05, Analytical Task Group (Reactor Grade
Graphite), Committee C-5, Manufacture of Carbon and
Graphite Products, ASTM
Member: Committee C-26, Nuclear Fuel Cycle, and C-26:05, Test
Methods, Task Group Leader, Micro Analytical Techniques,
ASTM
ORNL Management and Supervisory Development Program Advisory
Committee
ORNL Personnel Development Chemistry Course
Technical Chairman: 34th Annual Summer Symposium on Analytical Chemistry,
Pittsburgh, PA
Advisory Board American Chemical Society
Laboratory Coordinator: ISPO Programs

CATON, J. E.

Co-Chairman: Vibrational Spectroscopy Subgroup of UCC Analytical
Specialists Group
Member: Analytical Chemistry Division Long Range Planning Committee
NBS/DOE Analytical Characterization Group

CHRISTIE, W. H.

Member: ASTM Committee E-42, Surface Analysis
Organization and Symposium on Use of Surface Science Techniques in Solving
Session Chairman: Analytical Problems, 181st National Meeting, ACS, Atlanta,
GA, March 29 - April 3, 1981

Symposium on Surface Techniques, Inorganic and Organic, 34th
Annual Summer Symposium on Analytical Chemistry, University
of Pittsburgh, Pittsburgh, PA, June 30 - July 2, 1981

CORBIN, L. T.

Fellow:

Member:

American Society for Testing and Materials

Committee E-10, Nuclear Applications and Measurement of
Radiation Effects, ASTM

Subcommittee E-10:01, Fuel Burnup

Subcommittee E-10:02, Radiation-Induced Changes in Metallic
Materials

Chairman:

Committee C-26, Fuel, Control, and Moderator Materials for
Nuclear Reactor Applications, ASTM

Coordinator:

Analytical Chemistry Division Quality Assurance Program
Analytical Chemistry Division Energy Conservation Program

Division Representative:

ORNL Job Opportunity System
ORNL Labor Relations

COSTANZO, D. A.

Member:

Committee C-26, Fuel, Control, and Moderator Materials for
Nuclear Reactor Applications, and Subcommittee C-26:05,
Methods of Test, ASTM

DYER, F. F.

Lecturer:

Member:

ORAU Traveling Lecture Program

Analytical Chemistry Division Seminar Committee

ELDRIDGE, J. S.

Member:

Subcommittee No. 9 (Environmental Radiological Surveillance:
Mechanisms for Information Exchange), Health Physics
Society

EMERY, J. F.

Consultant:

Tennessee Valley Authority

FELDMAN, C.

Fellow:

Scientific Editor:

Member:

American Society for Testing and Materials

Optics and Spectroscopy (English translation of Russian
journal, *Optika i Spektroskopiya*)

Committee E-2 on Emission Spectroscopy, ASTM; Subcommittees
on Fundamental Methods, Editorial Practices; and
Nomenclature

Atomic Absorption and Emission Spectroscopy Subgroup, Union
Carbide Corporation

GRIEST, W. H.

Consultant:

Member:

National Cancer Institute

DOE Oil Shale Working Group

GUERIN, M. R.

Consultant:

Environmental Protection Agency

Working Meeting on the Master Analytical Scheme, Athens,
GA, January 8, 1981

	Review of Diesel Exhaust Program, RTP, NC, February 24, 1981
	Review of Synfuels Utilization Program, Alexandria, VA, May 12-14, 1981
	Review of Risk Analysis Category Concept, Alexandria, VA, July 21-22, 1981
	Federal Trade Commission
	Deliberations on Low Tar Cigarettes, 1981
	Department of Energy
	Review of Inhalation Toxicology Research Institute OHER Program, Albuquerque, NM, February 25-27, 1981
	Review of Interlaboratory Shale Oil OHER/FE Program, Los Alamos, NM, April 1-2, 1981
	Review of OHER Synfuels Chemistry Program, Seattle, WA, November 4-7, 1981
	National Cancer Institute
	Program Plan for Sidestream Tobacco Smoke, Bethesda, MD, September 30, 1981
	American Cancer Society
	Smoking or Health Program Plan, New York, NY, November 18-20, 1981
Member:	EPRI Source Assessment Subcommittee
Invited Speaker:	Department of Chemistry, Univ. of Illinois, Champaign, IL, April 24, 1981
	Joint US/USSR Shale Oil Program, Tallinn, Estonia/SSR, June 20-30, 1981
 HIGGINS, C. E.	
Coordinator:	Analytical Chemistry Division Affirmative Action Program
 HOLMBERG, R. W.	
Coordinator:	Analytical Chemistry Division Metric Coordinator
 HULETT, L. D.	
Organization and Session Chairman	Symposium on Use of Surface Science Techniques in Solving Analytical problems, 181st National Meeting, ACS, Atlanta, GA, March 29 - April 3, 1981
 JENKINS, R. H.	
Invited Participant:	NIH Core Grant Review, American Health Foundation, Valhalla, NY, October 19-21, 1981
Lecturer:	ORAU Traveling Lecture Program
 KLATT, L. N.	
Associate Editor:	<i>Chemical, Biomedical, and Environmental Instrumentation</i>
Member:	Fellowship Committee of Analytical Chemistry Division of American Chemical Society
	Committee C-26, Fuel, Control and Moderator Materials for Nuclear Reactor Applications, ASTM

LAING, W. R.

Chairman: Subcommittee C26:05, Test Methods, Nuclear Fuel Cycle, ASTM
 UCCND Analytical Committee
 Division Representative: UCCND Environmental Analysis Committee
 Member: ORNL Pregrievance Committee
 ACD Long Range Planning Committee

LYON, W. S.

Member: Committee D-5, Coal and Coke, Subcommittee on Methodology,
 Task Group on Trace Elements, ASTM
 Committee E-10, Nuclear Applications and Measurement of
 Radiation Effects, ASTM
 Subcommittee E-10:01, Fuel Burnup
 Subcommittee E-10:05, Dosimetry
 Organizing Committee, 6th International Conference on Modern
 Trends in Activation Analysis
 Executive Committee, Isotopes and Radiation Division, ANS
 Scientific Committee 25 on Radiation Protection in the Use of
 Small Neutron Generators, NCRP
 Regional Editor: *Journal of Radioanalytical Chemistry*
 Associate Editor: *Radiochemical and Radioanalytical Letters*
 Advisory Board: *The Analyst*
 Chairman: Isotopes and Radiation Division, ANS
 Honors and Awards

MASKARINEC, M. P.

Chairman-Elect: UCCND Gas Chromatography Specialists' Group
 Member: Analytical Chemistry Division Seminar Committee
 ASTM E-19 Committee on Chromatography
 UCCND Liquid Chromatography Specialists' Group
 ASTM D-34 on Waste Disposal
 American Chemical Society
 ORNL Hazardous Waste Task Force

MUELLER, T. R.

Coordinator: ACD Awards Coordinator

RAMSEY, J. M.

Member: Analytical Chemistry Division Seminar Committee

ROGERS, C. W.

Instructor: ORNL In-House Continuing Education Program

ROSS, H. H.

Chairman: ORNL Graduate Student Selection Panel
 Education Committee, ORNL Credit Union
 Member: ORNL Technology Utilization Committee
 Editorial Advisory Board, *Journal of Radioanalytical
 Chemistry*
 Board of Directors, ORNL Credit Union

SCOTT, T. G.
Member: Committee D-19, Water, ASTM, and Subcommittee 4, Methods of Radiochemical Analysis

SHULTS, W. D.
Chairman: Division of Analytical Chemistry, ACS Analytical Specialist Group, UCC
Co-Chairman: ORNL United Way Campaign Committee, 1981
Member: ORNL In-House Continuing Education Committee
Technical Services Committee, UCCND
Editorial Board, *Analytica Chimica Acta*
Advisory Committee, "Analytical Approach," *Analytical Chemistry*
Program Chairman: Analytical Chemistry Program, National ACS Meeting, Atlanta, 1981
Consultant: UCC Career Planning Program

SMITH, D. H.
Coordinator: Analytical Chemistry Division PhD Recruiting

STEWART, J. H., Jr.
Member: ASTM C-26 Nuclear Fuel Cycle Task: Group Leader for X-Ray Fluorescence Methods
Invited Speaker: 1981 Winter Meeting of ASTM C-26 Nuclear Fuel Cycle, Ft. Myers, Florida, January 19, 1981

STOKELY, J. R.
Coordinator: Analytical Chemistry Division MS/BS Recruiting

TODD, P. J.
Member: Analytical Chemistry Division Seminar Committee

TOMKINS, B. A.
Coordinator: Analytical Chemistry Division University Relations

ANALYTICAL CHEMISTRY DIVISION SEMINARS AT ORNL

Speaker	Title	Date
Dessy, R. E. Virginia Poly. Institute Blacksburg, VA	"Laboratory Automation - A Rational Approach"	Apr. 15, 1981
Poole, C. Wayne St. Univ. Detroit, MI	"High Performance Thin Layer Chromatography - An Acceptable Alternative to HPLC"	May 5, 1981
Milne, G. W. A. Natl. Inst. of Health, Bethesda, MD	"The NIH/EPA Chemical Information System"	May 21, 1981

Hagemann, R. Centre D'Etudes De Saclay, Saclay, France	"OKLO Natural Reactor in Gabon"	June 25, 1981
Fleming, R. F. Natl. Bureau of Standards, Washington, DC	"Analytical Chemistry at the NBS Reactor"	July 17, 1981
Conzemius, R. Ames Laboratory, Iowa State Univ. Ames, IA	"Laser Mass Spectrometry of Solids"	Sep. 17, 1981
Blount, H. Univ. of Delaware Newark, DE	"Derivative Optical Methods in Spectro- electrochemistry"	Oct. 19, 1981
VanLoon, J. C. Univ. of Toronto Toronto, Canada	"The Expanding Frontiers of Analytical Atomic Spectrometry-Interfaces with Other Analytical Techniques"	Nov. 10, 1981

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8. PRESENTATION OF RESEARCH RESULTS

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

PUBLICATIONS

Contributions to Books, Proceedings, and Reports

AUTHOR	AUTHOR(S) AND TITLE	PUBLISHED IN
Buchanan, M. V.	Buchanan, M. V.; Ho, C.-h.; Clark, B. R.; Guerin, M. R. "Chemical Characterization of Mutagenic Nitrogen-Containing Polycyclic Aromatic Hydrocarbons in Fossil Fuels"	<i>Chem. Anal. and Biol.</i> <i>Fate: Polynucl. Aromat.</i> <i>Hydrocarbons</i> , M. Cooke and A. J. Dennis, Eds., Battelle Press, Colum- bus, Ohio, 1981, p. 133
	Smith, G. P.;* Buchanan, A. C.;* Dworkin, A.;* Sorlie, M.;* Buchanan, M. V. "Cata- lytic Chemistry and Spectroscopic Studies of SbCl_3 Melts Containing Polycyclic Aromatic Hydrocarbons"	<i>Proc., 3rd Int. Symp. on</i> <i>Molten Salts, Phys.</i> <i>Electrochem. Div.,</i> <i>Electrochem. Soc.,</i> Pennington, N.J., Vol. 81-9, 1981, p. 393
Carter, J. A.	Walton, J. R.; Smith, D. H.; McKown, H. S.; Carter, J. A. "A Quadrupole Mass Spectrom- eter for a Mobile Laboratory to Measure Isotopic Ratios"	<i>29th Annu. Conf. on Mass</i> <i>Spectrom. and Allied</i> <i>Topics</i> , Minneapolis, Minn., May 24-29, 1981, p. 246
	Walker, R. L.; Smith, D. H.; Carter, J. A. "Resin Bead Mass Spectrometry"	<i>Proc., 4th SALE Program</i> <i>Participants Meet.</i> , Argonne, Ill., 1981, p. 208
Caton, J. E.	Griest, W. H.; Caton, J. E. "Studies of the Interaction of Polycyclic Aromatic Hydrocarbons with Fly Ash"	<i>Chem. Anal. and Biol.</i> <i>Fate: Polynucl. Aromat.</i> <i>Hydrocarbons</i> , M. Cooke and A. J. Dennis, Eds., Battelle Press, Colum- bus, Ohio, 1981, p. 719
	Caton, J. E.; Barnes, Z. K.;* Kubota, H.; Griest, W. H.; Maskarinec, M. P. "Compari- son of Several Methods for Collection and Quantification of Polycyclic Aromatic Hydro- carbons from Aqueous Media"	<i>Advances in the Identifi-</i> <i>cation and Anal. of Org.</i> <i>Pollut. in Water Vol. 1</i> , L. H. Keith, Ed., Ann Arbor Sci. Pub., Ann Arbor, Mich., 1981, p. 329

AUTHOR	AUTHOR(S) AND TITLE	PUBLISHED IN
Christie, W. H.	Narayan, J.;* Fletcher, J.;* Appleton, B. R.;* Christie, W. H. "Enhanced Diffusion and Formation of Defects During Thermal Oxidation"	<i>Defects in Semiconductors</i> , ed. by J. Narayan and T. Y. Tam, Elsevier North-Holland, 1981, p. 355
	Loundes, D. H.;* Cleland, J. W.;* Christie, W. H.; Eby, R. E. "Pulsed Ruby Laser Annealing of Zn, Mg, Se and Si Ion Implants in Semiconductivity GaAs"	<i>Laser and Electron Beam Solid Interactions and Mater. Processing</i> , J. F. Gibbons, L. D. Hess, and T. W. Sigmon, Eds., Elsevier North Holland, Vol. 1, New York, N.Y., 1981, p. 223
	Wood, R. F.;* Loundes, D. H.; Christie, W. H. "Pulsed Laser Annealing of Ion-Implanted GaAs: Theory and Experiment"	<i>Laser and Electron Beam Solid Interactions and Mater. Processing</i> , J. F. Gibbons, L. D. Hess, and T. W. Sigmon, Eds., Elsevier North Holland, Vol. 1, New York, N.Y., 1981, p. 231
	Christie, W. H.; Eby, R. E.; Loundes, D. H.;* Young, R. T.;* Wood, R. F.;* White, C. W.* "The Use of SIMS to Measure Dopant Profile Changes in Semiconductor Materials Caused by Pulsed Laser Annealing"	<i>Proc. of Electrochem. Soc. 81-2</i> , Denver, Colo., 1981, p. 883
	Eby, R. E.; Christie, W. H. "The Determination of B and Li in Nuclear Materials by Secondary Ion Mass Spectrometry"	<i>29th Annu. Conf. on Mass Spectrom. and Allied Topics</i> , Minneapolis, Minn., May 24-29, 1981, p. 242
	Narayan, J.;* Fletcher, J.;* White, C. W.;* Eby, R. E.; Christie, W. H. "Melting Phenomena and Laser Annealing in Semiconductors"	<i>Proc., Conf. on Lasers and Electro Optics, IEE</i> , 81CH1655-0, New York, N.Y., 1981, p. 94
	Young, R. T.;* Wood, R. F.;* Jellison, G. E.;* Christie, W. H. "Laser Processing for High Efficiency Solar Cells"	<i>3rd E.C. Photovoltaic Solar Energy Conf.</i> , Reidel, Boston, Mass., 1981, p. 703
	Bittner, H. F.;* Bell, J. T.;* White, C. W.;* Christie, W. H. "Steam Oxidation of Laser-Annealed Incoloy 800"	<i>Proc. of Electrochem. Soc. 80-2</i> , 1980, p. 653

AUTHOR	AUTHOR(S) AND TITLE	PUBLISHED IN
Christie, W. H.	Loundes, D. H.;* Cleland, J. W.;* Fletcher, J.;* Narayan, J.;* Westbrook, R. D.;* Wood, R. F.;* Christie, W. H.; Eby, R. E. "Pulsed Laser Annealing of Ion-Implanted Semi-Conducting GaAs for Heterojunction Solar Cells"	<i>Proc., 15th IEEE Photovoltaic Spec. Conf.</i> , 81CH1644-4, New York, N.Y., 1981, p. 45
	Smith, D. H.; Christie, W. H.; Eby R. E. "A SIMS Study of the Resin Bead as a Thermal Ion Source"	<i>29th Annu. Conf. on Mass Spectrom. and Allied Topics</i> , Minneapolis, Minn., May 24-29, 1981, p. 244
Clark, B. R.	Buchanan, M. V.; Ho, C.-h.; Clark, B. R.; Guerin, M. R. "Chemical Characterization of Mutagenic Nitrogen-Containing Polycyclic Aromatic Hydrocarbons in Fossil Fuels"	<i>Chem. Anal. and Biol. Fate: Polynuc. Aromat. Hydrocarbons</i> , M. Cooke and A. J. Dennis, Eds., Battelle Press, Columbus, Ohio, 1981, p. 133
	Rao, T. K.;* Epler, J. L.;* Guerin, M. R.; Clark, B. R.; Ho, C.-h. "Mutagenicity of Nitrogen Compounds from Synthetic Crude Oils: Collection, Separation, and Biological Testing"	<i>Environ. Sci. Res., Vol. 22</i> , M. D. Waters, S. Sandhu, J. L. Huisinigh, L. Claxton, S. Nesnow, Eds., Plenum Press, 1981, p. 243
Cooper, J. H.	Gilai, D.;* Cooper, J. H.; Laing, W. R.; Raman, S.;* Stelson, P. H.;* Walker, R. L.; Williams, M. L.* "Experimental and Calculational Results of an Irradiation Experiment in EBR-II"	<i>Trans. Am. Nucl. Soc.</i> , 38, 1981, 669
Eby, R. E.	Loundes, D. H.;* Cleland, J. W.;* Christie, W. H.; Eby, R. E. "Pulsed Ruby Laser Annealing of Zn, Mg, Se and Si Ion Implants in Semiconductivity GeAs"	<i>Laser and Electron Beam Solid Interactions and Mater. Processing</i> , J. F. Gibbons, L. D. Hess, and T. W. Sigmon, Eds., Elsevier North Holland, Vol. 1, New York, N.Y., 1981, p. 223
	Christie, W. H.; Eby, R. E.; Loundes, D. H.;* Young, R. T.;* Wood, R. F.;* White, C. W.* "The Use of SIMS to Measure Dopant Profile Changes in Semiconductor Materials Caused by Pulsed Laser Annealing"	<i>Proc. of Electrochem. Soc. Meet. 81-2</i> , Denver, Colo., 1981, p. 883
	Narayan, J.;* Fletcher, J.;* White, C. W.;* Eby, R. E.; Christie, W. H. "Melting Phenomena and Laser Annealing in Semiconductors"	<i>Proc., Conf. on Lasers and Electro Optics, IEE</i> , 81CH1655-0, New York, N.Y., 1981, p. 94

AUTHOR	AUTHOR(S) AND TITLE	PUBLISHED IN
Eby, R. E.	Loundes, D. H.;* Cleland, J. W.;* Fletcher, J.;* Narayan, J.;* Westbrook, R. D.;* Wood, R. F.;* Christie, W. H.; Eby, R. E. "Pulsed Laser Annealing of Ion-Implanted Semi-Conducting GaAs for Homojunction Solar Cells"	<i>Proc., 15th IEEE Photovoltaic Spec. Conf.</i> , 81CH1644-4, New York, N.Y., 1981, p. 45
	Eby, R. E.; Christie, W. H. "The Determination of B and Li in Nuclear Materials by Secondary Ion Mass Spectrometry"	<i>29th Annu. Conf. on Mass Spectrom. and Allied Topics</i> , Minneapolis, Minn., May 24-29, 1981, p. 242
	Smith, D. H.; Christie, W. H.; Eby, R. E. "A SIMS Study of the Resin Bead as a Thermal Ion Source"	<i>29th Annu. Conf. on Mass Spectrom. and Allied Topics</i> , Minneapolis, Minn., May 24-29, 1981, p. 244
Glish, G. L.	Glish, G. L.; Todd, P. J. "SIMS + MS/MS = SIMS/MS"	<i>29th Annu. Conf. on Mass Spectrom. and Allied Topics</i> , Minneapolis, Minn., May 25-29, 1981, p. 675
	Glish, G. L.; McLucky, J. H.;* Cooks, R. G.;* "Description and Applications of a Hybrid MS/MS Instrument of BQQ Design"	<i>29th Annu. Conf. on Mass Spectrom. and Allied Topics</i> , Minneapolis, Minn., May 25-29, 1981, p. 666
Griest, W. H.	Griest, W. H.; Maskarinec, M. P.; Herbes, S. E.;* Southworth, G. R.* "Multicomponent Methods for the Identification and Quantification of Polycyclic Aromatic Hydrocarbons in the Aqueous Environment"	<i>Anal. of Waters Assoc. with Fuel Production</i> , L. P. Jackson and C. C. Wright, Eds., 1981, p. 167
	Griest, W. H.; Caton, J. E. "Studies of the Interaction of Polycyclic Aromatic Hydrocarbons with Fly Ash"	<i>Chem. Anal. and Biol. Fate: Polynuc. Aromat. Hydrocarbons</i> , M. Cooke and A. J. Dennis, Eds., Battelle Press, Columbus, Ohio, 1981, p. 719
	Caton, J. E.; Barnes, Z. K.;* Kubota, H.; Griest, W. H.; Maskarinec, M. P. "Comparison of Several Methods for Collection and Quantification of Polycyclic Aromatic Hydrocarbons from Aqueous Media"	<i>Advances in the Identification and Anal. of Org. Pollut. in Water Vol. 1</i> , L. H. Keith, Ed., Ann Arbor Sci. Pub., Ann Arbor, Mich., 1981, p. 329
Guerin, M. R.	Buchanan, M. V.; Ho, C.-h.; Clark, B. R.; Guerin, M. R. "Chemical Characterization of Mutagenic Nitrogen-Containing Polycyclic Aromatic Hydrocarbons in Fossil Fuels"	<i>Chem. Anal. and Biol. Fate: Polynuc. Aromat. Hydrocarbons</i> , M. Cooke and A. J. Dennis, Eds., Battelle Press, Columbus, Ohio, 1981, p. 133

AUTHOR	AUTHOR(S) AND TITLE	PUBLISHED IN
Guerin, M. R.	Dalbey, W.;* Lock, S.;* Holmberg, R. W.; Moneyhun, J. H.; Guerin, M. R. "Acute Exposure of Rats to an Inhaled Aerosol of Diesel Fuel"	<i>Proc., Smoke/Obscurants Symp. V</i> , Harry Diamond Labs., Adelphi, Md., Apr. 28-30, 1981, p. 759
	Rao, T. K.;* Epler, J. L.;* Guerin, M. R.; Clark, B. R.; Ho, C.-h. "Mutagenicity of Nitrogen Compounds from Synthetic Crude Oils: Collection, Separation, and Biolog- ical Testing"	<i>Environ. Sci. Res., Vol. 22</i> , M. D. Waters, S. Sandhu, J. L. Huisingh, L. Claxton, S. Nesnow, Eds., Plenum Press, 1981, p. 243
	Guerin, M. R.; Ho, C.-h.; Rao, T. K.;* Epler, J. L.* "Chemistry and Mutagenicity of Shale Oil"	<i>Proc., 2nd Joint US-USSR Workshop on the Health and Environ. Effects of Oil Shale Develop.</i> , Tallinn, USSR, NTIS, PC A02/MF A01, 12 p.
Ho, C.-h.	Buchanan, M. V.; Ho, C.-h.; Clark, B. R.; Guerin, M. R. "Chemical Characterization of Mutagenic Nitrogen-Containing Polycyclic Aromatic Hydrocarbons in Fossil Fuels"	<i>Chem. Anal. and Biol. Fate: Polynucl. Aromat. Hydrocarbons</i> , M. Cooke, and A. J. Dennis, Eds., Battelle Press, Colum- bus, Ohio, 1981, p. 133
	Rao, T. K.;* Epler, J. L.;* Guerin, M. R.; Clark, B. R.; Ho, C.-h. "Mutagenicity of Nitrogen Compounds from Synthetic Crude Oils: Collection, Separation, and Biolog- ical Testing"	<i>Environ. Sci. Res., Vol. 22</i> , M. D. Waters, S. Sandhu, J. L. Huisingh, L. Claxton, S. Nesnow, Eds., Plenum Press, 1981, p. 243
	Guerin, M. R.; Ho, C.-h.; Rao, T. K.;* Epler, J. L.* "Chemistry and Mutagenicity of Shale Oil"	<i>Proc., 2nd Joint US-USSR Workshop on the Health and Environ. Effects of Oil Shale Develop.</i> , Tallinn, USSR, NTIS, PC A02/MF A01, 12 p.
Holmberg, R. W.	Dalbey, W.;* Lock, S.;* Holmberg, R. W.; Moneyhun, J. H.; Guerin, M. R. "Acute Exposure of Rats to an Inhaled Aerosol of Diesel Fuel"	<i>Proc., Smoke/Obscurants Symp. V</i> , Harry Diamond Labs., Adelphi, Md., Apr. 28-30, 1981, p. 759
	Keller, J. M.; Holmberg, R. W.; Jones, C. M.* "The Problem of Sulfur Hexafluoride Impur- ities in the Operation of Large Electrostatic Accelerators"	<i>Proc. of EPRI Workshop on Arc By-Products in Gas- Insulated Equip.</i> , June 1980, Chicago, Ill., EPRI EL-80-8-LD, Dec. 1980, p. 15

AUTHOR	AUTHOR(S) AND TITLE	PUBLISHED IN
Holmberg, R. W.	Holmberg, R. W.; Moneyhun, J. H.; Dalbey, W. E.* "An Exposure System for Toxicological Studies of Concentrated Oil Aerosols"	<i>Proc., Symp. on Inhalation Toxic. and Techn.</i> , Oct. 23-24, 1980, Kalamazoo, Mich., 1981, p. 53
	Jenkins, R. A.; Holmberg, R. W.; Moneyhun, J. H.; Wike, J. S. "Chemical and Physical Characteri- zation of a Diesel Fuel Obscurant for Inhala- tion Toxicology Studies"	<i>Proc., Smoke/Obscurants Symp. V</i> , Harry Diamond Labs., Adelphi, Md., Apr. 28-30, 1981, p. 739
Jenkins, R. A.	Jenkins, R. A.; Holmberg, R. W.; Moneyhun, J. H.; Wike, J. S. "Chemical and Physical Characteri- zation of a Diesel Fuel Obscurant for Inhala- tion Toxicology Studies"	<i>Proc., Smoke/Obscurants Symp. V</i> , Harry Diamond Labs., Adelphi, Md., Apr. 28-30, 1981, p. 739
Keller, J. M.	Keller, J. M.; Holmberg, R. W.; Jones, C. M.* "The Problem of Sulfur Hexafluoride Impur- ities in the Operation of Large Electrostatic Accelerators"	<i>Proc. of EPRI Workshop on Arc By-Products in Gas- Insulated Equip.</i> , June 1980, Chicago, Ill., EPRI EL-80-8-LD, Dec. 1980, p. 15
Kubota, H.	Caton, J. E.; Barnes, Z. K.;* Kubota, H.; Griest, W. H.; Maskarinec, M. P. "Compari- son of Several Methods for Collection and Quantification of Polycyclic Aromatic Hydro- carbons from Aqueous Media"	<i>Advances in the Identifi- cation and Anal. of Org. Pollut. in Water Vol. 1</i> , L. H. Keith, Ed., Ann Arbor Sci. Pub., Ann Arbor, Mich., 1981, p. 329
Laing, W. R.	Gilai, D.;* Cooper, J. H.; Laing, W. R.; Raman, S.;* Stelson, P. H.;* Walker, R. L.; Williams, M. L.* "Experimental and Calculational Results of an Irradia- tion Experiment in EBR-II"	<i>Trans. Am. Nucl. Soc.</i> , 38, 1981, 669
Layton, F. L.	Lackey, W. J.;* Angelini, P.;* Layton, F. L.; Stinton, D. P.;* Vavruska, J. S.* "Sol-Gel Technology Applied to Glass and Crystalline Ceramics"	<i>Waste Manage. '80, The State of Waste Disposal Techn., Mill Tailings, and Risk Anal.</i> , Vol. 2, R. G. Post, Ed., Univ. of Arizona, Tuscon, Ariz., 1980, p. 391
Lyon, W. S.	Lyon, W. S. "Nuclear Methods in Coal Research"	<i>4th Int. Conf. on Nucl. Methods in Environ. and Energy Res.</i> , Columbia, Mo., Apr. 14-17, 1980, p. 587

AUTHOR	AUTHOR(S) AND TITLE	PUBLISHED IN
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	Arnold, S.;* Whitten, W. B. "Temperature Dependence of the Triplet Exciton Yield in Fission and Fusion in Tetracene"	<i>J. Chem. Phys.</i> , 75, 1166, 1981
Young, J. P.	Ensor, D. D.;* Peterson, J. R.;* Haire, R. G.;* Young, J. P. "Absorption Spectrophotometric Study of Berkelium (III) and (IV) Fluorides in the Solid State"	<i>J. Inorg. Nucl. Chem.</i> , 43, 1000, 1981
	Ensor, D. D.;* Peterson, J. R.;* Haire, R. G.;* Young, J. P. "Absorption Spectrophotometric Study of $^{253}\text{EsF}_3$ and Its Decay Products in the Bulk-Phase Solid State"	<i>J. Inorg. Nucl. Chem.</i> , 43, 2425, 1981
	Young, J. P.; Haire, R. G.;* Peterson, J. R.;* Ensor, D. D.;* Fellows, R. L.* "Chemical Consequences of Radioactive Decay. II. Spectrophotometric Study of the Ingrowth of Bk-249 and Cf-249 into Halides of Es-253"	<i>Inorg. Chem.</i> , 20, 3979, 1981

Norvell, V. E.

Norvell, V. E. "Spectroelectrochemical Investigations in Molten Salts and Aqueous Media"

Dissertation, Ph.D.,
Univ. of Tennessee,
Knoxville, Tenn.

ROSS, H. H.

Ross, H. H.; Strain, J. E. "Continuous-Flow Free Acid Monitoring Method and System"

U.S. Patent No. 4,272,247
issued June 9, 1981

Reports

AUTHOR	AUTHOR(S) AND TITLE	REPORT NO. AND DATE
Carter, J. A.	Carter, J. A. <i>Analytical Chemistry Division, Research and Development Summary for January 1981: Mass and Emission Spectrometry Section</i>	ORNL/CF-81/17 (Feb. 1981)
	Smith, D. H.; Walker, R. L.; Carter, J. A. <i>Progress Report for the Office of Safeguards and Security for FY 1980</i>	ORNL/TM-7673 (Jan. 1981)
	McKown, H. S.; Walker, R. L.; Smith, D. H. Carter, J. A. I. <i>Thermal Emission Resin Bead Mass Spectrometric Two-Filament Arrangement Evaluation. - A.82</i> II. <i>The Rapid Bulk Resin Bead Method of Sample Preparation for Mass Analysis of Plutonium and Uranium in Spent Reactor Fuel - A.56</i>	ORNL/TM-7834 (ISPO-152) (May 1981)
	Carter, J. A. <i>Analytical Chemistry Division Research and Development Summary for May 1981: Mass and Emission Spectrometry Section</i>	ORNL/CF-81/175 (June 1981)
	Carter, J. A. <i>Analytical Chemistry Division, Research and Development Summary: Mass and Emission Spectrometry Section</i>	ORNL/CF-81/279 (Oct. 1981)
	Smith, D. H.; Walton, J. R.; McKown, H. S.; Walker, R. L.; Carter, J. A. <i>Progress Report for the Office of Safeguards and Security for FY 1981</i>	ORNL/TM-8097 (Nov. 1981)
	Walton, J. R.; Smith, D. H.; McKown, H. S.; Walker, R. L.; Carter, J. A. <i>Acquisition and Performance Evaluation of a Quadrupole Mass Spectrometer in a Mobile Laboratory</i>	ORNL/TM-8027 (Dec. 1981)
	Warmack, R. J.; Landau, L.; Christie, W. H.; Carter, J. A. <i>A Spark-Source Mass Spectrometric Facility to Analyze Highly Radioactive Samples</i>	ORNL/TM-8019 (Dec. 1981)
Christie, W. H.	Warmack, R. J.; Landau, L.; Christie, W. H.; Carter, J. A. <i>A Spark-Source Mass Spectrometric Facility to Analyze Highly Radioactive Samples</i>	ORNL/TM-8019 (Dec. 1981)
Corbin, L. T.	Corbin, L. T. <i>ACD Monthly Report for March 1981: Analytical Technical Support Section</i>	ORNL/CF-81/73 (Apr. 1981)
	Corbin, L. T. <i>ACD Monthly Report for July 1981: Analytical Technical Support Section</i>	ORNL/CF-81/219 (Aug. 1981)
	Corbin, L. T. <i>Quality Assurance Report of the Analytical Chemistry Division, October 1980 through September 1981</i>	ORNL/CF-81/275 (Oct. 1981)

AUTHOR	AUTHOR(S) AND TITLE	REPORT NO. AND DATE
Corbin, L. T.	Corbin, L. T. <i>ACD Monthly Report for November 1981: Analytical Technical Support Section</i>	ORNL/CF-81/315 (Dec. 1981)
Eldridge, J. S.	Oakes, T. W.;* Montford, M. A.;* Shank, K. E.;* Wagner, E. B.;* Scott, T. G.; Eldridge, J. S. <i>Methods and Procedures Utilized in Environmental Environmental Management Activities at Oak Ridge National Laboratory</i>	ORNL/TM-7212 (Mar. 1981)
Goeringer, D. E.	Goeringer, D. E.; Klatt, L. N. <i>A Microcomputer Controlled Remote Pipetter: Design and Evaluation of a Prototype System</i>	ORNL/TM-7608 (Apr. 1981)
Guerin, M. R.	Guerin, M. R. <i>Analytical Chemistry Division Research and Development Summary: February 1981, Bio/Organic Analysis Section</i>	ORNL/CF-81/48 (Mar. 1981)
	Guerin, M. R. <i>Analytical Chemistry Division Research and Development Summary: June 1981, Bio/Organic Analysis Section</i>	ORNL/CF-81/199 (July 1981)
	Guerin, M. R. <i>Analytical Chemistry Division Research and Development Summary: September 1981, Bio/Organic Analysis Section</i>	ORNL/CF-81/297 (Nov. 1981)
Keller, J. M.	Keller, J. M.; Rickard, R. R. <i>The Determination of Dibutylphosphoric Acid in Carbonate, Oxalate, or Nitrate Solutions by Ion Chromatography</i>	ORNL/TM-7569 (Jan. 1981)
	Keller, J. M. <i>Modification of a Dionex Ion Chromatograph for Higher Sensitivity, Increased Sample Throughput, and Glove-box Operation</i>	ORNL/CFRP-81/2 (Feb. 1981)
	Keller, J. M. <i>Determination of Spark-Induced By-Products of Sulfur Hexafluoride in High-Voltage Accelerators</i>	ORNL/CF-81/44 (Feb. 1981)
	Keller, J. M.; Corder, G. E.;* Mach, G. R.* <i>Baseline-Drift Compensator for a Perkin-Elmer DSC-1B Differential Scanning Colorimeter</i>	ORNL/CFRP-81/5 (Apr. 1981)
Klatt, L. N.	Goeringer, D. E.; Klatt, L. N. <i>A Microcomputer Controlled Remote Pipetter: Design and Evaluation of a Prototype System</i>	ORNL/TM-7608 (Apr. 1981)
Laing, W. R.	Laing, W. R.; Rickard, R. R. <i>Analysis of ORNL Process Water from 1960 to Present</i>	ORNL/CF-81/74 (Apr. 1981)
Landau, L.	Warmack, R. J.; Landau, L.; Christie, W. H.; Carter, J. A. <i>A Spark-Source Mass Spectrometric Facility to Analyze Highly Radioactive Samples</i>	ORNL/TM-8019 (Dec. 1981)

AUTHOR	AUTHOR(S) AND TITLE	REPORT NO. AND DATE
Lannom, K. C.	Marley, J. L.; Lannom, K. C. <i>The Decomposition of Hydrogen Peroxide in a Nitric Acid System: Spectrophotometric Determination of Hydrogen Peroxide</i>	ORNL/CFRP-81/10 (June 1981)
Lee, D. A.	Knipschild, K. E.;* Lee, D. A. <i>Thermal Analysis of SYNROC Gel Microspheres</i>	ORNL/TM-7930 (Nov. 1981)
Lyon, W. S.	Lyon, W. S. <i>Analytical Chemistry Division Research and Development Summary for April 1981: Analytical Methodology Section</i>	ORNL/CF-81/79 (Apr. 1981)
	Lyon, W. S. <i>Analytical Chemistry Division Research and Development Summary for August 1981: Analytical Methodology Section</i>	ORNL/CF-81/253 (Aug. 1981)
	Lyon, W. S. <i>Analytical Chemistry Division Research and Development Summary for December 1981: Analytical Methodology Section</i>	ORNL/CF-81/336 (Dec. 1981)
Marley, J. L.	Marley, J. L.; Lannom, K. C. <i>The Decomposition of Hydrogen Peroxide in a Nitric Acid System: Spectrophotometric Determination of Hydrogen Peroxide</i>	ORNL/CFRP-81/10 (June 1981)
Maskariniec, M. P.	Brown, D. K.;* Francis, C. W.;* Maskariniec, M. P.; Larimer, F. W.* <i>Comparison of EP Extracts and Landfill Leachates</i>	ORNL/TM-7563 (June 1981)
McKown, H. S.	Walton, J. R.; Smith, D. H.; McKown, H. S.; Walker, R. L.; Carter, J. A. <i>Acquisition and Performance Evaluation of a Quadrupole Mass Spectrometer in a Mobile Laboratory</i>	ORNL/TM-8027 (Dec. 1981)
	McKown, H. S.; Walker, R. L.; Smith, D. H. Carter, J. A. I. <i>Thermal Emission Resin Bead Mass Spectrometric Two-Filament Arrangement Evaluation - A.82</i> II. <i>The Rapid Bulk Resin Bead Method of Sample Preparation for Mass Analysis of Plutonium and Uranium in Spent Reactor Fuel - A.56</i>	ORNL/TM-7834 (ISPO-152) (May 1981)
	Smith, D. H.; Walton, J. R.; McKown, H. S.; Walker, R. L.; Carter, J. A. <i>Progress Report for the Office of Safeguards and Security for FY 1981</i>	ORNL/TM-8097 (Nov. 1981)
Mueller, T. R.	Mueller, T. R. <i>Precision Triangular Waveform Generator Model 79-11965</i>	ORNL/TM-7311 (May 1981)
Reynolds, S.	Drury, J. S.;* Reynolds, S.; Owen, P. T.;* Ross, R. H.;* Ensminger, J. T.* <i>Uranium in U.S. Surface, Ground, and Domestic Waters, Volume 1</i>	EPA-570/9-81-001 ORNL/EIS-192/VI (Apr. 1981)

AUTHOR	AUTHOR(S) AND TITLE	REPORT NO. AND DATE
Rickard, R. R.	Keller, J. M.; Rickard, R. R. <i>The Determination of Dibutylphosphoric Acid in Carbonate, Oxalate, or Nitrate Solutions by Ion Chromatography</i>	ORNL/TM-7569 (Jan. 1981)
	Laing, W. R.; Rickard, R. R. <i>Analysis of ORNL Process Water from 1960 to Present</i>	ORNL/CF-81/74 (Apr. 1981)
Scott, T. G.	Oakes, T. W.;* Montford, M. A.;* Shank, K. E.;* Wagner, E. B.;* Scott, T. G.; Eldridge, J. S. <i>Methods and Procedures Utilized in Environmental Management Activities at Oak Ridge National Laboratory</i>	ORNL/TM-7212 (Mar. 1981)
Shults, W. D.	Shults, W. D. <i>Analytical Chemistry Division Annual Progress Report for Period Ending December 31, 1980</i>	ORNL-5738 (May 1981)
Smith, D. H.	Smith, D. H.; Walker, R. L.; Carter, J. A. <i>Progress Report for the Office of Safeguards and Security for FY 1981</i>	ORNL/TM-7673 (Jan. 1981)
	Smith, D. H.; Walton, J. R.; McKown, H. S.; Walker, R. L.; Carter, J. A. <i>Progress Report for the Office of Safeguards and Security for FY 1981</i>	ORNL/TM-8097 (Nov. 1981)
	Walton, J. R.; Smith, D. H.; McKown, H. S.; Walker, R. L.; Carter, J. A. <i>Acquisition and Performance Evaluation of a Quadrupole Mass Spectrometer in a Mobile Laboratory</i>	ORNL/TM-8027 (Dec. 1981)
	McKown, H. S.; Walker, R. L.; Smith, D. H. Carter, J. A. I. <i>Thermal Emission Resin Bead Mass Spectrometric Two-Filament Arrangement Evaluation - A.82</i> II. <i>The Rapid Bulk Resin Bead Method of Sample Preparation for Mass Analysis of Plutonium and Uranium in Spent Reactor Fuel - A.56</i>	ORNL/TM-7834 (ISPO-152) (May 1981)
Stewart, J. H.	Stewart, J. H.; Sparks, C. J.* <i>Design Proposal for an X-Ray Fluorescence Analyser System for Determining Uranium and Plutonium in Gamma-Active Dissolver Solutions</i>	ORNL/CFRP-81/15 (July 1981)
Walker, R. L.	Smith, D. H.; Walker, R. L.; Carter, J. A. <i>Progress Report for the Office of Safeguards and Security for FY 1980</i>	ORNL/TM-7673 (Jan. 1981)
	Walton, J. R.; Smith, D. H.; McKown, H. S.; Walker, R. L.; Carter, J. A. <i>Acquisition and Performance Evaluation of a Quadrupole Mass Spectrometer in a Mobile Laboratory</i>	ORNL/TM-8027 (Dec. 1981)

AUTHOR	AUTHOR(S) AND TITLE	REPORT NO. AND DATE
Walker, R. L.	McKown, H. S.; Walker, R. L.; Smith, D. H. Carter, J. A. I. <i>Thermal Emission Resin Bead Mass Spectrometric Two-Filament Arrange- ment Evaluation - A.82 II. The Rapid Bulk Resin Bead Method of Sample Preparation for Mass Analysis of Plutonium and Uranium in Spent Reactor Fuel - A.56</i>	ORNL/TM-7834 (ISPO-152) (May 1981)
	Smith, D. H.; Walton, J. R.; McKown, H. S.; Walker, R. L.; Carter, J. A. <i>Progress Report for the Office of Safeguards and Security for FY 1981</i>	ORNL/TM-8097 (Nov. 1981)
Walton, J. R.	Walton, J. R.; Smith, D. H.; McKown, H. S.; Walker, R. L.; Carter, J. A. <i>Acquisition and Performance Evaluation of a Quadrupole Mass Spectrometer in a Mobile Laboratory</i>	ORNL/TM-8027 (Dec. 1981)
	Smith, D. H.; Walton, J. R.; McKown, H. S.; Walker, R. L.; Carter, J. A. <i>Progress Report for the Office of Safeguards and Security for FY 1981</i>	ORNL/TM-8097 (Nov. 1981)
Warmack, R. J.	Warmack, R. J.; Landau, L.; Christie, W. H.; Carter, J. A. <i>A Spark-Source Mass Spectro- metric Facility to Analyze Highly Radioactive Samples</i>	ORNL/TM-8019 (Dec. 1981)
Wilson, G. R.	Wilson, G. R. <i>The Art of Column Cleaning Mercury</i>	ORNL/CF-81/295 (Oct. 1981)

ORAL PRESENTATIONS

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

At Meetings of Professional Societies, Conferences, and the Like

SPEAKER	AUTHOR(S) AND TITLE	PRESENTED AT
Barkenbus, B. D.	Barkenbus, B. D.; MacDougall, C. S.; Tomkins, B. A.; Griest, W. H.; Caton, J. E. "Methodology for the Extraction and Analysis of Air Particulate Organics"	FACSS VIII Symp., Recent Advances in Environ. Anal. Chem. of Priority Pollut., Philadelphia, Pa., Sept. 20-25, 1981
Bate, L. C.	Bate, L. C. "Iodine-129 Separation and Determination by Neutron Activation Analysis"	6th Int. Conf., Modern Trends in Activation Anal., Univ. of Toronto, Toronto, Canada, June 15-19, 1981
Bostick, D. T.	Bostick, D. T.; McCue, D. D.;* Strain, J. E.; Bauer, M. L.;* Dixon, D. M.* "An In-Line Multiwavelength Photometer for the Determination of Heavy Metal Concentrations"	25th Conf. on Anal. Chem. in Energy Techn., Gatlinburg, Tenn., Oct. 6-8, 1981
Brazell, R. S.	Brazell, R. S.; Maskarinec, M. P. "Dynamic Headspace Analysis of Solid Wastes"	Expochem 81, Houston, Tex., Sept. 1-3, 1981
Brooksbank, R. D.	Brooksbank, R. D.; Stewart, J. H. "Analysis of Aqueous and Organic Fission Product Solutions by Energy Dispersive X-Ray Fluorescence"	25th Conf. on Anal. Chem. in Energy Techn., Gatlinburg, Tenn., Oct. 6-8, 1981
	Brooksbank, R. D.; Stewart, J. H. "The Determination of Ultratrace Concentration of Uranium and Thorium in Natural Waters by X-Ray Fluorescence"	5th Symp. on X- and Gamma-Ray Sources and Appl., Ann Arbor, Mich., June 12, 1981
Buchanan, M. V.	Buchanan, M. V. "Gas Chromatography/Mass Spectrometry as an Analytical Tool"	Invited, Univ. of Mexico, Mexico City, Mexico, Jan. 11-17, 1981
	Buchanan, M. V. "Derivatization and Chemical Ionization Techniques in Mass Spectrometry"	Invited, Univ. of Mexico, Mexico City, Mexico, Jan. 11-17, 1981
	Buchanan, M. V. "Characterization of Mutagenic Nitrogen-Containing Compounds in Fossil Fuels Using Ammonia Chemical Ionization Mass Spectrometry"	Invited, Univ. of Mexico, Mexico City, Mexico, Jan. 11-17, 1981

SPEAKER	AUTHOR(S) AND TITLE	PRESENTED AT
Buchanan, M. V.	Buchanan, M. V.; Ho, C.-h.; Clark, B. R.; Guerin, M. R. "Spectroscopic Characteriza- tion of Neutral Aromatic Polar Fractions of Synthetic Fuels"	Anal. Chem. Soc. Symp., Spectrosc. Charac. of Complex Organic Samples, New York, N.Y., Aug. 1981
	Buchanan, M. V.; Guerin, M. R.; Rubin, I. B.; Caton, J. E. "Comparative Spectroscopic Characterization of Synthetic Fuels,"	Workshop on Chem. Charac. of Hazardous Substances in Synf., Battelle Seattle Conf. Center, Seattle, Wash., Nov. 4- 8, 1981
Carter, J. A.	Carter, J. A.; Walker, R. L. "Multielement Isotope Dilution Measurements by Mass Spec- trometric Techniques"	Invited, 33rd South- eastern Reg. Meet., Am. Chem. Soc., Lexington, Ky., Nov. 4-6, 1981
Caton, J. E.	Caton, J. E.; Harmon, S. H.; Griest, W. H.; Guerin, M. R. "Diffuse Reflectance FTIR Spectra of Fossil Fuel Derived Liquids and the Correlation of Spectral Features with Observed Bioactivity"	FACSS-VII Symp., Recent Advances in Environ. Anal. Chem. of Priority Pollut., Philadelphia, Pa., Sept. 20-25, 1981
	Caton, J. E.; Griest, W. H. "Sorption and Release of Organic Molecules by Fly Ash During and After Heat Treatment"	FACSS-VII Symp., Recent Advances in Environ. Anal. Chem. of Priority Pollut., Philadelphia, Pa., Sept. 20-25, 1981
Christie, W. H.	Christie, W. H.; Eby, R. E.; Warmack, R. J.; Landau, L. "Secondary Ion Mass Spectrometry: Some Applications in the Analysis of Nuclear Materials"	25th Conf. on Anal. Chem. in Energy Techn., Gat- linburg, Tenn., Oct. 6- 8, 1981
	Christie, W. H.; Eby, R. E.; Lowndes, D. H.* Young, R. T.* Wood, R. F.* White, C. W.* "The Use of SIMS to Measure Dopant Profile Changes in Semiconductor Materials Caused by Pulsed Laser Annealing"	Invited, The Electrochem. Soc. Fall Meet., Denver, Colo., Oct. 11-16, 1981
	Christie, W. H. "Quantitative Aspects of SIMS as a Depth Profiling Technique: Some Appli- cations in Energy Research Programs"	2nd Annu. Symp. and Equip. Exhibit - Am. Vacuum Soc., Knoxville, Tenn., Oct. 20-22, 1981
	Christie, W. H. "Secondary Ion Mass Spectrom- etry: Empirical Quantification Techniques and Their Application"	181st Am. Chem. Soc. Nat. Meet., Atlanta, Ga., Mar. 29-Apr. 3, 1981
Clark, B. R.	Clark, B. R. "DOE Briefing - An Overview of Coal Conversion"	Invited, Two Year College Chem. Conf., Atlanta, Ga., Mar. 27, 1981

SPEAKER	AUTHOR(S) AND TITLE	PRESENTED AT
Costanzo, D. A.	Costanzo, D. A. "CFRP Analytical Chemistry Development"	CFRP Mid-Year Techn. Rev. Meet., Oak Ridge Nat. Lab., Oak Ridge, Tenn., Mar. 12, 1981
	Costanzo, D. A. "CFRP Analytical Chemistry Development"	Semiannu. Techn. Rev. of CFRP Process and Eng. R&D Act., Oak Ridge Nat. Lab., Oak Ridge, Tenn., Aug. 19, 1981
Cowser, K. E.*	Cowser, K. E.*; Epler, J. L.*; Gehrs, C. W.*; Guerin, M. R.; Klein, J. A.* "Health and Environmental Studies of H-Coal Process"	EPA Symp. on Environ. Aspects of Fuel Conversion Techn. VI, Denver, Colo., Oct. 26-30, 1981
Dalbey, W. E.*	Dalbey, W. E.*; Lock, S.*; Holmberg, R. W.; Moneyhun, J. H.; Guerin, M. R. "Acute Toxicity of an Aerosol of Diesel Fuel"	Soc. of Toxicol. Annu. Conf., San Diego, Calif., Mar. 1-5, 1981
	Dalbey, W. E.*; Lock, S.*; Holmberg, R. W.; Moneyhun, J. H.; Guerin, M. R. "Acute Exposure of Rats to an Inhaled Aerosol of Diesel Fuel"	Smoke/Obscurants Symp. V, Harry Diamond Labs., Adelphi, Md., Apr. 28-30, 1981
	Dalbey, W. E.*; Lock, S.*; Garfinkel, S.*; Jenkins, R. A. "Effects of Repeated Exposures to Aerosolized Diesel Fuel"	Soc. of Toxicol. Annu. Conf., San Diego, Calif., Mar. 1-5, 1981
Dale, J. M.	Dale, J. M.; Hulett, L. D.; Pendyala, S.* "Low Energy Positron Spectroscopy of Solid Surfaces"	Anal. Chem. Soc. Spring Meet., Atlanta, Ga., Mar. 30, 1981
	Dale, J. M.; Hulett, L. D.; Pendyala, S.* "Slow Positrons from a Tungsten Moderator"	Positron Workshop, Brookhaven Nat. Lab., Brookhaven, Ill., Sept. 13-15, 1981
Dyer, F. F.	Dyer, F. F.; Emery, J. F.; Northcutt, K. J.; Scott, R. M.* "Determination of Uranium and Thorium in Computer Memory Materials by High Fluence Neutron Activation Analysis"	6th Int. Conf., Modern Trends in Activation Anal., Univ. of Toronto, Toronto, Canada, June 15-19, 1981
Eatherly, W. P.*	Eatherly, W. P.*; Lee, D. A. "Corrosion-Induced Micro-Structural Changes in U.S. Core Graphite"	Colloq. on the Transport of Fusion Products in Matrix and Graphite, Berlin, FRG, Nov. 9-11, 1981
Eby, R. E.	Christie, W. H.; Eby, R. E. "The Determination of B and Li in Nuclear Materials by Secondary Ion Mass Spectrometry"	29th Anu. Conf. on Mass Spectrom. and Allied Topics, Minneapolis, Minn., May 24-29, 1981

SPEAKER	AUTHOR(S) AND TITLE	PRESENTED AT
Eldridge, J. S.	Eldridge, J. S.; Oakes, T. W.* "Insects as Bio-Indicators for Radionuclides"	Health Phys. Soc. Annu. Meet., Louisville, Ky., June 21-25, 1981
Epler, J. L.*	Epler, J. L.*; Larimer, F. W.*; Maskarinec, M. P.; Francis, C. W.* "Ecological and Health Effects Bioassay Results from Coal Conversion Solid Wastes"	EPA Symp. on Environ. Aspects of Fuel Conversion Techn. VI, Denver, Colo., Oct. 26-30, 1981
Gilai, D.*	Gilai, D.*; Cooper, J. H.; Laing, W. R.; Raman, S.*; Stelson, P. H.*; Walker, R. L.; Williams, M. L.* "Experimental and Calculational Results of an Irradiation Experiment in EBR-II"	Trans. Am. Nucl. Soc., 38, 669, 1981
Gleason, G. I.	Gleason, G. I. "Advanced Gamma-Ray Spectroscopy Using Ge(Li) Detectors"	ORAU Training Div. for Annu. Ten-Week Health Phys. Course spons. by NRC, Oak Ridge, Tenn., Apr. 1, 1981
	Gleason, G. I. "Gamma-Ray Spectroscopy I and II"	ORAU Training Div. for Annu. Five-Week Health Phys. Course spons. by NRC, Oak Ridge, Tenn., Oct. 12-14, 1981
Glish, G. L.	Glish, G. L.; Todd, P. J. "SIMS + MS/MS = SIMS/MS"	29th Annu. Conf. on Mass Spectrom. and Allied Topics, Minneapolis, Minn., May 24-29, 1981
	Glish, G. L. "Desorption Ionization Methods for Mass Spectrometry"	UCCND Anal. Spec. Meet., Oak Ridge Nat. Lab., Oak Ridge, Tenn., Oct. 20-21, 1981
Goeringer, D. E.	Goeringer, D. E.; Klatt, L. N. "Design and Evaluation of a Microcomputer-Controlled Remote Pipetter System"	25th Conf. on Anal. Chem. in Energy Techn., Gatlinburg, Tenn., Oct. 6-8, 1981
Griest, W. H.	Griest, W. H.; Higgins, C. E.; Barkenbus, B. D.; Caton, J. E.; MacDougall, C. S. "Quantitative Aspects of Vapor and Particulate Phase Organic Analysis"	EPA Nat. Symp. on Monitoring Hazardous Organic Pollut. in Air, Raleigh, N.C., Apr. 28-May 1, 1981
	Griest, W. H.; Guerin, M. R.; Yeatts, L. B. "Repository Support of Synthetic Health Effects Research"	EPA Workshop on Combustion Products of Synf., Alexandria, Va., May 13-14, 1981
	Griest, W. H. "Issues and Requirements in Atmospheric PAH Emission Control Technology"	OECD Workshop on PAH, Paris, France, Oct. 19-21, 1981

SPEAKER	AUTHOR(S) AND TITLE	PRESENTED AT
Guerin, M. R.	Guerin, M. R. "Integrated Biological and Chemical Analysis"	Invited, Chem. Dept., Univ. of Illinois, Champaign, Ill., Apr. 24, 1981
	Guerin, M. R.; Ho, C.-h.; Rao, T. K.* Epler, J. L.* "Chemistry and Mutagenicity of Shale Oil"	Invited, 2nd Joint US-USSR Workshop on the Health and Environ. Effects of Oil Shale Devel., Tallinn, Estonian SSR, June 17-30, 1981
Haire, R. G.*	Haire, R. G.*; Young, J. P.; Peterson, J. R.* "The Effect of Structural Environment on the Absorption Spectra of Selected Lanthanide Sesquioxides"	15th Rare Earth Res. Conf., Rolla, Mo., June 15-18, 1981
Heine, D. R.	Heine, D. R. "Inductively Coupled Plasma Instrumentation and Applications"	UCCND Anal. Com. Meet., Oak Ridge Nat. Lab., Oak Ridge, Tenn., June 9, 1981
Henry, C. J.*	Henry, C. J.*; Lopez, A.*; Dansie, D. R.*; Avery, M. D.*; Whitmire, C. E.*; Caton, J. E.; Stokely, J. R.; Guerin, M. R.; Curren, R. D.*; Kouri, R. E.* "Distribution and Clearance of Three Cigarette Smoke Constituents, Dehydro- epiandrosterone (DHEA), Nicotine, (NIC), and Benzo(a)pyrene (BP) After Exposure of Mice to Whole Cigarette Smoke"	Soc. of Toxicol. Annu. Conf., San Diego, Calif., Mar. 1-5, 1981
Hieftje, G. M.*	Hieftje, G. M.*; Haugen, G. R.*; Russo, R. E.*; Ramsey, J. M.; Lilley, T.* Use of Linear Response Theory in High-Speed Measurements on Chemical Systems"	8th Annu. Meet. of the Fed. of Anal. Chem. and Spectrosc. Soc., Philadelphia, Pa., Sept. 20-25, 1981
Higgins, C. E.	Higgins, C. E.; Griest, W. H.; Olerich, G. "Application of Tenax Trapping to the Analysis of Gas Phase Organics in Cigarette Smoke"	Tobacco Chemists' Res. Conf., Winston-Salem, N.C., Oct. 7-9, 1981
Ho, C.-h.	Ho, C.-h.; Clark, B. R.; Guerin, M. R. "Chemical and Biological Characterization of Synthetic Fuels"	Invited, Dept. of Chem., Tenn. Techn. Univ., Cookeville, Tenn., Feb. 6, 1981
	Ho, C.-h.; Guerin, M. R.; Buchanan, M. V. "Isolation and Identification of Bioactive Constituents in Synfuels"	Invited, Workshop on Chem. Charac. of Hazardous Substances in Synf., Seattle, Wash., Nov. 4-6, 1981

SPEAKER	AUTHOR(S) AND TITLE	PRESENTED AT
Holmberg, R. W.	Holmberg, R. W.; Moneyhun, J. H.; McCann, M. P. "An Exposure System for Studies of Concentrated Diesel Fuel Aerosols"	14th Aero. Techn. Meet., Johns Hopkins Univ., Baltimore, Md., Aug. 24-26, 1981
	Holmberg, R. W. "The New Department of Defense Program"	Anal. Chem. Div. Info. Meet., Oak Ridge Nat. Lab., Oak Ridge, Tenn., July 27-28, 1981
Hulett, L. D.	Hulett, L. D. "Positron Spectroscopy"	Chem. Dept., Univ. of Tenn., Knoxville, Tenn., Feb. 24, 1981
	Hulett, L. D. "Monoenergetic Positron Spectroscopy"	Physics Dept. Colloq., Univ. of Tenn., Knoxville, Tenn., Apr. 14, 1981
	Hulett, L. D. "Chemical Species and Structures in Fly Ash"	Louisiana State Univ. Mech. Eng. Dept., Baton Rouge, La., Dec. 7, 1981
	Hulett, L. D.; Dale, J. M.; Pendyala, S.* "Energy Loss Spectroscopy of Positrons Scattered from Solid Surfaces"	Positron Workshop, Brookhaven Nat. Lab., Brookhaven, Ill., Sept. 13-15, 1981
	Hulett, L. D. "Spectroscopy with Monoenergetic Positrons"	Southeastern Louisiana State Univ., Hammond, La., Dec. 8, 1981
	Hulett, L. D. "Spectroscopy with Monoenergetic Positrons"	Louisiana State Univ. Chem. Dept., Baton Rouge, La., Dec. 7, 1981
	Hulett, L. D. "Scanning Electron Microscopy"	ORAU Short Courses, Oak Ridge, Tenn., Jan. 20, 1981
Jenkins, R. A.	Jenkins, R. A.; Holmberg, R. W.; Moneyhun, J. H.; Wike, J. S. "Chemical and Physical Characterization of a Diesel Fuel Obscurant for Inhalation Toxicology Studies"	Proc., Smoke Obscurants Symp. V, Harry Diamond Labs., Adelphi, Md., Apr. 28-30, 1981
	Jenkins, R. A.; Gayle, T. M.;* Wike, J. S. "Sampling and Chemical Characterization of Concentrated Smokes"	ASTM Symp. on Sampling and Anal. of Toxic Mater. in the Atmos., Boulder, Colo., Aug. 2-6, 1981

SPEAKER	AUTHOR(S) AND TITLE	PRESENTED AT
Jenkins, R. A.	Jenkins, R. A. "Analytical Chemistry of Tobacco Smoke"	ORAU Traveling Lecture, Arkansas State Univ., Jonesboro, Ark., Mar. 23, 1981
Keller, J. M.	Keller, J. M. "Determination of Spark-Induced By-Products of Sulfur Hexafluoride"	23rd Rocky Mountain, Conf., Ion Chromatogr. Symp., Denver, Colo., Aug. 3-6, 1981
	Keller, J. M. "Ion Chromatography"	Chem. Tech. Div. Sem., Oak Ridge Nat. Lab., Oak Ridge, Tenn., July 29, 1981
	Keller, J. M. "Ion Chromatography"	UCCND Anal. Spec. Meet., Oak Ridge, Nat. Lab., Oak Ridge, Tenn., Sept. 20, 1981
Klatt, L. N.	Klatt, L. N.; Walia, D.* "Analytical Instruments in Coal Preparation Industry - Current Status and Development Needs"	Invited, Am. Chem. Soc. Meet., Atlanta, Ga., Mar. 1981
	Klatt, L. N. "Hot-Cell Instrumentation"	Semi-Annu. Meet., ASTM C-26 Comm., Reno, Nev., July 20, 1981
Laing, W. R.	Laing, W. R. "Computer-Assisted Management"	182nd Am. Chem. Soc. Nat. Meet., New York, N.Y., Aug. 23-28, 1981
	Laing, W. R.; Corbin, L. T. "National Standards for the Nuclear Industry"	25th Conf. on Anal. Chem. in Energy Techn., Gatlinburg, Tenn., Oct. 6-8, 1981
	Laing, W. R. "Activities of ASTM Subcommittee C26.05"	ASTM Comm. C-26 Winter Meet., Ft. Myers, Fla., Jan. 19-23, 1981
	Laing, W. R. "Computer-Assisted Management"	UCCND Anal. Spec. Meet., Oak Ridge Nat. Lab., Oak Ridge, Tenn., Oct. 20-21, 1981
Loundes, D. H.*	Loundes, D. H.*; Cleland, J. W.*; Fletcher, J.*; Narayan, J.*; Westbrook, R. D.*; Wood, R. F.*; Christie, W. H.*; Eby, R. E. "Pulsed Laser Annealing of Ion-Implanted Semiconducting GaAs Homojunction Solar Cells"	15th Photovoltaic Spec. Conf., Orlando, Fla., May 11-15, 1981

SPEAKER	AUTHOR(S) AND TITLE	PRESENTED AT
Lyon, W. S.	Lyon, W. S. "Institutional and National Representation, Speakers and Topics at Five Modern Trend Conferences"	6th Int. Conf., Modern Trends in Activation Anal., Univ. of Toronto, Toronto, Canada, June 15-19, 1981
	Lyon, W. S. "Nuclear and Activation Methods"	Invited, Symp. on Resonance Ionization Spectroscopy and its Applications, Gatlinburg, Tenn., June 1-5, 1981
	Lyon, W. S. "Project Selection in Analytical Chemistry Division"	Invited, Selection of Projects Seminar, Oak Ridge Nat. Lab., Oak Ridge, Tenn., Dec. 3, 1981
Kramer, S. D.*	Kramer, S. D.;* Bemis, C. E.;* Beene, J. R.;* Young, J. P. "Laser-Induced Nuclear Polarization "	Symp. on Resonance Ionization Spectros. and Its Appl., Gatlinburg, Tenn., June 1-5, 1981
Maskarinec, M. P.	Maskarinec, M. P. "Environmental Applications of High Resolution Chromatography"	ORAU Traveling Lecture, Tenn. Techn. Univ., Cookeville, Tenn., Jan. 16, 1981
	Maskarinec, M. P.; Harvey, R. W. "Analytical Chemistry of Solid Wastes"	UCCND/GAT Environ. Protection Seminar, Barkley Lake Lodge, Ky., Apr. 21-22, 1981
	Maskarinec, M. P.; Brown, D. K.* "Analysis of Treated Sludges and Associated Leachates from Coal Conversion Facilities"	Am. Chem. Soc. Meet., Symp. on Water Manage. and Pollut. Control for Coal Gasification and Liquefaction, New York, N.Y., Aug. 25, 1981
	Maskarinec, M. P. "Methods for the Analysis of Energy-Related Solid Wastes"	CONFAB '81, Saratoga, Wyo., July 21-24, 1981
	Maskarinec, M. P.; Buchanan, M. V.; Ho, C.-h. "Chemical Characterization of the Neutral Polar Fractions of Fossil-Derived Oils"	CONFAB '81, Saratoga, Wyo., July 21-24, 1981
	Maskarinec, M. P. "On-Column Injection in the Analysis of Environmental Materials by Capillary Gas Chromatography"	Am. Chem. Soc. Meet., Symp. on Water Manage. and Pollut. Control for Coal Gasification and Liquefaction, New York, N.Y., Aug. 25, 1981

SPEAKER	AUTHOR(S) AND TITLE	PRESENTED AT
Narayan, J.*	Narayan, J.;* Fletcher, J.;* White, C. W.;* Eby, R. E.; Christie, W. H. "Melting Phenomenon and Laser Annealing in Semiconductors"	Opt. Soc. of Am. Symp. on Laser Annealing and Mater. Processing, June 9-12, 1981
O'Kelley, G. D.*	O'Kelley, G. D.;* Auble, R. L.;* Hulett, L. D.; Kim, H. J.;* Milner, W. T.;* Raman, S.;* Shahal, O.;* Vane, C. R.;* Young, J. P.; Lapicki, G.* "Yields of X-Rays Produced by Bombardment with Energetic ^{40}Ar Ions"	33rd Annu. Southeastern Reg. Meet., Am. Chem. Soc., Lexington, Ky., Nov. 4-6, 1981
Peterson, J. R.*	Wood, A. B.;* Young, J. P.; Peterson, J. R.* "Absorption Spectrophotometric Characterization of Sm(II), Sm(III), and Sm(II/III) Oxybromide in the Solid State"	15th Rare Earth Res. Conf., Rolla, Mo., June 15-18, 1981
	Peterson, J. R.;* Young, J. P.; Ensor, D. D.;* Haire, R. G.* "Absorption Spectrophotometric Characterization of Berkelium-249 and Californium-249 Trichlorides and Determination of Their Relationships Through Beta Decay"	Actinides-1981, Asimolar Conf. Grounds, Pacific Grove, Calif., Sept. 10-15, 1981
Pippin, C. G.*	Pippin, C. G.;* Ensor, D. D.;* Young, J. P.; Ramsey, J. M. "Selective Laser Excitation of Terbium (III) in Lanthanum (III) Fluoride Precipitates"	33rd Annu. Southeastern Reg. Meet., Am. Chem. Soc., Lexington, Ky., Nov. 4-6, 1981
Ramsey, J. M.	Ramsey, J. M.; Whitten, W. B. "Delayed Lasing Measurements: A New Approach to Absorption Spectroscopy"	Pittsburgh Conf. on Anal. Chem. and Applied Spectros., Atlantic City, N.J., Mar. 9-13, 1981
	Ramsey, J. M. "Laser-Based Analytical Activities"	Anal. Chem. Div. Info. Meet., Oak Ridge Nat. Lab., Oak Ridge, Tenn., July 27-28, 1981
Rao, T. K.*	Rao, T. K.;* Ho, C.-h.; Allen, B. E.;* Cox, J. T.;* Epler, J. L.* "Relationships Between Mutagenic Activity in the Salmonella Assay and Chemical Structures with a Group of Nitrogen-Containing Chemicals Relevant to Synfuels"	12th Annu. Meet. of the Environ. Mutagen Soc., San Diego, Calif., Mar. 5-9, 1981
Ross, H. H.	Ross, H. H. "The ACD Long Range Plan"	Anal. Chem. Div. Info. Meet., Oak Ridge Nat. Lab., Oak Ridge, Tenn., July 27-28, 1981
	Ross, H. H. "Liquid Scintillation Counting: Recent Trends in the Assay of Nuclides of Biological Interest"	Invited, Am. Nucl. Soc. Winter Meet., San Francisco, Nov. 29-Dec. 3, 1981

SPEAKER	AUTHOR(S) AND TITLE	PRESENTED AT
Shaw, R. W.	Shaw, R. W. "Simple Optoacoustic Energy Meter for Pulsed Lasers"	2nd Int. Topical Meet. on Photoacoustic Spectros., Univ. of California, Berkeley, Calif., June 22-25, 1981
	Shaw, R. W.; Howell, H. E.* "First Piezo-electric Detection of Optoacoustic Spectra at Cryogenic Temperatures: Nd_2O_3 Below 10K"	2nd Topical Meet. on Photoacoustic Spectros., Berkeley, Calif., June 22-25, 1981
	Shaw, R. W.; Howell, H. E.* "First Piezo-electric Detection of Optoacoustic Spectra at Cryogenic Temperatures"	Workshop on Advanced Laser Techn. for Chem. Measure., Ames Lab., Ames, Iowa, Oct. 6-7, 1981
	Shaw, R. W. "Piezoelectric Detection of Optoacoustic Spectra"	UCCND Anal. Spec. Meet., Oak Ridge Nat. Lab., Oak Ridge, Tenn., Oct. 19-21, 1981
Shults, W. D.	Shults, W. D.; Carter, J. A.; Emery, J. F.; Laing, W. R. "Responsive Analytical Chemistry: Three-Mile Island"	Chem. Dept., College of Charleston, Charleston, S.C., Apr. 23, 1981
	Shults, W. D. "Analytical Chemistry is Alive and Well at ORNL"	Southeastern Assoc. of Anal. Chem., Charleston, S.C., Apr. 24, 1981
	Shults, W. D. "The Gatlinburg Conference: Barometer of Progress in Analytical Chemistry"	25th Conf. on Anal. Chem. in Energy Techn., Gatlinburg, Tenn., Oct. 6-8, 1981
Smith, D. H.	Smith, D. H.; Christie, W. H.; Eby, R. E. "A SIMS Study of the Resin Bead as a Thermal Ion Source"	29th Conf. on Mass Spectrom. and Allied Topics, Minneapolis, Minn., March 1981
	Smith, D. H.; Walker, R. L.; Carter, J. A. "The Modified Resin Bead Technique - Its Application to Safeguards"	25th Conf. on Anal. Chem. in Energy Techn., Gatlinburg, Tenn., Oct. 6-8, 1981
	Smith, D. H. "Mass Spectrometry Developments"	UCCND Anal. Meet., Oak Ridge Nat. Lab., Oak Ridge, Tenn., June 9, 1981
	Smith, D. H. "Resin Bead Mass Spectrometry in a Mobile Laboratory"	UCCND Anal. Spec. Meet., Oak Ridge Nat. Lab., Oak Ridge, Tenn., Oct. 20-21, 1981

SPEAKER	AUTHOR(S) AND TITLE	PRESENTED AT
Stewart, J. H.	Stewart, J. H. "Analytical Chemistry Division Emergency Response System"	Invited, 9th Nucl. Div. Anal. Meet., Oak Ridge Nat. Lab., Oak Ridge, Tenn., June 9, 1981
	Stewart, J. H. "Analytical Chemistry Divisions Quick Response System"	Anal. Chem. Div. Info. Meet., Oak Ridge Nat. Lab., Oak Ridge, Tenn., July 27, 1981
	Stewart, J. H. "Impurities and Their Effects in the Gaseous Diffusion Plant"	Invited, ASTM C-26 Nucl. Fuel Cycle Meet., Ft. Myers, Fla., Jan. 19, 1981
Tomkins, B. A.	Tomkins, B. A.; Caton, J. E. "Rapid, Sequential, HPLC Determination of Benzo(a)pyrene in Natural Synthetic, and Refined Crudes"	1981 Pittsburgh Conf., Atlantic City, N.J., Mar. 9-13, 1981
	Tomkins, B. A.; Griest, W. H.; Caton, J. E.; Wike, J. S.; Reagan, R. R. "Multicomponent Isolation and Analysis of Polycyclic Aromatic Hydrocarbons"	6th Int. Symp. on Polynuclear Aromatic Hydrocarbons; Battelle Columbus Labs., Columbus, Ohio, Oct. 27-29, 1981
Walker, R. L.	Walker, R. L.; Smith, D. H.; Musick, W. R.; Carter, J. A.; Donohue, D. L.;* Deron, S.*; Asakura, Y.*; Kagami, K.*; Irinouchi, S.*; Masui, J.* "Part 1. Resin Bead Mass Spectrometry. Part 2. Results of a Resin Bead Field Experiment - TASTEX-J"	Safeguards Anal. Lab. Eval. (SALE) Program, Argonne, Ill., July 8-9, 1981
Walton, J. R.	Walton, J. R.; Smith, D. H.; McKown, H. S.; Carter, J. A. "A Quadrupole Mass Spectrometer for a Mobile Laboratory to Measure Isotopic Ratios"	29th Annu. Conf. on Mass Spectrom. and Allied Topics, Minneapolis, Minn., May 24-29, 1981
Wade, J. W.	Wade, J. W. "Instrumental Neutron Activation Analysis on Environmental Samples"	UCCND Anal. Comm., Paducah, Ky., Sept. 14-15, 1981
	Wade, J. W. "Instrumental Neutron Activation Analysis at ORNL"	UCCND Anal. Spec. Meet., Oak Ridge Nat. Lab., Oak Ridge, Tenn., Oct. 21, 1981
Warmack, R. J.	Warmack, R. J.; Landau, L.; Christie, W. H.; Carter, J. A. "A New Mass Spectrometric Facility for the Analysis of Highly Radioactive Samples"	25th Conf. on Anal. Chem. in Energy Techn., Gatlinburg, Tenn., Oct. 6-8, 1981

SPEAKER	AUTHOR(S) AND TITLE	PRESENTED AT
White, C. W.*	White, C. W.;* Christie, W. H. "Use of RBS and SIMS to Investigate the Near Surface Region of Laser Annealed Semiconductors"	181st Am. Chem. Soc. Nat. Meet., Atlanta, Ga., Mar. 29-Apr. 3, 1981
Whitten, W. B.	Whitten, W. B. "Time-of-Flight Optical Spectrometry with Fiber Optic Waveguides"	Workshop on Advanced Laser Techn. for Chem. Measure., Ames Lab., Ames, Iowa, Oct. 6-7, 1981
	Whitten, W. B. "Time-of-Flight Optical Spectrometry"	ORAU Traveling Lecture, Univ. of Arkansas, Fayetteville, Ark., Mar. 27, 1981
Young, J. P.	Young, J. P. "RIS for Analytical Chemistry"	Invited, Symp. on Resonance Ionization Spectros. and its Appl. Gatlinburg, Tenn., June 1-5, 1981
	Young, J. P.; Haire, R. G.;* Peterson, J. R.;* Ensor, D. D.* "Spectrophotometric Determination of the Structure of Transcurium Ions in Solid-State Compounds: Analytical Applications of the Technique"	182nd Am. Chem. Soc. Nat. Meet., New York, N.Y., Aug. 23-28, 1981
	Young, J. P.; Haire, R. G.;* Peterson, J. R.;* Ensor, D. D.* "Chemical Consequences of the Decay of ^{253}Es Ions in Solid State Compounds: Effects of Heredity and Environment"	182nd Am. Chem. Soc. Nat. Meet., New York, N.Y., Aug. 23-28, 1981
	Young, J. P.; Haire, R. G.;* Peterson, J. R.;* Ensor, D. D.* "Effects of Heredity and Environment on the Chemical Consequences of the Decay of ^{253}Es Ions in Solid State Compounds"	Actinides, Pacific Grove, Calif., Sept. 10-15, 1981
	Young, J. P.; Auble, R. L.;* Hulet, L. D.; Kahane, S.;* Kim, H. J.;* Milner, W. T.;* O'Kelley, G. D.;* Raman, S.;* Rosseel, T. M.; Slaughter, G. G.;* Vane, C. R.* "Chemical Information from Heavy-Ion Induced X-Rays"	Phys. Div. Info. Meet., Oak Ridge Nat. Lab., Oak Ridge, Tenn., Nov. 11-12, 1981

ARTICLES REVIEWED OR REFERRED FOR PERIODICALS

	<i>Anal. Chem.</i>	<i>Anal. Chim. Acta</i>	<i>Environ. Sci. Technol.</i>	<i>J. Polym. & Colloid Sci.</i>	<i>J. Radioanal. Chem.</i>	<i>Nucl. & Chem. Waste Manag.</i>	<i>Nucl. Instrum. & Meth.</i>	<i>Opt. Lett.</i>	<i>ORNL Reports</i>	<i>Proceedings</i>	<i>Proposals</i>	<i>Radiochem. Radiocnol. Lett.</i>	<i>Radiochim. Acta</i>	<i>Other</i>	<i>Total</i>
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Carter, J. A.	1										2				3
Christie, W. H.														2	2
Clark, B. R.											5				5
Costanzo, D. A.						1	1						1		3
Dyer, F. F.		1							1						2
Emery, J. F.	1														1
Feldman, C.	4										1				5
Goeringer, D. E.		1													1
Griest, W. H.	3										2			2	7
Guerin, M. R.									1	1	4			4	10
Holmberg, R. W.			1												1
Hulett, L. D.			3	3	1										7
Keller, J. M.	1													1	2
Klatt, L. N.	1	1													2
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MacDougall, C. S.	2														2
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Stewart, J. H.														1	1
Stokely, J. R.														1	1
Tomkins, B. A.	5	1													6
Young, J. P.	2							1			2				5
Total	28	5	5	3	4	2	1	1	2	2	23	1	1	14	92

DIVISIONAL MAN-POWER AND FINANCIAL SUMMARY
FY 1981

Source	Funding (in thousands)	Man-years
DOE programs		
Environmental Research and Development	1,232	15.4
Basic Energy Sciences	1,067	11.2
Consolidated Fuel Reprocessing Program	300	2.6
Fossil Energy	179	1.8
Nuclear Materials Security and Safeguards	125	1.2
Reactor Research and Development	80	0.9
Defense Waste Management	<u>55</u>	<u>0.6</u>
Total DOE programs	3,038	33.7
Work for others - federal agencies		
Department of Defense (U.S. Army)	712	7.3
National Cancer Institute	533	5.5
Environmental Protection Agency	411	4.5
State Department (I.S.P.O.)	114	1.5
Miscellaneous	<u>10</u>	<u>0.1</u>
Total work for others - federal agencies	1,780	18.9
Work for others - nonfederal agencies		
Electric Power Research Institute	274	3.5
Council for Tobacco Research	100	0.7
Protective Coating Companies	75	0.8
E. I. DuPont	66	0.7
Rockwell International	40	0.5
IBM	23	0.3
Miscellaneous	<u>85</u>	<u>0.9</u>
Total work for others - nonfederal agencies	663	7.4
ORNL divisions - support	6,424 ^a	88.0
Division administration	<u> </u>	<u>12.0</u>
Total financial plans	11,905	160.0

^aReflects Laboratory general and administration expenses and general plant services.

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