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ANALYTICAL CHEMISTRY DIVISION ANNUAL PROGRESS REPORT

For Period Ending December 31, 1982

W. D. Shults, Director

W. S. Lyon, Editor

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INTRODUCTION AND SUMMARY

W. D. Shultz, Director

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 26% of the FY 1982 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 DO programs and from "Work-for-Others" agreements. Emphasis here is on "applied" chemistry. This type of activity accounted for approximately 23% of the division's budget in FY 1982.
- 3. Technical Support.** The division performs chemical and physicochemical analyses 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. Much of this work is short-term in nature and comes from other divisions and programs within ORNL, but a significant fraction originates outside of ORNL and involves the use of talent and/or facilities in which the division is particularly strong or unique. This type of work accounted for approximately 51% of the budget during FY 1982.

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, 1982 to December 31, 1982. 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 61 articles, 32 proceedings publications and 37 reports have been published, and 107 oral presentations were given during this reporting period.

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SOME TECHNICAL HIGHLIGHTS

We instituted an informal "exchange" program with the Center for Analytical Chemistry of the National Bureau of Standards in CY 1982. In the first collaborative study, an ORNL researcher spent two weeks at the Bureau, where he used the CAMECA IMS-3f secondary ion mass spectrometer to examine the distribution of uranium around a "resin bead" source as a function of temperature. These studies indicated that the bead does not act as a point source as previously thought, but that it provides diffusion-controlled release of neutral uranium atoms. The released uranium then moves with high mobility on the filament surface.

Several collaborative programs with the Consolidated Fuel Reprocessing Program (CFRP) have resulted in significant accomplishments. A prototype of our multi-wavelength photometric in-line monitor, developed with Instruments and Controls Division staff for the CFRP, was installed at Y-12. This application calls for monitoring 1-30 g/L of uranium in an aqueous stream. The photometer has performed beautifully for several months and other units are to be constructed and installed in the plant. This photometric monitor is applicable to U, Pu(III), and Pu(IV) in aqueous and organic solutions.

In another project supported by CFRP, a prototype microcomputer-based remote titration system was designed, built and is now under test in our mock-up hot cell. Titrations can be as precise as $<0.05\%$ under favorable conditions and typically require about 5 minutes. This state-of-the-art titrator constitutes the second step in our program for modernizing remote analytical instrumentation. The first step was development of our microcomputer-based pipettor, and was reported in previous annual reports.

A joint venture between ACD, CFRP, and the Metals and Ceramics Division involves development of an x-ray fluorescence system for measuring uranium and plutonium in a highly gamma-active background. The system has been conceptualized and carried through proof-of-principle experiments. Preliminary data show resolution to be 10-20 eV and sensitivity to be about 0.25 cps/ppm uranium. The system has been successfully tested in the presence of high radiation fields with a 1.9×10^{12} Bq Ir-192 isotopic source and by focusing a silver target x-ray tube on the entrance slit of the spectrometer.

We continued to team with members of the Physics Division to develop applications of heavy ions. The importance of various parameters -- projectile, x-ray shell, target makeup -- is being studied. This effort has shown that fine structure in the x-ray spectra produced by bombardment with energetic ions is indicative of the valence electron density of the target material, hence may provide useful information about the composition of compounds and alloys. Long range ordered (LRO) alloys exhibit physical properties that are dependent on small changes in composition. We initiated a collaborative effort with members of the Metals and Ceramics Division, to study heavy ion induced x-ray chemical shifts of LROs.

Development of an extrusion-combustion generator for producing H_3PO_4 aerosols from a red phosphorus/butyl rubber formulation was completed. The generator produces aerosol continuously and at uniform concentration for several hours, as is required for inhalation toxicology studies. We constructed five of these generators plus aerosol monitoring equipment and installed them at the Illinois Institute of Technology Research Institute. This activity is part of a multi-institution program in inhalation toxicology, sponsored by the Department of Defense.

Working with the Laboratory Protection staff, we established an analytical system for non-destructive assay of special nuclear materials, to be used primarily for inventory audits. It is a mobile gamma spectrometer with 100 mm diameter intrinsic germanium detector. The system can be used for inventory verification of precious metals via radionuclide-excited x-ray fluorescence, and has been used for assay and/or identity verification of silver, gold, platinum, and uranium in various forms. It also can be used in response to unusual occurrences within ORNL, hence constitutes a triple-purpose mobile analytical device.

Collaborative work between ACD and Environmental Sciences Division has shown that an impurity in commercial acridine results in cricket teratogenicity. This compound has now been identified as benzo(g)isoquinoline-5,10-dione; it constitutes only 20 ppm of the commercial product. Unambiguous identification required the synthesis of model compounds, preparative scale isolation, high resolution chromatographic purification, high resolution mass spectrometry, diffuse

reflectance, Fourier transform infrared spectrometry, and multiple chromatographic retention measurements. The diode has been made available for testing in other biological systems and is being sought in coal-derived oils.

A new concept, Fourier transform microwave spectrometry, was proved. An electronic method had to be devised to introduce a temporal delay into the system thereby accessing the transform. Spatial displacement is used ordinarily to get a temporal delay, but this is impractical in the case of microwaves. Feasibility was demonstrated with carbonyl sulfide. This work was supported in part by the ORNL Exploratory Studies Program.

We developed laser-based resonance ionization mass spectrometry (RIMS) as a means for markedly improving the selectivity of isotope ratio measurements. Feasibility was demonstrated with mixtures of neodymium and samarium, which are difficult to analyze by thermal ionization MS because of isobaric interferences. RIMS has proved to be a fertile research area in analytical MS. We have now used the RIMS technique for other elements, including cerium, praseodymium, europium, and plutonium.

A "mixed spike" technique for uranium analysis has been developed. Mixtures of uranium isotopes (^{233}U & ^{236}U) were synthesized and characterized for use as "spikes" in isotope dilution mass spectrometric analysis. A mixed spike of plutonium isotopes (^{242}Pu & ^{244}Pu) has also been synthesized. Use of such spikes provides an improvement in precision of about five, i.e., standard deviations of $\leq 15\%$ for uranium at the nanogram level. Much larger (μg) quantities are necessary to obtain this level of performance by conventional spiking methodology.

Studies of the mass spectrometry of non-volatile organic compounds have continued. The MS source that we designed for research in organic secondary ion mass spectrometry (SIMS) has been combined with our three-sector MS/MS instrument. This union provides unparalleled sensitivity. Initial experiments into thermal desorption/thermal ionization of organic materials of low volatility were also successful. This technique was combined with our MS/MS system; it promises to be of great analytical utility.

Research into the use of labelled reagents in chemical ionization mass spectrometry continued. We are able to distinguish, by this technique, isomers of several different types of compounds. Methanol and methanol- D_4 can now be used to differentiate a phenol, aromatic ether or aromatic substituted alcohol of the same formula. Similarly, this approach has now been extended to the characterization of isomeric sulfur-containing compounds.

Studies of open tubular liquid chromatography (OTLC) were initiated this year. These included investigations into techniques for column preparation, sample injection, and UV absorption and laser-excited fluorescence detection. The use of electrokinetic phenomena in OTLC is also under study. We have initiated development of an orifice for OTLC/MS. The OTLC technique offers significant advantages as a complement to existing chromatographic methodology.

Finally, the division performed some 266,000 determinations during CY 1982, primarily in support of experimental programs at the Laboratory. Approximately 1550 control analyses were processed as part of the quality control program in the service/support groups, with an overall quality level of 91.3%.

1. ANALYTICAL METHODOLOGY

W. S. Lyon

Analytical Methodology is composed of two groups: Analytical Instrumentation and Analytical Physics. Each performs research and development that is primarily funded by the Office of Basic Energy Sciences of the Department of Energy. Some applied work is also done.

In the Analytical Instrumentation Group emphasis is primarily on the use of lasers or laser based systems for analytical purposes. Research is carried through the proof of principle and development stage after which, in collaboration with other sections within the division or other divisions within ORNL, instrumentation is applied to processes or problems. Particular success has been achieved in applying systems to uranium fuel monitoring.

The Analytical Physics Group has a small but innovative program that collaborates with other physics oriented divisions at ORNL. At present the emphasis is on fundamental studies directed toward understanding low energy positron scattering and heavy ion induced fluorescence. The ultimate objective is to use these techniques for materials characterization.

ANALYTICAL INSTRUMENTATION

H. E. Ross

Spectroscopy Research

The spectroscopy research carried out in the Instrumentation Group follows along lines similar to those described last year. The common thread in much of this work is the laser, used as either a light source or as an intense thermal source. Several projects were completed this year and a few new tasks were initiated. Our general approach remains unchanged: develop the idea and/or theory, test the system experimentally, demonstrate the practical analytical potential, and define those analytical problems or application areas where the work could have a significant impact. We are particularly attentive to maintaining a broad spectrum (pun intended) of expertise in the area of atomic and molecular spectroscopy; the following description of accomplishments for the year attests that we are meeting well this objective.

A program initiated last year (with the help of an ORNL seed money grant) concerned the development of a new analytical concept - Fourier transform microwave spectroscopy. The measurement apparatus is essentially a microwave Michelson interferometer making the experiment similar to that performed in Fourier transform infra-red spectroscopy. However, a subtle difference exists in our system. In a conventional interferometer, a needed time delay is implemented by means of a path length change in one arm. In the microwave region, adequate resolution would require a 3 km path length differential! To circumvent this awkward spatial requirement, we have devised an electronic way to implement a temporal delay that simulates a path length change. To prove that such an approach is feasible, we have constructed a demonstration measurement system and observed the J=0 to 1 transition of carbonyl sulfide. Work this year will be directed toward improving the signal-to-noise ratio; we will also test Stark modulation of the sample to reduce non-stationary noise sources.

Many of the problems in our program to develop matrix-isolation optoacoustic spectroscopy have been solved, and we have now acquired spectra for several N-heterocycle polynuclear aromatic compounds. Sub-microgram detection limits have been measured for several members of the quinoline family in rare gas matrices at 10-20K.

For these compounds, the spectral resolution is enhanced by a factor of 3 to 5 over that obtained at room temperature. This improvement allows identification and quantitation of mixtures impossible with conventional room temperature techniques. Solid xenon has been used as the host matrix for these experiments rather than the more usual argon. We feel that the xenon host serves as an external heavy atom catalyst and enhances our lower detection limit. A new acoustic detector has been designed and is presently under construction. It should lead to a 100-fold improvement in our detection limit, that is, to levels below about ten nanograms.

Another significant advance this year has been the use of the resonance ionization technique to generate ions for mass spectrometry. The technique of resonance ion mass spectrometry (RIMS) is described in detail in the Mass Spectrometry section of this report. Other resonance ionization spectroscopy (RIS) methods were also explored. To apply RIS to certain ultra-low level radioactive counting determinations, the daughter species from the decay process must thermalize to a neutral atom to be detected by an RIS process. We recently completed a study of the ^{218}Po daughter of ^{222}Rn . We found that several gas mixtures of organic vapors in helium effectively neutralize the polonium recoils; these include helium plus 1-3% isobutane, 0.5% triethylamine, 1-5% cyclohexane, or 1-5% ethylene. Other gases and gas mixtures that were found to be ineffective include air, argon, argon-(10%) methane, argon-isobutane, and helium-NO. We have made some progress in our cooperative project with EG&G Corporation to detect single atoms of plutonium coming from the decay of ^{244}Cm . A laser test system and sample chamber have been built and are now ready to use at the EG&G site. Detection will be attempted in an atmosphere of helium-isobutane, which has already been shown to allow the neutralization of recoil species.

We continue to study time-of-flight optical spectrometry and have made initial tests to evaluate this technique for application in the ultra-violet spectral region. The short optical fibers, required by the high UV attenuation, still have useful dispersion because of the rapid change of refractive index with wavelength in this region. We have measured the attenuation of three different fibers from 900 to less than 300 nm, and also measured the dispersion and pulse broadening in the near UV. The most significant result is that for some step index

fibers, the pulse broadening in the UV is only half that observed in the visible region, presumably because of enhanced attenuation of rays which make a large angle with the fiber axis. This increased bandwidth may permit the use of step index fibers with high purity fused silica cores for UV time-of-flight spectrometry.

We have started a new project to investigate the flame optogalvanic determination of cesium in brines. Determination of the cesium content of brine solutions is of interest for leaching studies related to nuclear waste isolation. The high salt level of these NaCl/KCl/MgCl₂·6H₂O brines (ca. 20 g/L each) interferes with most atomic spectroscopy techniques, including those that employ flame or graphite furnace atom reservoirs, yet direct analysis of these brines is desirable. A flame optogalvanic spectroscopy apparatus was assembled using a modified atomic absorption slot burner and our pulsed dye laser. In the sample-seeded flame gases, there exists a dynamic equilibrium between neutral cesium atoms and cesium ions. When laser light resonant with cesium atoms illuminates the flames, this ionization equilibrium is disturbed toward additional ionization; the resulting pulse of current is detected.

For distilled water solutions our cesium detection limit is 60 ppB. Cesium has the lowest ionization potential of any element and spontaneous cesium ionization at our flame temperature is almost complete. Because this method can only detect neutral atomic species, we actually only measure a small percentage of the total cesium aspirated into the flame. The effects of the Na/K/Mg brine matrix on the cesium detection limit and response linearity have been studied. For various brine concentrations, the cesium sensitivity factor is approximately constant. For a 5 g/L concentration of each of the three brine components (the highest tolerable concentration for reproducible nebulization), the cesium detection limit is approximately 7 ppm. Our approach has been to handle brine samples by diluting them to a reasonable working salt level and accepting the consequent dilution factor for the cesium content. Because the cesium detection limit for distilled water samples has been extended as far as possible, this brine sample dilution does not degrade our overall detection limit to an unacceptable level. Thus, the brine matrix problem is severe for these solutions, but at salt levels approaching 300 g/L NaCl, we can still detect cesium concentrations as low as 3 ppm.

We have continued to explore, both experimentally and theoretically, the use of the time delay in the emission of a laser pulse for determining the absorbance of a sample within the laser cavity. The delay can be interpreted in terms of gain and cavity build-up times. There is a fundamental uncertainty or jitter in the cavity build-up time due to the statistical properties of light. Detection limits and accuracy should be improved if the gain build-up time is made much larger than the cavity build-up time. For practical applications, it would be extremely desirable for the gain medium to be tunable in wavelength. We have been experimenting with an optical cavity in which one mirror is a phase-conjugate reflector with gain. This mirror uses nonresonant degenerate four-wave mixing in a single domain crystal of barium titanate. A volume hologram is formed at the wavelength of the pump beams, furnished by a CW dye laser. The gain of the mirror builds up with a time constant which depends on the intensity of the three incident light beams. We have set up such an oscillator and shown that the gain build-up time is a function of cavity losses.

The reflected wave from a phase-conjugate mirror is a "time-reversed" replica of the incoming wave. This implies that if a uniform beam propagates through a distorter into the mirror, the reflected beam will have the original uniformity after passing back through the distorter. We have demonstrated that such distortion correction can be obtained by directing a single pump beam into a barium titanate crystal with no other mirrors. Other applications of real-time volume holography are being investigated.

We are presently exploring a new method for the generation of molecular beams: laser ablation of solids into a high vacuum. The isolation of large molecules in ultracold molecular beams can be accomplished by expanding a high pressure gas (such as helium) seeded with an analyte gas through a nozzle into a high vacuum chamber. The spectra of molecules so isolated are very sharp and simple due to greatly reduced collisional and Doppler broadening. These sharp spectra are very useful analytically for identification of individual compounds in complex mixtures. We are attempting to generate similar beams using a pulsed laser to flash evaporate the surface layer from a solid sample located in a vacuum chamber. The focal spot of the laser beam will simulate the orifice of a conventional

molecular beam machine. A vacuum chamber for these experiments has been assembled and has been included in an optical arrangement whereby three laser beams can be impinged on a single sample spot. The lasers are a Nd:YAG pulsed laser (for ablation), a frequency-doubled flash-lamp dye laser (for excitation of fluorescence), and a He-Ne laser (for visualization of the target spot and alignment purposes). Initial experiments indicate that our 1 mJ ablation laser is not intense enough to evaporate sufficient material from a tetracene film for spectral observation. Further experiments with different targets, excitation geometries, and more powerful ablating pulses are currently in progress.

Further tests have been conducted on the phase fluorimeter that uses a self-modulated argon-ion laser as the excitation source. As we reported last year, this argon-ion laser can be operated such that the output intensity is nearly 100% modulated at any of five frequencies between 408 and 737 MHz. This is an attractive source for making high temporal resolution measurements by the phase-shift method because of the very high modulation frequencies. The phase-shift measurements can be made in an analog fashion using a double-balanced mixer to compare the phase of the fluorescence signal and the exciting light. Originally, an optical delay line was used to adjust the phase of the signals at the mixer to obtain zero output signal (signals 90° out of phase). From the relative positions of the delay line element for a scattering solution (zero lifetime) and the fluorescing sample, the lifetime of the sample can be calculated. The precision of this approach proved to be poor. To improve the precision, the optical delay line was scanned through one full period of the modulation frequency and the output of the mixer recorded. The response function of a scattering solution and a fluorophor can now be compared to yield a phase shift of higher precision. An improvement was the replacement of the high-gain photomultiplier tube (PMT) by a moderate gain tube; the high-gain PMT does not allow sufficient sampling of the modulated fluorescence signal.

An excellent test for a fluorescence lifetime measuring apparatus is a bimolecular quenching study. A Stern-Volmer plot should yield a line whose slope gives the quenching rate constant. We used a series of 1 μ mol/L R6G/H₂O solutions quenched by KI. The fluorescence lifetimes for these solutions range from 3 ns to 30 ps. Lifetime measurements below \sim 300 ps could be made

reliably. Lifetimes longer than 300 ps have phase shifts which decrease with increased photomultiplier current. Similar results have been observed when this experiment is performed with a time-correlated single-photon counting apparatus. Plans are being made to incorporate high speed counting equipment into the latter experiment to help understand these problems.

In a cooperative project with the University of Tennessee, optically transparent thin layer electrochemical (OTTLE) cells have been found to have wide analytical application. This is because they can simultaneously acquire spectroscopic and electrochemical data. However, because of the small optical path length, the ability to observe, spectroscopically, species possessing small molar absorption coefficients is severely limited. We are developing a multipass capability that can be used in aqueous as well as molten salt media and can be coupled to a multichannel optical detection system. The technique has been successfully demonstrated with an aqueous system using a tungsten filament radiant energy source. Deviations between theoretical and experimental gains are less than 0.5%. Work is in progress to demonstrate the technique with a molten salt OTTLE in a high temperature furnace.

Finally, we note that study of the selected laser excited fluorescence determination of Tb(III) coprecipitated with LaF₃ has been completed and a paper describing this study is in press in *Analytical Chemistry*. Using Er(III) as an internal standard, Tb(III) can be determined in LaF₃ with a precision of better than \pm 4%. The detection limit is 1 \times 10⁻⁶ mole % Tb(III) in LaF₃; this mole ratio corresponds to a Tb concentration of 3 \times 10⁻¹⁰ M in the sample solution before precipitation. The procedure is insensitive to anionic interference from nitrate or chloride and is less sensitive to common cationic interferences than is a similar laser fluorescence technique which utilizes coprecipitation of lanthanides in CaF₂. Attempts to apply this technique to trivalent actinides has only been marginally successful. Expected sample fluorescence does not always occur, and it is presently felt that the problem may lie in crystal symmetry effects which are not properly controlled.

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Applied Instrumentation

For the past four years, we have been heavily committed to a cooperative program with the Instrumentation & Controls Division to develop in-line chemical instrumentation for a demonstration fuel reprocessing plant. Major efforts were directed toward the continuous measurement of uranium, plutonium, certain organic reagents, and free acid concentration. Process conditions included both organic and aqueous streams and concentrations that ranged from trace to major component. This year a prototype multiwavelength photometer, incorporating a multiplexing mechanism, was fabricated and installed in a U²³⁵ uranium recovery process. The instrument will ultimately be capable of monitoring three process points simultaneously and report real-time uranium concentration in both aqueous and organic streams for process control. To date, a three-wavelength calibration algorithm has been developed for monitoring 1-30 g/L uranium in an aqueous raffinate stream having a variable optical background. The photometer will also be calibrated for uranium measurement in an aqueous feed and an organic strip column.

A description of the photometric analysis of Pu(IV) in organic Purex solutions was also completed. Optimum wavelengths for plutonium determination have now been selected and optical fiber purchased so that the prototype photometer can also be applied to the determination of U, Pu(III), Pu(IV) in both aqueous and organic solutions.

In-line electrochemical reduction of U(VI) was assessed in an effort to extend the photometric analysis down to 0.2 g/L in highly colored aqueous process solutions. U(VI) absorbs light at several wavelengths that are not influenced by the presence of the usual impurities found in reprocessing streams. This slight optical background can be accurately measured prior to U(VI) reduction. The U(IV) molar absorptivity is also a factor of 23 times higher than that of U(VI); U(IV) measurement would therefore provide a more sensitive indication of aqueous uranium concentration. Prepared U(VI) standards can be reduced in <5 min using a platinum cathode and a reticulated vitreous carbon anode. If the sample solution is highly contaminated, a chemical reductant, such as hydrazine nitrate, must be added to the solution to prevent reoxidation of U(IV). A report is being prepared that further describes the electrochemical procedure.

We also have a significant effort to develop advanced instrumentation for use in hot-cell environments. A prototype of a remote titration system suitable for such use was designed and constructed. The in-cell components consist of the titrant delivery unit, which features interchangeable burets, and the preamplifier circuit for the transducer electrodes. The buret is driven by a stepping motor. The out-of-cell components include the microcomputer controller, plotter, and terminal. Laboratory evaluation of the system is complete. With the 25 mL buret, the precision of volume delivery is 0 μ L and is independent of the aliquot size. Based upon acid-base titration data, a precision and accuracy better than 0.05% can be achieved if the equilibrium constant for the titration reaction is $\geq 10^6$. The end-point is obtained from the zero-crossing point of the second derivative. A digital filter calculates the second derivative while the incremental addition of titrant is controlled automatically by the microcomputer (calculated via a predictive least-squares algorithm). A typical titration requires ~100 data points and is completed in approximately five minutes. Testing of the system in a mock-up hot-cell is underway.

An interface to an Apple II computer for a two-dimensional position sensitive detector was designed. The outputs from the detector are two analog voltages which correspond to the coordinate where a particle impacted upon the detector. These coordinate data may represent energy or positional information depending upon the particular experimental situation. The coordinate data are digitized into two 6-bit addresses which represent elements of a 64 x 64 matrix whose magnitude corresponds to the number of events that occurred at that particular coordinate. The interface consists of the analog-to-digital converter, the digital memory and its control logic, and the logic necessary to operate the unit under computer control. Construction and testing of the system are underway. Plans for the system include its use as a positron detector for ongoing work in the Analytical Physics group.

Work on the glow discharge detector for oxygen detection has been completed. A major problem was the reduction of the oxygen background in the helium carrier gas. With the use of a trap containing BASF R3-11 catalyst, the oxygen signal emitted by a glow discharge in laboratory-grade helium was kept below 0.1% of its original intensity for 2 hours, and below

1.0% for 6 hours. Passage of peaks of oxygen-bearing compounds through the glow discharge was simulated by injecting such compounds into the gas stream through an injection loop placed upstream from the capillary column. The height (and sign) of the peak produced depended on the composition of the compound. To convert all oxygen to a single chemical form (CO), a quartz tube containing carbon at 900°C was inserted between the GC column and the glow detector. All oxygen-bearing compounds then gave positive peaks.

(L. A. Postirk, C. Feldman, L. D. Hulett, L. N. Klatt, S. Pendyal, * J. E. Strain)

ANALYTICAL PHYSICS

L. D. Hulett, Jr.

Problem Solving with Electron and X-ray Methods

Solid specimens can be characterized by their composition, crystal structure, morphology and the chemical state of the constituents. Knowledge of several or all of these properties is often required to solve real materials problems. When used in a matrix approach, electron and x-ray methods can solve complex problems by providing the critical information about the properties of a material. Elemental composition from major to trace levels can be determined by x-ray fluorescence (XRF). Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) yield information on surface morphology and the shape and size of submicron particles. With an energy dispersive x-ray fluorescence detector (EDX), the SEM can scan across a sample to provide an elemental map. Diffraction patterns produced by x-rays or electrons are used to determine crystal structures of bulk materials or particles as small as 1 μ m. Examples of how these techniques have been applied to problems from various laboratory programs are described in this section.

The Chemistry Division has an ongoing program for developing methods of tritium containment by producing oxide films on container materials. Tritium rapidly diffuses through clean metals and alloys and its containment is a major problem in developing controlled thermonuclear reactors. We have recently characterized films produced on 310 stainless steel which have good

tritium barrier characteristics. Based on SEM, TEM, and XRD results, it was concluded that the material was possibly a spinel compound having properties similar to $MnCrO_4$ which is a superior containment material. Another problem involved the characterization of a composite sample containing less than 1% uranium. This was related to a program for characterizing uranium-bearing refractory solid residues to enable development of improved techniques for complete uranium recovery from such residues.

We were recently requested to make on-site sampling of residues which had collected on some automobiles in the ORNL West Parking Lot. A few automobiles in this location had developed rust spots and it was necessary to determine the presence or absence of fossil fuel plant fly ash particles. Using SEM and SEM-EDX it was determined that the particles removed had compositions which resembled soil; there was no evidence that fly ash particles caused the corrosion.

The correlation of air-borne asbestos fibers with lung cancer has resulted in requirements imposed by the Environmental Protection Agency (EPA) to limit human exposure. Because compliance with these health standards requires a method for accurate asbestos analysis, we have been working with EPA to develop a sensitive x-ray diffraction (XRD) technique for the quantitative determination of asbestos in building materials. As previously reported, the ORNL approach uses an internal standard to determine concentrations from 0.5% to 60%. Initial testing on EPA samples indicated that the XRD method was more sensitive to asbestos than either polarized light microscopy (PLM) or thermogravimetric analysis (TGA). Further evaluation has shown, however, that the internal standard, aspirin, may decompose in some materials. Work is in progress to find a stable substitute for aspirin.

Work is also progressing on modifications to the computer controlled diffractometer and eight-position sample changer which, when completed, will allow overnight and weekend operation. Continuous operation will be required for the anticipated high sample load from state agencies and local governments attempting to evaluate public health hazards. In conjunction with a more efficient diffractometer, a number of techniques are being evaluated as pre-screening methods to help process samples more rapidly.

*State University of New York, Fredonia.

(P. L. Ball, H. W. Dunn, J. H. Stewart)

Chemical Analysis by Positrons and Heavy Ions

Investigations of the characteristics of low energy positrons for revealing various properties of matter has shown a dramatic increase in the last few years. It was made evident that the field is growing at a rapid pace by the number of papers presented on the subject this year at the Sixth International Positron Annihilation Conference in Fort Worth, Texas. The growth of this science has been made possible by the relatively recent breakthroughs in the development of two high efficiency positron moderators, copper and tungsten. The copper moderator was developed by workers at Bell Laboratories and the tungsten moderator by our group here at Oak Ridge. These moderators have the necessary properties for thermalizing high energy positrons which are then accelerated to low velocities for controlled low energy positron interactions with materials under study. Our work on the development of the tungsten moderator and its use for positron energy loss experiments at metal surfaces were reported at this conference. Further attempts at moderator improvements have been made. Tungsten with an increased surface area was tested and specific alloys were also tried for theoretical reasons. The efficiencies of these moderators approached but did not exceed that of the high temperature treated single crystal and polycrystalline tungsten.

Our positron program has induced other workers in the ORNL Physics Division to become interested in monoenergetic positrons. They have carried out initial experiments for creating high energy positrons by proton bombardment of ^{13}C to produce ^{11}C which is a positron emitter. Although the yields were low in these first attempts to produce positrons by this technique, due to moderator contamination, we still expect to achieve a factor of ten greater intensity over that from the ^{22}Na source presently being used. We will use this facility to do high energy positron diffraction experiments at energies normally used in the transmission electron microscope. A simplified dynamic diffraction theory has been formulated to compare positron and electron diffraction. The theory predicts that positrons will travel between rows of atoms in crystals and that measurements in intensity loss of the diffracted beams will provide information about electron densities between atomic sites. Positron diffraction data are therefore expected to be richer in chemical bond information than ordinary electron diffraction data.

Positron and electron scattering spectra from a polished annealed polycrystalline molybdenum surface are currently being measured with a low pass/high pass differential retarding field spectrometer. This spectrometer was designed to provide the high throughput necessary for positron scattering experiments combined with the direct measurement of differential energy loss and diffraction spectra. A report of possible invention for this spectrometer has been filed with the ORNL patent department. A position sensitive detector with a resistive strip readout has been purchased for recording positron diffraction patterns. An interface has been designed and is being constructed for storing these data in a microcomputer.

An invited paper, reviewing the applications of monoenergetic positrons for the characterization of solids has been completed and submitted to *Applied Spectroscopy Reviews*. A combined symposium-workshop on the implementation of monoenergetic positrons in fundamental and applied science has been organized and will be held at the annual meeting of AAAS in Detroit, May 26-31, 1983.

The evaluation of heavy ion induced x-ray fluorescence (HIXRF) as a technique for the direct determination of the bulk chemical environment of an element in a solid has branched successfully in several directions. In the past year, we have explored methods to increase the sensitivity, expanded the range of elements which can be analyzed and initiated collaborative efforts to evaluate material science applications.

To increase the sensitivity of HIXRF, we examined the effects of using a heavier projectile for the excitation of target K α x-ray satellites at ORIC. The results indicated that the average L-shell vacancy fraction, i.e. the degree of multiple ionization in the L-shell, increased with increasing projectile atomic number. The relative change in the L-shell vacancy fraction, i.e. the chemical shift, remained constant with atomic number. More encouraging was the discovery that the cross section for x-ray emission increased by a factor greater than Z^2 . This means that in the absence of any loss of chemical sensitivity or significant decrease in projectile current density, the heaviest projectiles available for a given velocity should be used for excitation.

Because the sensitivity of the K α satellite lines to the chemical environment decreases as the number of shells between the K and valence shell increase, we focused our effort to expand

the range of elements which can be analyzed by evaluating the chemical shifts of other x-ray transitions. For example, the molybdenum L x-ray satellites produced by the bombardment of compounds and alloys with energetic chlorine ions from the EN-tandem displayed intensity variations indicative of the valence electron density of the material. Using molybdenum foil data as a subtrahend, amplification of those variations indicative of the valence electron density of the material. Using molybdenum foil data as a subtrahend, amplification of those variations was obtained by calculating difference spectra. Analysis of the spectra produced two significant observations. First, the larger the difference in the valence electron density of the compounds or alloys, the greater the variation in the difference spectra. Second, the difference spectra displayed an oscillatory fine structure. These oscillations appear to occur at energies corresponding to the $L_{2,3}^{M\alpha}$ satellite transition energies of molybdenum. Dirac-Fock calculations of the binding energies of multiply ionized molybdenum are being used to ascertain the transition energies and to aid the study of the oscillatory effect.

Based on the molybdenum results which demonstrated our ability to observe chemical shifts in alloys, we have begun a collaborative effort with the Metals and Ceramics Division to study x-ray chemical shifts in long range ordered (LRO) alloys. These alloys, of composition $V(Co_xFe_yNi_z)_3$, exhibit different physical properties such as crystal structure and ductility with small changes in composition. In addition to the LRO alloys we have begun to evaluate the $K\alpha$ and $K\beta$ satellite transitions of vanadium and titanium compounds and alloys. Preliminary results indicate that the $K\beta$ satellite lines are much more sensitive to the chemical environment of these elements than $K\alpha$ lines. Future experiments will continue to evaluate transitions which may increase the sensitivity and range of HIXRF and to explore potential materials applications.

(J. M. Dale, P. F. Dittner,* H. W. Dunn, L. D. Hulet, S. Kahane, H. J. Kim,* L. N. Klatt, H. F. Kruse,* P. D. Miller,* W. T. Milner,* S. Raman,* T. M. Rossel, C. R. Vane,* J. P. Young)

*Physics Division

2. MASS AND EMISSION SPECTROMETRY

J. A. Carter

Research, development, and support analyses are carried out in three topical areas: Inorganic Mass Spectrometry, Organic Mass Spectrometry, and Elemental and Secondary Ion Mass Spectrometry. Each area has many opportunities to develop techniques that support laboratory programs more effectively and to pursue research aimed at extending mass spectrometry into new areas. 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; analysis of special nuclear materials for various safeguarding applications, domestic and international; organic MS/MS and GC/MS analysis; isotopic abundance measurements of actinides and burnup analysis; and stable isotopes mass spectrometry.

Some 30% of our R&D support is received 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 programs. Another 10% of our R&D support comes through the Office of Safeguards and Security for domestic and international activities. The remaining support comes from several other laboratory programs and from various Work-for-Others programs, as, for example, the analytical efforts for NRC, waste isolation work at Hanford and Sandia, and support for the Three-Mile Island reactor cleanup.

RESEARCH IN INORGANIC MASS SPECTROMETRY

R. L. Walker and D. H. Smith

Resonance ionization mass spectrometry (RIMS) is being developed for potential use in isotope ratio measurements where isobaric (same mass) overlap between elements has been a problem for conventional thermal ionization. RIMS uses a finely tuned, high intensity laser to ionize atoms of a chosen element with high selectivity and without interference from other elements present in the sample. Proof of the selectivity of RIMS was obtained with the elements neodymium and samarium in an equimolar mixture. Using laser excitation at two different wavelengths, either neodymium or samarium could be measured with essentially no interference, whereas thermal ionization gave unacceptably large errors for certain isotope ratios. The RIMS technique has been used with other elements of interest, including cerium, praseodymium, europium, and plutonium.

To improve precision and accuracy of isotopic ratio measurements, two internal standards of uranium have been synthesized, characterized, and evaluated. One of the standards is used for isotope dilution measurements for uranium concentration; the other is used to refine $^{235}\text{U}/^{238}\text{U}$ measurements. Analysis of a number of isotopic standards and other samples whose content is well known led to improvements in precision of factors ranging from 3 to 7. In the $^{235}\text{U}/^{238}\text{U}$ measurements we are limited by the purity of enriched ^{236}U materials available to make the spike; they are all high in ^{235}U content. Relative precisions of about 0.1% are obtained with these new standards. An internal standard for plutonium is presently being developed.

Software programs have been written and tested for burn-up calculations. The ^{148}Nd (ASTM E-321) method was chosen for this work, but any neodymium isotope may be substituted for 148 if desired. The programs are written to correct for cerium at m/z 142 and for normal neodymium contamination in fission-produced neodymium.

The quadrupole mass spectrometer in its mobile laboratory made a number of field trips in the last year in support of the Oak Ridge Operations Office of DOE. Environmental losses were monitored at several DOE facilities. The Savannah River Plant was visited in support of the Office of Safeguards and Security, where

both uranium and plutonium in implant streams were monitored for accountability.

A new ion source for the quadrupole was designed and installed, resulting in an improvement in sensitivity by about a factor of 10. The instrument was remounted to reduce shock-induced problems during travel.

(D. L. Donohue, H. S. McLean, L. K. Bertram, C. A. Pritchard, R. L. Sherman, J. R. Sites, J. P. Young).

ORGANIC MASS SPECTROMETRY

G. L. Glish and P. J. Todd

Research has been initiated into thermal desorption/ionization of involatile and/or thermally labile organic compounds. Good success has been achieved with quaternary ammonium and phosphonium salts. It has been demonstrated that at low filament temperatures (<700°C) direct desorption of the intact cation occurs with no fragmentation. At high filament temperatures (>2000°C), thermal ionization occurs, again giving the intact cation with little or no fragmentation. Both processes can give very intense ion beams, suitable for application of the MS/MS technique. However, the higher temperature signal is usually more stable and is more reproducible.

The joint research project with Exxon involving pyrolysis/GC/MS of specially treated coals is essentially complete. Using a statistical model for hydrogen/deuterium scrambling it has been determined that on the average there are two radical sites per phenyl ring in a compound and an additional site for each methyl group. Also, several compounds were observed to be formed by complete methyl group transfer while other similar compounds existed in the coal structure.

A project studying the fragmentation of chalcogen-containing fatty acids and methyl esters (in conjunction with F. F. Knapp of Health and Safety Research Division) has been completed. High resolution measurements and several different metastable scanning techniques were performed with the MS50 to elucidate various decomposition pathways. In the course of this study B/E linked scan capabilities were added to the MS50.

Because Secondary Ion Mass Spectrometry (SIMS) involves the ionization of both analyte

and matrix, analysis is always a mixture problem. The key advantage to possessing a substantial secondary ion signal is that it permits use of MS/MS--a powerful mixture analysis technique. Thus, the structure of organic species can be determined in the presence of vastly more concentrated matrix. Species investigated to date include alkyl ammonium salts, arginine, phosphate esters and psychoactive substances, in addition to metal-organic ion complexes.

The organic secondary ion source is operational and being used with the MS/MS spectrometer to study the chemistry of secondary ion emission. We developed this source originally to generate secondary ions from involatile organic analytes. It has been quite successful, yielding ion currents of the order of $\sim 600 \times 10^6$ ions/sec at the detector after transmission through the three sectors of the MS/MS spectrometer.

Of particular significance is the observation that, in addition to a generally intense, intact cation, the dominant fragment ions formed from the analyte samples are ions which are formed by gas phase decompositions of the initially formed secondary ions. Such evidence indicates minimal surface damage to the organic analyte. A further observation concerns the matrix containing the analyte species. Solid matrices such as aluminum are substantially less productive than liquids such as glycerol. Surface concentration enhancement has long been studied by colloid scientists, and the effect is to be expected. Further enhancement by the matrix, e.g., by sulfuric or polyphosphoric acid, is being investigated. The interest here is in generating secondary ions from weak bases such as hydrocarbons.

(E. H. McBay, L. K. Bertram, H. S. McKown, W. H. Christie, D. H. Smith.)

ELEMENTAL AND SECONDARY ION MASS SPECTROMETRY

W. H. Christie

SIMS was used to help evaluate XeCl excimer laser annealing as a processing step in the manufacture of photovoltaic devices. Pulsed ruby and Nd:YAG lasers used for the removal of ion implantation damage in solid state devices have numerous disadvantages that have limited their commercial application. Among these disadvantages are: 1) spatial beam nonuniformity

resulting in local "hot" or "cold" spots; 2) low repetition rates for high energy pulses; 3) high spatial coherences, which cause interference effects ("speckle") extending over the entire illuminated area.

In contrast, workers in the Solid State Division (SSD) have found pulsed excimer lasers to provide relatively large rectangular annealing areas within these areas the pulsed energy density is highly uniform at high repetition rates. The purpose of the SIMS study was to examine dopant redistribution in terms of the required laser threshold energy density. These studies would help establish the optimum energy density for annealing of ion implantation lattice damage, and allow us to examine the effect on dopant distribution resulting from the use of multiple overlapping pulses at high repetition rate in a scanning mode.

Boron was implanted at 5 and 20 keV and arsenic at 20 and 110 keV in single crystal silicon corresponding to projected implantation ranges of 16 and 65 nm. Dose levels for both dopants were 2 and 6×10^{15} ions cm^{-2} . SIMS measurements of redistribution following pulsed excimer laser annealing showed a first observed departure from the as-implanted dopant profile at 1.15 J cm^{-2} for boron and at 0.98 J cm^{-2} for arsenic. Model calculations by SSD personnel predict 1.03 J cm^{-2} for melting to a depth of 50 nm in crystalline silicon. This result is in good agreement with the SIMS measured boron redistribution to a depth of 50 nm at 1.15 J cm^{-2} .

The smoothness of the silicon surface after excimer laser annealing allowed unusually high depth profiling resolution to be preserved down through the back side of the profile. This improved resolution made it possible to observe a slope change in the profile that corresponds directly to the melting depth during laser annealing.

In a collaborative program with the National Bureau of Standards, some aspects of resin bead-enhanced thermal ionization of uranium were studied. This project involved a truly national collaboration as the secondary ion mass spectrometer (CAMECA IMS-3f) was located at the Bureau, the thermal ionization stage for this instrument was borrowed from the Solar Energy Research Institute in Golden, CO, and an ORNL worker traveled to the Bureau to participate in the research.

Initial experiments attempted to visualize in real time the thermal emission of U^+ ions

directly from resin beads loaded on a rhenium filament, using the CAMECA instrument's position sensitive electro-optical detector. However, the ion currents produced from the 150 μm diameter fields of view were too low to produce such images. Photographic integration was required. The most interesting experiment utilized the combined thermal/SIMS mode of operation. The distribution of uranium around a resin bead was studied as a function of temperature. At low temperatures the uranium signal was localized to the resin bead and the area immediately around it. As the temperature was increased, it was observed that the uranium covered a wider and wider area around the bead and was diffusing away in a nonuniform manner. In this study two dynamic processes were occurring simultaneously: the diffusion of uranium away from the resin bead and sputtering of the surface.

These experiments are extremely provocative. They suggest that the resin bead does not provide a point source of uranium ions for a mass spectrometer as previously envisioned. The bead does appear to provide a temperature dependent, diffusion controlled release of neutral uranium atoms, which have high mobility on the surface of the rhenium filament. Many future experiments can be envisioned to utilize the unique capabilities of the CAMECA ion microscope to investigate fundamental processes of thermal and secondary ionization.

In collaboration with researchers at the University of Tennessee Chemistry Department, we have used SIMS to study the concentration vs depth distribution of anions and cations electrolytically deposited into thin-organic-film modified electrodes. Tetracyanoquinodimethane (TCNQ) modified platinum or gold electrodes were prepared using a mixture of oligomers in which the TCNQ electron exchange sites are incorporated into the polymer's molecular backbone. In TCNQ this results in electron rich sites throughout the depth of the polymer film that can act as cation acceptor sites. Early experiments used sodium and potassium perchlorate solutions from which either cation was electrolytically deposited into the film. Early SIMS work demonstrated the unacceptability of the ubiquitous Na^+ and K^+ cations from a contamination standpoint and less common cations (Li^+ and Rb^+) were used in subsequent work. Depth profiling of these materials demonstrated a difference in the Rb^+ concentration gradient for a film reduced at -0.3 V and for the same film which was then oxidized. Although we are

currently analyzing "raw" SIMS data, it appears that the $(\text{TCNQ-Rb}^+)_x$ film has been oxidized in an outside-inside fashion; i.e., the surface of the film in contact with the electrolyte solution has been depleted in Rb^+ upon being cycled back to the initial potential (oxidized). This is consistent with rapid electron hopping and slow ion migration in the charge transport process. The neutral "virgin film" gave Rb^+ signals approximately 100 times lower than the exposed film.

These early experiments are interesting and have turned up a number of phenomena that will be investigated in future work. One exciting possibility is that the polymer film electrochemistry technique may also prove to be of value to the surface scientist since it might provide a means of fabricating standards useful in quantitative biological "tissue" studies.

(R. E. Eby, L. Landau, P. J. Todd).

3. RADIOACTIVE MATERIALS ANALYSIS

W. R. Laing

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

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

PROGRAMS AND PROJECTS

D. A. Costanzo

High-Temperature Gas-Cooled Reactor Program
Particle Technology Studies

A method has been developed to measure micro- and meso-pore volumes and surface area distributions in solids, especially in reactor grade core graphite samples. Nitrogen adsorption-desorption isotherms showed hysteresis loops characteristic of porous materials. From these isotherms and applications and the theories of a number of specialists writing in the literature, we have been able to write computer programs to calculate and plot pore surface area and volume distributions in the range of 1 to 100 nm for the hydraulic pore radii. Above 100 nm the pore distributions were determined by mercury porosimetry.

A continuing program of determining BET surface areas and helium densities of solid samples has been pursued. This has included the improvement of the apparatus and the computer software to make hardcopy of the data in tabular and graphical form. The samples analyzed have been both solid cylinders and powders.

Great Lakes-grade graphite for the high-temperature gas-cooled reactor (HTGR) has been submitted to oxidation testing to simulate the effect of an inleakage of moisture in the secondary helium coolant flowing in the HTGR. The analytical techniques used to characterize oxidation effects upon the pore structure of the graphite include BET surface area analyses by nitrogen adsorption at 77°K, helium pycnometry, and mercury porosimetry. Changes in accessible and inaccessible void volumes with oxidation of the graphite by water vapor in helium at 900-1000°C were demonstrated. Significant changes in BET surface areas were also noted as the oxidation increased. Changes in pore-size distributions were determined from gas adsorption and mercury porosimetry isotherms. Evidence of catalytic oxidation was noted. The surface area and helium pycnometer apparatus is a fully automated system controlled by an HP9835A microcomputer. The computer also processes the data, stores it on tape, calculates the surface areas and helium densities with appropriate statistics, prints hardcopy, and plots isotherms using an HP9872A plotter. Mercury porosimetry data are also processed by the computer, and pore distribution curves are charted by the plotter.

(D. A. Lee and J. M. Keller)

Thermal Analyses

Miscellaneous samples have been analyzed by thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) analysis. These included uranyl nitrate solids from uranium processing lines, cesium chloride and nitrate and technetium oxalate. Durez resin samples were analyzed by evolved gas analysis using MS.

(D. A. Lee and J. M. Keller)

Software Development for ICP

Numerous new features were added to the inductively coupled plasma (ICP) spectrometer software, these included utility programs for monitoring system performance and tabulation of data, automatic update of calibration curves, enhancement of techniques employed for interference corrections, and improvements in data formatting for customer's print-outs. Programs were developed for the direct transfer of data from the ICP computer to the Laboratory DEC-10 System for integration into the Data Management System (DMS). TSX-Plus, a multi-user operating system, was fully implemented in the ICP computer.

Preliminary investigation into simultaneous multispectrometer operation of the ORNL ICP were completed. A satellite microcomputer (AIM-65) was used to control and take data from the ICP scanning monochromator. The microcomputer communicated directly with the main ICP computer (LSI-11/23) which would allow combining data from both the polychromator and monochromator. Continued work on this project will require development of software to integrate the multispectrometer functions into a fully operational package.

(J. M. Keller)

XRF Development

Working with C. J. Sparks, Jr. (ORNL Metals and Ceramics Division), we have designed an x-ray fluorescence (XRF) system capable of quantitatively detecting less than 0.4 mg/mL of uranium, plutonium, or other elements in a high gamma radiation active background. A basic feature incorporated into the XRF spectrometer is simplicity. This design employs pyrolytic graphite for the x-ray optics; this has the advantages of being highly resistant to

radiation and having high diffraction efficiency. Because of the nearly ideal mosaic structure of highly oriented pyrolytic graphite, the entire energy region from PuL_{α} to UL is diffracted simultaneously with good efficiency and sufficient wavelength dispersion to permit the use of fixed optics in the spectrometer. The excitation source for the XRF system uses a graphite barrel monochromator to provide sufficient intensity with a standard diffraction x-ray tube to achieve the quantitative detection limits of <0.4 $\mu\text{g/mL}$ for uranium and plutonium.

For proof-of-principle testing, a laboratory version of the XRF design has been assembled with borrowed components. Preliminary investigations with this laboratory version indicate that it will have an energy resolution of 10-20 eV and will exceed the original sensitivity criteria of 0.05 cps/ppm uranium or plutonium by a factor of five or more. Current work on this project is directed toward the development of a prototype system to be located in a hot-cell at building 2026. Once the prototype is located in a hot-cell, the system performance will be evaluated with uranium and plutonium determinations on spent nuclear fuel processing samples.

(J. M. Keller)

Multi-element Analyses by ICP-AES

The inductively coupled plasma atomic emission spectrometer (ICP-AES) continues to provide support for a variety of Environmental Sciences Division programs concerning trace metals in the environment. Routine monitoring of local ponds, streams, and laboratory effluents for trace and toxic metals has been very successful.

An investigation of the uptake and distribution of trace metals in different types of trees is another project that relied heavily on the cost effective, multi-element capability of this technique. The concentration of various metals in tree rings provides a chronological record of the abundance of these elements in the environment. One goal of this study is the determination of the effects of coal-fired steam plants on trace metal uptake in surrounding vegetation such as trees. The tree ring core samples are prepared by low temperature ashing prior to analysis by ICP-AES; approximately 30 elements are determined at concentration levels below one part-per-million.

The leachability of toxic substances from municipal waste sites into ground water supplies is an EPA-funded project that also depends upon

the cost-effective, multi-element capabilities of the ICP-AES. Simulated matrices prepared from a number of organic acids were used to leach materials from various waste sites. The leachates were analyzed directly by ICP-AES to determine a number of trace metals.

Several other research projects were supported by the ICP multi-element analyses technique. Two of the more difficult samples, in terms of both sample preparation and analysis, are the mineral and shale under study by geologists interested in the geochemistry of organic-containing rocks, especially Chattanooga shale. The very large concentration of organic compounds along with inorganic complexes, which are difficult to dissolve, required a multistep digestion procedure. Wet ashing in concentrated HNO_3-HClO_4 followed by digestion in concentrated $HF-HNO_3$ was used to prepare solution samples for analyses.

(D. R. Reine and J. C. Price)

RADIOACTIVE MATERIALS ANALYTICAL LABORATORIES

D. A. Costanzo

X-ray fluorescence analysis continued to be applied to many types of samples. During this reporting period, 2000 XRF determinations were made compared with 1000 during the previous period. The majority of sample types were aqueous and organic solutions containing uranium and plutonium; other samples were analyzed for thorium, zirconium, technetium, molybdenum, and rhenium. Additional applications included analysis of plating bath solutions, organophosphorus extractants, neutron poisons (cadmium and gadolinium) in U-233 solutions, and qualitative scans of deposits, residues, and alloys.

Ninety-six solutions containing statistically varying amounts of uranium, plutonium, and acid were prepared for use in a matrix study on the x-ray fluorescence analyzer. All components were measured by both volume and weight, an internal standard was added, and the x-ray fluorescence was measured. Statistical evaluation of the data is in process.

A software program for XRF matrix correction has been obtained from the manufacturer for use with our instrument. Initially, the program could not be loaded because of insufficient memory capacity. After some hardware changes were made to provide additional memory, the program was successfully loaded. Now problems within the program will not allow proper

execution, the manufacturer is assisting in finding the source of this error.

To provide uranium determinations with a precision of 0.5%, we routinely use the Davies and Gray volumetric procedure. Recently, a need has arisen for more precise measurements with a relative standard deviation of 0.05%. The procedure has been changed to a gravimetric basis by weighing sample aliquots and the dichromate solution used in the titration. Careful preparation of reagents and calibration with NBS standard reference materials were necessary to ensure freedom from bias.

At the Three Mile Island reactor site, clean-up continued with the processing of water from the containment building. Fission products were removed by passage through zeolite and ion exchange columns. Chemical, radiochemical, and mass spectrometric assays were performed before and after treatment to determine the efficiency of the removal process.

The design basis accident (DBA) apparatus is used for the testing of nuclear coatings (paints) under simulated reactor accident conditions. Early this year it became increasingly difficult to duplicate the standard temperature curve (75 to 170°C in 20 s) in the initial phase of the test. Replacement of several steam valves produced no improvement. Insulation was removed from the autoclave, and the resistance heaters were checked. As a result, additional heaters were added, and replacement insulation was increased from 8 to 15 cm. To permit easier removal of condensate, an improvement in valving to the condensate leg of the steam lines was made. These improvements were successful, and the standard 170°C DBA curve was obtained.

Several new pieces of equipment have been installed: a remote pipet for hot-cell use, two electronic balances, a glove-box fume scrubber, a programmable hot plate-stirrer, and an environmental cabinet to house the XRF electronics.

The planned second-story addition to building 2026 is at the 90% design review level. The current schedule calls for the contract to be bid in December and awarded in March 1983; construction would begin in April.

Other work in the Radioactive Materials Analytical Laboratories has been of a general nature and has included tests of solvent extraction equipment, dissolution of 3.7×10^{13} Bq of ^{65}Zn for radiochemical analysis, and preparation of ^{240}Pu , ^{241}Pu , and ^{242}Pu solutions for analysis. Samples from the gunnite tanks in the old tank farm have been

processed for density, total solids, acid, anions, and radiochemical scans.

(R. P. Apple, J. L. Botts, R. D. Brooksbark, R. E. Jones)

CFRP Support Laboratory

Approximately 4000 analyses were performed by the satellite service laboratory in building 7501 in support of Consolidated Fuel Reprocessing Program (CFRP) process and engineering studies. The samples were generated in fuel dissolver, iodine trapping, nitric acid recycle, and NO_x off-gas studies. Analyses included free acid by potentiometric and thermometric titrimetry, alcohol by refractometry, iodine by volumetry and spectrophotometry, oxides of nitrogen by acidimetry and spectrophotometry, and solids by gravimetry. In addition, a laboratory study was completed in the density of the $\text{Mg}(\text{NO}_3)_2\text{-HNO}_3\text{-H}_2\text{O}$ system over the range 30 to 70 wt % $\text{Mg}(\text{NO}_3)_2$, 0-15 wt % HNO_3 and at 50 to 150°C. The $\text{Mg}(\text{NO}_3)_2$ system is being considered for the recycle and production of high-concentration (>90%) nitric acid.

(F. L. Layton and J. L. Marley)

TRANSURANIUM ANALYSIS LABORATORY

J. H. Cooper

The Transuranium Analysis Laboratory (TRU) provided support primarily for the production of transplutonium elements. This year the sample load decreased 7% while the staff decreased 9%. The major effort was directed toward transuranium processing campaigns in which about one gram of ^{252}Cf was produced. More than one third of the analyses were for the Operations Division in support of waste monitoring.

This year many special short-term projects were undertaken. For measuring uranium concentrations in power reactor processing solutions, we sought a method whereby smaller amounts of radioactive sample could be used but which would still provide the accuracy needed. A method reported by R. T. Chitnis et al was investigated and modified for use. To determine plutonium in solutions containing greater than 5 g-L^{-1} , an amperometric titration method using a recording titrator fitted with a polarizer was adopted.

Samples of wastes for injection into the Shale Hydrofracture Facility were submitted for determination of heavy elements. The samples were highly contaminated with long-lived fission products and had to be diluted before they could be analyzed. The levels of alpha activity from plutonium, americium, and curium were of the order of 5-100 Bq-mL⁻¹ in these dilutions. Anion resin exchange, accompanied with solvent extraction, was used to separate the various nuclides of interest. All aliquots were spiked with tracers to determine the recovery yields.

In TRU processing, fission product zirconium is removed from process streams by scrubbing with oxalic acid. In order to use this radioactive zirconium in tracer studies, the oxalate complex must be destroyed. In the tested method a solution of 4 M HNO₃, saturated with oxalic acid, was heated to 80-90°C. Hydrogen peroxide (30%) was added at a rate of 50-60 mL-h⁻¹. Samples were taken hourly and analyzed for nitric and oxalic acids. These analyses showed that the oxalic acid was destroyed after 5 h.

and the results are calculated. A chemist then reviews the results on a video screen and authorizes their transfer to the DMS.

The Analytical Chemistry Division Analysis Catalog is now available to all PDP-10 users. This feature will be useful to ACD customers for the determination of availability and cost of analytical measurements.

A new format has been developed to allow the inclusion of text (comments) with a customer report. An existing text-editing program, SOS, was used to collect, edit, and store the written information. When the customer report is completed, the text information is printed along with the individual results and will also appear on the weekly microfiche cards.

(W. R. Laing, J. M. Keller, P. A. Tilson*)

*Computer Sciences Division.

DATA MANAGEMENT SYSTEM

This year work began on the analysis of special samples under an WRC contract. In order to facilitate the preparation of reports required by WRC, a separate area (lab 2), within the Data Management System data bank, isolated from ORNL work, was assigned to this project. The reporting system has now been evaluated and is in full operation. An additional report format, developed for compiling WRC results, allows the inclusion of additional material such as results on standards, relative standard deviations, and comments on samples.

We have written and tested a new program to transfer sample results from the DMS to customer files. It is more general and more easily adapted to customer format than the previous one.

Recently, current work order listings became available on the PDP-10. These files are now included in the timekeeping program along with current charge accounts. All customer charges entered through the timekeeping system are checked for valid accounts against this list, which is updated weekly.

Computer Sciences Division personnel have completed their part of the programming for data transfer from the ICP system to the DMS. In this approach, samples are processed by the ICP,

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 of analysis. Applied programs emphasize environmental and health issues associated with human exposure to smokes, with synthetic fossil fuels technologies, and with environmental pollution. Approximately 30 people, including 20 professionals, are engaged in work supported by the Environmental Protection Agency, Electric Power Research Institute, National Cancer Institute, Department of the Army, and the Department of Energy.

ANALYTICAL CHROMATOGRAPHY

W. H. Griest

Gas and liquid chromatographic methods development and applications to environmental and synfuels material are the main activities underway in this group. Smaller efforts address fundamental analytical research in liquid chromatography, and advanced organic analytical services for in-house clients. The main projects in this group are funded by the U.S. Department of Energy, the U.S. Environmental Protection Agency, and the Electric Power Research Institute. The National Cancer Institute and the Department of Defense also have supported work.

Fundamental analytical research included studies of open tubular liquid chromatography (OTLC). An efficient capillary column was drawn and coated, and techniques for sample injection and UV absorbance detection of separated components were developed. On-column laser fluorescence detection also was devised and was shown to provide femtogram sensitivity for certain classes of compounds. The columns in current use are complementary to those employed in open tubular GC, providing increased separation power at the expense of analysis time. For example, all three ethylphenol isomers can be separated with near baseline resolution in a six hour run. Development of an orifice for OTLC/MS has begun. Studies have been initiated to take advantage of electrokinetic phenomena (electroosmosis and electrophoresis) as applied to OTLC. Electroosmosis, rather than hydrostatic force, has been used to migrate mobile phases in open tubes. Electroosmosis provides a flat flow profile in contrast to the parabolic profile of hydrostatic flow. This can lead to substantially higher efficiencies and can greatly reduce instrumental constraints. Electroosmotic flow has been used with mechanically fragile stationary phases (e.g., Sephadex LH-20) to achieve three to four times higher linear velocities than that by gravity flow.

Studies of the interaction of organic compounds with highly sorptive particulate samples are being conducted to determine the cause of low analytical extraction recoveries of some PAHs. Benzo(a)pyrene spiked onto coal fly ash is found to be incompletely extracted and to partly degrade into more polar species. However, by use of radiotracers we are able to quantitatively correct for the decomposition.

The sorptivity of fly ash for PAHs appears to be associated with carbonaceous particles, but the mineral portion of fly ash also may be active in PAH sorptivity and decomposition. Serious discrepancies between static sorptivity measurements and analytical extraction recoveries suggest that how the PAHs are contacted with the fly ash surface may critically control the surface interaction.

Acids have received particular attention in HPLC methodology development and applications. C₁-C₈ carboxylic acids, benzoic acid, and phenols are determined by reversed phase liquid chromatographic procedures utilizing conductimetric, ultraviolet absorbance, and fluorescence detectors, respectively. The methodology includes efficient solvent partitioning methods for extraction of the analytes from aqueous and organic liquid matrices.

The normal phase semipreparative scale HPLC method described in last year's report has been automated and incorporated into a sequential HPLC/HPLC or HPLC/GC method for the fractionation and analysis of complex mixtures. The fractionation procedure has been extended to include nitro-PAH, but was found to suffer from co-elution of oxygenated-PAH. Chemical derivatization of the oxygenated-PAH was only partially successful in facilitating the resolution of these two PAH classes.

A high performance size exclusion chromatography capability was set up, and found to offer a very useful adjunct to partition chromatography. We are the first group to use this technique to examine solvent extracts of air particulate matter and coal fly ash for "gas chromatographic intractable matter". Constituents with molecular weights up to approximately 500 AMU were found in the former, and up to 900 AMU in the latter.

Developmental activities in GC included sampling and thermal desorption GC methodology for the collection and analysis of semivolatile or volatile organic species in a wide range of matrices, from cigarette whole smoke to coal liquid headspace vapors and coal fly ash. A dual Tenax/charcoal adsorbent trap was found to provide improved collection efficiency for volatile organic compounds in workplace atmospheres, and yet allow the sensitive determination of compounds with boiling points up to approximately 300°C.

A GC service laboratory was set up this year to consolidate GC activities and more efficiently handle the increased commitment to

components of coal liquids. Carcinogenicity is known to be associated with the polyaromatic components of the neutral fraction. Mutagenicity is found to be concentrated in the moderately polar component of the neutral fraction. This neutral fraction has been further separated by high performance preparative liquid chromatography to yield a highly mutagenic subfraction that is enriched in polyaromatic carbonyl compounds. Although this subfraction accounts for only 0.14% of the original synfuel product, it was so enriched in carbonyl content that the presence of benz(de)anthracenone, C₁-benz(de)anthracenone, C₂-benz(de)anthracenone, benzo(cd)pyrenone, and C₁-benzo(cd)pyrenone was established.

Carbonyl compounds also appear to be important teratogens as indicated by results generated in the ongoing study to isolate the teratogenic components in selected lots of commercially-available acridine. In this case, the combined use of both normal phase and reversed phase high performance preparative liquid chromatography effected the isolation of a single component from the acridine impurity fraction. This single component [benz(g)quinoline,5,10-dione] appeared to account for much of the teratogenic activity even though it was present at the 20 ppm level in the original product. Although this compound caused gross teratogenesis in crickets, its isomers such as benzo(g)quinoline,5,10-dione and benzo(h)quinoline,5,6-dione show significantly lower and no teratogenic activity in crickets. Such results confirm that subtle differences in molecular structure may give rise to significant differences in bioactivity.

During this year, a project to characterize potential synfuel process streams was initiated. The beginning approach was to "scope" a maximum number of treatments in order to ascertain any process which might improve the quality of the final product. To this end, a wide range of treatments was surveyed. Preliminary results have indicated that processes such as extraction with certain solvents, AlCl₃ chemical alteration, acid washing, and high boiling point compound removal may be quite promising approaches to improving quality of the fuels while at the same time reducing potential bio/environmental hazards. Future efforts on this study will be directed toward the detailed characterization of refinery process streams with the objective of

defining procedures that can minimize the environmental risk associated with the utilization of these products.

Applications of diffuse reflectance infrared fourier transform (DRIFT) spectrometry were continued. A catalog of the DRIFT spectra of pure compounds was assembled. Similarly, the spectra of a series of synfuel products representative of different processes and treatments have been collected. Using unbiased computational techniques, the relative intensity of several spectral regions (1600 to 1900 cm⁻¹, 750 to 900 cm⁻¹, and 1525 to 1600 cm⁻¹) appeared to be highly associated with observed mutagenic activity. DRIFT spectra were also utilized to easily distinguish several different sets of isomers including o-, m-, and p-phthalate esters, six different acridine (C₁₃H₉N) isomers, and three different acridine dione isomers (C₁₃H₇O₂N).

Nuclear magnetic resonance (NMR) has been used extensively this year. Carbon and proton NMR has been used routinely to confirm the structures of synthesized compounds, and also in the identification of trace components isolated from biological matrices. For example, a compound isolated from the retina of drosophila (fruit fly) was unambiguously identified using sub-milligram quantities of the compound. Phosphorus NMR was used extensively in the qualitative and quantitative studies of phosphate species trapped on glass filter pads from phosphorus combustion studies. Fluorine NMR has been used for the quantitative analysis of primary, secondary heterocyclic and secondary N-alkyl substituted amines in complex mixtures.

A number of special projects were completed this year. Included are the synthesis and characterization of six different phthalate compounds for a toxicity study carried out elsewhere; the isolation and detection of amines from water samples associated with synfuel processes which aided in the explanation of a corrosion mechanism by the metallurgists; and the characterization of two colored smokes used in military obscurants to complete a study supported by the Department of Defense.

Spectroscopic services in support of Laboratory-wide programs has continued with some small growth. Data and in some cases interpretation have been provided to several divisions, including Health and Safety Research, Biology, Industrial Safety and Applied Health Physics,

organic analytical services. GC methods for determining volatile organic solvents were used extensively to monitor stream and well water samples for the Laboratory Environmental Coordinator's Office, and to determine selected constituents in solid waste leachates. Routine methods employing GC also have been set up for determining phthalates in aqueous toxicity exposure systems, and decachlorobiphenyl in tissue samples. Requested determinations of volatile indoor air pollutants were facilitated by methodology developed earlier for the sorbent resin collection and thermal-desorption GC analysis of vapor phase atmospheric pollutants.

Research sample management also has been an important continuing activity of this group, supporting several projects and programs. The direction of the Synfuels Repository has now changed to the careful archival and stability characterization of bulk drum quantities of several synfuels. Included are coal liquids from the H-Coal, EDS, and SRC-II processes, a shale oil, a petroleum crude oil, and diesel fuels refined from petroleum and also shale oil. Selected samples are being analyzed for bioactive constituents, including PAH, phenols, and nitrogen-containing aromatics.

(J. F. Attrill, B. D. Barkenbus, S. H. Harmon, R. W. Harvey, C. E. Higgins, D. L. Manning, M. P. Maskarinec, R. R. Reagan, M. E. Roth**, B. A. Tomkins, J. D. Vargo+, and L. B. Yeatts)*

SEPARATIONS AND SPECTROSCOPY

J. E. Caton

Research and technical support functions are carried out in the areas of gas chromatography/mass spectrometry, nuclear magnetic resonance spectrometry, and infrared spectrometry. Preparative scale separation procedures are developed and applied to complex organic mixtures in order to identify bioactive constituents and to support biological/environmental testing in collaborating Laboratory divisions. Organic compounds are synthesized to produce standards and compounds for biological testing.

The research on chemical ionization techniques to distinguish isomeric compounds by mass

spectrometry was continued. Methanol and methanol-d₄ have been shown to be useful in distinguishing isomeric oxygen-containing compounds. When the methanol spectrum of a compound is compared with its methanol-d₄ spectrum, a change of two, one, or zero mass units unequivocally identifies the compound as a phenol, aromatic ether, or aromatic substituted alcohol. Methanol chemical ionization has been extended to the differentiation of isomeric sulfur containing compounds as well, and isomeric thiophenols, aromatic thioethers, and aromatic substituted thiols can be distinguished by changes in mass of two, one, or zero mass units, respectively. Additional studies have been conducted to address questions on the effects of pressure and concentration on chemical ionization as well as to test the selectivity of the chemical ionization reagent in the analysis of complex mixtures.

The methanol chemical ionization technique used in conjunction with the previously developed ammonia chemical ionization was systematically applied to two subfractions from a series of four coal oils. The crude oil and low, medium, and high severity hydrotreated products of the oils were studied to determine whether hydroreatment resulted in a selective reduction of a few components or a general reduction of all components. For all compounds identified and monitored including primary aromatic amines, aromatic ethers, azarenes, and phenols, there was a general and uniform decrease in concentration with increasing hydrotreatment; but no selective reduction was observed.

Past studies have shown that nitrogen-containing aromatic compounds contribute significantly to the biological activity of fossil derived fuels. In addition, the activity of a compound often has been found to be dependent not only on the type of nitrogen substitution within the compound, but also on the overall geometry of the molecule and the position of substituents. For example, *β*-naphthylamine is more active than *α*-naphthylamine. In order to get more definitive identifications of these isomeric compounds, a mass spectral library of nearly two hundred nitrogen-containing compounds is being compiled. The electron impact spectra as well as the gas chromatographic retention indices will be compiled as an aid in the identification of isomeric nitrogen containing compounds.

Research to identify mutagens, carcinogens, and teratogens has focused on the neutral

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Environmental Sciences, Chemistry, Chemical Technology, and Metals and Ceramics.

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SPECIAL PROJECTS

R. W. Holmberg

The efforts of this group have centered on studies of the chemical and physical properties of dense aerosols and on techniques to generate them for toxicological studies. Principal projects include continued studies of cigarette smoke sponsored by the National Cancer Institute and a Department of Defense (DOD) sponsored study on the toxicology of military obscurants. Activities include routine analyses of cigarette smoke for selected organic constituents, methods and instrumentation development to more adequately generate and characterize the aerosols, inorganic and organic chemical studies on the nature of the smoke produced from burning phosphorus formulations, and chemical analyses to characterize a broad range of starting materials used in these projects.

Development work on the extrusion-combustion generator for producing phosphoric acid aerosols from a military red phosphorus-butyl rubber (RPBR) formulation have been completed. This generator produces the aerosol continuously and at uniform concentration for several hours such as is required for inhalation toxicology studies. Five of these generators plus aerosol monitoring equipment developed here have been constructed and installed at the Illinois Institute of Technology Research Institute for their use in a DOD sponsored inhalation toxicology program.

Both the phosphorus aerosol and the starting RPBR are being characterized chemically to identify constituents which may have toxicological significance in the exposure studies, to identify those parameters which are most useful for monitoring the exposure environment, and to define the starting material. Both the particulate phase and the vapor phase of the aerosol have been examined. Various polyphosphate species up to the P_{13} polymer have been determined in the particulate phase by HPLC

using a flow injection analysis (FIA) as a specific post-column detector. The FIA system is also used directly for the rapid batch analysis of total phosphate. Methods of handling and storing particulate samples that preserve the integrity of the linear polyphosphates have also been determined. The composition as a function of aerosol concentration was found to remain constant. Gas chromatographic analysis of the vapor phase provides some evidence of the presence of organic phosphates. The major organic components present in the vapor phase are found to be C_6 hydrocarbons. The concentration of these hexane isomers is well below permissible exposure limits.

Active chemical and physical support continues to be given to two programs assaying the toxicology of military petroleum-based smokes. One, in the ORNL Biology Division, is investigating diesel fuel smokes. The other, at EPA, Research Triangle Park (RTP), is investigating a higher boiling petroleum fraction, Fog Oil.

Levels of respiratory tract deposition of the diesel fuel smoke were determined for the various smoke concentration/time conditions under which the animals were exposed. This was accomplished by adding an inert tracer, deca-chlorobiphenyl (DCBP), to the fuel prior to aerosolization, and measuring the amount of tracer in the appropriate organs. The studies indicated that virtually all of the respiratory tract deposition occurs in the lungs. Also, for a given exposure duration, animals exposed at higher smoke concentration had higher levels of tracer in their lungs. This particular technique should be applicable to deposition studies with other petroleum based aerosols.

Physical and chemical studies of the diesel fuel smoke were virtually completed this year. It was found that, as smoke concentration increased, the average smoke particle size increased and the proportion of fuel remaining in the vapor phase decreased. We could find no compounds present in the smoke generated under laboratory conditions that were not present in the original fuel. However, smoke generated with oxygen containing carrier gas (so as to more closely mimic diesel exhaust gases) contained 100 ppm of carbon monoxide and 5 ppm of NO_x . These levels were not considered to be toxicologically significant, in light of the contribution of normal exhaust gases to the concentrations of CO and NO_x .

High resolution gas chromatography employing direct, on-column injection of tri-methyl

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silylated cigarette smoke particulate matter appears to be very promising for the reproducible, semiquantitative profiling of "tar". An initial survey of commercial cigarette brands uncovered few significant differences in the relative proportions of several of the major tar constituents. We have recently acquired a 20-port analytical smoking machine, designed to increase sample throughput and improve quality control in smoke room operations. Verification of its accurate smoking performance has been a more lengthy procedure than originally envisioned.

Projects addressing human smoking issues have centered around development of hardware to generate or characterize cigarette smoke under nonstandard puffing conditions. A variable puff smoking machine, which will smoke cigarettes at any desired puff profile and set of parameters, has been designed and constructed. Initial programming and validation studies are now underway. In addition, an instrumental cigarette smoke monitor, which simultaneously measures smoke flow rate and concentration immediately downstream of a cigarette butt, has been developed. The device quantitatively determines the amount of smoke that an individual actually generates on puffing a cigarette. An extensive effort to determine smoke retention in laboratory animals culminated with the analysis of blood samples from over 200 smoke exposed beagles for nicotine and its major metabolite, cotinine. Although animal-to-animal variation was large, blood nicotine levels increased with increasing exposure to cigarette smoke. This increase, however, was not directly proportional to either the amount of nicotine in the smoke or the number of cigarettes smoked.

Field sampling is an important focus of the Special Projects group. We have continued to provide guidance and hardware in support of studies to determine the composition of air in the home (Health and Safety Research Division). Two trips were also made to the K-Coal Pilot Plant in Catlettsburg, KY, (Synfuels Life Sciences Program) to obtain vapor and particle phase samples of point source fugitive emissions and high volume ambient air samples from several locations within the plant. In addition, one trip was made to the Veterans Administration Hospital, East Orange, NJ, to conduct blood sampling experiments to determine the time course of nicotine and its major metabolite, cotinine, in beagle dogs following cigarette smoke exposure.

Studies initiated last year to make greater use of computer-based data acquisition and manipulation have continued. A prototype of our portable data logger for peak producing instruments has been completed and is being laboratory tested. It is based on an AIM-65 single board microcomputer acquiring data through a 12 bit analog to digital converter and uses a Phillips type minicassette for temporary data storage. Each minicassette tape will hold about 20K data points, sufficient for example, for acquiring approximately 6 hours of data from a gas chromatograph. Interfacing and programs have been developed to transfer this to a larger computer for archiving, plotting and analysis. Programs have been developed on the LSI 11/23, RT11/TSX+ sectional computer system to rapidly and interactively prepare viewgraphs and data plots using a HP 7221C plotter. A virtual terminal program has been developed so that any of the four terminals of the system can communicate with the DEC System 10 and other compatible Laboratory computers. ASCII data files may be automatically transferred to and from these computers.

(R. S. Brazell, T. M. Gayle^a, D. E. Goeringer, R. A. Jenkins, J. H. Moneyhun, G. M. Henderson, D. D. Pair, and S. K. White)

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5. GENERAL AND ENVIRONMENTAL ANALYSIS

J. R. Stokely

The General and Environmental Analysis Section is composed of five groups: Chemical and Physical Analysis, Environmental Analysis, Activation Analysis, Low-Level Radiochemical Analysis, and Special Projects. The section supports a large number of DOE programs at ORNL and other Nuclear Division facilities. A small amount of work is also done for other government agencies and private corporations.

The section's work is quite varied and involves application of physical and chemical techniques to obtain analytical results and/or to solve problems. Both instrumental and classical chemical methods are utilized. Inductively coupled plasma spectroscopy has been put into routine operation this year to complement existing multielement analytical techniques such as neutron activation analysis. A new transmission electron microscope is being put into routine use to improve physical analysis capabilities. The Special Projects group is involved primarily in developmental work related to the measurement of radionuclides at ultra-low levels for environmental studies. Other development activities within the section are directed at improvement of capabilities and/or introduction of new techniques.

CHEMICAL AND PHYSICAL ANALYSIS

J. H. Stewart

The Chemical and Physical Analysis group provides a diversified base of technical support for both the Laboratory and other approved agencies. During this year; approximately 70,000 analyses were completed with fewer staff members than the previous year.

The data management and sample transaction system permitted control of the large numbers of specimens and accurate data transmittal. The very large data banks produced by the inductively coupled plasma spectrometer (ICP) are now transferred directly from the ICP computer into the ORNL DEC-10 computer. This significantly reduces transcription errors, reduces the cost per analysis, and increases the speed of reporting the data.

The ICP has revolutionized the ORNL trace metals analysis program. It made possible multiple metal analyses for the ORNL surface water characterization program, which required some 10,000 individual determinations. The same instrument provided multielement analysis of iron-cement aerosol particles collected during a nuclear safety evaluation test. The ICP is now the major instrument for multielement trace metal routine analysis of both surface waters and well waters from test wells that monitor ORNL burial grounds.

In the area of physical analysis, x-ray diffraction measurements provided insight into the mechanisms of TiB_2 formation in studies of synthesis of ultra-hard materials as replacements of industrial diamonds in drilling materials. Photovoltaic cells were examined by scanning electron microscopy x-ray fluorescence (SEM-XRF) to determine failure modes. A large tungsten valve was studied by XRF, using special masking techniques, to determine the cause of failure.

The staff also devised an on-site instrumental capability for measuring LiBr in heat exchanger fluid using an Abbe refractometer, developed a successful sealed-tube method for dissolving iridium prior to mass spectrometric study, measured low concentrations of ferrous-ferric iron to estimate its influence on uranium extraction coefficients; and provided both consultation and analysis of materials leading to a successful method for removing silver from photoreproduction wastes.

A vastly improved method for dissolving shale, mineral, and refractory materials for ICP and atomic absorption analysis was completed and placed in operation.

The MIT School of Engineering Practice requested support for student experiments in low-temperature pyrolysis of coal and in measuring characteristics of diesel-synfuel blends as possible fuel for diesel engines.

An environmental specimen coordinator was appointed to receive and transmit specimens to the proper area and to evaluate data prior to reporting. Also, the ACD emergency response teams for non-nuclear situations have responded to a variety of unplanned events involving oil, PCB, fluorescein dye and glycol releases.

(C. S. Alley, J. E. Attrill, P. L. Ball, H. G. Davis, H. W. Dunn, J. H. Hackney, D. R. Heine, B. Philpot, J. C. Price, W. P. Rogers, K. I. Webb)

ENVIRONMENTAL ANALYSIS LABORATORY

N. M. Ferguson

The Environmental Analysis Laboratory (EAL) provides technical support for the Environmental Sciences Division (ESD), other ORNL divisions, and outside organizations. The EAL has been extremely busy during this report period. Approximately 40,000 analytical results were reported. Two persons were lost by retirement, one was added to the staff, and whenever possible, personnel trained in environmental type of analyses were borrowed to provide added support to the staff. Some of the analytical projects undertaken are reported here.

A large portion of EAL work for the report period came from the Terrestrial Ecology Section of ESD. Projects associated with Walker Branch Watershed, elemental leaching from forest soil, and whole tree harvesting generated a large number of samples. Water, soil, and plant tissue samples were received from these programs. Water samples were routinely analyzed for calcium, sodium, potassium, magnesium, ammonia, phosphorus, nitrate, chloride, sulfate, and Kjeldahl nitrogen. Soil and plant tissues were analyzed for sodium, potassium, calcium, magnesium, phosphorus, and Kjeldahl nitrogen.

The determination of mercury continues to be a constant challenge. The EAL analyzed

approximately one hundred effluent samples from an industrial site. The effluents ranged in concentration from 0.1-4 ppb. Organic mercury was determined in sediment samples from the same site. The Virginia Water Control Board continued to submit water samples from the Abingdon and Bridgewater areas for mercury analyses. Streams near industrial sites were monitored for mercury.

Technical support has continued during the report period for the Industrial Hygiene Department. Lead was determined in urine and blood. Air filters were analyzed for several metals. Two factors are important for this work: fast turnaround time and a high level of quality assurance.

An ambitious sampling program was initiated by the ESD in August to examine the toxicity of leachates. This project required the analysis of large numbers of samples. Municipal and industrial waste leachates were analyzed for arsenic, selenium, lead, cadmium, chromium, nickel, and barium. Matrices varied considerably and concentrations ranged from sub ppb to ppm. Samples were screened, and elements determined by flame atomic absorption when present in appropriate concentrations. Trace elements were determined by flameless atomic absorption (graphite furnace), and the arsine accumulation helium glow detector method.

A sizeable effort was expended to determine sulfate, nitrate, ammonia, calcium, and potassium in dry deposition, rain, and throughfall samples for the forest canopy acid deposition program. The determination of sulfate by the methyl thymol blue colorimetric method made up a large portion of the work. Quality assurance for this work required comparison of results by an alternate method, ion chromatography, and an outside laboratory, Illinois State Water Laboratory.

Development work was required in several areas. Dry ashing and wet ashing methods were evaluated for the dissolution of plant tissues for the determination of major, minor, and trace elements. A dissolution method for soils and sediments was also developed. The Kjeldahl nitrogen determination was expanded to include soils, sediments, and vegetation. The Technicon block digester was used for the wet digestion of the samples to convert organic compounds to ammonia. Ammonia was determined by a colorimetric ammonia-salicylate method. There had been no interest in methyl mercury analysis for

approximately two years. Last year the instrumentation for this analysis was dismantled. New interest in organic mercury in sediments during this report period required some development work.

A Perkin Elmer hydride system (MHS-20) is being evaluated for arsenic and selenium analysis. This technique may improve our present capabilities. It should have fewer interferences, require less analysis time, and be better suited for routine analyses.

(L. S. Barringer, S. J. Morton, W. R. Musick, J. W. Wade, C. L. Watson)

ACTIVATION ANALYSIS

J. F. Emery

This group provides general neutron activation analysis for a number of Laboratory divisions and programs. Multielement neutron activation analysis was used to establish the elemental content of samples from the H-Coal Program. We have also determined uranium by the delayed neutron counting method in almost 2,000 samples from the FUSRAP program. We continue to provide radiochemical analysis for the Laboratory's decontamination and decommissioning of old nuclear facilities. In the area of fuel reprocessing, we provided gamma-ray spectrometric and neutron activation analysis of fuel dissolver residues. We have continued routine assays of radioisotope products produced by the Operation Division.

Work for other government agencies and private organizations was performed. Work with the U.S. Geological Survey carried over into the first quarter. This work involved multielement analysis of soil samples. We have continued to perform radiochemical analysis for General Public Utilities (TMI-2) and Florida Power and Light Co. to assist them in monitoring radionuclides at their nuclear reactor facilities. We have provided irradiation facilities for Battelle Northwest Laboratory personnel for the determination of ^{129}I while their facilities are shut down. We are continuing our development activities in determining uranium and thorium in semiconductor material by analyzing device-encapsulating materials for Texas Instruments, Inc. Technical support and ^3H analysis for a fusion blanket experiment have also been provided. This is a collaborative effort

between Argonne National Laboratory and the Engineering Technology Division of ORNL.

Several data acquisition systems were moved to better utilize their capabilities. The 8 K PDP-15 at the High Flux Isotope Reactor (HFIR) was replaced with a 32 K PDP-15. This reduced the processing time for a gamma-ray spectrum by a factor of approximately 3. Our Nuclear Data (ND-6620) acquisition system was moved to the ORR neutron activation analysis laboratory. We now have two 20% Ge(Li) detectors for low-level analysis and two 10% Ge(Li) detectors for high count rate gamma-ray spectrometry at the Oak Ridge Research Reactor (ORR). We also have an intrinsic Ge x-ray detector. All of these detectors may be operated simultaneously on the ND-6620. Several new programs were written for the ND-6620. These programs were for determining the neutron flux in the pneumatic tube at the ORR and for monitoring the quality of data from Ge(Li) detector systems.

We have also provided technical guidance to two trainees this year. F. P. Vercelez, an IAEA Fellow from the Philippines, performed multi-element analysis of commercial fertilizers. Approximately 33 elements were determined. J. R. Parrish, an ORAU participant from the University of Alabama, studied trace element profiles in Peregrine falcon feathers. This study will be utilized in predicting origins of migrant falcons sampled enroute to South American wintering grounds.

(L. C. Bate, P. P. Dyer, J. W. Wade, K. J. Northcutt)

LOW-LEVEL RADIOCHEMICAL ANALYSIS

T. G. Scott

The Low-Level Radiochemical Analysis group provides support for members of UCC-ND and outside customers who require radioanalysis for natural and man-made radionuclides at background or slightly elevated levels. The portion of our support efforts allotted for environmental monitoring programs increased from 40 to 50% this year due primarily to the monitoring of burial grounds. The level of support given to surveillance programs decreased slightly from 30 to 25%; research and development support efforts decreased from 25 to 15%; and work for others increased from 5 to 10%.

The materials commonly analyzed for environmental monitoring were charcoal and paper filters, water from various sources, soil and vegetation from selected monitoring sites, sediments from local streams, fish from local streams and streams near another Nuclear Division facility, vegetables and small animals from areas in the Nuclear Division facility, and insects trapped at ORNL. The frequency of sample collections for these materials varied from daily (e.g., charcoal and paper filters collected during campaigns at the TRU facilities) to annually (e.g., fish collected at the end of the calendar year from the Clinch River).

The volume of materials received from the Formerly Used Sites, Remedial Action Program (FUSRAP) declined steadily throughout the year to about six samples per month. Salt Lake City and St. Louis were the only sites represented in this year's work for FUSRAP. Samples of water and sediment from the Salt Lake City site were received at random. St. Louis-site samples, submitted monthly, consisted of water and related particulates from six wells located at the St. Louis airport. Five of the six wells were accidentally destroyed about mid-year and have only recently been redrilled. All samples from FUSRAP were analyzed routinely for ^{226}Ra , ^{210}Pb , ^{238}U , and occasionally for ^{232}Th .

This year we made a total of approximately 1800 determinations on water, fish, and sediment samples for plutonium, americium, curium, uranium, and neptunium isotopes in support of a program conducted by Environmental Sciences Division. This program, designed to study the ecology of the aquatic system in Pond 3513 over the past few years, was concluded at the end of fiscal year 1982.

As a special project for the Department of Environmental Management of the Industrial Safety and Applied Health Physics Division, we conducted a study to evaluate the performance of ion-exchange resin columns in concentrating radionuclides from very large (50-liter) volumes of natural (lake and river) water. The objective of the study was to furnish information for a qualified procedure to be used in monitoring large bodies of water for radionuclides. During our study, we experienced a problem with the quantitative recovery of cobalt, which is apparently chemically bound in a manner that makes it intractable. Further work is planned to resolve this problem.

The increase in work for others this year was mainly due to work from Florida Power and Light Co., the Tennessee Department of Public Health, and a special project for the Nuclear Regulatory Commission. Work for Florida Power and Light Co. involved radioanalyses of water concentrates, smears, crud, and ion-exchange resins from two power reactors. Radioanalyses for fission products and activation products were required on these materials. Fish samples collected by the Department of Public Health from the Little Limestone Creek in upper East Tennessee were analyzed for uranium and thorium isotopes. The results of these analyses will determine if there are any significant levels of uranium and thorium in the fish. The intent of the NRC project is to identify and evaluate promising methods for determining uranium, plutonium, and thorium at the parts-per-trillion level in urine and feces. Our involvement in this project is to check procedure for "loading" the mentioned elements onto a few (3-10) resin beads which are subsequently analyzed by isotope dilution mass spectrometry.

(L. D. Bible, G. I. Gleason, P. S. Gouge, C. C. Granger, W. C. Massey, M. P. May, S. H. Prestwood, L. M. Roseberry, A. J. Weinberger)

SPECIAL PROJECTS

J. S. Eldridge

The Special Projects Group provides technical support and conducts applied research in a variety of Laboratory programs. Instrumental methods for radioactivity determination make up the base for the efforts, with the major thrust involving low-level gamma-ray spectrometry. Funding is provided by the Department of Environmental Management (DEM) of the Industrial Safety and Applied Health Physics Division with ancillary programs in the Laboratory Protection Division.

We have worked mainly this year on radioactivity determinations in a variety of sample matrices in support of environmental surveillance projects of the DEM. Final sample collections and analyses were made in a radionuclide transfer study (via aquatic insects) at the old hydrofracture disposal site. Results from those studies yielded almost "textbook" pathways: collector-gatherer organisms such as mayfly

nymphs contained the highest concentrations of radionuclides followed by predator engulfer (dragonfly nymphs) and then by piercer-predators. Emergent adult dragonflies and damselflies contained the highest concentrations of radionuclides ($4 \text{ kBq} \cdot \text{g}^{-1} \text{ }^{137}\text{Cs}$) in organisms leaving the pond. Second phase investigations are underway to determine the total biomass leaving the aquatic habitat.

A facility was constructed in the field for the study of biological half-lives of ^{137}Cs in those emergent species. A number of dragonflies and damselflies were captured shortly after emerging from the pond. The live insects were immediately transported to a laboratory spectrometer designed for whole-body counting. After the initial radioactivity measurement, the individual insects were marked with a code for future identification and released in the enclosure at the study site. The capture, radioactivity measurement, and release procedures were repeated at 1-2 day intervals as long as any study insects survived. By this technique we determined the biological half-life of ^{137}Cs to be 19 days and 7.5 days in dragonflies and damselflies, respectively.

Honeybee hives placed near former radioactive waste disposal sites were sampled during times of major nectar flows to determine whether bees or honey would accumulate radioactivity. Small but measurable quantities of ^3H , ^{60}Co and ^{137}Cs were determined in those hives closest to contaminated sites. Control samples showed ^{137}Cs levels attributable to global fallout. It was concluded that honey produced by honeybees at the most contaminated site was still below the applicable concentration guides for these three nuclides.

A new measurement system was instituted for performing nondestructive assay procedures (NDA) in conjunction with inventory audits for special nuclear materials. In addition, the system functions as an NDA system for precious metal inventory verifications with the simple addition of a radionuclide source exciter for characteristic x-rays. The detector in the new system contains an intrinsic germanium planar crystal with 100 mm cross-section and 10 mm depletion depth. The detector has good efficiency and excellent resolution for the 185-keV γ -rays from ^{235}U sources as well as excellent response from the K-series x-rays from precious metals. Recent applications of the precious metal system have involved the quantitative determination of

silver in various photographic wastes as well as the qualitative verification of gold in a massive collimator. Identity verification was accomplished on a series of 3-4 m length thermocouple probes thought to be platinum.

A new portable spectrometer system has been acquired for use in a variety of programs where in situ analyses are required. The analyzer portion of the spectrometer contains 2048 channels of memory, display, amplifier, battery pack, and cassette tape storage in a single 8 kg package. The detector portion of the spectrometer consists of a portable cryostat containing a 30% (relative efficiency) intrinsic germanium detector. This combined assembly is easily transported to any number of field locations for immediate in situ determinations. Projected use of the assembly involves rapid emergency response, qualitative identification of radioactive contaminants in decontamination and decommissioning activities, and field spectroscopy for more efficient sample collection.

(N. A. Teasley)

6. QUALITY ASSURANCE, SAFETY, AND TABULATION OF ANALYSES

W. R. Laing
F. L. Layton
A. L. Harrod

QUALITY ASSURANCE

Responsibility for the quality of work done within the five sections of the Analytical Chemistry Division falls to the respective supervisors. Because of the diversity in types and scopes of work done in these sections, various means are used to assure quality. The annual quality assurance report comprises a collection of statements from each group and/or laboratory, encompassing (1) scope of work, (2) quality assurance program and procedures, (3) quality assurance compliance information and data, and (4) standard reference materials and their sources. Included is a statement summarizing control samples prepared and distributed during the reporting period and the quality level obtained with these internal control (reference) standards. Table 6.1 shows the quality level for each of the participating laboratories within the division and compares the results with those from the previous year. Lists of the different control programs and the number of results reported for each are given in Table 6.2. During the past year there has been a 3% increase in the number of control determinations reported and a 5% increase in the number of control programs.

During this reporting period three internal quality assurance audits of AC^D sections on April 21, 1982; and the Nuclear and Radiochemical Analysis Section on June 11, 1982.

A Laboratory quality assurance audit of the division was made on July 19, 1982. Other audits included one by the UCCND Environmental Programs Review Committee on September 19, 1982, and an external audit of the Bio/Organic Analysis Section by the Environmental Protection Agency on February 3, 1982.

Table 6.1. Distribution of control tests by laboratory (October 1981 through September 1982)

Laboratory	Number of control results			
	Total	Fixed limits	1981	1982
Chemical & Physical				
Analysis	428	37	96.82	91.36
Environmental				
Analysis	542	70	90.00	87.08
Radioactive Matls				
Analytical	451	13	95.99	97.12
Transuranium	123	15	93.00	87.80
Total	1544	135	92.98	91.76

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

Table 6.2 Distribution of control results (by method)
for October 1981 through September 1982

Method	Constituent	Number of programs	Number of results	Total
Atomic absorption spectrometry	Aluminum	1	18	
	Cadmium	1	5	
	Calcium	1	65	
	Chromium	1	7	
	Copper	1	6	
	Iron	1	32	
	Lead	1	4	
	Lithium	1	3	
	Magnesium	2	47	
	Nickel	1	6	
	Potassium	1	44	
	Sodium	1	36	
	Zinc	1	8	281
Colorimetry	Chemical Oxygen Demand (COD)	1	9	
	Chloride	1	42	
	Molybdenum	1	2	
	Nitrogen	2	64	
	Phosphorus	1	48	
	Sulfate	1	58	
	Uranium	2	78	301
Coulometry	Uranium	1	18	18
Count, Gross Alpha	Plutonium	1	8	8
Fluorimetry	Uranium	2	187	187
Gravimetry	Carbon	1	21	
	Hydrogen	1	21	42
Infrared absorption spectrophotometry	Carbon	1	53	53
Inductively coupled plasma spectrometry	Aluminum	1	6	
	Antimony	1	3	
	Arsenic	1	2	
	Beryllium	1	6	
	Boron	1	4	
	Caesium	1	6	
	Chromium	1	6	
	Cobalt	1	6	
	Copper	1	6	
	Iron	1	6	
	Lead	1	6	
	Manganese	1	6	
	Molybdenum	1	4	
	Nickel	1	6	
	Selenium	1	6	
	Tin	1	1	
	Titanium	1	4	
	Vanadium	1	6	
Zinc	1	6	96	

(cont'd)

Table 6.2 Distribution of control results (by method)
for October 1981 through September 1982 (Cont'd)

Method	Constituent	Number of programs	Number of results	Total
Ion chromatography	Bromide	1	6	
	Chloride	1	6	
	Fluoride	1	6	
	Nitrate	1	6	
	Nitrite	1	6	
	Phosphate	1	6	
	Sulfate	1	6	42
Leco	Carbon	1	33	
	Sulfur	1	48	81
Volumetric titrimetry	Acid	3	126	
	Chloride	1	1	
	Magnesium	1	10	
	Nitrate	1	35	
	Silver	1	6	
	Uranium	2	223	401
X-ray fluorescence	Plutonium	1	13	
	Thorium	1	6	
	Uranium	1	15	34
Total		70		1544

SAFETY

Employees of the division have accumulated 2,305,477 workday hours since the last lost-time accident in 1972. This record is possible because of the excellent support of the safety program within the division. Approximately ninety percent of employees attended a safety meeting each quarter.

Quarterly safety audits were made by the division safety committee and division safety officer. Over eighty five percent of the committee findings were corrected and the needed action was taken.

All members of the division's local emergency organization (squad) are trained in firstaid and CPR. We conducted one in-house CPR recertification training class and held one in-house workshop on laboratory emergency procedures. 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 6.3 Summary of analytical work

Organization	Number of results reported by					Total
	Mass and Emission Spectrometry	Chemical, Physical, and Environmental Analysis Laboratories	Transuranium Laboratory	Radioactive Materials Analytical Laboratory	Analytical Physics Analysis	
ORNL Unit						
Analytical Chemistry	1,053	7,994		565	36	14,623
Biology	398	22				420
Central Management		591				591
Chemical Technology	3,132	1,822	17,876	6,088	275	30,535
Chemistry	542	251	85	48	399	1,979
Energy		30				30
Engineering Physics	171	1			2	174
Engineering Technology	239	3,875			123	4,450
Environmental Sciences		2,157			121	8,903
Finance and Materials		202			40	242
Fuel Recycle		46,890	8	4,045	126	51,292
Fusion Energy	477	26				503
Health	856	753				1,609
Health and Safety Research		270	1	7	254	3,427
Industrial Safety and Applied Health Physics		1,770		2	54	8,274
Instrumentation and Controls	924	13	2	544	22	1,685
Metals and Ceramics	18,486	1,524			376	20,675
Neutron Physics		1				1
Operations	9,271	638	9,396	1,242	36	26,995
Physics			34			17
Plant and Equipment	215	149				392
Quality Assurance and Inspection	183					183
Solid State	2,447	404		18	123	4,666
Others						
Protective Coating Testing				347		347
NURE ^a						18,706
K-25 and Y-12	4,536		15		29	4,744
Miscellaneous	8,188	245		299		56,533
Savannah River Laboratory	1,291					1,291
Work for Others					44	467
TOTAL	52,409	69,626	27,417	13,205	2,060	265,906

^a National Uranium Resource Evaluation.

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 available for student and faculty research and training programs.

ADVISORY COMMITTEE

This year the division Advisory Committee was composed of:

- M. V. Mozotny, Chemistry Department, Indiana University, Bloomington, IN.
- H. B. Mark, Jr., Chemistry Department, University of Cincinnati, Cincinnati, OH.
- J. W. Taylor, Chemistry Department, University of Wisconsin, Madison, WI.
- J. G. Grasselli, Standard Oil Company, Molecular Spectroscopy Section, 4440 Warrensville Center Road, Cleveland, OH.

CONSULTANTS

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

- L. A. Carreira, University of Georgia, Athens, GA.
 - B. Freiser, Purdue University, West Lafayette, IN.
 - A. Rosencwaig, Thermawave, Inc., Fremont, CA.
 - J. C. Travis, National Bureau of Standards, Washington, DC.
 - M. Wightman, Indiana University, Bloomington, IN.
- G. Mamantov and M. J. Sepaniak of the University of Tennessee continued working in the division as Adjunct Research Participants this year.
- S. Pendyala worked with L. D. Hulett on research in positron spectroscopy through the ORAU travel contract program.

PARTICIPATION IN ORNL IN-HOURS PROGRAM

The following divisional people completed courses during the 1982 winter term of the Continuing Education Program for Scientific and Technical Personnel:

M-650C. Computer Methods for Mathematical Computations: T. G. Scott.

The following courses were completed during the spring 1982 term:

Introduction to System 1022 Data - Base Management System: M. P. May.

Courses completed during the fall 1982 term were:

M-310H. Introduction to DEC System-10: D. M. Watson

M-800B. Practical Solution of Nonlinear Partial Differential Equations: L. K. Bertram.

S-205. Grammar for the Technical Writer: D. M. Watson.

M-600F. Introduction to Practical Statistics: S. K. White.

GUEST ASSIGNMENTS

Two predoctoral students from the University of Tennessee worked in the Analytical Methodology Section this year. Brisco L. Harvard worked with L. N. Klatt on rapid scan spectrometry and Huston E. Howell worked with R. W. Shaw on opto-acoustic spectrometry. Howell holds an ORAU fellowship for this work.

The division maintains a subcontract with the University of Tennessee to enable advanced graduate students to participate in environmental analysis programs. Donna Taylor Dixon worked in the Bio/Organic Analysis Section and in the Mass Spectrometry Section at Y-12 under this contract.

Arlene Garrison came to work in the Bio/Organic Analysis Section for a one-month period on a DOE contract.

John Vargo was granted an ORAU fellowship and is assigned to the Bio/Organic Analysis Section.

The Nuclear and Radiochemical Analysis Section was host to four research scientists funded by Battelle Northwest Laboratories. They worked for two weeks conducting experiments which involved irradiations at HFIR.

SCUU PROGRAM

D. R. Ross, Centre College, was selected for the 1982 SCUU Science Semester program. Her assignment involved working with M. P. Maskarinec in the Bio/Organic Analysis Section on the analysis of wastewaters and sludges using gas and high-pressure liquid chromatography.

ANALYTICAL CHEMISTRY DIVISION SEMINARS AT ORNL

Speaker	Title	Date
Carreira, L. A. Univ. of Georgia Athens, GA	"Analytical Applications of Coherent Raman Spectroscopy"	Feb. 1, 1982
Freiser, B. Purdue Univ. W. Lafayette, IN	"Atomic Metal Ion Chemical Ionization Using Fourier Transform Mass Spectrometry"	April 20, 1982
Rosencwaig, A. Thermawave, Inc. Freemont, CA	"Recent Developments in Photoacoustics"	April 30, 1982
Travis, J. C. NBS Washington, DC	"The Characterization and Application of Laser Enhanced Ionization in Flames"	May 3, 1982
Wightman, R. M. Indiana Univ. Bloomington, IN	"Electroanalysis with Microvoltammetric Electrodes"	Nov. 8, 1982

ACD SUMMER LECTURE SERIES

The division initiated a summer lecture series this year to present in-depth lectures on current analytical techniques.

Speaker	Title	Date
Hulett, L. D.	"Electron Microscopy and X-Ray Methods"	June 18, 1982
Christie, W. H.	"Surface Analysis and Other Aspects of Secondary Ion Mass Spectrometry"	June 25, 1982
Donohue, D. L.	"Spark Source Mass Spectrometry"	July 9, 1982
Lyon, W. S.	"Neutron Activation Analysis"	July 16, 1982
Heine, D. R.	"Trace Elemental Analysis by Inductively Coupled Plasma and Atomic Absorption"	July 23, 1982
Eldridge, J. S.	"Radiochemical Analysis"	July 30, 1982
Maskarinec, M. P.	"Preparative and Analytical Organic Separations"	Aug. 6, 1982
Buchanan, M. V.	"Spectroscopic Organic Analysis"	Aug. 13, 1982

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.

M. E. Roth, Kenyon College, worked in the Bio/Organic Analysis Section on the separation of nitro-PAHs.

L. Williams, Colgate University, was assigned to work on the development of a multicomponent analytical procedure for cigarette smoke particulate matter using fused silica capillary column gas chromatography in the Bio/Organic Analysis Section.

ORNL CO-OP PROGRAM

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

SPECIAL AWARDS

W. R. Laing received the ASTM Award of Merit at the Knoxville meeting in August. Mr. Laing received the award for his leadership in the development of test methods for nuclear fuel cycle and his contributions to standards for fuels, absorbers, and moderators.

L. T. Corbin also received an ASTM award, the Charles B. Dudley Medal, at the August meeting in Knoxville. This award is made for an outstanding contribution which has widely recognized impact on a particular field of ASTM interest, and which has been documented in the ASTM literature.

J. H. Stewart, Jr. and Ronald T. Roseberry, Sr. received the IR-100 award for the ORNL-Inductively Coupled Plasma (ICP) Spectrometer (Model JY-85CP).

The division has received the Laboratory's Environmental Protection Award for the past two years. In 1981 the following people received the award for outstanding achievement in environmental protection for 1980:

H. G. Davis	For the development of a procedure for the analysis of PCB in
R. R. Rickard	oils
J. H. Stewart, Jr.	For liaison and coordination of specific environmental analysis
	problems
B. R. Clark	For the development of the ORNL oil characterization program
W. F. Rogers	
H. W. Dunn	For the development of a system for quantitative determinations
J. H. Stewart, Jr.	of asbestos in construction materials
R. L. Sherman	
J. S. Eldridge	For developments in the detection of radioactivity in environ-
T. G. Scott	mental samples
J. R. Stokely	

In 1982 the following people were awarded for 1981:

J. E. Attrill	For emergency analytical response system to oil spill incident
H. G. Davis	
K. I. Webb	
W. F. Rogers	

M. M. Ferguson	For the inductively coupled plasma and improved atomic
D. R. Heine	absorption spectrometer capabilities to provide rapid low cost
J. H. Stewart, Jr.	determination of metallic contaminants in surface water.
M. P. Maskarinec	For the qualitative and quantitative determination of ultra-
M. V. Buchanan	trace concentrations of multiple organic constituents in surface
B. A. Tomkins	waters.
G. Olerich	
R. W. Harvey	
S. H. Harmon	

ADDITIONAL PROFESSIONAL ACTIVITIES

ATTRILL, J. E.	
Member:	ASTM Committee E-38 on Resource Recovery ORNL Emergency Oil Spill Response Team
Advisory Committee:	Subcommittee E-38.01 on Energy, ASTM
Sample Coordinator:	Analytical Chemistry Division/Department of Environmental Management
Division Representative:	Environmental Protection Officer
BALL, F. L.	
Secretary:	Electron Microscopy Society of America Analytical Chemistry Division Safety Committee
BRAZELL, R. S.	
Lecturer:	ORAU Traveling Lecture Program
BROOKSBANK, R. D.	
Member:	ASTM Committee D-33, Protective Coating and Lining Work for Power Generation Facilities
Chairman:	Task Group on Methods of Coatings Analysis, ASTM D-33
BUCHANAN, M. V.	
Member:	Analytical Chemistry Division Seminar Committee
Treasurer:	East Tennessee Section, American Chemical Society
CARTER, J. A.	
Secretary:	Subcommittee C-5:05, Analytical Task Group (Reactor Grade Graphite), Committee C-5, Manufacturer 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
Symposium Organizer:	Symposium on Analytical Chemistry of Nuclear Safeguards 184th National ACS Meeting, Kansas City, MO, Sept. 1982.
Advisory Board:	<i>Analytical Chemistry</i>
Laboratory Coordinator:	ISPO Programs
Advisory Panel Member:	DOE/ISA Laboratory Advisory Group for Sample Analysis (LAGSA)
CATON, J. E.	
Co-Chairman:	Vibrational Spectroscopy Subgroup of UCC Analytical Specialists Group
Member:	Analytical Chemistry Division Long Range Planning Committee Analytical Chemistry Division Computer Utilization Committee

CHRISTIE, W. H.
 Member: ACD Long Range Planning Committee
 ASTM Committee E-42, Surface Analysis

COOPER, J. H.
 Member: ORNL Management Information Committee

CORBIN, L. T.
 Fellow: American Society for Testing and Materials
 Member: 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: ACD Quality Assurance Program
 Committee C-26, Fuel, Control, and Moderator Materials for
 Nuclear Reactor Applications, and Subcommittee C-26:05,
 Methods of Test, ASTM

DONOHUE, D. L.
 Lecturer: ORAU Traveling Lecture Program
 Member: Analytical Chemistry Division Ad Hoc Committee on
 Computerization

DYER, F. F.
 Member: Analytical Chemistry Division Seminar Committee
 Session Chairman: "New Directions in Nuclear and Radiochemistry," at Winter
 Meeting of American Nuclear Society held in Washington, DC,
 November 13-18, 1982.

ELDRIDGE, J. S.
 Member: Analytical Chemistry Division Long Range Planning Committee

EMERY, J. F.
 Consultant: Tennessee Valley Authority

FEIDMAN, C.
 Fellow: American Society for Testing and Materials
 Scientific Editor: *Optics and Spectroscopy* (English translation of Russian
 journal, *Optika i Spektroskopiya*)
 Member: Committee E-2 on Emission Spectroscopy, ASTM, Subcommittees
 on Fundamental Methods, Editorial practices, and
 Nomenclature
 Atomic Absorption and Emission Spectroscopy Subgroup, Union
 Carbide Corporation.

CRIEST, W. H.
 Consultant: EPRI: Research Agenda Planning for Potential Human Health
 Effects of Inhalable Fly Ash
 National Cancer Institute
 Member: DOE Oil Shale Working Group

CHERIN, H. R.
 Consultant: National Institute of Drug Abuse
 Synthesis of Drugs of Abuse (RTI)
 Synthesis of Cannabinoids (ADL)
 National Cancer Institute
 ^{210}Po in Cigarette Smoke
 Department of Energy (ONER)
 Chemical Separations for Biological Testing (Argonne)
 Biological Test Matrix for Complex Mixtures (Johns
 Hopkins)
 Analytical Chemistry of Airborne Organics (Gettysburg)
 Coal Gasification Research Progress (DOE)
 Environmental and Health Research in Synfuels (Airlie
 House)
 Shale Oil Chemistry (LBL)
 Federal Trade Commission
 Low Tar Cigarettes
 Advisor: NAS panel

HOLMBERG, R. W.
 Coordinator: Analytical Chemistry Division Metric Coordinator

BOLETT, L. D.
 Coordinator: Solids and Surface Analysis Team

JENKINS, R. A.
 Coordinator: ORNL World's Fair Exhibit

KLATT, L. H.
 Associate Editor: *Chemical Biomedical, and Environmental Instrumentation*
 Member: Fellowship Committee of Analytical Chemistry Division of
 American Chemical Society
 Committee C-26, Fuel, Control and Moderator Materials for
 Nuclear Reactor Applications, ASTM

LAINC, W. R.
 Vice-chairman: ASTM Committee C26, Nuclear Fuel Cycle
 Member: ASTM Committee on Technical Committee Operations
 ISO Technical Committee 85, Subcommittee 5
 UCCND Analytical Committee
 ORNL Grievance Committee
 ACD Long Range Planning Committee
 Chairman: Subcommittee C26:05, Test Methods, Nuclear Fuel Cycle, ASTM
 Division Representative: UCCND Environmental Analysis Committee

LYON, W. S.

Member:

Committee E-10, Nuclear Technology and Applications, 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
Journal of Radioanalytical Chemistry
Radiochemical and Radioanalytical Letters
Analyst
 Isotopes and Radiation Division, ANS
 Honors and Award Committee

Regional Editor:

Associate Editor:

Advisory Board:

Vice-chairman:

Chairman:

MASKARINEC, M. P.

Member:

ASTM E-19 Committee on Chromatography
 UCCND Liquid Chromatography Specialists' Group
 UCCND Gas Chromatography Specialists' Group
 ASTM D-34 on Waste Disposal
 ORNL Hazardous Waste Task Force

MUELLER, T. R.

Coordinator:

ACD Awards Coordinator

RANSBY, J. H.

Chairman:

Analytical Chemistry Division Seminar Committee

ROSS, H. H.

Chairman:

ORNL Graduate Student Selection Panel
 Nominating Committee, ORNL Credit Union

Member:

ORNL Technology Utilization Committee
 ACD Long Range Planning Committee
 Editorial Advisory Board, *Journal of Radioanalytical
 Chemistry*
 ORNL Proposal Review Committee
 Scientific Advisory Committee, International Conference on
 Scintillation Spectroscopy, Canada, 1982-1983

SCOTT, T. G.

Member:

Committee D-19, Water, ASTM, and Subcommittee 4, Methods of
 Radiochemical Analysis

SHULTS, W. D.

Chairman:

ORNL United Way Campaign Committee, 1982

Past Chairman:

Division of Analytical Chemistry, ACS
 Analytical Specialist Group, UCC

Vice-President

Sigma Xi RESA

Member:

ORNL In-House Continuing Education Committee
 Technical Services Committee, UCCND
 Editorial Board, *Analytica Chimica Acta*
 Advisory Committee, "Analytical Approach," *Analytical
 Chemistry*

Committee on the Scientific Aspects of Regulatory
 Measurements

Convening Committee for the ACS Award for Creative Advances
 in Environmental and Technology

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
International Working Group "Analytical Standards of
Minerals, Ores and Rocks"
Analytical Chemistry Division Technical Ombudsman

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

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

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

WALKER, R. L.
Member: DOE/ISA Laboratory Advisory Group for Effluent Research

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

YOUNG, J. P.
Fellow: American Association for Advancement of Science
Member: Site Review Committee - DOE-OHES Review of the Environmental
Research Program at Aerospace Corporation, El Segundo, CA

8. PRESENTATION OF RESEARCH RESULTS

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

PUBLICATIONS

Contributions to Books, Proceedings, and Reports

AUTHOR	AUTHOR(S) AND TITLE	PUBLISHED IN
Bostick, D. T.	Strain, J. E.; Bostick, D. T.; McCue, D. D.*; Harper, R. E.* "In-Line Instrumentation for Control of Nuclear Fuel Reprocessing - I: In-Line Neutron Poison and Free Acid Monitor"	<i>Trans. of Amer. Nucl. Soc.</i> , Washington, DC, Nov. 14-18, 1982, Vol. 43, p. 275
Buchanan, M. V.	Walton, B. T.*; Buchanan, M. V.; Ho, C.-h. "Comparative Embryotoxicity and Teratogenicity of Petroleum and Petroleum Substitutes to Insects Developing in Contaminated Substrates"	<i>Energy and Environmental Chemistry, Vol. 1, Fossil Fuels</i> , L. H. Keith, Ed., Ann Arbor Sci., 1982, pp. 249-257
	Ho, C.-h.; Buchanan, M. V.; Clark, B. R.; Guerin, M. R. "Chemical Characterization of Synfuel Neutral Fractions"	<i>Coal Conversion and the Environment</i> , D. Mahlum, R. H. Gray, and W. D. Felix, Eds., Tech. Inf. Cent., USDOE, 1981, p. 34
Carter, J. A.	Carter, J. A.; Smith, D. H.; Walker, R. L. "Mass Spectrometric Measurements for Nuclear Safeguards"	<i>Proc., 30th Annu. Conf. on Mass Spectrom. and Allied Topics</i> , Honolulu, HI, June 6-11, 1982, pp. 432-433
	Warmack, R. J.*; Landau, L.; Christie, W. H.; Carter, J. A. "A New Mass Spectrometric Facility for the Analysis of Highly Radioactive Samples"	<i>Analytical Chemistry in Nuclear Technology</i> , W. S. Lyon, Ed., Ann Arbor Sci., 1982 pp. 195-202
	Smith, D. H.; Walker, R. L.; Carter, J. A. "The Modified Resin Bead: Its Application to Safeguards"	<i>Analytical Chemistry in Nuclear Technology</i> , W. S. Lyon, Ed., Ann Arbor Sci., 1982 pp. 139-143
Caton, J. E.	Griest, W. H.; Higgins, C. E.; Holmberg, R. W.; Moneyhun, J. H.; Caton, J. E.; Wike, J. S.*; Reagan, R. R. "Characterization of Vapor- and Particulate Phase Organics from Ambient Air Sampling at the Kosovo Gasifier"	<i>Energy and Environmental Chemistry, Vol. 1, Fossil Fuels</i> , L. H. Keith, Ed., Ann Arbor Sci., 1982, pp. 395-410

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	Guerin, M. R. <i>Analytical Chemistry Division Research and Development Summary: September, 1982, Bio/Organic Analysis Section</i>	ORNL/CF-82/292 (Nov. 1, 1982)
Harmon, S. H.	Harmon, S. H.; Caton, J. E. <i>Diffuse Reflectance Infrared Fourier Transform Spectra of Selected Organic Compounds</i>	ORNL/TM-8321 (May 1982)
Heine, D. R.	Keller, J. M.; Heine, D. R. <i>Summary of Software/Hardware Development for the ORNL ICP System</i>	ORNL/CF-82/241 (Aug. 1982)
Keller, J. M.	Keller, J. M.; Heine, D. R. <i>Summary of Software/Hardware Development for the ORNL ICP System</i>	ORNL/CF-82/241 (Aug. 1982)
	Keller, J. M. <i>TSX-Plus for Laboratory Data Acquisition and Control</i>	ORNL/TM-8507 (Oct. 1982)
	Keller, J. M. <i>Development of Techniques for the Determination of Hexamethylenetetramine, Formaldehyde, and Urea in Internal Gelation Process Waste</i>	ORNL/CFRP-82/23 (Oct. 1982)
Klatt, L. N.	Allgood, G. O.*; Anderson, R. L.*; Andrews, W. H.*; Bradley, N. C.*; Brown, C. H.*; Canright, G. S.*; Caton, G. M.*; Hamel, W. R.*; Hutton, J. T.*; Jansen, J. M., Jr.*; Klatt, L. N.; Moore, R. L.*; Moyers, J. C.*; Ottinger, L. E.*; Rohrer, E. R.*; Seaver, G. V.*; Wallis, D. S.*; Wetherington, G. R., Jr.*; Carr, K. R.* <i>State-of-the-Art Assessment of Coal Preparation Plant Automation</i>	ORNL-5699 (Feb. 1982)
Laing, W. R.	Laing, W. R. <i>Analytical Chemistry Division Report for November 1982: Radioactive Materials Analysis Section</i>	ORNL/CF-82/320 (Dec. 1, 1982)
	Gilai, D.*; Williams, M. L.*; Cooper, J. H.; Laing, W. R.; Walker, R. L.; Raman, S.*; Stelson P. H.* <i>Experimental and Computational Analyses of Actinide Samples Irradiated in EBR-II</i>	ORNL/5791 (Oct. 1982)
Lee, D. A.	Lee, D. A.; Knipschild, K. E.* <i>Thermal Analysis of Synrock Gel Microspheres</i>	ORNL/TM-7930 (Nov. 1981)
Lyon, W. S.	Lyon, W. S. <i>Analytical Chemistry Division Research and Development Summary for December 1981; Analytical Methodology Section</i>	ORNL/CF-81/336 (Jan. 4, 1982)
	Lyon, W. S. <i>Analytical Chemistry Division Research and Development Summary for April 1981; Analytical Methodology Section</i>	ORNL/CF-82/74 (May 5, 1982)

AUTHOR	AUTHOR(S) AND TITLE	REPORT NO. AND DATE
Lyon, W. S.	Lyon, W. S. <i>Analytical Chemistry Division Research and Development Summary for August 1982; Analytical Methodology Section</i>	ORNL/CF-82/259 (Sept. 1, 1982)
McBay, E. H.	Raine, W. T.*; McBay, E. H.; Glish, G. L. <i>Procedure for the Pyrolysis/GC/MS Analysis of Coal</i>	ORNL/TM-8257 (Apr. 1982)
McKown, H. S.	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)
	Smith, D. H.; McKown, H. S.; Walker, R. L.; Sherman, R. L.; Pritchard, C. A.; Carter, J. A. <i>Progress Report for the Office of Safeguards and Security for FY 1982</i>	ORNL/TM-8557 (Dec. 1982)
Pritchard, C. A.	Smith, D. H.; McKown, H. S.; Walker, R. L.; Sherman, R. L.; Pritchard, C. A.; Carter, J. A. <i>Progress Report for the Office of Safeguards and Security for FY 1982</i>	ORNL/TM-8557 (Dec. 1982)
Quinby, T. C.*	Quinby, T. C.*; Adair, H. L.*; Kobisk, E. H.*; Ramey, D. W.*; Setaro, J. A.*; Botts, J. L.; Cooper, J. H.; Walker, R. L.; Bigelow, J. E.*; Gibson, J. R.*; Martin, W. T.*; Pope, R. G.*; Raman, S.* <i>Preparation of Actinide Specimens for the US/UK Joint Experiment in the Downreay Prototype Fast Reactor</i>	ORNL-5858 (May 1982)
Scott, T. G.	Oakes, T. W.*; Ohnesorge, W. F.*; Eldridge, J. S. Scott, T. G.; Parsons, D. W.*; Hubbard, H. M.*; Sealand, O. M.*; Shank, K. E.*; Eyman, L. D.* <i>Technical Background Information for the Environmental and Safety Report, Vol. 5: The 1977 Clinch River Sediment Survey--Data Presentation</i>	ORNL-5878 (Nov. 1982)
Shults, W. D.	Shults, W. D. <i>Analytical Chemistry Division Annual Progress Report for Period Ending December 31, 1981</i>	ORNL-5873 (April 1982)
Smith, D. H.	Smith, D. H.; McKown, H. S.; Walker, R. L.; Sherman, R. L.; Pritchard, C. A.; Carter, J. A. <i>Progress Report for the Office of Safeguards and Security for FY 1982</i>	ORNL/TM-8557 (Dec. 1982)
	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)

AUTHOR	AUTHOR(S) AND TITLE	REPORT NO. AND DATE
Smith, D. H.	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. <i>New Portran Programs to Acquire and Process Isotopic Mass Spectrometric Data</i>	ORNL/TM-8356 (Aug. 1982)
Stokely, J. R.	Stokely, J. R. <i>Analytical Chemistry Division Summary Report: Nuclear and Radiochemical Analysis Section</i>	ORNL/CF-82/3 (Jan. 1, 1982)
	Stokely, J. R. <i>Analytical Chemistry Division Summary Report: Nuclear and Radiochemical Analysis Section</i>	ORNL/CF-82/61 (Apr. 1, 1982)
	Stokely, J. R. <i>Analytical Chemistry Division Summary Report: Nuclear and Radiochemical Analysis Section</i>	ORNL/CF-82/240 (Aug. 1, 1982)
Wade, J. W.	Wade, J. W.; Emery, J. F. <i>Computer Programs for Absolute Neutron Activation Analysis on the Nuclear Data 8620 Data Acquisition System</i>	ORNL/TM-8208 (March 1982)
Walker, R. L.	Quinby, T. C.*; Adair, H. L.*; Kobisk, E. H.*; Ramey, D. W.*; Setaro, J. A.*; Brctts, J. L.; Cooper, J. H.; Walker, R. L.; Bigelow, J. E.*; Gibson, J. R.*; Martin, W. T.*; Pope, R. G.*; Raman, S. <i>Preparation of Actinide Specimens for the US/UK Joint Experiment in the Downreay Prototype Fast Reactor</i>	ORNL-5858 (May 1982)
	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)
	Gilai, D.*; Williams, M. L.*; Cooper, J. H.; Laing, W. R.; Walker, R. L.; Raman, S.*; Stelson, P. H.* <i>Experimental and Calculational Analyses of Actinide Samples Irradiated in EBR-II</i>	ORNL/5791 (Oct. 1982)
	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.; McKown, H. S.; Walker, R. L.; Sherman, R. L.; Pritchard, C. A.; Carter, J. A. <i>Progress Report for the Office of Safeguards and Security for FY 1982</i>	ORNL/TM-8557 (Dec. 1982)
Walton, J. R.	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)

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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/TN-8027 (Dec. 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
Anderson, R. L.*	Anderson, R. L.*; Kollie, T. G.*; Christie, W. R.; Eby, R.; Lyons, J. D.* "Decalibration of Sheathed Thermocouples"	6th Symp. on Temperature, Washington, DC, Mar. 16-18, 1982
Attrill, J. E.	Attrill, J. E.; Clark, B. R.; Davis, H. G.; Eisenhower, B. M.*; Stewart, J. H., Jr. "Industrial Oil Spill Identification Procedure at ORNL"	183rd Am. Chem. Soc. Meet., Las Vegas, NV, Mar. 28-Apr. 2, 1982
Beene, J. R.*	Beene, J. R.*; Bemis, C. E., Jr.*; Kramer, S. D.*; Young, J. P. "Laser-Optical Studies of the Spontaneous-Fission Isomer Am-240m"	Conf. on Lasers in Nucl. Phys., Oak Ridge, TN, Apr. 21-23, 1982
Bostick, D. T.	Bostick, D. T.; Strain, J. E.; McCue, D. D.*; Harper, R. E.*; Bauer, M. L.* "In-Line Instrumentation for Control of Nuclear Fuel Reprocessing II. In-Line Heavy Metals and Oxidant-Reductant Monitors"	Invited, 1982 Winter Meet., Am. Nucl. Soc., Washington, DC, Nov. 14-19, 1982
Brazell, R. S.	Brazell, R. S.; Bayne, C. K.* "Analysis of Volatile Constituents in Physiological Fluids from Tobacco Smoke Exposed Animals"	Int. Symp. on Advances in Chromatogr., Las Vegas, NV, Apr. 7, 1982
Brown, D. K.*	Brown, D. K.*; Maskarinec, M. P.; Francis, C. W.* "An Evaluation of Extraction Techniques to Determine Extractable Organics in Solid Wastes"	Am. Chem. Soc. Meet., Las Vegas, NV, Mar. 28-Apr. 2, 1982
Buchanan, M. V.	Buchanan, M. V.; Kao, G. L. "Mass Spectral Characterization of Isometric Oxygen-Containing Aromatic Compounds Using Methanol Chemical Ionization"	Am. Chem. Soc. Meet., Kansas City, MO, Sept. 12-17, 1982
	Buchanan, M. V.; J. Flanagan*; Rubin, I. B.; Guerin, M. R. "Effects of Hydrotreatment on the Composition of Mutagenic Fractions of a Cosl-Derived Petroleum Substitute"	7th Int. Symp. on Polynuclear Aromatic Hydrocarbons, Battelle's Columbus Laboratories, Columbus, OH, Oct. 26-28, 1982
	Buchanan, M. V. "Chemical Ionization Mass Spectrometry"	UCND Anal. Comm. Meet., Oak Ridge, TN, June 23, 1982

SPEAKER	AUTHOR(S) AND TITLE	PRESENTED AT
Buchanan, M. V.	Buchanan, M. V. "Spectroscopic Organic Analysis"	Anal. Chem. Div. Summer Lecture Series, Oak Ridge Nat. Lab., Oak Ridge, TN, Aug. 13, 1982
	Buchanan, M. V.; Kao, G. L. "Characterization of Isomeric Oxygen- and Nitrogen Containing Compounds Using Chemical Ionization Mass Spectrometry"	Anal. Chem. Div. Infor. Meet., Oak Ridge Nat. Lab., Oak Ridge, TN, July 13, 1982
Carter, J. A.	Carter, J. A.; Smith, D. H.; Walker, R. L. "Mass Spectrometric Measurements for Nuclear Safeguards"	Invited, 30th Annu. Conf. on Mass Spectrom. and Allied Topics, Honolulu, HI, June 6-11, 1982
	Donohue, D. L.; Young, J. P.; Carter, J. A. "Ultra-Trace Analysis by Resonance Ionization Mass Spectrometry"	ISA Tech. Rev. Meet., Washington, DC, Nov. 17-18, 1982
	Carter, J. A. "Measurements Using Resin Bead Technique"	MC&A Representatives Meet., DOE/ORD, Oak Ridge, TN, Dec. 4-15, 1982
Caton, J. E.	Caton, J. E.; Hurst, G. B.*; Olerich, G.; Buchanan, M. V. "Analysis of Water Associated with Coal Liquefaction Systems for Alkaline Organic Components"	FACSS IX Symp., Philadelphia, PA, Sept. 19-24, 1982
	Caton, J. E. "Predictions of Potential Bioactivity from Diffuse Reflectance Spectra of Fossil Fuel Derived Liquids"	Digilab Users Group, Southeast Reg. Meet., Knoxville, TN, Oct. 14, 1982
	Caton, J. E. "Correlation of Chemical Spectroscopic Measurements with Bioassay Test Results"	Anal. Chem. Div. Infor. Meet., Oak Ridge Nat. Lab., Oak Ridge, TN, July 13, 1982
Christie, W. H.	Christie, W. H.; Eby, R. E. "Quantitative SIMS Analysis of Tritium Permeation Barrier Oxide Films Grown on Laser Modified Incoloy Surfaces"	30th Annu. Conf. on Mass Spectrom. and Allied Topics, Honolulu, HI, June 6-11, 1982
	Christie, W. H. "Surface Analysis and Other Aspects of Secondary Ion Mass Spectrometry"	Anal. Chem. Div. Summer Lecture Series, Oak Ridge Nat. Lab., Oak Ridge, TN, June 25, 1982
Costanzo, D. A.	Costanzo, D. A. "CFRP Analytical Chemistry Development"	Semiannual Tech. Rev. of CFRP Process and Eng. R&D Activities, ORNL, March 18, 1982

SPEAKER	AUTHOR(S) AND TITLE	PRESENTED AT
Costanzo, D. A.	Costanzo, D. A. "Analytical Chemistry Development in Support of the Consolidated Fuel Reprocessing Program"	UCND Anal. Comm. Meet., Oak Ridge, TN, June 23, 1982
	Costanzo, D. A. "Analytical Chemistry Development for CFRP"	CFRP Tech. Rev. Meet., Oak Ridge Nat. Lab., Oak Ridge, TN, Aug. 24, 1982
Dalbey, W.*	Dalbey, W.*; Lock, S.*; Garfinkel, S.*; Jenkins, R.; Holmberg, R.; Guerin, M. "Effects of Repeated Exposures to Aerosolized Diesel Fuel"	Soc. Toxicol., Boston, MA, Feb. 22-26, 1982
	Dalbey, W.*; Lock, S.*; Garfinkel, S.*; Jenkins, R.; Holmberg, R.; Guerin, M. "Inhalation Exposures of Rats to Aerosolized Diesel Fuels"	Symp. Toxicol. of Pet. Hydrocarbons, Washington, DC, May 11-13, 1982
Dale, J. M.	Dale, J. M.; Hulett, L. D.; Pendysala, S.* "Energy Loss Spectra of Positrons Scattered from Solid Surfaces"	6th Int. Conf. on Positron Annihilation, The Univ. of Texas at Arlington, TX, Apr. 3-7, 1982
Donohue, D. L.	Donohue, D. L. "Resonance Ionization Mass Spectrometry"	UCND Anal. Comm. Meet., Oak Ridge, TN, June 23, 1982
Dumont, J. W.*	Dumont, J. W.*; Schultz, T. W.*; Buchanan, M. V.; Kao, G. L. "Frog Embryo Teratogenesis Assay: Xenopus (PETAX) - A Short Term Assay Applicable to Complex Environmental Mixtures"	Symp. Application of Short-Term Bioassays in the Analysis of Complex Environ. Mixtures, Chapel Hill, NC, Jan. 25-27, 1982
Dyer, F. F.	Dyer, F. F.; Emery, J. F.; Northcutt, K. J.; Scott, R. M.* "High Fluence Neutron Activation Analysis of Subnanogram Levels of U and Th in High Purity Aluminum and Silicon"	Anal. Chem. Div. Annu. Info. Meet., Oak Ridge Nat. Lab., Oak Ridge, TN, July 12-13, 1982
Eldridge, J. S.	Eldridge, J. S.; Oakes, T. W.*; Hubbard, H. M.* "Off-Site Response for Radiological Emergencies"	1982 UCC-ND and GAT Environ. Prot. Semin., Gatlinburg, TN, Apr. 5-7, 1982
	Eldridge, J. S.; Oakes, T. W.*; Parsons, D. W.*; Fell, R. D.* "Radionuclide Concentrations in Honey and Bees Near Radioactive Waste Disposal Sites"	Annu. Meet. Health Phys. Soc., Las Vegas, NV, June 27-July 1, 1982
	Eldridge, J. S. "Modern Equipment for Combination Use: Precious Metals Inventory Verification, Safeguards Audits, and Emergency Response"	UCND Anal. Comm. Meet., Oak Ridge, TN, June 23, 1982

SPEAKER	AUTHOR(S) AND TITLE	PRESENTED AT
Eldridge, J. S.	Eldridge, J. S. "Radiochemical Analysis"	Anal. Chem. Div. Summer Lecture Series, Oak Ridge National Lab., Oak Ridge, TN, July 30, 1982
	Eldridge, J. S. "Nondestructive Assay Measurements"	1982 DOE Survey of Oak Ridge National Labs. Accountability and Control (Special Mater. Management Dep.), Oak Ridge, TN, June 14, 1982
	Eldridge, J. S. "Measurement Capabilities: Past, Present, and Future"	Industrial Safety and Appl. Health Phys. Div. Staff Meet., Nov. 11, 1982
Gleason, G. I.	Gleason, G. I. "NaI Gamma Spectrum Analysis I and II"	Oak Ridge Assoc. Univ. Training Lecture, Oak Ridge, TN, Feb. 9-10, 1982
	Gleason, G. I. "Gamma Counting Efficiency and Absolute Assay"	Oak Ridge Assoc. Univ. Training Lecture, Oak Ridge, TN, Feb. 11, 1982
	Gleason, G. I. "Advanced Gamma-Ray Spectroscopy Using Ge(Li) Detectors"	Oak Ridge Assoc. Univ. Training Lecture, Oak Ridge, TN, Mar. 16, 1982
Glish, G. L.	Glish, G. L.; McBay, E. H.; Rainey, W. T.*; Liotta, R.* "Pyrolysis GC/MS of Specifically Treated Coals"	30th Annu. Conf. on Mass Spectrom. and Allied Topics, Honolulu, HI, June 6-11, 1982
Griest, W. H.	Griest, W. H.; Tomkins, B. A.; Maskarinec, M. P.; Caton, J. E.; Higgins, C. E.; Rao, T. K.* "Analytical Methodology for Coal Fly Ash Organics and Comparison of Mutagenic and Nonmutagenic Fly Ashes"	Third Symp. on Environ. Anal. Chem., Brigham Young Univ., Provo, UT, June 16-18, 1982
Guerin, M. R.	Guerin, M. R.; Rubin, I. B.; Caton, J. E.; Bayne, C. K.*; Tomkins, B. A.; Ma, C. Y.; Buchanan, M. V.; Griest, W. H.; Ho, C.-h. "Spectroscopic and Chromatographic Screening of Fossil-Derived Oils for Bacterial Mutagenicity"	FACSS IX Symp., Philadelphia, PA, Sept. 19-24, 1982
	Guerin, M. R. "Role of Analytical Chemistry in Toxicology"	University of Georgia, Athens, GA, Feb. 18, 1982

SPEAKER	AUTHOR(S) AND TITLE	PRESENTED AT
Heine, D. R.	Heine, D. R. "Developments and Applications of Inductively Coupled Plasma Emission Spectrometry"	Anal. Chem. Div. Annu. Info Meet., Oak Ridge Nat. Lab., Oak Ridge, TN, July 13, 1982
	Heine, D. R. "Trace Elemental Analysis by Inductively Coupled Plasma and Atomic Absorption Spectrometry"	Anal. Chem. Div. Summer Lecture Series, Oak Ridge Nat. Lab., Oak Ridge, TN, July 23, 1982
Higgins, C. E.	Higgins, C. E.; Griest, W. H.; Guerin, M. R. "Sampling and Analysis of Cigarette Smoke Using Solid Adsorbents"	Am. Chem. Soc. Meet., Kansas City, MO, Sept. 13-17, 1982
Ho, C-h.	Ma, C. Y.; Ho, C.-h.; Guerin, M. R.; Rao, T. K.*; Epler, J. L.* "Evaluation of Procedures Employed for Preparing Samples for Mutagenicity Testing"	Poster Session, Symp. on Application of Short-Term Bioassays in the Analysis of Complex Environ. Mixtures, sponsored by US EPA, Research Triangle Park, NC, Jan. 25-27, 1982
	Jones, A. R.; Guerin, M. R.; Ho, C.-h.; Maskarinec, M. P.; Rao, T. K.* "Removal of Solutes from DMSO Extracts for Chemical Characterization"	Poster Session, Symp. on Application of Short-Term Bioassays in the Analysis of Complex Environ. Mixtures, sponsored by US EPA, Research Triangle Park, NC, Jan. 25-27, 1982
Holmberg, R. W.	Holmberg, R. W.; Moneymun, J. H. "A System for the Continuous Generation of Phosphorus Aerosols from Red Phosphorus-Butyl Rubber"	Smokes/Obscurants Symp. VI, Harry Diamond Laboratories, Adelphi, MD, Apr. 27-29, 1982
Howell, H. E.*	Howell, H. E.*; Mamantov, G.; Wehry, E. L.*; Shaw, R. W. "Photoacoustic Spectroscopy of Matrix-Isolated Polycyclic Aromatic Compounds"	Southeastern Regional Am. Chem. Soc. Meet., Birmingham, AL, Nov. 4, 1982
Hulett, L. D.	Hulett, L. D. "Scanning Electron Microscopy"	Oak Ridge Assoc. Univ. Training Lecture, Oak Ridge, TN, Jan. 14, 1982
	Hulett, L. D. "Scanning Electron Microscopy and X-ray Fluorescence"	Anal. Chem. Div. Summer Lecture Series, Oak Ridge Nat. Lab., Oak Ridge, TN, June 18, 1982
	Hulett, L. D.; Dale, J. M; Pendysal, S.* "The Generation and Detection of	6th Int. Conf. on Positron Annihilation, The

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Hulett, L. D. (cont'd)	Mono-energetic Positrons"	Univ. of Texas at Arlington, TX, Apr. 3-7 1982
	Hulett, L. D. "The Generation of Monoenergetic Positrons and Their Energy Loss and Diffraction Processes in Solids"	Chem. Phys. Semin., Oak Ridge Nat. Lab., Oak Ridge, TN, May 24, 1982
Jenkins, R. A.	Jenkins, R. A.; Gayle, T. H.* "Instrumental Measurement of the Total Particulate Matter of Cigarette Smoke"	Tobacco Chemists' Res. Conf., Raleigh, NC, Oct. 25-27, 1982
Kao, G. L.	Kao, G. L.; Bucharan, M. V.; Guerin, M. R. "Determination of Oxygen-Containing Compounds by Gas Chromatography/Chemical Ionization Mass Spectrometry"	CONFAB "82", Saratoga, NY, July 20-23, 1982
Keller, J. M.	Keller, J. M.; Heine, D. R.; Riegel, C.* "Effects of Inorganic Acids on ICP Analysis"	Am. Chem. Soc. Meet., Las Vegas, NV, Mar. 28- Apr. 2, 1982
	Keller, J. M.; Heine, D. R.; Rogers, C. W. "Simultaneous Operation of a Polychromator and Monochromator on a Single Source In- ductively Coupled Plasma Emission Spectrometer"	1982 Spring DECUS U.S. Symp., Atlanta GA, May 10-14, 1982
	Keller, J. M. "Determination of U and Pu in Nuclear Fuel Reprocessing Solutions"	Tech. Rev. of CFRP Process and Eng. R&D Activities, Oak Ridge Nat. Lab., Oak Ridge, TN, December, 1982
Lapicki, G.*	Lapicki, G.*; Auble, R. L.*; Hulett, L. D.; O'Kelly, G. D.*; Kim, H. J.*; Milner, W. T.*; Raman, S.*; Shahal, O.*; Vane, C. R.*; Young, J. P. "Extraction of X-Ray Production Cross Sections by 36-103 MeV Ar Ions in Thick Targets of V, Cu, Ni, Ta, and Pt"	Int. Conf. X-Ray and Atomic Inner-Shell Physics, Eugene, OR, Aug. 23-27, 1982
Lee, D. A.	Lee, D. A.; Eatherly, W. P.*; Kintigh, J. D.* "Analytical Evaluation of Corrosion-Induced Microstructural Changes in a U.S. Core Graphite"	24th Rocky Mountain Conf., Denver, CO, Aug. 1-5, 1982
Lowndes, D. H.*	Lowndes, D. H.*; Cleland, J. W.*; Christie, W. H.; Eby, R. E.; Narayan, J.*; Wood, R. D.*; Nilson, J. A.*; Dass, S.* "Pulsed Excimer Laser (308 nm) Annealing of Ion Implanted Silicon"	Mater. Res. Soc. Meet., Boston, MA, Nov. 1-4, 1982

SPEAKER	AUTHOR(S) AND TITLE	PRESENTED AT
Lyon, W. S.	Bulet, L. D.; Dale, J. M.; Pendyala, S.*; Chang, Y. K.*; Lyon, W. S. "Positron Spectroscopy Applied to the Analysis of Surface"	Int. Symp. on Applications and Technol. of Ionizing Radiations, Univ. of Riyadh Campus, Malaz, Riyadh, Saudi Arabia, Mar. 12-17, 1982
	Lyon, W. S. "Practical Applications of Activation Analysis and Other Nuclear Techniques"	Invited, Int. Symp. on Applications and Technol. of Ionizing Radiations, Univ. of Riyadh Campus, Malaz, Riyadh, Saudi Arabia, Mar. 12-17, 1982
	Lyon, W. S. "A Review of Energy Related Research in Activation Analysis and X-Ray Fluorescence"	Invited, Am. Nucl. Soc., 1982 Annu. Meet., Los Angeles, CA, June 6-11, 1982
	Lyon, W. S.; Bujdoso, E.*; Noszlopi, I.* "Direction and Growth in Prompt Nuclear Analysis"	Invited, Am. Nucl. Soc., 1982 Winter Meet., Washington, DC, Nov. 14-18, 1982
	Lyon, W. S. "Activation Analysis"	Anal. Chem. Div. Summer Lecture Series, Oak Ridge Nat. Lab., Oak Ridge, TN, July 16, 1982
Ma, C. Y.	Ma, C. Y.; Ho, C. -h.; McBay E. H.; Buchanan, M. V. "Identification of Carbonyl Compounds in a Mutagenic Neutral Coal Oil Fraction"	Am. Chem. Soc., 34th Annu. Southeastern Reg. Meet., Birmingham, AL, Nov. 3-5, 1982
Manning, D. L.	Manning, D. L.; Maskarinec, M. P. "The Analysis of Low Molecular Weight Carboxylic Acids by HPLC with Conductivity Detection,"	Am. Chem. Soc. Meet., Las Vegas, NV, Mar. 29-Apr. 2, 1982
Maskarinec, M. P.	Maskarinec, M. P.; Brazell, R. S.; Brown, D. K.* "Analytical Chemistry of Solid Wastes: From Methods Development to Quality Assurance"	Invited, Soc. of Anal. Chemists of Pittsburgh, PA, Jan. 4, 1982
	Maskarinec, M. P.; Larimer, F. W.*; Francis, C. W.* "Preparation of Solid Wastes and Associated Leachates for Chemical and	Symp. on Appl. of Short-Term Bioassays in the Analysis of Complex

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Maskarinec, M. P. (cont'd)	Biological Analysis"	Environ. Mixtures, Chapel Hill, NC, Jan. 25-27, 1982
	Maskarinec, M. P.; Brown, D. K.* "Batch and Column Extraction Procedures for Organics in Solid Wastes"	1982 Am. Soc. Test Mater. D-34 Meet., Orlando, FL, Jan. 28-29, 1982
	Maskarinec, M. P.; Brown, D. K.*; Harvey, R. W. "Comparison of Extraction Procedures for Solid Wastes: Organic Materials"	Invited, 1982 Am. Soc. Test. Mater. D-34 Meet., Orlando, FL, Jan. 28- 29, 1982
McKown, H. S.	McKown, H. S. "Applications of Thermal Ionization Mass Spectrometry at ORNL"	15th Annu. Carbide Nucl. Div. Anal. Meet., Lexington, KY, Dec. 1, 1982
Moneyhun, J. H.	Moneyhun, J. H. "Army Smoke Obscurant Program"	UCND Anal. Comm. Meet., Oak Ridge, TN, June 23, 1982
Peterson, J. R.*	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	12 emes Journees des Actinides, Orsay, FRANCE, May 24-25, 1982
Ramsey, J. M.	Ramsey, J. M.; Whitten, W. B. "Recent Advances in Delayed Lasing Measurements"	Pittsburgh Conf., Atlan- tic City, NJ, Mar. 8- 12, 1982
	Ramsey, J. M. "Subnanosecond Phase Fluorimetry With a Self-Modulated Argon-Ion Laser"	Pittsburgh Conf., Atlan- tic City, NJ, Mar. 8- 12, 1982
	Ramsey, J. M. "Fluorescence Lifetime Measure- ments Using Self-Modulated Argon-Ion Lasers"	Invited, FACSS Conf., Philadelphia, PA, Sept. 20-24, 1982
	Ramsey, J. M.; Whitten, W. B. "Fourier Trans- form Microwave Spectroscopy"	Anal. Chem. Div. Infor- Meet., Oak Ridge Nat. Lab., Oak Ridge, TN, July 13, 1982
	Ramsey, J. M.; Whitten, W. B. "A New Approach to Fourier Transform Spectroscopy"	Invited, 1982 Gordon Res. Conf. on Anal. Chem., New Hampton, NH, Aug. 16-20, 1982
Rao, T. K.*	Ma, C. Y.; Rao, T. K.*; Griest, W. H.; Ho, C.-h; Guerin, M. R.; Hardigree, A. A.*; Epler, J. L.* "Preparative Scale Organic Extraction and Mutagenicity Analysis of Fly Ash"	13th Annu. Meet. of the Environ. Mutagen Soc., Boston, MA, Feb. 26- Mar. 2, 1982

SPEAKER	AUTHOR(S) AND TITLE	PRESENTED AT
Rogers, C. W.*	Rogers, C. W.*; Keller, J. M.; Warmack, R. J. "Selection of a Multi-User Operating System for the Laboratory"	1982 Spring Decus U.S. Symp., Atlanta, GA, May 10-14, 1982
Ross, H. H.	Ross, H. H. "Liquid Scintillation Counting: Recent Trends in the Assay of Nuclides of Biological Interest"	Am. Nucl. Soc. Winter Meet., San Francisco, CA, Nov. 29-Dec. 4, 1982
Rosseel, T. M.	Rosseel, T. M.; Auble, R. L.*; Hulett, L. D.; Kahane, S.*; Kim, H. J.*; Milner, W. T.*; O'Kelley, G. D.*; Raman, S.*; Slaughter, G. G.*; Vane, C. R.*; Young, J. P. "Ana- lytical Applications of Heavy Ion Induced X-Ray Fluorescence and Auger Electrons"	24th Rocky Mt. Conf., Denver, CO, Aug. 1-5, 1982
	Rosseel, T. M.; Dale, J. M.; Dunn, H. W.; Hulett, L. D.; Kim, H. J.*; Milner, W. T.*; Raman, S.*; Slaughter, G. G.*; Vane, R.*; Young, J. P. "Analytical Implications of Heavy-Ion Induced X-ray Fluorescence"	Anal. Chem. Div. Infor- Meet., Oak Ridge Nat. Lab., Oak Ridge, TN, July 13, 1982
Sepaniak, M.*	Sepaniak, M.*; Maskarinec, M. P. "Open Tubular Capillary Liquid Chromatography With Rectangular and Concentric Columns"	1982 Pittsburgh Conf., Atlantic City, NJ, Mar. 8-13, 1982
Scott, T. G.	Scott, T. G. "The Determination of Ra-226 by the Rn-222 Emanation Technique"	UCND Anal. Comm. Meet., Oak Ridge, TN, June 23, 1982
Shaw, R. W.	Shaw, R. W. "Lasers in Analytical Chemistry"	UCND Anal. Comm. Meet., Oak Ridge, TN, June 23, June 23, 1982
	Shaw, R. W. "Optoacoustics at Cryogenic Temperatures"	Invited, Seminar at Transuranium Res. Lab., Oak Ridge Nat. Lab., Oak Ridge, TN, Feb. 26, 1982
Shultz, W. D.	Shultz, W. D.; Vogt, J. J.* "Career Paths for Technicians at a National Laboratory"	Invited, Am. Chem. Soc. Meet., Las Vegas, NV, Mar. 28-April 1, 1982
Smith, D. H.	Smith, D. H.; Walker, R. L.; Carter, J. A.; "Mass Spectrometry in Safeguards"	Am. Chem. Soc. Meet., Kansas City, MO, Sept. 12-17, 1982
	Smith, D. H. "Applications of Mass Spectrometry to Waste Solution"	UCND Anal. Comm. Meet., Oak Ridge, TN, June 23, 1982

SPEAKER	AUTHOR(S) AND TITLE	PRESENTED AT
Strain, J. E.	Strain, J. E.; Bostick, D. T.; McCue, D. D.* Harper, R. E.* "In-Line Instrumentation for Control of Nuclear Fuel Reprocessing Part I. In-Line Neutron Poison and Free Acid Monitor"	Invited, 1982 Winter Meet., Am. Nucl. Soc., Washington, DC, Nov. 14-19, 1982
Stewart, J. H.	Stewart, J. H., Jr.; Dunn, H. W. "The Quantitative Determination of Chrysotile Asbestos by X-Ray Diffractometry"	1982 UCC-ND and GAT Environ. Prot. Semin., Gatlinburg, TN, Apr. 5- 7, 1982
Tomkins, B. A.	Tomkins, B. A.; Reagan, R. R.; Maskarinec, M. P.; Harmon, S. H.; Griest, W. H.; Caton, J. E. "Analytical Chemistry of Polycyclic Aromatic Hydrocarbons Present in Coal-Fired Power Plant Fly Ash"	7th Int. Symp. on Poly- nuclear Aromatic Hydro- carbons, Columbus, OH, Oct. 26-28, 1982
	Tomkins, B. A.; Ostrum, V. H.*; Caton, J. E. "A Rapid HPLC/HPLC Screening Procedure for 2-Aminonaphthalene in: Natural, Synthetic, and Refined Crudes"	1982 Pittsburgh Conf., Atlantic City, NJ, Mar. 8-13, 1982
Todd, P. J.	Todd, P. J.; Warmack, R. J. "Calculated Angular Distributions of Fragment Ions from Polyatomic Ion Collisions"	30th Annu. Conf. on Mass Spectrom and Allied Topics, Honolulu, HI, June 6-11, 1982
Vane, R.*	Vane, R.*; Ahle, R. L.*; Hullett, L. D., Jr.; Kahane, S.*; Kim, H. J.*; McDaniel, F. D.*; Milner, W. T.*; O'Kelley, G. D.*; Raman, S.*; Rosseel, T. M.; Slaughter, G. G.*; Varghese, S. L.*; Young, J. P. "Chemical Sensitivity of Heavy-Ion Induced X-Ray Spectra"	Am. Phys. Soc. Meet., Washington, DC, Apr. 26- 29, 1982
Van Hoesen, S. D.*	Van Hoesen, S. D.*; Cowser, K. E.*; Cushman, R. M.*; Dreibelbis, W. G.*; Griest, W. H.; Klein, J. A.*; Larimer, F. W.*; Meyer, H. R.*; Strand, R. H.* "Health and Environ- mental Assessment of Low BTU Gasification"	5th Miami Int. Conf. Alternative Energy Sources, Miami beach, FL, Dec. 13-15, 1982
Voshell, J. R.*	Voshell, J. R., Jr.*; Eldridge, J. S.; Oakes, T. W.* "Radionuclide Transfer by Aquatic Insects in a Contaminated Pond"	Health Phys. Soc. Annu. Meet., Las Vegas, NV, June 27-July 1, 1982
Whitten, W. B.	Whitten, W. B.; O'Lear, J. R.* "Time-of-Flight Optical Spectrometry With Optical Fibers in the Near UV"	Invited, Am. Chem. Soc. Meet., Kansas City, MO, Sept. 12-17, 1982
	Whitten, W. B. "Time-of-Flight Optical Spectrometry"	Invited, Piedmont Sec- tion, Appl. Spectrosc. Soc., Oak Ridge, TN, Dec. 9, 1982

SPEAKER	AUTHOR(S) AND TITLE	PRESENTED AT
Whitten, W. B.	Whitten, W. B. "Time Domain Optical Spectrometry with Fiber Optic Waveguides"	Am. Chem. Soc. Meet., Kansas City, MO, Sept. 12-17, 1982
Young, J. P.	Young, J. P.; Hurst, G. S.*; Kramer, S. D.* "Analytical Applications of Resonance Ionization Spectroscopy"	Invited, Fed. of Anal. Chem. and Spectrosc. Soc., Philadelphia, PA Sept. 19-24, 1982
	Young, J. P. "Resonance Ionization Mass Spectrometry"	Health and Safety Res. Div. Semin., June 4, 1982
Young, R. T.*	Young, R. T.*; Narayan, J.*; Christie, W. H. "Effect of Pulse Duration on the Annealing of Ion Implanted Silicon With a XeCl Excimer Laser"	Mater. Res. Soc., Boston, MA, Nov. 1-4, 1982

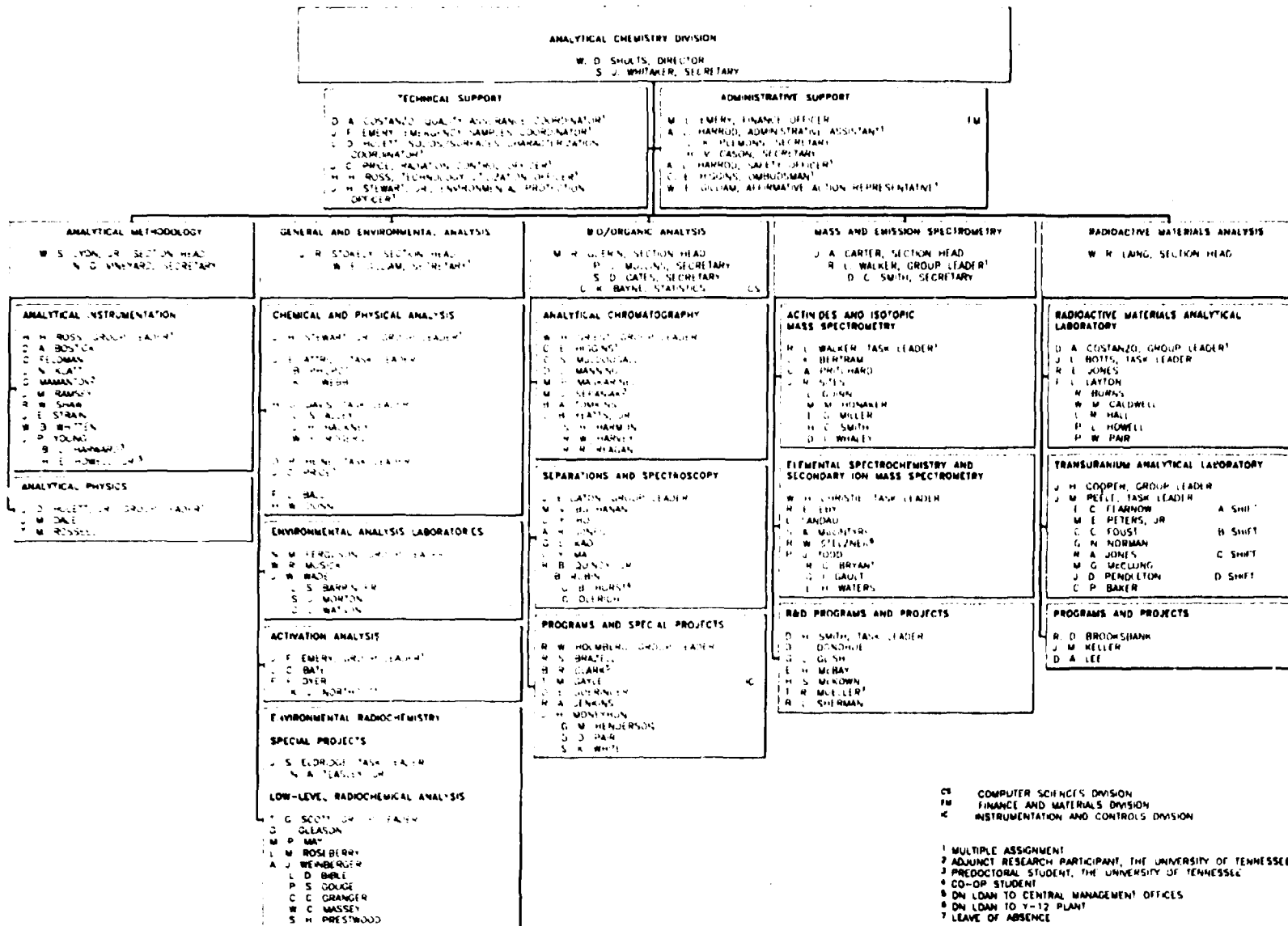
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	Ana'lum	Ana'l. Chem.	Ana'l. Chim. Acta	Ana'l. Lett.	Health Physica	J. Radioanal. Chem.	ORNL Reports	Proposals	Radiochem. Radioanal. Lett.	Other	Total
Attrill, J. E.										2	2
Brazell, R. S.		4								2	6
Brooksbank, R. D.										1	1
Buchanan, M. V.		1						1		1	3
Carter, J. A.		1		1				2			4
Caton, J. E.			1					1		1	3
Donohue, D. L.				1							1
Eldridge, J. S.					2						2
Goeringer, D. E.							1	1		1	3
Griest, W. H.		2					1	2		1	6
Guerin, M. R.								6		2	8
Ho, C.-h.							1				1
Klatt, L. N.			1				1			1	3
Laing, W. R.							1			1	2
Lyon, W. S.	2	2	2			5		2	8	7	28
Manning, D. L.		4									4
Maskarinec, M. P.			1							3	4
Ramsey, J. M.		2								1	3
Ross, H. H.		1								1	2
Rosset, T. R.										1	1
Scott, T. G.							1				1
Shaw, R. W.								1		2	3
Stewart, J. H.		2									2
Tomkins, B. A.		5	1							1	7
Young, J. P.		3						1		2	6
Whitten, W. B.								1			1
Total	2	27	6	2	2	5	6	18	8	31	107

DIVISIONAL MAN-POWER AND FINANCIAL SUMMARY
FY 1982

Source	Funding (in thousands)	Man-years
DOE programs		
Health and Environmental Research	1,131	11.6
Basic Energy Sciences	1,101	10.7
Consolidated Fuel Reprocessing Program	200	2.0
Fossil Energy	234	2.5
Nuclear Materials Security and Safeguards	140	1.4
Reactor Research and Development	<u>70</u>	<u>0.8</u>
Total DOE programs	2,876	29.0
Work for others - federal agencies		
Department of Defense (U.S. Army)	870	7.1
National Cancer Institute	450	3.7
Environmental Protection Agency	395	3.8
State Department (I.S.P.O.)	66	0.7
National Regulatory Commission	88	0.6
Miscellaneous	<u>10</u>	<u>0.1</u>
Total work for others - federal agencies	1,879	16.0
Work for others - nonfederal agencies		
Electric Power Research Institute	145	1.6
Battelle Pacific NW Laboratory	36	0.4
Protective Coating Companies	112	1.2
E. I. DuPont, Savannah River Laboratory	55	0.5
Sandia National Laboratory	40	0.4
General Public Utilities	225	2.3
Bendix Corporation	25	0.2
Miscellaneous	<u>35</u>	<u>0.4</u>
Total work for others - nonfederal agencies	673	7.0
ORNL divisions - support	5,860*	81.0
Division administration	<u> </u>	<u>14.0</u>
Total financial plans	11,288	147.0

*Reflects Laboratory general and administration expenses and general plant services.



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