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ANALYTICAL CHEMISTRY LABORATORY
Progress Report for FY 1994

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I. INTRODUCTION

The purpose of this report is to summarize the activities of the Analytical Chemistry Laboratory (ACL) at Argonne National Laboratory (ANL) for Fiscal Year (FY) 1994 (October 1993 through September 1994). This annual report is the eleventh for the ACL and describes continuing effort on projects, work on new projects, and contributions of the ACL staff to various programs at ANL.

The Analytical Chemistry Laboratory is a full-cost-recovery service center, with the primary mission of providing a broad range of analytical chemistry support services to the scientific and engineering programs at ANL. The ACL also has a research program in analytical chemistry, conducts instrumental and methods development, and provides analytical services for governmental, educational, and industrial organizations. The ACL handles a wide range of analytical problems. Some routine or standard analyses are done, but it is common for the Argonne programs to generate unique problems that require significant development of methods and adaption of techniques to obtain useful analytical data.

The ACL is administratively within the Chemical Technology Division (CMT), its principal ANL client, but provides technical support for many of the technical divisions and programs at ANL. The ACL has four technical groups -- Chemical Analysis, Instrumental Analysis, Organic Analysis, and Environmental Analysis -- which together include about 45 technical staff members (see Fig. 1 for the organization chart at the end of FY 1994). Talents and interests of staff members cross the group lines, as do many projects within the ACL. The Chemical Analysis Group uses wet-chemical and instrumental methods for elemental, compositional, and isotopic determinations in solid, liquid, and gaseous samples and provides specialized analytical services. Major instruments in this group include an ion chromatograph (IC), an inductively coupled plasma/atomic emission spectrometer (ICP/AES), spectrophotometers, mass spectrometers (including gas-analysis and

thermal-ionization mass spectrometers), emission spectrographs, autotitrators, sulfur and carbon determinators, and a kinetic phosphorescence uranium analyzer.

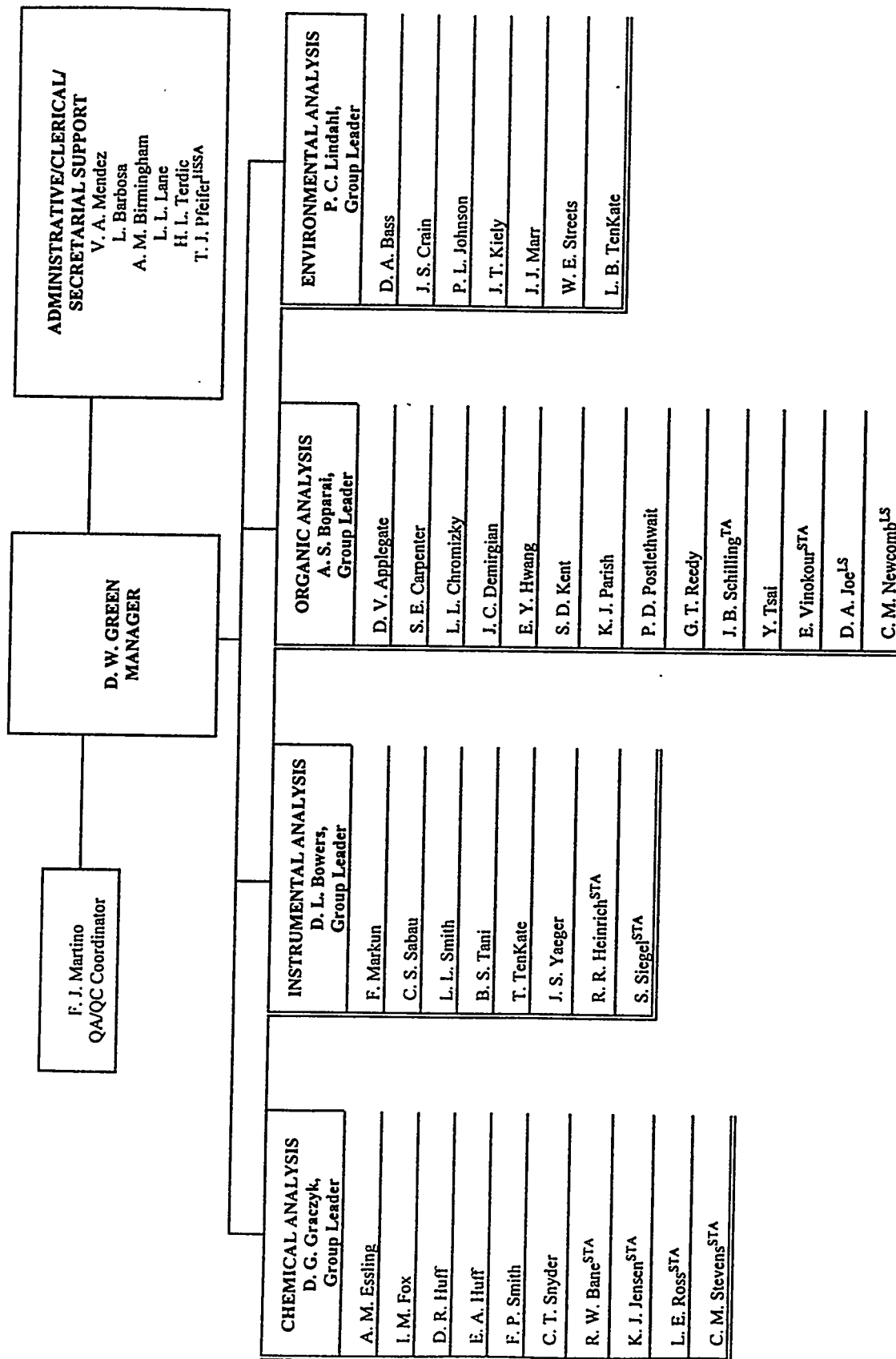
The Instrumental Analysis Group uses nuclear counting techniques to determine the radiochemical constituents in a wide range of sample types, from environmental samples with low radioactivity to samples with high radioactivity that require containment. Other types of determinations use X-ray diffraction and X-ray fluorescence of solids. Major instrumentation includes nuclear detectors, a variety of multichannel analyzers and associated counting equipment, X-ray diffractometers, energy dispersive X-ray analyzers, and a scanning electron microscope with energy dispersive X-ray fluorescence capability. Radioactive samples can be analyzed by using ICP/AES or IC.

The Organic Analysis Group uses a number of complementary techniques to separate organic compounds and measure them at trace levels. Samples that can be analyzed include complex organic mixtures and compounds, pesticides, potentially hazardous wastes, and drugs. Major instrumentation in this group includes gas chromatograph/mass spectrometer (GC/MS) systems, Fourier transform infrared (FTIR) spectrometers, a GC/matrix-isolation FTIR/MS (GC/MI-IR/MS) instrument, GCs, an FTIR microscope, high-performance liquid chromatographs, a supercritical fluid extraction (SFE) apparatus, liquid chromatography/mass spectrometry (LC/MS) system, and a supercritical fluid chromatograph (SFC). Facilities exist for analysis of radioactive samples. Highly radioactive samples can be treated to separate the organic and radioactive constituents. Personnel in the Organic Analysis Group also develop methods for such purposes as (1) continuously monitoring incinerator emissions, (2) detecting organic compounds remotely and performing rapid on-site determination of organic constituents with FTIR DOE waste containers. Chemometrics techniques are used for extracting useful information from sensor response data.

The Environmental Analysis Group analyzes environmental, hazardous-waste, and coal

Figure 1. ANALYTICAL CHEMISTRY LABORATORY
Organization Chart

9/30/94



^{LSA} = High School Student Aide; ^{STA} = Special Term Appointee; ^{LS} = Lab Support.

absorption spectrophotometers (AAS) equipped with flame, flameless, cold-vapor mercury, and hydride-generation capabilities; toxicity characteristic leaching procedure (TCLP) extraction facilities; an automated carbon/hydrogen/nitrogen (CHN) analyzer; an inductively coupled plasma/mass spectrometer (ICP/MS); and a sulfur determinator. The group also provides quality assurance support to DOE and other clients for various kinds of analytical data.

In FY 1994, the ACL continued to receive samples requiring multigroup efforts. Typical of those samples are "orphan" waste samples whose ownership and composition are unclear, and samples for analyses in support of ANL's decontamination, decommissioning, and cleanup activities.

The ACL has a sample receiving system that allows efficient processing of environmental samples and hazardous and mixed-waste samples, including chain-of-custody procedures. The ACL also has quality assurance (QA) and quality control (QC) systems in place to produce data packages that meet the reporting requirements of the U.S. DOE, the U.S. Environmental Protection Agency (EPA), and other organizations.

II. ADMINISTRATIVE HIGHLIGHTS

The ACL is both a full-cost-recovery service center in support of ANL programs and an organization doing research and development in support of DOE programs. Generally, operating funding for ACL research is included as part of CMT's total budget, while the ACL effort on these programs is also included as ACL income. In FY 1994, the total funding for the ACL increased to its highest level, as shown in Fig. 2.

The ACL recovered 101.9% of its total operating costs in FY 1994. Over the last two years, the ACL has recovered 101.1% of its costs. Approximately 60% of the income of the ACL in each of the last two years came from service work through ANL programs, and

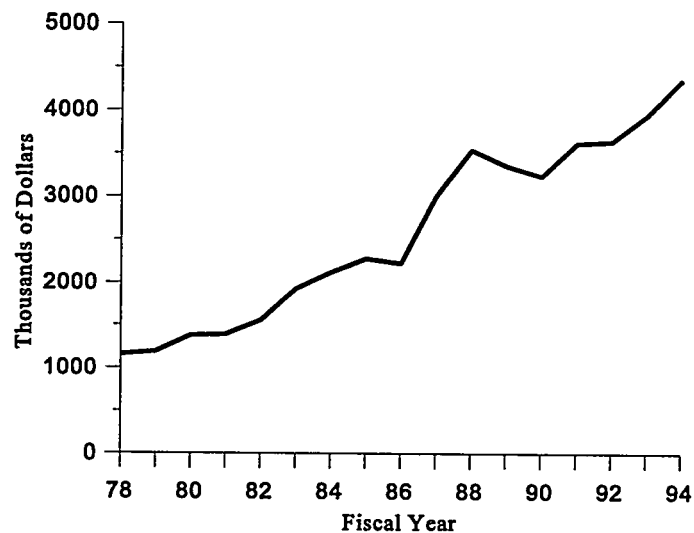


Fig. 2. Total ACL Funding History

the remainder came directly to the ACL from DOE, other DOE sites than ANL, other federal agencies, and external users of ACL services. The ACL effort, including divisional administration, includes about one-half recoverable time (sales to users in Fig. 3). Figure 4 gives a breakdown of the total operating costs for FY 1994. The majority of those costs are effort-related.

In FY 1994, the ACL obtained valuable help from seven Special Term Appointees (STAs) and three people from Lab Support, a commercial laboratory personnel organization, to assist with load leveling. The STAs, which include several retired ACL staff, provided more than one full-time equivalent of assistance in FY 1994. In addition, nine students and co-ops, one high-school faculty

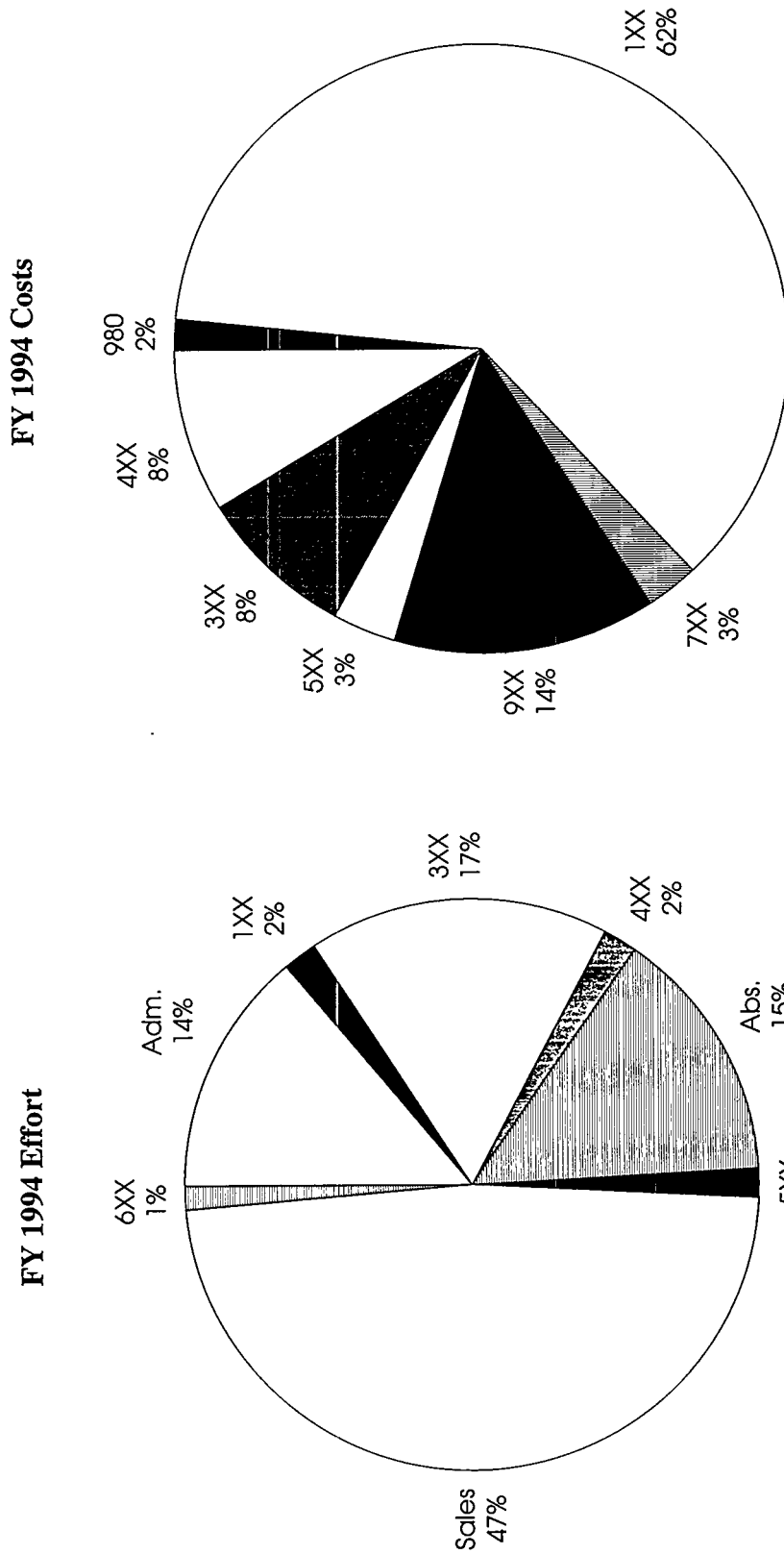


Fig. 3 Analytical Chemistry Laboratory
FY 1994 Effort

Adm. = administration
Abs. = absences
1XX = operations administration
3XX = analytical overhead; e.g., instrument maintenance
4XX = non-analytical overhead; e.g., safety
5XX = professional activities
6XX = experimentation

Fig. 4. Analytical Chemistry Laboratory
FY 1994 Total Operating Costs

1XX = salaries and fringes
3XX = materials and services
4XX = services, subcontracts, etc.
5XX = shopwork and work projects
7XX = redistributions
9XX = indirect costs
980 = intradivisional allocation

member, and two college faculty members supplemented the regular ACL staff in FY 1994 (not all are included in Fig. 1 because they were not in ACL at the end of the fiscal year).

New ACL capabilities established in FY 1994 include an automated flashpoint tester to meet an EPA method requirement, a platinum-lined oxygen combustion bomb for more accurate determination of trace metals in waste and environmental samples, an α/β discriminating liquid scintillation counter with a low-background for screening environmental samples, and a high-efficiency (~50%), high-purity germanium detector for gamma-emitting nuclides. In addition, a new scanning electron microscope user facility was opened for operation this year.

The ACL worked on three projects in FY 1994 with ANL Support Development Funds. The ACL established the capability to analyze mixed-waste samples by means of graphite furnace atomic absorption spectrophotometry. With this added capability, the ACL can now determine the full set of metals of interest in the analysis of waste samples containing radioactive materials. Work was initiated to add a flow injection nebulizer system to our ICP/MS instrument. In addition, work was initiated on a fully automated high-performance gel permeation chromatograph (GPC) system. The new system will be used for cleaning up environmental and mixed-waste samples.

Four projects were funded in FY 1994 by Analytical Development Funds, which were established several years ago to support analytical effort on small projects that are likely to result in publication and/or technical presentation. Development work was done on procedures for using high-pressure microwave dissolution to make gross α/β determinations for environmental and mixed-waste samples. Development work was done on preparation of waste oil, a relatively common waste form from ANL work, for determination of both the hazardous metals and radioactivity. Certain EPA methods were evaluated for implementation using the ICP/MS system. Some development work was done to allow the analysis of opaque samples by reflectance FTIR microscopy. Results from these projects are given later in this report.

The ACL continues to improve in the areas of safety, quality assurance, and operation efficiency. Through the ANL "orphan waste" program, the ACL continued to remove waste materials and samples for which no "owner" could be identified. The ACL orientation for new CMT employees (and others as requested) now includes information about sample transportation. Regular inspections of work areas by ACL personnel, as well as other ANL and DOE personnel, resulted in improvements in ACL facilities and operating procedures with respect to environment, safety, and health. Quality of analytical results continues as a high priority for the ACL; quality assurance activities are covered in Section IV of this report. Several improvements were made in the computer system during FY 1994 to improve the efficiency and accuracy with which samples are tracked and results are reported.

The remainder of this report is a collection of short descriptions of the analytical work performed by the staff of the ACL in FY 1994 on selected projects. Although the ACL receives about 1500 different jobs annually, this report covers approximately 60 topics. Many other contributions by ACL staff are not included because they involved generation of analytical data on only a relatively small number of samples. Despite their small scope, the analytical results often make an important contribution to the overall success of the project that received the data. These contributions to projects can easily be overlooked, but collectively they form an important part of the mission of the ACL.

III. TECHNICAL HIGHLIGHTS

Engineering Studies of Pyrochemical Processes for Integral Fast Reactor Fuels (C. S. Sabau, D. L. Bowers, F. P. Smith, J. T. Kiely, and J. S. Crain)

In the electrorefining of spent fuels for the Integral Fast Reactor (IFR), metallic fuel pins (U, Pu, Zr) are chopped into small segments and placed in a basket that is introduced into molten salt ($\text{LiCl} + \text{KCl}$) at 500°C . The basket is connected to a dc power supply and made anodic; nearly pure uranium is removed from the spent fuel by electrotransport to solid cathodes, followed by electrotransport of the plutonium and any remaining uranium to liquid cadmium cathodes. Engineering-scale studies are being conducted in CMT to develop processes to recover transuranic (TRU) elements from the spent electrolyte salt and metal discharged from the electrorefiner and to separate the fission products so that the treated salt and metal can be recycled. The ACL has contributed to this effort by determining elements of interest in samples from the halide-salt electrolyte, cadmium anode, and the cathode product. Special dissolution procedures are followed for each type of sample matrix, and separation schemes based on solvent extraction and ion exchange are used to isolate the desired elements from matrix components and, subsequently, to separate these elements. Cations, including uranium and plutonium, are determined in samples with an ICP/AES instrument configured to analyze radioactive solutions. The mass spectrometric isotope dilution (MSID) technique is used whenever higher precision and accuracy than attainable with ICP/AES are required. An additional instrument, an ICP/MS, was used to determine the amount of those metals that were low (<10 ppm) in concentration.

Experiments to which ACL contributed during the past year include studies on the extraction efficiency of a specially designed centrifugal contactor ("pyrocontactor") for carrying out salt/metal extractions. The ACL's measurement of cerium in the cadmium and LiCl-KCl salt after processing demonstrated the effectiveness of the pyrocontactor. Further tests with the pyrocontactor used

cerium in the cadmium phase and Y and La in the eutectic salt. The cerium is then used to separate La from Y in the salt. The analytical results showed good separation between the La and Y. These tests demonstrated the feasibility of extracting TRU from the rare earth chlorides and uranium.

Studies were also undertaken to demonstrate the feasibility of electrochemically reducing UCl_3 and PuCl_3 in the electrorefiner salt by using Li-Cd alloy (5.9 wt% Li) as an anode material. Analytical results showed that the U and Pu content in the salt had been reduced by a factor of 150, and the rare earth elements (Y, Sm, Gd, Nd, Ce, and La) had been reduced by a factor of four. These results demonstrate the possibility of salt purification using this process.

Analytical Certification of IFR Special Reference Materials (A. M. Essling, D. G. Graczyk, F. P. Smith, I. M. Fox, C. S. Sabau, and D. L. Bowers)

The Analytical Laboratory in the Fuels and Engineering Division at ANL-West has developed methods for analyses that will be required for process control and materials accountability during the planned demonstration of the IFR fuel cycle. To help in their methods development/validation and to provide a source of quality-control samples for use during the fuel-cycle demonstration, the CMT (J. Ackerman et al.) has prepared two sets of "Special Reference Materials," each consisting of specified analytes in a matrix typical of samples that will be generated during electrorefiner operations at the IFR Fuel Cycle Facility of ANL-West. It is the task of the ACL to characterize each batch of materials by chemical analysis and provide reference values for the concentrations of individual constituents in each matrix.

In FY 1993, the CMT researchers prepared nominal 1-g samples of two special reference materials. One of these, designated SRM-A, contains enriched uranium in a LiCl-KCl salt matrix. The other, SRM-B, has the same salt matrix but contains U, Pu, Am, and a mixture of simulated fission-product and process elements such as rare earths, alkali metals, and transition metals. To

provide accurate and precise U and Pu assays in this work, we applied the mass spectrometric isotope dilution method with a special internal-standard technique for the mass-spectrometric uranium measurements. Americium was determined by gamma spectrometry; metals and rare earths were measured by ICP/AES; Cs and Rb were determined by flame emission or atomic absorption after separation from U and Pu.

All of the uranium isotopic and compositional analyses on SRM-A have been completed. The results of uranium assays on 10 SRM-A samples analyzed in duplicate show a mean concentration of 2.5058 wt% U, with a standard deviation of 0.0022 wt%. Our detailed report on SRM-A, which includes the analysis results, discussion of the method protocols, and an evaluation of the data obtained, is almost completed. All of the remaining SRM-A "standards" were sent to ANL-West, and the analytical laboratory there is currently testing and validating a methodology for comparable uranium assays.

The plutonium isotopic and compositional analyses on seven SRM-B samples analyzed in duplicate are practically complete (some rechecks are in progress), as are the uranium isotopic and compositional analyses. All of the other elements of SRM-B designated to be measured (e.g., americium, rare earths, alkali metals, and transition metals) have been measured. When all the results are in, our data will be submitted to ANL-West. As with SRM-A, the data obtained on SRM-B by ANL-East and ANL-West will be compared to validate this more complicated, multiple-element reference material.

Analysis of Soil Samples for Special Volatile Organics (D. V. Applegate, S. M. Macha, A. S. Boparai, M. A. Gritters, and L. L. Chromizky)

The ACL analyzed soil samples taken by the Environment and Waste Management (EWM) Program during the closure of the 317 Area at ANL. This closure required determination of several

special volatile organic compounds (VOCs) i.e., compounds not on the target compound list of the EPA Contract Laboratory Program. However, these special analytes could not be determined by the traditional purge-and-trap methods because their purge efficiencies from water are very poor. Two methods were developed for determination of the special VOCs. These methods allow for fast analysis while still meeting the traditional quality assurance requirements of the Resource Conservation and Recovery Act (RCRA). One method involved a modified purge-and-trap procedure, and the other involved a decane extraction.

Several data packages were prepared from analysis of nearly 50 samples, which were mostly soil or sand. The analytical data helped EWM characterize the 317 Area soil for several unique VOCs and determine what cleanup was needed.

Analysis of Highly Acidic Mixed Waste (D. V. Applegate, L. L. Chromizky, P. D. Postlethwait, S. D. Kent, Y. Tsai, A. S. Boparai, and D. L. Bowers)

The ACL assisted the Environment and Waste Management Program in developing methods for analyzing some unique mixed-waste samples. These samples are contaminated with americium at a level that exceeds the maximum level of radioactivity for analysis in our laboratory. In addition, the sample matrix is highly acidic. Because of the contamination level and acidity, we had to develop new ways of analyzing these samples for volatile and semivolatile organic compounds (SVOCs). For volatile organic compounds (VOCs), we are developing an off-line purge-and-trap method for use in a laboratory configured for work with radioactive materials in a glovebox. This method involves purging the VOCs from corrosive aqueous sample onto a trap. The traps may then be surveyed and sent to our normal organic analysis laboratory. For determination of SVOCs, we have focused on solid-phase extraction, which is a method that, compared to traditional separatory funnel methods, is much less complicated and time consuming. This method will reduce the

analyst's exposure during sample preparation and will allow for quick and easy setup in a laboratory that is equipped for handling high-level radioactive samples.

Analysis of Environmental Samples for the U.S. Department of Agriculture (L. L. Chromizky and P. D. Postlethwait)

Over the past six years, the ACL has participated in a collaborative effort with the Environmental Research Division (M. C. Hansen) that involves monitoring the removal of contaminants (carbon tetrachloride and chloroform) from groundwater under a former grain storage facility in Waverly, NB, which is operated by the U.S. Department of Agriculture. Contaminants in the Waverly aquifer are being removed by groundwater extraction and air stripping. Carbon tetrachloride contamination in the Waverly aquifer decreased during the year, from approximately 47 ppb at the end of FY 1993 to 25 ppb at the end of FY 1994. The aquifer will continue to be sampled monthly in FY 1995 because the concentration of carbon tetrachloride is still above the Federal health standard of 5 ppb. Since October 1993, aliquots from three new monitoring wells have been submitted monthly. One of these wells has consistently shown high concentrations of carbon tetrachloride (approximately 1800 ppb) and chloroform (approximately 120 ppb). In FY 1994, the ACL analyzed more than 100 aqueous samples for this project.

Analytical Procedures for Waste Minimization and Pollution Prevention (D. W. Green L. L. Smith, J. S. Yaeger, J. S. Crain, A. S. Boparai, P. C. Lindahl, C. M. Newcomb, and J. B. Schilling)

Procedures exist for waste characterization, but waste minimization and pollution prevention have not usually been considered in the development of these procedures. The DOE expects to require several million characterizations over a 30-year period to accomplish the Department's goals for its environmental restoration and waste management program. The waste generated by the

analytical procedures used for characterizations is a significant source of new DOE waste. Success in reducing the volume of secondary waste and the costs of handling this waste would significantly decrease the overall cost of this DOE program. We have begun investigating ways to reduce these secondary wastes, focusing on three areas: (1) development of microanalytical methods to use flow injection; (2) development of methods to reduce the volume of solvent required for dissolution of waste samples for radiochemical analysis; (3) and development of alternative sample preparation methods for determination of organic constituents in waste samples.

Determination of the inorganic constituents of environmental and waste samples usually involves the use of multielement techniques such as inductively coupled plasma/atomic emission spectrometry or inductively coupled plasma/mass spectrometry. These techniques provide simultaneous determination of inorganic constituents, such as heavy metals, in aqueous solution. Both techniques use a nebulizer that converts the sample into an aerosol that is injected into the high-temperature plasma. In the standard method, the sample is continuously pumped into a nebulizer having 1% utilization efficiency. We are investigating the combination of a flow injection technique and a direct-injection nebulizer, which will allow introduction of sample aliquots of less than a milliliter with a 100% utilization efficiency. For such a system, all of the sample aliquot will be converted to an aerosol for analysis. Work has just been initiated on this task.

Progress on the other two tasks (reducing solvent volume for radiochemical analysis and developing alternative sample preparation methods for determination of organic constituents in waste samples) is described in separate sections of this report.

Analytical Laboratory Status Reports (D. A. Bass, P. L. Johnson, P. C. Lindahl, and W. E. Streets)

In FY 1994, the ACL assisted personnel from ANL's Environmental Assessment Division [J. Hensley, Gaithersburg Office (ANL-GAI)] and the DOE's Office of Environmental Management, Northwest Area Programs, EM-44 (S. Warren), in preparing status reports for laboratories providing analytical services to EM programs. The reports summarize the performance of these laboratories in performance evaluation programs. They present information in brief and concise formats and provide general interpretations of the results for three levels of DOE-EM management: the DOE Operations Office, the Office of Area Programs, and the Offices of the Deputy Assistant Secretaries for Waste Management (EM-30) and Environmental Restoration (EM-40). These reports have been well received by the targeted DOE management levels and will become part of the Integrated Performance Evaluation Program (IPEP), which is discussed elsewhere in this report.

The process of generating the reports also clarified some of the political and operational issues impeding full implementation of the IPEP. A pilot production of all IPEP reports will be conducted during FY 1995 to help resolve some of these issues.

Quality Assurance Plan for Aberdeen Proving Ground Analytical Laboratory (D. A. Bass, L. B. TenKate, and F. J. Martino)

During FY 1994, the ACL collaborated with Energy Systems Division (K. Brubaker) and with U.S. Army and U.S. Army contractor staff in preparing a laboratory quality assurance plan for the Chemical Material Screening Laboratory at Aberdeen Proving Ground. This laboratory, which will support environmental restoration activities at Aberdeen, will be used primarily to screen soil samples for chemical warfare agents to ensure they are safe to be shipped to commercial laboratories. The laboratory will also perform inorganic and organic analyses using the EPA's Contract Laboratory Program (CLP) protocols. Because the ACL staff has experience in quality

assurance activities and is familiar with the CLP, the ACL has been assigned the lead responsibility in developing the quality assurance plan. The project was begun in FY 1994 and will continue into FY 1995.

Review of Quality Assurance Documents for the Aberdeen Proving Ground (D. A. Bass)

In FY 1994, the ACL assisted Environmental Assessment Division personnel (S. S. Prasad, ANL-DC) in the technical review of two quality assurance documents for the U.S. Army at Aberdeen Proving Ground. These documents were the Aberdeen Proving Ground Directorate of Safety, Health, and Environment Quality Assurance Plan and Recommended Analytical Methods for Obtaining Adequate Quantitation Limits for Use in Risk Assessments at the Aberdeen Proving Ground. These reviews drew upon the ACL's familiarity and experience with quality assurance and with environmental analysis requirements. The primary areas of review focused on activities associated with inorganic analysis. The comments from these reviews were incorporated into the documents.

Evaluation and Implementation of U.S. EPA Methods for Inductively Coupled Plasma/Mass Spectrometry (D. A. Bass, J. S. Crain, and F. C. Laquer)

The EPA has proposed standard methods for the analysis of environmental samples using inductively coupled plasma/mass spectrometry (ICP/MS). The proposed methods for aqueous systems, including groundwaters and wastewater, and for solid waste are not currently used by the ACL, but we have begun to optimize and evaluate the performance of our ICP/MS equipment when operated according to the EPA methods. The ACL has procured the standards and quality control materials needed to perform these methods. The ACL staff have become familiar with the methods and their requirements and have established preliminary instrument protocols for performing them.

Additional work is required to determine analyte concentration ranges, optimize and demonstrate instrument performance, and write a standard operating procedure.

Development of Mixed-Waste Analysis Capability for Graphite Furnace Atomic Absorption Spectrophotometry (D. A. Bass, L. B. TenKate, and A. M. Wroblewski)

The ACL has been expanding its capabilities to analyze mixed-waste samples (i.e., radioactive hazardous-waste materials). In FY 1994, this capability was expanded to include analysis by graphite furnace atomic absorption (GFAA) spectrophotometry. This technique allows the determination of trace metals at the low parts-per-billion level and meets U.S. EPA requirements for detection limits. Typical metals determined by GFAA include Sb, As, Pb, Se, Ag, and Tl. In a typical installation of a graphite furnace instrument, an exhaust vent is located directly over the furnace area. However, some vaporized material produced during graphite furnace analyses can diffuse from the furnace area before reaching the exhaust vent. Therefore, greater containment of the vaporized material is required during analysis of mixed-waste samples. We modified a GFAA instrument to better contain vaporized sample aliquots. With this modification, the radioactivity of the samples can be approximately 10 times higher, or the exposure can be 10 times lower. The actual level of radioactivity that can be analyzed will depend on the radionuclide and the estimated number of mixed-waste samples analyzed per year.

Determination of Arsenic Species in Soil Samples from the Rocky Mountain Arsenal (D. A. Bass, L. B. TenKate, K. J. Parish, and J. S. Crain)

In FY 1994, the ACL began a collaborative research effort with the Energy Systems Division (M. Gowdy) and the U.S. Army at the Rocky Mountain Arsenal. In this effort, we are developing a method to distinguish among arsenic(III), arsenic(V), and organoarsenic compounds in soil samples. The Army wants to distinguish between the arsenic originating from lewisite, a chemical

warfare agent, and that originating from other sources. They will use this information to examine the impact of the chemical form of the arsenic on remediation activities. The approach chosen was a combination of high-performance liquid chromatography (HPLC) and ICP/MS. This permits separation of the arsenic species by HPLC, followed by sensitive detection of As by ICP/MS. This project, which will continue in FY 1995, utilizes the ACL's experience in soil extractions, chromatographic separations, and atomic spectroscopy.

Cleanup of Environmental Samples by High-Resolution Gel Permeation Chromatography
(K. J. Parish, S. D. Kent, A. S. Boparai, and C. M. Newcomb)

Gel permeation chromatography (GPC) is a size-exclusion procedure for cleaning environmental samples before analysis. It is required by the U.S. EPA Contract Laboratory Program for all soil samples that are being analyzed for semivolatile organic and polychlorinated biphenyl/pesticide compounds. The cleanup procedure is designed to remove high molecular weight contaminants that interfere with the determination of the target analytes. The sample extracts are passed through a column packed with a porous gel that separates compounds on the basis of molecular weight and size. The pore size of the packing material determines which compounds are removed.

The ACL currently uses a semiautomated, low-pressure GPC system. This system uses significant amounts of methylene chloride as the mobile solvent and is cumbersome to operate. In addition, because of the large plumbing volumes of the low pressure system, only half the sample can be loaded onto the column. The other half is lost in the GPC plumbing.

The new high-resolution GPC system also separates compounds on the basis of size, but the column is packed more tightly with smaller particles. As a result, it performs separations more efficiently. The high-resolution column requires higher operating pressures, but it reduces run times

and solvent usage by more than half. Furthermore, up to 80% of the total sample may be loaded onto the column, an increase of about 30%.

The high-resolution system was tested with two different brands of columns. The extraction solvent was spiked with semivolatile surrogate and matrix spike compounds, injected onto the column, and collected. The recoveries of the spiked compounds were found to be 100% or greater. More tests will be run to investigate the few recoveries that were greater than 100%. The two brands of columns produced similar results.

The ACL is further comparing the low-pressure and high-resolution GPC systems by comparing analytical results from two environmental samples, one cleaned in each system. Results of the analyses are pending. It is expected that the two systems will be comparable.

Development of a Continuous Emission Monitor for Thermal Treatment Systems (J. C. Demirgian, E. Y. Hwang, G. T. Reedy, C. T. Snyder, and S. E. Carpenter)

The objective of this project, which is a collaboration with Energy Systems Division (Z. Mao), is to develop a continuous emission monitor based on FTIR technology. This device will be used to monitor stack gases on-stream for volatile organic and selected inorganic compounds.

The primary benefit of an on-stream monitor for stack effluent is the near real-time determination of performance of the thermal treatment systems being monitored. On-stream analysis of the stack effluent would satisfy the requirements of the Clean Air Act of 1990 and address public concern about incinerator safety. Another benefit is that such a monitor eliminates the need for the expensive techniques currently used to characterize the effluent, such as a volatile organic sampling train followed by GC/MS laboratory analysis. This method, which is required under current procedures, only provides a one-time analysis.

Development of the monitor has progressed in three focus areas. The first is technical development in instrument optimization, automation software, sampling, and advanced data analysis. The second focus is obtaining EPA regulatory approval. The third focus is commercialization of the instrument. At the successful completion of this project, all data would be EPA-compliant and the instrument would be packaged as a complete unit.

Instrument development has progressed from a large assemblage of commercially available instrumentation, through a "breadboard" unit with interchangeable parts, to a small cabinet unit that contains all the hardware for sampling. This unit can easily be rolled to different locations. A complete sampling system has been designed and integrated into the FTIR spectrometer. The sampling system is automated, and it interacts with the spectrometer through the "TEAM" software package, which runs in a Microsoft Windows environment. The "TEAM" software controls the spectrometer and the sampling system. The designs for the infrared cell and transfer optics have been greatly simplified. The entire system, including the spectrometer, has been moved on a forklift without requiring subsequent optical alignment.

Analysis software has also progressed. Initially, data were analyzed by single classical and partial least squares methods. Since then, parallel processing of individual methods for each component has been implemented. Data analysis is currently being enhanced with time-domain and Fourier-domain methods that use peak width to mathematically separate components with overlapping bands. This combined approach will enhance the system's ability to detect substances that have overlapping absorbances and allow us to digitally filter system noise.

Our efforts to obtain regulatory acceptance have focused on working with the EPA. We contributed to an EPA protocol for continuous emission monitoring using FTIR technology. The EPA also asked us to prepare and submit a procedure to them for validation (this procedure is described separately in this report). The EPA procedures are not required; however, when they are

followed and quality control is within the specified limits, the EPA guarantees acceptance of the data. The procedure was submitted in September 1994, and validation is underway. We anticipate that data generated with this system will be EPA-compliant in FY 1995.

A Cooperative Research and Development Agreement (CRADA) to commercialize the system was completed in October 1994 with Clean Air Engineering and Martin Marietta Energy Systems, the operators of the DOE's Toxic Substances Control Act incinerator at the K-25 site in Oak Ridge, TN. The CRADA, in combination with EPA acceptance of the data, will make the system commercially available and facilitate acceptance in the user's community. We are also participating in the American Society for Testing and Materials task committee to develop an FTIR continuous emission monitoring method, with one team member (J. Demirgian) serving as task committee chairman.

Procedure for the Continuous Emission Monitoring of Incinerator Effluents Using Fourier Transform Infrared Technology (E. Y. Hwang and J. C. Demirgian)

A procedure has been written for the continuous monitoring of incinerator effluents using Fourier transform infrared (FTIR) technology. This procedure combines an Environmental Protection Agency protocol and ANL standard operating procedure. It is based on the removal and transport of a slip stream of incinerator exhaust through a heated transfer line to the heated long-path cell of the FTIR instrument. The acquired interferograms are Fourier transformed. Qualitative and quantitative analyses of resultant spectra are performed using an appropriate algorithm with prevalidated training sets of target analytes. This procedure is applicable to the determination of benzene, toluene, chlorobenzene, 1,1,1-trichloroethane, trichlorethylene, tetrachloroethylene, methane, hydrogen chloride, and carbon monoxide in incinerator effluents. In addition to the operating procedure, this document also establishes test requirements and quality assurance/quality

control criteria to be met for validation of training sets, verification of method performance, and reproducibility of monitoring results.

Counter-Narcotics Program Support (J. C. Demirgian and E. Y. Hwang)

The ACL is providing support to the Office of the National Drug Control Policy (ONDCP) in a collaborative program with the Technology Development (TD) Division (T. J. Yule). The ACL provides analytical support. We have supported ONDCP activities in the planning and testing of interdiction technology. The ACL has performed field tests that help to determine the optimum use criteria for analytical instruments that will detect narcotics in various interdiction scenarios.

Transition Metal Speciation in Textile Mill Wastewater (J. S. Crain, A. S. Boparai, A. M. Essling, D. G. Graczyk, D. R. Huff, E. A. Huff, P. C. Lindahl, J. B. Schilling, and F. P. Smith)

Under the new Stream Quality Standards enforced by the U. S. Environmental Protection Agency, certain metals discharged into receiving streams from industrial facilities will be sharply curtailed. This reduction is especially significant to the U.S. textile industry because many of the important dyes used in textile processing are "pre-metallized"; that is, the dye molecules contain either Cu, Cr, or Co as part of the chromophore. With support from the AMTEX™ partnership (an initiative based on a CRADA between the U.S. Department of Energy and the textile industry), the ACL is developing analytical procedures that will be used to characterize the lability of these metals as they appear in textile mill wastewater. These data will be used to assess the bioavailability (and environmental impact) of the metals. These data will also provide a basis for formulating an effective process by which bioavailable toxic metals can be removed from the wastewater.

Determination of Uranium in Dissolver Solutions for Production of ^{99}Mo from Low-Enrichment Uranium (J. S. Crain and J. T. Kiely)

Molybdenum-99 is a precursor to a radioisotope $^{99\text{m}}\text{Tc}$, which is used frequently in nuclear medicine. The production of this ^{99}Mo is currently accomplished by neutron irradiation of targets containing high-enrichment uranium (HEU). However, the use of HEU is being curtailed to reduce the risk of further nuclear weapons proliferation. It is possible to produce ^{99}Mo from targets containing low-enrichment uranium (LEU), but the process is only economical if the yield of ^{99}Mo is comparable to that obtained from HEU targets. High-density uranium silicide (U_3Si_2) targets, developed at ANL in 1985, offer a practical HEU substitute, and CMT staff (J. Hutter, B. Srinivasan) are investigating the recovery of ^{99}Mo from these materials. The ACL is supporting these investigations by using inductively coupled plasma/mass spectrometry to determine the uranium present in alkaline hydrogen peroxide dissolver solutions. These uranium data have been used to determine the chemical conditions (i.e., temperature and solvent composition) that are optimal for target dissolution; future experiments will examine the recovery of molybdenum from the dissolved targets.

Determination of Volatile Organic Compounds in Headspace for WIPP Project (D. V. Applegate, Y. Tsai, K. J. Parish, G. T. Reedy, A. S. Boparai, and S. E. Carpenter)

An important part of the analytical work that must be done as part of the Waste Isolation Pilot Plant (WIPP) project involves the analysis of material destined for the WIPP site to show that it meets the requirements of regulating agencies with respect to toxic constituents and does not constitute mixed waste that must be handled separately. Of particular interest are volatile organic compounds (VOCs), which have appreciable vapor pressure at room temperature and, if released, would tend to migrate quickly from a point of confinement. To test the level of VOCs in waste drums, gas samples are drawn from the drums into special sample canisters and shipped to

laboratories for analysis. The ACL is one of several laboratories participating in a performance demonstration program that results in qualification to do analysis of the drum headspace gas. The analysis for VOCs involves making quantitative additions of diluent and standards to the gas sample, trapping the analytes on a four-phase adsorbent, and heating the adsorbent to release the compounds to a gas chromatograph/mass spectrometer, where they are identified and measured quantitatively. The ACL has maintained qualification as a participating laboratory in this program.

Development of an Analytical Scheme to Determine Actinides in Soil by Alpha Spectrometry or Inductively Coupled Plasma/Mass Spectrometry (L. L. Smith, J. S. Crain, and J. S. Yaeger)

The DOE's Office of Environmental Restoration and Waste Management is sponsoring a multitask program to develop methods for the chemical characterization of environmental samples. A primary focus of this project is to develop faster and cheaper radioanalytical methods. As part of this project, the ACL, in collaboration with groups from the Chemistry Division (E. P. Horwitz, H. Diamond, and R. Chiarizia) and the Environmental Research Division (J. S. Alvarado and M. D. Erickson), has developed an improved method to isolate and separate the actinide elements from soil for analysis. This method satisfies the requirements of both alpha spectrometry and inductively coupled plasma/mass spectrometry (ICP/MS).

The new analytical scheme uses a series of extraction chromatographic materials, such as Tru•Spec and Teva•Spec, and ion exchange resins, such as Diphonix and BioRad AG 1-X8. These materials are used to perform sequential isolations of the actinides for alpha spectrometry and group separations for ICP/MS. Sequential isolation is necessary for alpha spectrometry because of limits on instrument resolution. However, for ICP/MS determinations it is only necessary to isolate uranium from plutonium and thorium to remove potential hydride interferences. Hydride adducts, $^{238}\text{UH}^+$ at $m/z = 239$ and $^{232}\text{ThH}^+$ at $m/z = 233$, were found to interfere with the determination of

^{239}Pu and ^{233}U by ICP/MS. Because of the relatively short half-life of ^{241}Am , ICP/MS is not a suitable detection method for this isotope at environmental levels. Therefore, the trivalent actinides are isolated for alpha spectrometry determinations prior to further group separation for ICP/MS measurements. Additionally, the separation schemes were designed to allow determination analysis of the actinides in soil, whether the soils had been acid-leached or had been totally dissolved through a sodium hydroxide fusion.

The utilization of the Diphonix resin offered several distinct advantages. Diphonix is selective for actinides in highly acidic solutions, even in the presence of complexing agents. The ability to utilize hydrochloric acid made it easier to maintain large soil samples in solution. Diphonix also remains effective in the presence of hydrofluoric acid, which makes it possible to process sample solutions that contain considerable quantities of silicon. In more traditional methods silicon is removed through volatilization of silicon tetrafluoride. However, in this analytical scheme, silicon is kept in solution, thereby reducing fume hood emissions.

Assessment of Gallium Hazards at the Advanced Photon Source (A. M. Essling, E. A. Huff, and D. R. Huff)

Argonne's Environment, Safety, and Health - Industrial Hygiene department (ESH-IH) is conducting an assessment of potential hazards associated with the use of liquid gallium for cooling targets in facilities at the Advanced Photon Source. This assessment includes an ongoing review of published information on gallium toxicity, together with smear sampling and workplace air monitoring of areas where gallium pumps are serviced. For this effort, the ACL put procedures in place for measuring any gallium that might be collected on the smear samples or air filters and analyzed samples collected by ESH-IH. In our method, the smear paper is dry ashed in a platinum beaker at 400°C , and the ashing residue is dissolved in nitric acid solution for determination of

gallium by inductively coupled plasma/atomic emission spectrometry. Results from spiked samples, to which known amounts of gallium were added, have demonstrated complete gallium recovery and accurate determination of the gallium present. Microgram quantities of gallium on a smear paper or air filter can be determined.

Preparation of Material for Tests of a Nonintrusive Contraband Detection System (K. J. Jensen, A. M. Essling, I. M. Fox, F. P. Smith, and D. G. Graczyk)

Researchers in ANL's Reactor Engineering Division (C. Dickerman, E. Rhodes et al.) are investigating the potential application of a nonintrusive method for detecting contraband materials that is based on neutron interrogation. Their system identifies and measures specific nuclides or chemical elements in a material by interpreting the spectrum of gamma rays that is emitted when 14-MeV neutrons from the Associated-Particle Sealed-Tube Neutron Generator (APSTNG) are inelastically scattered by the material. For tests of the APSTNG system's ability to detect illegal drugs, the investigators wished to prepare a noncontraband surrogate material that would have the same elemental composition as cocaine hydrochloride ($C_{17}H_{22}NO_4Cl$). They asked the ACL to prepare this material as a mixture of ground plastic polymers, which contained the elements of interest and could be combined in appropriate proportions to give the desired stoichiometry.

Under a tight schedule, ACL staff ground and blended appropriate quantities of five polymers by mixing them with dry ice and processing them through a Waring blender. Approximately 1.25 kg of surrogate product was generated. This mixture showed some nonuniformity because the blender was unable to pulverize all of the feedstock materials (e.g., polyethylene pellets were incompletely ground), but it proved satisfactory for the initial APSTNG tests. Subsequently, the mixture was sieved and the larger particles were put through a cutter mill (also with dry ice to embrittle the polymers). After blending the reground product on a roller mill,

we sampled and analyzed the flour-like powder to determine its as-prepared elemental composition. These analyses showed the material to be fairly uniform, with an average stoichiometry (equivalent to $C_{17}H_{22.3}N_{1.1}O_{4.0}Cl_{1.2}$) very close to the target composition.

Characterization of Actinide Materials Used for EBR-II Reactor Dosimetry (D. L. Bowers, T. TenKate, F. P. Smith, C. S. Sabau, and D. G. Graczyk)

Actinide dosimeters are used to measure the neutron flux and energy in a nuclear reactor. The material to be used as a dosimeter has to be of known isotopic and elemental content. However, little documentation existed on the purity of three metal actinides that Energy Technology Division personnel (A. Hins et al.) planned to encapsulate and insert in the EBR-II reactor. The ACL analyzed a sample of each metal actinide (^{237}Np , ^{241}Am , and ^{243}Am). Each sample was dissolved and analyzed for U, Pu, ^{237}Np , ^{241}Am , ^{243}Am , ^{242}Cm , and several alkali and transition elements. Chemical separation was performed by using TRU•Spec and AG 1 x 8 chromatographic resins. Several techniques were used to measure the desired constituents, including gamma, alpha, mass spectrometry, and ICP/AES. Results showed high purity in the ^{237}Np and ^{243}Am material, but the ^{241}Am metal contained large amounts of ^{237}Np . The materials were encapsulated and inserted into the EBR-II reactor by Fuels and Engineering Division personnel (C. Trybus et al.) for irradiation.

Determination of Ethylenediaminetetraacetic Acid in Water (J. B. Schilling)

In support of the Energy Systems Division's Mound Feasibility Study project (C. Negri), the ACL was asked to set up a method for the determination of ethylenediaminetetraacetic acid (EDTA) in water. This chemical is being used as a complexation reagent to remove plutonium and other metals from soil. Water leaching studies are carried out on the residual EDTA in the soil.

The EDTA concentration in water was determined by using high-performance liquid chromatography (HPLC) with UV detection. In this method, the EDTA is complexed with iron(III), separated under reversed-phase ion-pairing conditions, and detected with 254-nm UV light. The separations were carried out using a Hewlett Packard 1090 HPLC equipped with an Alltech 4.6 x 150 mm Spherisorb ODS 2.5 micron column and a UV-visible diode array detector. The mobile phase was a sodium acetate/acetic acid buffer (pH = 4) with tetrabutylammonium hydroxide as the ion-pairing agent. Without previous concentration of the sample, the detection limit for EDTA is approximately 5 ppm, with an analysis run time of less than 10 minutes per sample.

Analytical Support for the Energy Systems Division Mobile Laboratory at Aberdeen Proving Ground (D. V. Applegate)

In FY 1994, the ACL assisted the Energy Systems Division (J. F. Schneider) by providing analytical support at their mobile laboratory, which was located at Aberdeen Proving Ground. The purpose of the mobile laboratory was to screen samples taken from underground storage tanks and their surrounding soils as part of an effort to locate contaminant plumes. Qualitative and quantitative results were obtained with fast turn-around times to help in sampling at more than 100 underground storage tanks. Because the mobile laboratory allowed rapid analysis, the sampling team was able to use the results to direct sampling activities while sampling was in progress. The results from the mobile laboratory were used to determine what safety precautions were required and whether any additional analyses by commercial laboratories were needed. Analyses were performed by several screening methods, depending upon the type of matrix (water, soil, sludge, or organic liquid). Samples were analyzed using a gas chromatograph equipped with a mass selective detector and an electron capture detector, which gave information about a wide variety of analytes.

Methodology for Measuring Lead Sulfate in Mixtures with Lead Oxide (A. M. Essling)

Electrosorce, Inc., of Austin, TX, uses mixtures of lead sulfate and lead dioxide in the fabrication of advanced battery products. Electrosorce was having difficulty obtaining reproducible results from the methods that they were using to determine the lead sulfate content of these powder mixtures. Through the Electric Power Research Institute and CMT, the ACL was asked to analyze six powder samples having different lead sulfate contents (between 8 and 50%) and to provide details of the method we selected for the measurements. We determined lead sulfate in each mixture by measuring total sulfate according to an adaptation of American Society for Testing and Materials procedure ASTM D1301-91, *Chemical Analysis of White Lead Pigments*. By means of this procedure, total sulfate was measured gravimetrically as barium sulfate after the lead had been removed with a carbonate precipitation. The reliability of this method was verified by analyzing a synthetic mixture of lead dioxide/sodium sulfate and by replicate measurements on some of the Electrosorce samples. Uncertainty in our results was conservatively estimated at $\pm 0.5\%$ of each measured value.

Methods for Determining Metals in Waste Oil (A. M. Essling, E. A. Huff, D. R. Huff, I. M. Fox, and L. B. TenKate)

Many ANL programs depend on vacuum pumps, hydraulic systems, or machines that contain oil, which needs changing and, ultimately, disposal. To dispose of oil properly, a determination must be made as to whether it is contaminated with hazardous metals (e.g., As, Ba, Cd, Cr, Hg, Pb, Se, Ag) and/or radionuclides. Because commonly used methods for measuring these contaminants perform best with aqueous samples, we undertook a study of methods for decomposing oils to provide a single aqueous solution that could be used in all the hazardous metal determinations. We then applied two methods in the preparation of metal-containing oil standards (Conostan or National

Institute of Standards and Technology) to document the recovery of 20 metals. For this project, elemental measurements were by graphite furnace atomic absorption (GFAA), cold vapor atomic absorption (CVAA), or ICP/AES.

The two preparation techniques we examined were: (1) combustion of the oil with oxygen in a sealed pressure vessel and (2) decomposition/digestion of the oil with acid and microwave-energy heating in a closed vessel. The microwave digestion procedure proved to be more effective in recovering most metals (>90% recovery for every metal tested). It was also more efficient, since it allows six samples to be prepared together, while oxygen combustion allows only one sample at a time. As a general multielement preparation method, the oxygen combustion system was not satisfactory because several metals gave recoveries below 90%.

Unexpectedly, we found that the microwave digestate produced a large intensity enhancement in the ICP/AES spectrometer for many metals. The enhanced intensities produced apparent overrecoveries for these metals when the instrument was calibrated with aqueous standards that did not contain the oil decomposition products. By using matrix-matched standards, we were able to eliminate the enhancement interference. Following the microwave decomposition, the recoveries of all 20 elements (Al, As, B, Ba, Ca, Cd, Cr, Cu, Fe, Hg, Mg, Mn, Mo, Ni, Pb, Se, Ag, Ti, V, Zn) were within $\pm 10\%$ of the certified values. Because no oil standard is available with certified quantities of radionuclides, we are not able to evaluate radiological measurements at this time. However, we are confident that these measurements can be done reliably on a portion of the solution prepared by microwave digestion. Having this method available will help the ACL to provide reliable, cost-effective waste oil characterization to ANL's programs.

Support for High-Temperature Superconductor Development (A. M. Essling, E. A. Huff, D. R. Huff, I. M. Fox, L. E. Ross, F. P. Smith, B. S. Tani, and D. G. Graczyk)

The ACL continued providing extensive analytical support to high-temperature superconductivity programs in the Energy Technology and CMT Divisions. Much of this work involves analysis of starting materials, process samples, and products related to fabrication and characterization studies on various ceramic compositions, including $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ ("123-compound") and lead-doped bismuth/strontium/calcium/copper oxides ("BSCCO" ceramics). Our measurements include determining elemental composition by ICP/AES or classical methods, anions by ion chromatography, carbon with a LECO analyzer, and phase composition by X-ray powder diffraction. We also use an iodometric titration method to determine excess valence of the metals in a given ceramic (e.g., Cu^{3+}), which is related to the oxygen stoichiometry of the compound.

This past year, researchers have concentrated on the study of the BSCCO ceramics, which exhibit several superconducting phase compositions [e.g., the 2212 phase, $(\text{Bi,Pb})_2\text{Sr}_2\text{CaCu}_2\text{O}_x$, and the 2223 phase, $(\text{Bi,Pb})_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_x$]. Researchers at ANL, often in collaboration with scientists at universities and commercial organizations, are investigating new ways to prepare the BSCCO ceramics and are studying the chemical and physical properties of the specimens they obtain. New analytical challenges have arisen from these studies, especially in determining the oxygen coefficient by iodometric titration. It seems that in the BSCCO ceramics, bismuth may slowly form an iodobismuthite ion, which consumes titrant and causes an indistinct endpoint. We found that adding ethylenediaminetetraacetic acid to complex bismuth before titration greatly improved the endpoint behavior and allowed precise measurement of the oxygen coefficient.

In general, the BSCCO system exhibits potential for forming mixed phases and has a more complicated chemistry than the 123-compound. Compositional changes can arise from variations in metal stoichiometry (e.g., noninteger stoichiometry numbers for one or more metals) as well as

from variations in the valence of Pb, Bi, or Cu. This range of compositional variation in the BSCCO materials makes chemical analysis of individual specimens very important to understanding and comparing results. Our experience with these materials suggests that more precise methodology is needed for their compositional analysis, as well as new approaches to characterize the metal valences that occur in them. The ACL plans to pursue development of capabilities to satisfy these needs.

Characterization of Used HEPA Filters for Disposal (A. M. Essling, L. E. Ross, F. P. Smith, I. M. Fox, E. A. Huff, D. R. Huff, P. Melnikov, S. D. Kent, K. J. Parish, Y. Tsai, L. L. Chromizky, P. D. Postlethwait, E. Vinokour, L. B. TenKate, T. TenKate, J. S. Yaeger, L. L. Smith, F. Markun, D. L. Bowers, A. S. Boparai, and D. G. Graczyk)

High-efficiency particulate-air (HEPA) filters provide the final stage of containment on many laboratory exhaust systems in ANL facilities by preventing escape of chemical and radioactive materials entrained in the exhausted air. For a number of years, used HEPA filters removed from these systems have accumulated in the Environmental and Waste Management Program's Waste Management Department (EWM-WM) because no facility could be found that would accept them for disposal. In 1993, EWM-WM determined that these used filters would be accepted for disposal at DOE's Hanford Reservation if they could be shown to meet the relevant Hanford waste acceptance criteria. Procedures were proposed by EWM-WM for repackaging and composite sampling of the used HEPA filters, and the ACL established an analysis methodology for evaluating the composite samples against the Hanford criteria.

The composite samples generated during repackaging of the HEPA filters by EWM-WM consist of portions of 12 to 14 individual filters, which must be homogenized for analysis. Homogenizing the composite sample is quite difficult because of the fibrous, fabric-like nature of the HEPA filter media. After trying several techniques, we ultimately settled on a process wherein

the filter material is manually cut with a straight-bladed rotary cutter into small pieces (approximately 5 x 5 mm), which can be mixed and subsampled in a controlled manner. For containment during the cutting process, the manual operations are performed in a closed glovebag in a laboratory fume hood.

From the homogenized composite, representative subsamples are taken for determining specific characteristics or contaminants in the HEPA filter media by means of procedures established during FY 1993 in the ACL. These include procedures for evaluating pH, metals content, inorganic anions, sulfide and cyanide, semivolatile organic compounds, polychlorinated biphenyls, and radionuclides. For some samples, sufficiently high concentrations of heavy metals are present to require testing with the Toxicity Characteristic Leaching Procedure so that a proper hazard classification can be assigned to the waste.

During FY 1994, EWM-WM initiated a campaign to dispose of historical HEPA filter waste, which generated about 100 composite samples for analysis. By the end of the fiscal year, the ACL had completed characterization of roughly 60 samples, and EWM-WM had shipped 44 bins of used filters to Hanford. This campaign has provided a major reduction of the historical waste inventory at the ANL site.

Characterization of Products and Residues from Automobile Shredder "Fluff" Recycling
(A. M. Essling, F. P. Smith, I. M. Fox, L. B. TenKate, E. A. Huff, D. R. Huff, L. L. Chromizky, K. J. Parish, S. D. Kent, Y. Tsai, C. T. Snyder, G. T. Reedy, S. E. Carpenter, D. G. Graczyk, and A. S. Boparai)

About 225 kg (25%) of every junked car that is processed by automobile shredders to recover ferrous-metal scrap is made up of a mixture of plastics, glass, fibers, and foam. This mixture is referred to as "fluff" and represents a substantial waste side stream from the automobile shredder industry. At present, shredder fluff is sent to landfills. Researchers in the Energy Systems (ES)

Division (E. Daniels et al.) have developed and are testing technologies to segregate and recover the plastic and foam fluff components for recycling and are exploring alternatives to landfill disposal for residues ("fines") that are collected during segregation of the recyclable materials.

The ACL is providing chemical analyses to help characterize the various product streams from these processes, including oils, recovered plastics and foams, and the shredder fines. An FTIR microscope is used to examine plastics recovered by solvent extraction of shredder residues. With this technique, selectivity of the extraction and purity of the products are assessed by comparing spectra for polyvinylchloride and acrylonitrilebutadienestyrene polymer standards with spectra from reconstituted plastics from the process. Other analytical methods are applied to determine specific contaminants in the foams, oils, and fines. Heavy metals and polychlorinated biphenyls (PCBs) are common contaminants in the shredder fluff as a result of their presence in automotive components and fluids (e.g., lead from batteries, mercury from mercury switches, PCBs from capacitors or hydraulic fluids). After suitable treatment of samples to bring the metals into solution, all metals except mercury are determined by ICP/AES. Mercury is determined by cold vapor atomic absorption spectrophotometry. The PCBs are extracted with an appropriate solvent and measured by gas chromatography. We also determine the total chlorine content of many samples by a procedure that employs oxygen-bomb combustion and ion chromatography to measure the chlorine as chloride.

These and other analysis tools were used to provide the ES Division with data on the effectiveness of a new process to remove metals and oil contaminants from the polyurethane foams generated from the shredder fluff and to provide a comprehensive characterization of shredder fines to determine their suitability for disposal by incineration in a cement kiln.

National Acid Precipitation Assessment Program (F. P. Smith)

The National Acid Precipitation Assessment Program (NAPAP) was initiated in the early 1980s with the goal of quantifying the extent to which air pollution, including acid deposition, accelerates the weathering of materials used in the construction of cultural resources. Materials of particular interest are marble and limestone because they are widely used in exterior structures and monuments and because the calcium carbonate stones are especially sensitive to acid attack. In one NAPAP subprogram at ANL (K. J. Reimann, Energy Technology Division), limestone and marble briquettes are exposed to a variety of atmospheric conditions at a number of sites throughout the country. The weathered briquettes are brought to ANL for sampling and analysis to determine the surface chemistry and mineralogical changes in the test stones. In support of this program over the past few years, the ACL has analyzed hundreds of specimens for anions (F^- , Cl^- , NO_3^- , SO_4^{2-}) by ion chromatography and a smaller number for metal cations by ICP/AES. Cation concentrations changed little following exposure at the test sites. However, sulfate and nitrate concentrations were substantially elevated in some samples. These results have provided a quantitative measure of attack by atmospheric sulfur and nitrogen oxides. In FY 1994, the ACL performed anion measurements on approximately 156 specimens.

Orphan Waste Work Projects (D. G. Graczyk, A. M. Essling, I. M. Fox, D. R. Huff, E. A. Huff, F. P. Smith, C. T. Snyder, R. W. Bane, K. J. Jensen, L. E. Ross, D. L. Bowers, F. Markun, C. S. Sabau, L. L. Smith, B. S. Tani, T. TenKate, J. S. Yaeger, R. R. Heinrich, A. S. Boparai, D. V. Applegate, L. L. Chromizky, S. D. Kent, K. J. Parish, Y. Tsai, P. D. Postlethwait, G. T. Reedy, S. E. Carpenter, E. Vinokour, M. A. Gritters, S. M. Macha, P. C. Lindahl, J. S. Crain, J. J. Marr, and L. B. TenKate)

Many ANL facilities have waste materials left behind by programs that are no longer funded or materials whose origin is unknown or inadequately documented. Funding made available through ANL's Environment and Waste Management (EWM) Program has provided many of these facilities

the opportunity to undertake work projects directed at accumulation, characterization, and documentation of these so-called "orphan wastes" so they may be properly disposed of. In support of a number of these work projects, the ACL is conducting chemical and radiological analyses to provide the composition and hazards-classification data needed by treatment/storage/disposal (TSD) facilities that will ultimately process the wastes. Working closely with the EWM Program to ensure that information and procedural requirements of the appropriate TSD facility are satisfied, ACL staff have prepared detailed sampling and analysis plans for some projects, have fielded sampling teams to collect appropriate representative samples, and have collaborated with EWM staff in interpreting the analysis results for waste handling.

Wastes characterized by the ACL this fiscal year have included approximately 30 five-gallon pails of soil samples that were archived over decades of radiological characterization projects in ANL's Environment, Safety and Health Division, as well as hundreds of smaller waste items from various laboratories and storage areas. Many of these small items were unlabeled containers of solids, aqueous or organic liquids, and oils or sludges. When the material in these containers appeared to be pure chemical compounds or simple mixtures, we pursued an analytical approach that would provide a direct identification of the material and then relied on a material safety data sheet to obtain pertinent hazards information. For solids, X-ray fluorescence and X-ray diffraction analysis often gave the necessary information; for liquids and oils, several techniques (such as GC/MS and FTIR spectroscopy) were often employed. However, many wastes either could not be identified or contained complex mixtures of components; such wastes were evaluated for the Resource Conservation and Recovery Act characteristics of ignitability, corrosivity, toxicity, and reactivity. Wastes from radiological controlled areas required determination of the identity and quantity of radionuclides that might be present; these wastes were analyzed by gamma spectrometry, gas proportional counting, liquid scintillation counting, and/or alpha pulse height analysis. Through

its orphan waste work projects, EWM has helped ANL divisions dispose of tens of thousands of pounds of waste materials. The program is expected to continue through FY 1995.

Facility Closure Activities (D. G. Graczyk, P. C. Lindahl, A. S. Boparai, D. L. Bowers, F. J. Martino, L. B. TenKate, J. J. Marr, A. M. Essling, K. J. Jensen, I. M. Fox, F. P. Smith, R. W. Bane, D. R. Huff, E. A. Huff, D. V. Applegate, L. L. Chromizky, S. D. Kent, Y. Tsai, K. J. Parish, M. A. Gritters, P. L. Johnson, T. TenKate, J. S. Yaeger, L. L. Smith, F. Markun, and C. S. Sabau)

Under the Resource Conservation and Recovery Act (RCRA), facilities or areas that have been involved in the storage, treatment, or disposal of hazardous wastes must undergo a formal sequence of characterization, remediation, and recharacterization before they are returned to general use. This sequence, designated a "closure" of the affected area, is performed according to a predetermined plan prepared in collaboration with the appropriate regulatory agencies. Through its Environmental and Waste Management Program, ANL has initiated closure activities at several on-site locations. During FY 1994, the ACL provided both laboratory services and technical support to several of these activities.

In the 317 Area of the ANL site, RCRA closure of two areas was completed. One of these, the "Shoot-and-Burn Pile," consisted of a large mound of sand on which shock-reactive waste chemicals had been detonated by rifle shot in past years as a means to render the chemicals safe for disposal. The other area, the "Alkali Metal Passivation Tank," is a concrete structure that had held water for the purpose of neutralizing water-reactive chemicals. Chemical analysis of numerous samples of water, sand, soil, and sludge from these areas were required to complete their RCRA closures. Most of these analyses involved commonly applied EPA methods. However, for the Shoot-and-Burn Pile, special methods had to be devised to provide data on specific materials known to have been detonated on the sand pile.

Involvement by the ACL in these RCRA closures extended beyond performing laboratory measurements on samples. The ACL also participated in the selection and specification of methods for the analysis of samples, interacted with contractors that performed the sampling and remediation activities or provided third-party oversight of the closure process, and assisted in evaluation or interpretation of analysis results. The data that the ACL provided helped to determine how waste generated during the cleanup would be disposed and whether the remediation achieved decontamination standards established in the closure plan. The successful closure of these historical facilities is a significant milestone in ANL's efforts to remediate environmental concerns related to past operations.

Wastewater Treatability Studies (D. G. Graczyk, E. A. Huff, D. R. Huff, I. M. Fox, K. J. Jensen, L. B. TenKate, L. E. Ross, A. M. Essling, J. T. Kiely, F. P. Smith, S. D. Kent, L. L. Chromizky, Y. Tsai, and A. S. Boparai)

Wastewater from facilities on the ANL site is segregated into two distinct wastewater sewer systems, which collect either sanitary wastewater or laboratory wastewater for separate treatment and discharge. In coming years, ANL plans to upgrade its facilities for treating the effluents from these sewer systems. Among the planned improvements is construction of a laboratory wastewater treatment plant that would process the laboratory-system wastewater to remove potential contaminants such as heavy metals, volatile organic compounds, suspended solids, and other substances that are regulated under ANL's National Pollution Discharge Elimination System (NPDES) permit.

To help choose among alternative technologies and operational designs (e.g., batch-mode vs. continuous-mode processing) for the proposed plant, ANL's Environment and Waste Management (EWM) Programs commissioned an on-site engineering study of laboratory wastewater treatability. In this study, process engineers from the Chazen Companies (Poughkeepsie, NY)

examined the compatibility and effectiveness of various treatment processes with actual wastewater effluents from the ANL site. The ACL provided laboratory support to these studies by analyzing samples of treated and untreated water for the parameters of interest. Because adjustments to treatment conditions required feedback data from previous tests, short turnaround times and close cooperation were important to the study's success. In all, the ACL analyzed more than 500 samples in support of this effort in FY 1994. Parameters measured included transition metals (229 samples), mercury (164 samples), total suspended solids (58 samples), oil and grease (27 samples), organic contaminants (21 samples), and fluoride (16 samples). Process recommendations from the Chazen study will help ensure that the plant will operate effectively.

Analysis Support to Fabrication of Fusion Materials Test Capsules (R. W. Bane, E. A. Huff, D. R. Huff, F. P. Smith, C. T. Snyder, A. M. Essling, D. A. Bass, F. C. Laquer, and D. G. Graczyk)

To accomplish planned irradiation tests of vanadium alloys for ANL's Fusion Power development programs before the scheduled shutdown of the EBR-II reactor, two capsules containing the vanadium test materials had to be fabricated at ANL on short notice. Each capsule contained 12 subcapsules that were charged with 1.5 to 2.5 g of lithium metal, which acts as a heat-transfer medium for the specimens. More than 200 specimens were to be loaded into the 12 subcapsules.

One of the critical operations in this effort was purifying the lithium metal used in the subcapsules. This operation was carried out in a high-purity helium atmosphere glovebox by a team from the Energy Technology Division (A. Hins et al.). The ACL provided analysis support to this operation with short turnaround measurements to (1) monitor the purity of the glovebox atmosphere; (2) determine trace impurities (carbon, nitrogen, and metals) in the lithium at various stages in the process; and (3) verify the isotopic purity of the ^7Li (>99.9%) metal that was processed. The

operation was successful, with the final lithium product showing satisfactory low concentrations of nitrogen (15 ppm) and carbon (about 1 ppm).

The fabrication of the test capsules involved people from at least three ANL divisions as well as Central Shops and Inspection. The capsules were ultimately transferred to ANL-West and incorporated into a seven-capsule assembly in time for EBR-II's last run.

Measurement of Calcium Isotope Ratios in Canine Blood Serum by Thermal Ionization Mass Spectrometry (F. P. Smith, A. M. Essling, and D. G. Graczyk)

Researchers in ANL's Center for Mechanistic Biology and Biotechnology (M. Bhattacharyya, A. Wilson) are studying the effects of ovary removal and cadmium exposure on the metabolic uptake of calcium into the body. In one study designed to measure intestinal calcium uptake in canine subjects, isotopically enriched stable calcium tracers were administered orally (^{44}Ca) and intravenously (^{42}Ca) to groups of dogs having different treatment histories. Blood was drawn from each animal at an appropriate time after the tracers were administered. The relative amounts of each tracer present in the withdrawn blood provide the desired measure of calcium absorption through the digestive pathway.

Key to the application of this approach to measuring calcium uptake is the ability to precisely determine the extent of isotopic enrichment or depletion of calcium in the blood-serum samples from each subject. Last year, the ACL determined $^{44}\text{Ca}/^{42}\text{Ca}$ ratios on a number of samples by ICP/MS. In FY 1994, we established procedures for measuring calcium isotope ratios in the blood-serum samples by thermal ionization mass spectrometry (TIMS). For these measurements, an oxalate precipitation procedure was used to isolate calcium ($\sim 30\text{ }\mu\text{g}$) from a 0.3 mL portion of serum. For TIMS analysis, 3 μg of this Ca was loaded on a high-purity rhenium side filament and analyzed in triple-filament configuration with a rhenium ionizing filament. Instrumentally induced mass

fractionation was corrected for in each analysis run by normalizing all measured ratios to a reference value for $^{40}\text{Ca}/^{48}\text{Ca}$ in natural calcium. This normalization could be applied to the tracer-doped samples as well as to control samples because ^{40}Ca and ^{48}Ca were not present at significant levels in the tracers. With the normalization of ratios, run-to-run precision for ratios involving the tracer isotopes was quite good, showing a relative standard deviation of about 0.2% for duplicate measurements on 14 serum samples. Data obtained for natural calcium samples were in excellent agreement with literature values. The TIMS analysis uses considerably less sample than the ICP/MS measurements and provides precise data on all Ca isotopes in each sample. Agreement between the ICP/MS values and TIMS values for the $^{44}\text{Ca}/^{42}\text{Ca}$ ratios was extremely good, demonstrating that ICP/MS isotopic analysis for Ca is viable in appropriate cases.

Gross α/β Analysis of Environmental and Mixed-Waste Samples by High-Pressure Microwave Digestion (J. S. Yaeger and L. L. Smith)

In FY 1994, the ACL evaluated improved equipment and techniques for microwave digestion of samples for gross α/β analysis. Laboratories are receiving many requests for such analyses as a result of the DOE's environmental restoration and waste management activities. The new techniques could allow laboratories to respond to such requests more quickly.

Gross α/β measurements are used as a screening procedure before initiating other radioanalytical determinations, as well as before determining such contaminations as RCRA or volatile organics. Therefore, gross α/β determinations often become the rate-determining step in these analytical protocols. Traditional methods for the α/β analysis of environmental and mixed-waste samples require repetitive leaching, which is time-consuming and generates large volumes of secondary wastes.

An alternative to leaching is microwave digestion. In the past, microwave technology has had limited application in the radiochemical laboratory because of limitations on sample size and pressure. New microwave vessel designs, however, allow for pressures on the order of 1500 psi (~10,000 kPa). These high-pressure, closed-vessel systems achieve reliable sample digestion using larger sample sizes and shorter digestion times while also dramatically reducing secondary wastes. Results obtained by the ACL indicate that the microwave digestion procedure meets performance requirements for several matrix types and provides precision and detection limits that are equivalent to traditional hotplate methods. Moreover, analyst time is decreased by a factor of three, while turnaround time is improved by a factor of two. Secondary waste generation is dramatically reduced by a factor of nine or more. Therefore, this new digestion technique clearly allows the analyst to prepare the sample in a fraction of the time with significantly less acid and with a lower potential of cross-contamination.

Drum Headspace Sampling for WIPP Project (D. V. Applegate, G. T. Reedy, A. S. Boparai, C. T. Snyder, M. A. Gritters, and K. J. Parish)

In support of the WIPP project, the ACL has been preparing to collect and analyze headspace gas samples from 55-gal drums of transuranic waste stored at ANL-East. Some of these drums are equipped with charcoal filters which serve to vent any built-up pressure in the drum while retaining any harmful materials that could escape. The ACL has developed and tested a method to collect gas samples from drums that are not equipped with charcoal filters. In this method, a custom-made Tedlar bag is sealed over the drum, and a custom-made device is used to open the lid, releasing the gas into the bag. An evacuated SUMMA® canister is then used to draw the headspace gas sample from the bag for future analysis.

The ACL has also tested sampling equipment for collecting headspace gases from drums that are equipped with charcoal filters. Both sampling methods have been tested to verify that they meet the quality assurance guidelines of the WIPP project.

Currently, drums that are appropriate for sampling have been identified and set aside for sampling, which will begin once quality assurance documents are complete.

Holding Time Study of Volatile Organics in WIPP Sample Canisters (G. T. Reedy, D. V. Applegate, M. A. Gritters, S. E. Carpenter, and A. S. Boparai)

Before radioactive wastes can be transported from various DOE locations for burial at the WIPP underground repository near Carlsbad, NM, they must be analyzed to determine whether they pose a chemical hazard. To test for hazardous volatile organic compounds (VOCs), the gas present in the headspace of a waste container is sampled, and the sample is transported to a laboratory for analysis. Currently, no more than 28 days may elapse between collection and analysis of samples containing VOCs. This holding time is too short to be practical, given that time must often be allowed for transportation. However, before the holding time could be extended, more information was needed about how the VOC levels in the sample containers change over time.

In the ACL, the level of VOCs in SUMMA canisters, which are the specified containers for headspace gas collection, was measured over a period of 320 days. For each analyte in the canisters, the concentration data were fitted to a straight line of the form $y = mx + b$, where x is the time (day number), b is the concentration at day 0, y is the concentration at a designated day number, and m is the rate of change of concentration with time. Two statistical tests were applied to the data to check whether the changes in concentration were significant. The tests, known as "F-" and "G-" tests, check whether a better fit to the data is obtained by having $m = 0$ or $m \neq 0$. Of the compounds tested, the only compound found to have a statistically significant change was 1,1-dichloroethane,

whose concentration declined 1.6% for every 100 days. Since the accuracy with which a given concentration can normally be determined is $\pm 20\%$ for a single measurement, extension of the holding time up to a period of 100 days could certainly be justified.

Development of the Integrated Performance Evaluation Program for DOE Analytical Services Division (P. C. Lindahl, W. E. Streets, D. A. Bass, J. J. Marr, K. J. Parish, and P. L. Johnson)

In collaboration with DOE's Radiological and Environmental Sciences Laboratory (RESL) and the Environmental Measurements Laboratory (EML), the ACL is developing and implementing a comprehensive Integrated Performance Evaluation Program (IPEP) for DOE's Office of Environmental Management (EM), Analytical Services Division (EM-263). This program is designed to provide information on the quality of radiological and nonradiological analytical data being produced by all laboratories on which DOE is relying for environmental restoration and waste management sample analyses. The ACL has been active in this project since March 1991, and is assisting the two lead DOE laboratories in the development of the program requirements and strategies for implementation, especially in the nonradiological portions of the program. In collaboration with S. Hedayat (University of Illinois, Chicago) and ANL's Electronics and Computing Technologies Division (R. R. Krol and A. E. Scandora), the ACL is also developing the strategies for (1) compiling and analyzing performance evaluation (PE) results, and (2) monitoring to assure that needed corrective actions are taken.

The ACL also participates in Future Analytical Support Team activities in support of EM-263. This group, which is an outgrowth of a task group formed by EM, provides advice and needed data that assist EM-263 in meeting its objective of ensuring that the required capacity and capabilities exist to provide analytical support for EM Programs.

The Interagency Agreement (IAG) between ANL and Region V of the Environmental Protection Agency (EPA), which was renewed for FY 1994, establishes the IPEP at ANL as a pseudo-EPA Region. Thus, IPEP may nominate laboratories performing analyses for EM into the EPA's Water Supply (WS) and Water Pollution (WP) PE programs, and to access the relevant PE program information about these laboratories through the Environmental Monitoring Systems Laboratory, Cincinnati, OH (EMSL-Ci). Historical and current data from these programs are now routinely transferred by electronic means from the EPA data base at Research Triangle Park (North Carolina), to the data base at ANL.

Under another IAG, we have continued to receive from EMSL-Las Vegas (EMSL-LV) hard-copy information for DOE government-owned, contractor-operated (GOCO) participants in the Contract Laboratory Program (CLP) organic and inorganic PE programs. The EPA's Inspector General has impacted IPEP and DOE use of this PE program by allowing only those laboratories performing work for DOE Superfund sites to participate in this program. We have continued to work with the EPA to gain access to electronic information from the CLP and the EMSL-LV Performance Evaluation Studies Program, formerly the Radiological Intercomparison Studies; however, this issue is still pending. Meanwhile, we entered information from hard copy into a data base to make it available for the electronic generation of reports.

During this year, the following reports were prepared and submitted to the EM-263 and the QA Working Group: CLP organic and inorganic reports for the fourth quarter of 1993, a report for the Water Pollution PE Study number 31, and a prototype report for the Water Supply PE Program, a prototype report for the EML Quality Assurance Program, and a prototype report consolidating results from multiple PE programs. In addition, a report on the ranges of concentrations provided in the programs based on available historical data was prepared and distributed to EM-263 and the QA Working Group. This report will be used to assist development of new PE materials.

The IPEP is working to develop a performance-based statistical assessment and scoring scheme to address EM-263 criteria. Argonne developed a proposal for assessment and corrective action criteria to be used in IPEP and presented it to the EM-263 QA Working Group. The IPEP scoring procedures were modified to allow qualitative assessments of individual matrix analytes, and the evaluation of the laboratory performance assessments using statistical tools has begun.

The IPEP has continued to support EM-263 and RESL efforts to develop the Mixed Analyte Performance Evaluation Program (MAPEP). The MAPEP is currently focused on evaluating laboratory performance for inorganic and radiological analytes. A report on the MAPEP Water Pilot Study was completed and distributed to other members of the QA Working Group, which includes representatives of EML, RESL, Grand Junction Projects Office, and EM-263. A set of separate reports on results was prepared and submitted to RESL for distribution to each participant. In addition, packets of analysis and reporting information were prepared for the MAPEP Soil Pilot Study participants. Data returned by the field participants were compiled and assessed, and a draft report on the Soil Pilot Study was distributed to EM-263 and QA Working Group members for review and comment. Materials for the second MAPEP water study were prepared and forwarded to the participants. All MAPEP data have been entered into the IPEP data base.

In collaboration with the EM-263 Resource Management Group (RMG), we identified the ANL needs for information available from the RMG, as well as modes for computer communications and standardization of the RMG and IPEP data bases. Subsequently, laboratory identification data were transferred from the RMG into the IPEP data base.

Characterization of Waste from Liquid Scintillation Counters (A. S. Boparai, L. L. Chromizky, Y. Tsai, S. D. Kent, D. L. Bowers, and T. TenKate)

The Environment and Waste Management (EWM) Program at ANL needed to characterize liquid scintillation fluid waste for α - and β -emitting radionuclides, volatile organic compounds (VOCs), and semivolatile organic compounds (SVOCs) before shipping the waste to the Hanford Site. Workers in the EWM Program combined the contents of thousands of vials to obtain approximately 250 gal of liquid scintillation waste in several containers. The ACL analyzed the wastes in a timely manner and obtained the following results: toluene, xylenes, and ethyl benzene were the predominant VOCs; naphthalene and alkyl-substituted (alkyl substituents ranged from C-3 through C-12) benzenes were the major SVOCs; and radioactivity ranged from less than 0.3 pCi/mL to 20 pCi/mL for gross alpha and from 400 pCi/mL to 300,000 pCi/mL for gross beta. The β activity was attributed to tritium. The combined efforts of EWM and the ACL have allowed ANL to dispose of waste liquid scintillation fluids, thus reducing hazardous waste stored at ANL.

Review of Analytical Data from Subcontractor Analytical Laboratories (L. B. TenKate and J. J. Marr)

During FY 1994, the ACL assisted the Environment and Waste Management (EWM) Program Division and the Washington D.C. office of the ANL Environmental Assessment Division (EAD) in reviewing inorganic and organic data packages generated by DOE subcontractor laboratories. These packages reported data for 57 soil and 6 water samples taken as part of site characterization efforts at ANL-East (with EWM) and Aberdeen Proving Ground, MD (with EAD). This work included reviewing the data generated by subcontractor laboratories to ensure that the data were generated according to U.S. EPA Contract Laboratory Program guidelines, providing detailed reports of problems encountered with the data, and evaluating the usability of the data.

These efforts improved the usability and quality of the data gathered in the EWM and EAD investigations.

Analysis Support for the Mixed Analyte Performance Evaluation Program (A. M. Essling, I. M. Fox, D. G. Graczyk, D. R. Huff, E. A. Huff, P. L. Johnson , W. E. Streets, and L. B. TenKate)

The ACL continued to serve as an expert laboratory for inorganic analyses for the Mixed Analyte Performance Evaluation Program (MAPEP). This new performance evaluation program is being developed by DOE's Radiological and Environmental Sciences Laboratory (RESL) and the Grand Junction Projects Office (GJPO) for EM-263, Analytical Services Division, to meet the needs of laboratories analyzing mixed-waste samples. In FY 1994, the chemical analysis of materials for the MAPEP Soil Pilot Study materials was completed for the verification round. The method used was protocol ILM02 from the Contract Laboratory Program (CLP) . As specified in the Statement of Work (SOW) for analysis of this material, this protocol involved a mineral-acid digestion of the soil by EPA Method 3050A and subsequent analysis of the trace metals by ICP/AES and graphite furnace atomic absorption spectrophotometry. A full CLP data package was prepared to report these results, as requested in the SOW. Chemical analysis of the verification samples for the second MAPEP water study (MAPEP-94-W2) for the trace metal components was accomplished using a modified CLP protocol; no CLP data package was required for this study. The data were entered directly via computer into the Integrated Performance Evaluation Program (IPEP) data base maintained at ANL. The IPEP has the responsibility of collecting and collating the data from the MAPEP studies and has produced reports on the MAPEP pilot studies (see IPEP section in this report.)

Battery Program (J. J. Marr)

In FY 1994, the ACL continued to provide technical support to the CMT Battery Program (J. Smaga) in the area of post-test analysis of nickel-metal hydride batteries. The objective of the post-test analysis is to identify the existing and potential failure mechanisms that limit the performance and cycle life of the nickel-metal hydride cells. The information obtained from the analyses provides a measure of the technical progress made by battery developers and an indication of needed design or material changes.

During the past year, more than 40 nickel-metal hydride cells were examined. All of the cells were obtained from Ovonic Battery Company and tested at ANL in the CMT Analysis and Diagnostics Laboratory. Tests were conducted under a regimen wherein each cell is operated until it fails to meet performance standards. Post-test analysis results indicated that the primary causes of failure were, in descending frequency, electrolyte redistribution, excessive venting, mechanical degradation of the nickel electrodes, and internal shorting. Chemical and structure changes were also found in nickel electrodes for some of the batteries tested at high temperature. Results of the analyses have been documented and forwarded to the battery developer and program personnel for review.

Microwave-Assisted Extraction of Semivolatile Organic Compounds from Soil (J. B. Schilling and C. M. Newcomb)

Microwave-assisted extraction (MAE) has been investigated as an alternative to conventional Soxhlet and sonication methods for treating soils and extracting semivolatile organic compounds (SVOCs) regulated by the EPA Resource Conservation and Recovery Act. This method minimizes the amount of secondary waste generated during the extraction and analysis of environmental and

waste samples. Only 30 mL of solvent is needed for MAE, compared with up to 300 mL for the conventional techniques.

Extensive studies have been carried out to determine extraction efficiencies under a variety of conditions for both SVOCs spiked on blank soil and commercially available samples. The commercial samples included CRM103-100 that is soil contaminated with polynuclear aromatic hydrocarbons (PAHs) (from Resource Technology Corp.), SRM 1941 that is a contaminated PAHs marine sediment [National Institute of Standards and Technology (NIST)], and two soil samples contaminated with SOVCs [Environmental Resource Associates (ERA)]. Three solvent systems have been studied: methylene chloride, 50:50 methylene chloride/acetone, and 50:50 hexane/acetone. The effects of variations in temperature, microwave power, extraction time, and moisture content have also been studied.

Excellent extraction efficiencies are seen for CRM103-100 with all three solvent systems, compared to conventional extraction techniques. Methylene chloride extractions are improved by adding a small amount of water (10%) to the soil. For the NIST marine sediment, recoveries average only about 50% of the certified values. For the ERA soils, the results are mixed; for most compounds, recoveries were equal to or better than those obtained by using standard extraction techniques, but some recoveries were much lower.

When this method is applied to the whole range of SVOCs spiked on blank soil, extraction yields are comparable to Soxhlet and sonication. Extraction yields of many of the more polar acids and bases are poor; however, this is also true for the conventional techniques.

Microwave-Extraction of Polynuclear Aromatic Hydrocarbons from Coal Ash Samples
(E. Y. Hwang and K. R. Vanderveen)

Coal stack ash is a complex, inhomogeneous mixture of particle sizes, particle shapes, and both organic and inorganic constituents. Until very recently, research on coal ash had been centered on inorganic characterization. Remedial actions were primarily focused on reducing the inorganic constituents in fly ash. For example, to limit the emission of nitrogen oxides, the temperature was reduced for combustion of pulverized coal. Unfortunately, combustion at a decreased temperature and decreased total excess-air ratio dramatically increases organic emissions, including polynuclear aromatic hydrocarbons (PAHs). These compounds are emitted as a product of incomplete combustion of organic materials under such nonideal conditions.

The first step toward determining the PAH content in ash samples is the extraction process. Two microwave extraction techniques were tested as possible alternatives to the conventional Soxhlet and ultrasonic extraction methods. In this set of experiments, ash samples were mixed with methylene chloride in closed containers and subjected to controlled microwave radiation. The prepared sample extracts were analyzed by gas chromatograph/mass spectrometry. Sustained and pulsed-sequence microwave extraction methods were tested, and the results were compared with those from the same solvent in conventional Soxhlet and ultrasonic extraction methods. The data indicated that microwave extraction techniques result in comparable or better recoveries of PAHs. However, with certain ash matrices, chlorinated PAHs were observed in microwave-extracted samples, while none were found in samples prepared by using the other extraction methods. Preliminary investigation of the halogenation mechanism is inconclusive. Nonetheless, by optimizing temperature and microwave power used, the presence of halogenated PAHs can be minimized or eliminated.

Analysis of Polymers by Fourier Transform Infrared Microscopy (S. D. Kent, G. T. Reedy, and S. E. Carpenter)

Polymer samples submitted by the Energy Systems Division (E. Shoemaker) were analyzed using the Nicolet 60SX Spectrophotometer and Spectra-Tech IR Microscope. The instrument is equipped with an Olympus PM-10M camera for taking photographs of the sample in the microscope, as well as a Toshiba video camera and monitor for enhanced imaging of the sample. Preparation of the polymer sample is an important aspect of the analysis since thin films of the bulk polymers are required to obtain usable IR spectra. Polymers such as polyethylene, polypropylene, polystyrene, and polyvinyl chloride were prepared by cutting off a small section of the polymer sample and then flattening the sample into a thin film by using a roller knife. The thin polymer films were placed on potassium bromide disks, and the microscope was adjusted to a field of view of approximately 100 micrometers. The thicknesses of the polymer films were minimized to obtain proper light transmission and reduced scattering. The spectra obtained for the polymer samples were compared to those obtained from the Nicolet Hummel Polymer Library. Qualitative analysis was possible by choosing the closest match from a spectral library for each of the polymer films.

Support for Polyethylene Glycol Irradiation Studies (K. J. Parish, A. S. Boparai, L. L. Chromizky, and Y. Tsai)

The ACL provided analytical support to a project in which CMT researchers (D. Chaiko and Y. Vojta) are examining the use of polyethylene glycol to remove radioactive components from wastes currently stored at the Hanford Site. One objective of this project was to determine whether polyethylene glycol undergoes significant changes upon irradiation that would affect its function as an agent to remove radioactive components from waste. The ACL analyzed irradiated polyethylene glycol samples for volatile and semivolatile organic compounds that might have been formed during irradiation. The volatile organic compound analysis was performed using a purge-and-trap

technique, followed by gas chromatographic analysis with mass spectrometric detection. A semivolatile organic analysis was also performed on the samples. Prior to the semivolatile analysis, the samples were derivatized by using a silylation agent, a process that improves the stability and detectability of relatively non-volatile polar compounds. Results of the analyses were tabulated and submitted to the CMT researchers along with a report detailing the analytical procedures used for the project.

Reflectance FTIR Microscopy (S. E. Carpenter, G. T. Reedy, E. Y. Hwang, S. D. Kent, and C. T. Snyder)

Transmission FTIR microscopy has been successfully employed in the ACL to analyze samples that transmit, or can be made to transmit, infrared light; however, few studies of opaque samples, thin films, or coatings had been conducted in our laboratory until recently. Unlike samples that can be analyzed by transmission microscopy, materials such as opaque samples, thin films, and coatings must be analyzed by reflectance microscopy. Identification of reflectance spectra of unknown samples is difficult since they cannot be directly compared to libraries of absorbance spectra without mathematical transformation. For example, if the surface of an opaque sample is smooth, specular reflection may cause the spectrum to exhibit derivative-like curves. By applying a Kramers-Kronig transform to such a spectrum, an absorbance spectrum can be approximated. If the surface of the opaque sample is rough, diffuse reflectance may produce inaccuracies in the relative peak intensities. In this case, the absorption spectrum can be approximated by applying a Kubelka-Munk transform to the diffuse reflectance spectrum. Although the FTIR microscope in the ACL can be used to perform reflectance measurements, this capability was seldom used because ACL personnel had limited training with the technique, and software for performing some of the required mathematical transformations was not available in-house. However, the ACL is receiving

an increasing number of requests for FTIR microscopic analyses of samples requiring reflectance measurements. Therefore, ACL personnel received formal training from the FTIR microscope vendor on reflectance microscopy and sample preparation through an on-site, two-day training course. We have also purchased additional mathematical transformation software and new sample preparation equipment that will allow us to perform a full range of reflectance analyses.

Screening of Soil Samples at the Rocky Mountain Arsenal (L. L. Chromizky)

In FY 1994, the ACL collaborated with the Energy Systems Division (J. Schneider) to develop a method for screening of soil samples for pesticides at the Rocky Mountain Arsenal located in Denver, CO. Method development work involved extraction of spiked soils with methanol followed by detection and quantitation of pesticides using a gas chromatograph/mass spectrometer. Argonne personnel used the method at the Arsenal to determine the level of contamination in field samples. Results were generated within hours of sampling. The purpose of the screening was to identify soils suitable for a future demonstration of thermal desorption decontamination technologies.

Development of "Smart" Chemical Sensors (S. E. Carpenter, P. L. Johnson, and A. R. Demlow)

Current ACL research efforts in chemometrics (the use of mathematics, statistics, and formal methods of logic for analysis of chemical data) are focused on the development of an optimized software system for designing automated monitoring algorithms for spectroscopic, chromatographic, electrochemical, and radiological sensors that are of interest to the DOE Office of Arms Control and Nonproliferation. The objective of this work is to produce "smart" sensors that provide real-time data analysis and can be linked to multisensor data fusion systems. Smart sensors have obvious advantages for both invasive and noninvasive monitoring scenarios. The software system, referred

to as the Sensor Algorithm Generation Environment (SAGE), provides a unique "workbench" that will allow users to selectively combine and experiment with various types of signal processing procedures, including digital filtering, pattern recognition, and multivariate quantitative techniques, to produce customized qualitative and/or quantitative data analysis algorithms for a particular sensor. These algorithms can then be implemented in software via the computer controlling the sensor, or they can be implemented in hardware by encoding the algorithm onto a small digital signal processing board, which would become an integral part of the sensor electronics. The SAGE workbench is expected to save time, effort, and expense in producing optimized, smart sensors capable of automated data analysis and decision making. Although the SAGE workbench is still under development, novel algorithms have been successfully designed for qualitative processing of passive-remote FTIR sensor data and for quantitative processing of data from an extractive FTIR system used for continuous emissions monitoring.

Determination of Composition and Tritium Content for Gas from Sealed Device (C. T. Snyder, D. G. Graczyk, and D. L. Bowers)

Staff of ANL's Chemistry (CHM) Division (J. McCreary, J. Hughes) needed to perform destructive analysis of a sealed device to determine whether the device met fabrication specifications. Along with physical dimensions, the specifications included requirements for an amount of tritium that the device should contain, the distribution of tritium between elemental species and tritium oxide (i.e., T_2 vs. T_2O), and limits on the amounts of gases other than tritium that might be present. By breaking the glass shell of the device inside an evacuated system of known volume and measuring the pressure of the released gas, the CHM staff determined the total quantity of gas contained in the device. The ACL performed compositional analysis on a small portion of the gas by mass spectrometry, measuring elemental tritium species (HT and T_2), impurity gases

(such as N_2 and O_2), and the tritium decay product 3He . When taken together with the total gas content, these data permitted calculation of the tritium present in the device when it was broken as well as the quantity of tritium that had undergone radioactive decay before the device was opened. Because gas-phase analysis does not reliably measure hydrogen oxides (water), the tritium oxides were measured separately. The CHM staff added a known quantity of normal deionized water to the system that contained the broken device and collected the added water. By means of liquid scintillation counting, we measured the tritium oxides scavenged by the water that was added. All results obtained from these measurements were consistent with the expected characteristics of the gas with which the device was filled.

Determination of Low Molecular Weight Analytes in Waste-Drum Headspace Gas for WIPP Project
(C. T. Snyder, D. V. Applegate, and D. G. Graczyk)

Among the capabilities ACL has established for the WIPP Experimental Waste Characterization Program is a methodology for compositional analysis of waste-drum headspace samples to determine permanent gases and low molecular weight hydrocarbons (methane through propane). Volume-percent concentrations of each of 10 target-analyte gases (H_2 , N_2 , O_2 , Ar, CO, CO_2 , NO_x , CH_4 , C_2H_6 , and C_3H_8) are measured with a magnetic-sector, gas-analysis mass spectrometer having moderate resolution. Instrument stability and sensitivity have been demonstrated to meet program requirements regarding precision, accuracy, and detection limits for measuring the gases of interest.

In FY 1994, measurements were made to study the effectiveness of sampling procedures being developed for characterizing headspace gases in waste drums stored on the ANL-East site. In these studies, mixtures of the target analyte gases were analyzed before and after being transferred through a prototype of the sampling apparatus. These analyses demonstrated that the gas

composition was not changed by passage through the sampling device and confirmed the effectiveness of the proposed sampling approach.

Technical Support to the Department of Energy, Chicago Operations Office (C. T. Snyder, D. L. Bowers, and T. TenKate)

In 1991, DOE imposed an agency-wide moratorium on off-site shipment of hazardous wastes. To have the moratorium lifted, each DOE facility had to implement approved procedures for certifying, prior to shipment, that its hazardous waste contained "no DOE-added radioactivity." The ANL-East site satisfied this certification requirement with a system that drew on a trained, cognizant individual's assessment of the process by which the waste was generated to determine that specific wastes did not contain radioactive contaminants. This "process knowledge" certification procedure allowed ANL to ship most of its hazardous waste for disposal.

In cases where wastes might have been contaminated during their generation, additional measures were needed to complement the process-knowledge certification procedure. The ACL and ANL's Environment and Waste Management (EWM) Programs have provided the DOE Chicago Operations Office (DOE-CH) with technical support to establish a procedure in which the certification decision could be based on analysis data. In FY 1994, DOE-CH approved a "limit of sensitivity" approach for certifying wastes as free of added radioactivity, and EWM and the ACL began establishing a waste-certification process based on that approach. Draft operating procedures were evaluated by analyzing test-case wastes, and shortcomings that were identified have been addressed. By allowing waste generators to certify their waste as nonradioactive when no radionuclides are detected by specified analysis procedures, this alternative to process-knowledge certification will help ensure proper handling and disposal of ANL's hazardous wastes.

Stand-Off Detection (J. C. Demirgian, S. M. Macha, and G. F. Busse)

The U.S. Army's Edgewood Research, Development and Engineering Center has an ongoing program in stand-off detection of gaseous chemical plumes. The ACL contributes to the objectives of this program by testing instrumentation. This testing includes determining the sensitivity of newly designed passive-remote FTIR spectrometers and enhancing the ability of existing spectrometers to properly detect plumes in the presence of interferences.

We have constructed an analyte data base consisting of quantitative data at different black-body temperatures for dimethyl methylphosphonate and diisopropyl methylphosphonate. Data were collected in the laboratory and in the field using different backgrounds. We will use this data set to determine the ability of the FTIR instrumentation to detect analytes under differing background conditions. The black-body emission spectra will be correlated with different field emission data.

CMT/APS Thrust Group Activities (W. E. Streets and B. S. Tani)

This CMT intradivisional group was founded in January 1989 to explore possible interactions between the CMT and the Advanced Photon Source (APS) for current and future research programs. The group was directed (1) to gather and disseminate information on the application of synchrotron radiation (SR) in research areas of interest to CMT; (2) to familiarize CMT staff with the capabilities, procedures, special issues, and costs associated with research at the APS; (3) to arrange seminars on the use of synchrotrons in research areas germane to CMT; and (4) to create a link with the other divisional synchrotron thrust groups at ANL. The eventual goal of the group is to develop a plan for utilization of the APS by CMT. As specific interests within the division for use of SR have become better defined, the CMT/APS Thrust Group has again been reorganized to reflect those interests and to better serve the Division needs. R. Chiarello is being directly supported by CMT as an SR technical advisor. He also functions as chairman of the group,

which now consists of the following members: J. A. Fortner, C. E. Johnson, L. Leibowitz, M. A. Lewis, V. A. Maroni, W. E. Streets, and D. J. Zurawski. B. S. Tani continues to serve as back-up to W. E. Streets as ACL's representative.

The Thrust Group has continued to investigate ways that CMT as a division might participate in research at APS. Continuing negotiations with three existing APS Collaborative Access Teams (CATs) previously identified as potential partners have resulted in an agreement with the Materials Research Collaborative Access Team (MRCAT) to develop a sector at the APS with a \$1 million contribution from the CMT Division over five years. Other MRCAT collaborators include the University of Notre Dame, University of Florida, Illinois Institute of Technology, Northwestern University, AMOCO, and the Naval Research Laboratory.

The Associate Laboratory Director for Engineering Research is funding the ANL collaboration with the Louisiana State University (LSU) to construct a hard X-ray micro-analytical beamline at the Center for Advanced Microstructures and Devices (CAMD), a 1.5 GeV synchrotron radiation research center. The beamline will allow examination of small-scale structures using diffraction and fluorescence techniques.

IV. QUALITY ASSURANCE

Quality Assurance/Quality Control in the ACL (F. J. Martino)

Before sponsors will allow a laboratory to analyze samples, they often require the laboratory to demonstrate instrument and analyst performance through successful analysis of single blind performance evaluation samples. ("Single blind" means that the analyte values are not known in advance by the analyst.) The ACL continued its participation in multilaboratory performance assessment programs sponsored by the U.S. EPA and DOE, which provide a record of achievement on such blind samples. Programs in which the ACL participated in FY 1994 included:

- The U.S. EPA Environmental Monitoring Systems Laboratory-Cincinnati (EMSL-Ci) Water Pollution Performance Evaluation Study (WP). In this study, samples were analyzed for cyanide, oil and grease, total dissolved and suspended solids, and a variety of anions. The EMSL-Ci reported that analytical results submitted during FY 1994 for samples WP031 and WP032 showed excellent agreement with the EPA known values.
- The DOE Environmental Measurements Laboratory (EML) Quality Assessment Program and the U.S. EPA EMSL-Las Vegas Performance Evaluation Studies Program. Every six months, these programs provide participating laboratories with blind radiological performance evaluation samples. Water, soil, and air filter samples were analyzed for radiological parameters (e.g., gross alpha-beta, gamma, tritium, Ra, U, Pu, Np) throughout FY 1994. The comparison of ACL results to the accepted values showed that the ACL made each of the determinations correctly.

The ACL analyzed many hazardous and mixed-waste samples as part of site remediation and closure programs. Most of these samples are either from a site that is part of a U.S. Department of Agriculture monitoring program or from ANL-East programs for orphan waste cleanup and

decontamination and decommissioning. In analyzing these samples, the ACL used methods described in the U.S. EPA Contract Laboratory Program (CLP) Statement of Work or the EPA's "Test Methods for Evaluating Solid Waste" (SW-846), or approved alternatives. Approximately 50 CLP-type data packages, consisting of analytical results and raw data, were prepared and forwarded to the customers for data evaluation.

Because the ACL analyzes a large number of samples annually, we have implemented a computer-assisted bar code system that provides quick and error-free logging, distribution, and tracking of analytical samples. The system's capabilities include providing sample notification memos, chain-of-custody forms, and sample tracking reports. A bar code scanning station had been previously installed in the main sample receiving area; in FY 1994 a satellite scanning station was installed in the volatile organics laboratory in CMT. Another unit is in the process of being installed for use by the Chemical Analysis Group in Building 200, and additional scanning stations are planned for other ACL laboratories. The system is expected to be fully utilized in FY 1995.

Quality assessments are an important mechanism for detecting deficiencies in the ACL quality system and identifying areas needing improvement. In some cases, a sponsor requires the assessments. This year the ACL participated in assessments performed by teams representing Reynolds Electric and Engineering Co., Inc. (December 1993), the ANL-East Office of Quality Assurance (September 1994), and the State of Utah Department of Health, Division of Laboratory Services (August 1994). Teams assessed the ACL Quality Assurance Plan, standard operating procedures, training records, procurement practices, and sample custody and tracking, and they conducted interviews and laboratory tours. Most identified deficiencies have been resolved and the few remaining are to be resolved in FY 1995.

The DOE Chicago Field Office asked that the ACL obtain Utah certification so that it could provide analyses on mixed waste intended for disposal at Utah's Envirocare site. The certification

process included an on-site assessment (August 1994) of the ACL's analytical capabilities and the qualifications of its analysts. On August 15, 1994, the ACL obtained Utah certification to perform organics, inorganics, and radiological analyses required by the Resource and Conservation Recovery Act in support of DOE waste disposal efforts.

V. PROFESSIONAL ACTIVITIES

A. Publications and Reports

Advances in Passive-Remote and Extractive Fourier Transform Infrared Spectroscopic Systems

J. C. Demirgian

Proceedings of the Air and Waste Management Association's International Specialty Conference, Atlanta, GA, October 11-14, 1993 (1993)

Analytical Chemistry Laboratory Progress Report for FY 1993

D. W. Green, A. S. Boparai, D. L. Bowers, D. G. Graczyk, and P. C. Lindahl, with contributions from ACL Staff

Argonne National Laboratory Report No. ANL/ACL-93/2 (1993)

Analytical Procedures for Waste Minimization/Pollution Prevention

D. W. Green, L. L. Smith, J. S. Crain, A. S. Boparai, and J. B. Schilling

Proceedings of the Pollution Prevention Conference X, Denver, CO, May 3-5, 1994, pp. 1-7 through 1-11 (1994)

Determination of PCBs in Rocky Flats Type IV Waste Sludge by Gas Chromatography/Electron Capture Detection

K. J. Parish, D. V. Applegate, A. S. Boparai, and G. T. Reedy

Argonne National Laboratory Report No. ANL/ACL-93/1 (1993)

Effects of Radiation Exposure on Glass Alteration in a Steam Environment

D. J. Wronkiewicz, L. M. Wang, J. K. Bates, and B. S. Tani

Mater. Res. Soc. Symp. Proc. 294, 183-190 (1994)

Evaluation of a Fourier Transform Infrared Continuous Emission Monitor Field Test at a TSCA Incinerator

Z. Mao, J. C. Demirgian, and G. T. Reedy

Proceedings of the 1994 Incineration Conference, Houston, TX, May 4-8, 1994

Performance Specifications for Technology Development: Application for Characterization of Volatile Organic Compounds in the Environment

S. E. Carpenter, P. V. Doskey, M. D. Erickson, and P. C. Lindahl

Argonne National Laboratory Report No. ANL/ER/TM-3 (1994)

Radiochemical Method Development

M. D. Erickson, J. H. Aldstadt, J. S. Alvarado, J. S. Crain, K. A. Orlandini, and L. L. Smith

Proceedings of the Air and Waste Management Association's International Specialty Conference, Herndon, VA, March 22-25, 1994

The Stability of Calibration Standards for ICP/AES Analysis: A Two-Year Study

D. R. Huff and E. A. Huff

Proceedings of the Tenth Annual Waste Testing and Quality Assurance Symposium, Arlington, VA, July 11-15, 1994 (1994)

Stability of Low Concentration Calibration Standards for Graphite Furnace Atomic Absorption Spectrophotometry

D. A. Bass and L. B. TenKate

Argonne National Laboratory Report No. ANL/ACL-93/3 (1993)

Standoff Detection of Simulated Illicit Drug Laboratories

J. C. Demirgian and J. Fortuna

Proceedings of the Tactical Technologies and Wide Area Surveillance International Symposium, Chicago, IL, November 2-5, 1993 (1993)

B. Oral Presentations

Advances in Passive-Remote and Extractive Fourier Transform Infrared Spectroscopic Systems

J. C. Demirgian

Air and Waste Management Association's International Specialty Conference, Atlanta, GA, October 11-14, 1993

Analytical Electron Microscopy Examination of Solid Reaction Products in Long-Term Tests of SRL 200 Waste Glasses

E. C. Buck, J. A. Fortner, J. K. Bates, X. Feng, N. L. Dietz, C. R. Bradley, and B. S. Tani

Fall Meeting of the Materials Research Soc., Boston, MA, November 29-December 3, 1993

Analytical Procedures for Waste Minimization and Pollution Prevention

D. W. Green, L. L. Smith, J. S. Crain, A. S. Boparai, and J. B. Schilling

The Pollution Prevention Conference X, Denver, CO, May 3-5, 1994

Automated Sample Preparation of Radioactive Mixed Waste Samples for Organic Analysis

A. S. Boparai, K. J. Parish, D. V. Applegate, G. T. Reedy, and S. E. Carpenter

45th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, February 28-March 4, 1994

Biology's Animal Orphan Waste

F. Markun

19th ACL Technical Meeting, Argonne National Laboratory, Argonne, IL, November 9, 1993

Characterization of HEPA Filters for Disposal

A. M. Essling, I. M. Fox, F. P. Smith, and D. G. Graczyk

34th ORNL-DOE Conference on Analytical Chemistry in Energy Technology,
Gatlinburg, TN, October 5-7, 1993

Characterization of HEPA Filters for Disposal

A. M. Essling, I. M. Fox, F. P. Smith, D. G. Graczyk, and C. W. Grandy

ANL Technical Women's Symposium, Countryside, IL, September 13-14, 1994

Characterization of Organic Compounds in Coal Ashes

E. Y. Hwang

19th ACL Technical Meeting, Argonne National Laboratory, Argonne, IL,
November 9, 1993

A Comparison of Extraction of Soils Using the Toxicity Characteristic Leaching Procedure
and Total Metal Digestion

D. A. Bass

34th ORNL-DOE Conference on Analytical Chemistry in Energy Technology,
Gatlinburg, TN, October 5-7, 1993

A Comparison of Extraction of Soils Using the Toxicity Characteristic Leaching Procedure
and Total Metal Digestion

D. A. Bass

19th ACL Technical Meeting, Argonne National Laboratory, Argonne, IL,
November 9, 1993

Continuous Emission Monitoring of Incinerators Using Extractive and Passive-Remote
Fourier Transform Infrared Spectroscopy

J. C. Demirgian, C. L. Hammer, E. Y. Hwang, and Z. Mao

45th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied
Spectroscopy, Chicago, IL, February 28-March 4, 1994

Development of Prototype Management Reports on Analytical Laboratory Performance in
Performance Evaluation Programs for the DOE Office of Environmental Restoration and
Waste Management

S. Warren, J. Hensley, D. A. Bass, and A. Bunn

Waste Management 94 Conference, Tucson, AZ, February 27-March 3, 1994

Development of a Standard Operating Procedure for the Continuous Emission Monitoring of
Incinerator Effluents using FTIR Technology

E. Y. Hwang, J. C. Demirgian, Z. Mao, and G. Plummer

45th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied
Spectroscopy, Chicago, IL, February 28-March 4, 1994

Development of Self-Absorption Coefficients for the Determination of Gamma-Emitting Radionuclides in Environmental and Mixed Waste Samples

W. E. Streets

1994 Symposium on Radiation Measurements and Applications, Ann Arbor, MI,
May 16-19, 1994

Development of the Integrated Performance Evaluation Program (IPEP)

P. C. Lindahl and W. R. Newberry

International Association of Environmental Testing Laboratories Stakeholders
Workshop, Arlington, VA, April 20-22, 1994

DOE's PE Program Initiatives

P. C. Lindahl

8th Annual Workshop-Interagency Steering Committee for Quality Assurance for
Environmental Measurements, Gaithersburg, MD, August 23-24, 1994

Enhanced Detection of Long-Lived Actinides by Inductively Coupled Plasma-Mass Spectrometry

J. S. Crain and L. L. Smith

20th Annual Meeting of the Federation of Analytical Chemistry and Spectroscopy
Societies, Detroit, MI, October 17-22, 1993

Enhanced Measurement of Long-Lived Actinides by ICP-MS

J. S. Crain, L. L. Smith, J. S. Yaeger, and J. S. Alvarado

3rd International Conference on Methods and Applications of Radioanalytical
Chemistry, Kailua-Kona, HI, April 10-16, 1994

Environmental Radioanalytical Method Development: What's New

L. L. Smith

20th ACL Technical Meeting, Argonne National Laboratory, Argonne, IL,
February 22, 1994

Field-Analytical-Laboratory Screening of Soils and Underground Storage Tanks

J. F. Schneider, D. V. Applegate, N. A. Tomczyk, J. D. Taylor, and L. F. Sytsma

45th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied
Spectroscopy, Chicago, IL, February 28-March 4, 1994

Faster Radiochemical Methods

M. D. Erickson, J. H. Aldstadt, J. S. Alvarado, J. S. Crain, K. A. Orlandini, and

L. L. Smith

Cost-Efficient Acquisition and Utilization of Data in the Management of
Hazardous Waste Sites Conference, Herndon, VA, March 22-25, 1994

Fine Tuning the Gas Chromatography/Matrix Isolation-Infrared Spectrometry Technique

G. T. Reedy, S. E. Carpenter, and A. S. Boparai

45th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied
Spectroscopy, Chicago, IL, February 28-March 4, 1994

Improved Method for the Determination of Actinide Elements in Soils

L. L. Smith, J. S. Yaeger, and J. S. Crain

3rd International Conference on Methods and Applications of Radioanalytical Chemistry, Kailua-Kona, HI, April 10-16, 1994

New Isotopic Perspectives on Atmospheric Carbon Monoxide

C. M. Stevens

1993 American Geophysical Union 1993 Fall Meeting, San Francisco, CA, December 6-10, 1993

New Isotopic Perspectives on Atmospheric Carbon Monoxide

C. M. Stevens

20th ACL Technical Meeting, Argonne National Laboratory, Argonne, IL, February 22, 1994

PCB Determination in Rocky Flats Plant Mixed Waste Sludges

K. J. Parish

20th ACL Technical Meeting, Argonne National Laboratory, Argonne, IL, February 22, 1994

Performance Evaluation Programs - Alternatives Based on User Needs

P. C. Lindahl, W. E. Streets, R. Newberry, D. Bottrell, C. Klusek, S. Morton, and S. Hedayat

34th ORNL-DOE Conference on Analytical Chemistry in Energy Technology, Gatlinburg, TN, October 5-7, 1993

Proposed Scoring Protocols for the Integrated Performance Evaluation Program (IPEP)

W. E. Streets

20th ACL Technical Meeting, Argonne National Laboratory, Argonne, IL, February 22, 1994

Radioanalytical Methods Evaluation

J. S. Alvarado, J. S. Crain, J. H. Aldstadt, L. L. Smith, K. A. Orlandini, and M. D. Erickson

Outreach to Industry: Partnership for the Future Conference, Seattle, WA, January 25-27, 1994

Radiochemical Method Evaluation--Industrial Partner Opportunities

M. D. Erickson, M. A. Edgell, P. C. Lindahl, L. L. Smith, and M. H. Carter

International Association of Environmental Testing Laboratories Stakeholders Workshop, Arlington, VA, April 20-22, 1994

Removal of Radioactivity from Radioactive Mixed Waste Samples

A. S. Boparai, M. A. Gritters, Y. Tsai, and S. D. Kent

45th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, February 28-March 4, 1994

Sample Transportation

D. W. Green

11th DOE Analytical Laboratory Managers Meeting, Boulder, CO,
October 25, 1993

The Stability of Calibration Standards for ICP/AES Analysis: A Two-Year Study

D. R. Huff and E. A. Huff

10th Annual Waste Testing and Quality Assurance Symposium, Arlington, VA,
July 11-15, 1994

Stability of Graphite Furnace Calibration Standards

L. B. TenKate and D. A. Bass

Society for Applied Spectroscopy, Chicago Section, Villa Park, IL,
February 8, 1994

Standoff Detection of Simulated Illicit Drug Laboratories

J. C. Demirgian and J. Fortuna

Tactical Technologies and Wide Area Surveillance, International Symposium,
Chicago, IL, October 19-22, 1993

Time-Domain vs. Frequency Domain Analysis of FTIR Data

S. E. Carpenter

45th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied
Spectroscopy, Chicago, IL, February 28-March 4, 1994

TRU-Spec and RE-Spec Chromatography: Basic Studies and Applications

E. A. Huff and D. R. Huff

34th ORNL-DOE Conference on Analytical Chemistry in Energy Technology,
Gatlinburg, TN, October 5-7, 1993

TRU-Spec and RE-Spec Chromatography: Basic Studies and Applications

E. A. Huff

19th ACL Technical Meeting, Argonne National Laboratory, Argonne, IL,
November 9, 1993

C. Awards and Patents

Jack C. Demirgian

Pacesetter Award for his leadership on the continuous emission monitoring work,
February 1994.

Edmund A. Huff

With D. P. Peterson and M. H. Bhattacharyya, U.S. Statutory Invention Registration,
Reg. No. 41310, May 3, 1994. Process for Measuring Low Cadmium Levels in Blood
and Other Biological Specimens.

D. Meetings Attended

Dean A. Bass

45th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, February 28-March 4, 1994

10th Annual Waste Testing and Quality Assurance Symposium, Arlington, VA, July 11-15, 1994

Delbert L. Bowers

American Society for Testing and Materials, Committee C-26, San Francisco, CA, January 23-28, 1994

American Society for Testing and Materials, Committee C-26, Santa Fe, NM, July 24-29, 1994

Scott E. Carpenter

Joint Multisensor Systems Research (MSR) and Airborne Multisensor Pod System (AMPS) Working Group Meeting, EG&G Remote Sensing Laboratory, Nellis Air Force Base, NV, August 17-18, 1994

Jeffrey S. Crain

45th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, February 28-March 4, 1994

Alice M. Essling

45th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, February 28-March 4, 1994

David W. Green

34th ORNL-DOE Conference on Analytical Chemistry in Energy Technology, Gatlinburg, TN, October 5-7, 1993

45th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, February 28-March 4, 1994

Edmund A. Huff

45th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, February 28-March 4, 1994

Paul L. Johnson

Argonne National Laboratory Symposium for Undergraduates in Science, Engineering, and Mathematics, Argonne, IL, November 5-6, 1993, Session Chairman

Peter C. Lindahl

RADS Data Validation Workshop, Gaithersburg, MD, November 17-18, 1993

7th Annual Interagency Workshop on Quality Assurance for Environmental Measurements, New Orleans, LA, December 14-16, 1993

10th Annual Waste Testing and Quality Assurance Symposium, Arlington, VA, July 11-14, 1994

J. Bruce Schilling

42nd American Society for Mass Spectrometry (ASMS) Conference on Mass Spectrometry and Allied Topics, Chicago, IL, May 29-June 3, 1994

Florence P. Smith

45th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, February 28-March 4, 1994

Lesia L. Smith

34th ORNL-DOE Conference on Analytical Chemistry in Energy Technology, Gatlinburg, TN, October 5-7, 1993

39th Annual Conference on Bioassay, Analytical, and Environmental Radiochemistry, Colorado Springs, CO, October 11-15, 1993

45th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, February 28-March 4, 1994

Christine T. Snyder

Argonne National Laboratory Technical Womens Symposium, Countryside, IL, September 13-14, 1994

W. Elane Streets

39th Annual Conference on Bioassay, Analytical, and Environmental Radiochemistry, Colorado Springs, CO, October 11-15, 1993

7th Annual Workshop on Quality Assurance for Environmental Measurements, New Orleans, LA, December 14-16, 1993

5th International Environmental and Waste Management Conference (Energy and Environmental Quality Division of the American Society for Quality Control), Las Vegas, NV, April 17-20, 1994

10th Annual Waste Testing and Quality Assurance Symposium, Arlington, VA, July 11-15, 1994

8th Annual Workshop, Interagency Steering Committee for Quality Assurance for Environmental Measurements, Gaithersburg, MD, August 23-25, 1994

Lynn B. TenKate

45th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, February 28-March 4, 1994

Judith S. Yaeger

39th Annual Conference on Bioassay, Analytical, and Environmental Radiochemistry, Colorado Springs, CO, October 11-15, 1993

45th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, February 28-March 4, 1994

E. Professional Organizations and Activities

Dean A. Bass

American Chemical Society

Sigma Xi

Society for Applied Spectroscopy

Amrit S. Boparai

American Chemical Society

Sigma Xi

Delbert L. Bowers

American Chemical Society

American Society for Testing and Materials

Scott E. Carpenter

American Chemical Society

Air and Waste Management Association

North American Chapter of the International Chemometrics Society

Jeffrey S. Crain

American Chemical Society

Division of Analytical Chemistry

Jack C. Demirgian

American Chemical Society

Air and Waste Management Association

Technical Association of the Pulp and Paper Industry

Irene M. Fox

Society for Applied Spectroscopy

Association for Women in Science

Sigma Xi

Donald G. Graczyk

American Chemical Society

Sigma Xi

American Society for Mass Spectrometry

David W. Green

American Chemical Society

Division of Analytical Chemistry

Analytical Laboratory Managers Association, Board of Directors

Editorial Board of Managing the Modern Laboratory

Sigma Xi

Doris R. Huff

Phi Beta Kappa

Edmund A. Huff

American Chemical Society

American Society for Testing and Materials

Society for Applied Spectroscopy

Sigma Xi

Paul L. Johnson

American Crystallographic Association

Association for Computing Machinery

Sigma Xi, ANL Chapter Secretary

James T. Kiely

American Chemical Society

Peter C. Lindahl

American Chemical Society
Division of Analytical Chemistry

Society for Applied Spectroscopy

Francis Markun

Health Physics Society
Midwest Chapter of Health Physics Society

Gerald T. Reedy

American Chemical Society

Society for Applied Spectroscopy

Carmen S. Sabau

American Chemical Society (National)
Nuclear Chemistry and Technology (Division)
Chicago Section

American Nuclear Society (National)
Fuel Cycle and Waste Management Division
Chicago Section
Environmental Sciences Division

American-Romanian Academy of Arts and Sciences

Association of Women in Science (National)
Chicago Chapter

New York Academy of Sciences

Sigma Xi

J. Bruce Schilling

American Chemical Society

American Society for Mass Spectrometry

Madison-Chicago-Milwaukee Mass Spectrometry Discussion Group

Midwest Supercritical Fluid Chromatography User's Group

Florence P. Smith

National Organization of Black Chemists and Chemical Engineers

Christine T. Snyder

American Chemical Society

W. Elane Streets

American Society for Testing and Materials

American Society for Quality Control

Lynn B. TenKate

Society for Applied Spectroscopy

Sigma Xi

Tony TenKate

American Association of Physics Teachers

Sigma Xi

F. Professional Training

Lilia Barbosa

WordPerfect Desktop Publishing, Westmont, IL, October 14, 1993

Listen, Learn, and Live, Darien, IL, March 16, 1994

Alice M. Birmingham

Listen, Learn, and Live, Darien, IL, March 15, 1994

Life Balance and Stress Solution, Westmont, IL, June 22, 1994

Delbert L. Bowers

Environmental Radioactivity Quantification Workshop, Lisle, IL, March 24, 1994

Scott E. Carpenter

Advanced Laboratory Data Management Using a Personal Computer; Short Course,
45th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied
Spectroscopy, Chicago, IL, February 28-March 4, 1994

FTIR Microscopy Training Course, Argonne, IL, July 11-12, 1994

Alice M. Essling

Microwave Digestion Users Group Workshop, Gatlinburg, TN, October 4, 1993

Irene M. Fox

Life Balance and Stress Solution, Westmont, IL, June 22, 1994

Donald G. Graczyk

Priority Management Workshop, Oak Park, IL, October 26, 1993

David W. Green

Fundamentals of Radiochemistry, Las Vegas, NV, February 7-11, 1994

Elizabeth Y. Hwang

FTIR Microscopy Training Course, Argonne, IL, July 11-12, 1994

Paul L. Johnson

How to be a Better Technical Writer, Oak Lawn, IL, July 18, 1994

Stephen D. Kent

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James T. Kiely

ICP-MS Continuing Education Series, Omaha, NE, September 15-16, 1994

Linda L. Lane

Listen, Learn, and Live, Darien, IL, March 16, 1994

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Fundamentals of Radiochemistry, Las Vegas, NV, February 7-11, 1994

Vanessa A. Mendez

The Accounting Game, Oak Brook, IL, September 12, 1994

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FTIR Microscopy Training Course, Argonne, IL, July 11-12, 1994

Florence P. Smith

Introduction to Thermal Ionization Mass Spectrometry, Argonne, IL,
October 19-20, 1993

Lesla L. Smith

VG PlasmaQuad Basic Training Course: ICP-MS Theory and Operation, Argonne, IL,
July 11-15, 1994

Christine T. Snyder

FTIR Microscopy Training Course, Argonne, IL, July 11-12, 1994
How to be a Better Technical Writer, Oak Lawn, IL, July 18, 1994

W. Elane Streets

Project Management, Westmont, IL, August 4, 1994

Tony TenKate

Gamma Spectroscopy Using Germanium Detectors and Gammavision, Oak Ridge, TN,
September 26-30, 1994

Heidi L. Terdic

Listen, Learn, and Live, Darien, IL, March 15, 1994

Managing Time, Shorewood, IL, May 26, 1994

G. ACL Seminars

Applications of ICP-MS to the Analysis of Various Nuclear Waste Forms

Dr. Jose Ignacio Garcia Alonso, Institute for Transuranium Elements,
Karlsruhe, Germany

January 21, 1994

Neutron Activation Analysis of Bioenvironmental Samples

Dr. Sheldon Landsberger, University of Illinois, Champaign-Urbana, IL

February 16, 1994

The Use of Chemically Modified Electrodes in Liquid Chromatography and Flow Injection Analysis

Professor Erkang Wang, Chinese Academy of Sciences, Changchun, Jilin, China

March 2, 1994

Factors Controlling Supercritical Fluid Extraction (SFE) Recoveries of Organic Pollutants from Heterogeneous Environmental Solids

Dr. Steven B. Hawthorne, University of North Dakota, Grand Forks, ND

April 28, 1994

Radiochemical Characterization of Low Level Wastes at Chalk River Laboratories

Dr. Kerry I. Burns, Chalk River Laboratories, Chalk River, Ontario, Canada

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