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**ANALYTICAL CHEMISTRY LABORATORY**  
**Progress Report for FY 1996**

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## NOMENCLATURE

|         |   |
|---------|---|
| ACL     | Analytical Chemistry Laboratory                         |
| AGS     | Alternating Gradient Synchrotron                        |
| AMPS    | Airborne Multispectral Pod System                       |
| ANL     | Argonne National Laboratory                             |
| APS     | Advanced Photon Source                                  |
| BNL     | Brookhaven National Laboratory                          |
| BSCCO   | bismuth/strontium/calcium/copper oxides                 |
| CEM     | continuous emission monitor                             |
| CLP     | Contract Laboratory Program                             |
| CMT     | Chemical Technology Division                            |
| CST     | crystalline silicotitanate                              |
| CVAA    | cold vapor atomic absorption                            |
| DOE     | U.S. Department of Energy                               |
| EANO    | Entergy Arkansas Nuclear One                            |
| EDTA    | ethylenediaminetetraacetic acid                         |
| EM      | Environmental Management                                |
| EML     | Environmental Measurements Laboratory                   |
| EMO     | Environmental Management Operations                     |
| EPA     | U.S. Environmental Protection Agency                    |
| ESH-HP  | Environment, Safety and Health—Health Physics           |
| ET      | Energy Technology                                       |
| FTIR    | Fourier transform infrared                              |
| FY      | fiscal year   |
| GC/FPD  | gas chromatograph/with flame photometric detector       |
| GC/MS   | gas chromatography/mass spectrometry                    |
| GPC     | gel permeation chromatography                           |
| GPS     | Global Positioning System                               |
| HARC    | Houston Advanced Research Center                        |
| HEPA    | high-efficiency particulate air [filters]               |
| ICP/AES | inductively coupled plasma atomic emission spectrometer |
| ICP/MS  | inductively coupled plasma mass spectrometer            |
| IPC     | internal pair conversion                                |
| IR      | infrared  |
| MAPEP   | Mixed Analyte Performance Evaluation Program            |
| MCFC    | molten carbonate fuel cell                              |
| MSRE    | Molten Salt Reactor Experiment                          |
| M2SI    | matrix spectral imaging sensor                          |
| NERL-Ci | National Exposure Research Laboratory—Cincinnati        |
| NERL-LV | National Exposure Research Laboratory—Las Vegas         |
| PC      | personal computer                                       |
| PCB     | polychlorinated biphenyl                                |
| PDP     | Performance Demonstration Program                       |
| PMMA    | polymethyl methacrylate                                 |
| QA      | quality assurance                                       |
| QC      | quality control   |



## NOMENCLATURE (contd)

|          |   |
|----------|---|
| RA       | Reactor Analysis  |
| RCRA     | Resource Conservation and Recovery Act                            |
| RE       | Reactor Engineering   |
| SEM/EDAX | scanning electron microscopy/energy dispersive X-ray fluorescence |
| SOP      | standard operating procedure                                      |
| TCLP     | Toxicity Characteristic Leaching Procedure                        |
| TD       | Technology Development  |
| TFA      | Tanks Focus Area  |
| TIMS     | thermal ionization mass spectrometer                              |
| TMI-2    | Three Mile Island-2   |
| TRU      | transuranic   |
| VOC      | volatile organic compound   |
| WEPCo    | Wisconsin Electric Power Company                                  |
| WIPP     | Waste Isolation Pilot Plant                                       |
| WP       | Water Pollution Study   |
| WS       | Water Supply  |
| XRD      | X-ray diffraction   |

# ANALYTICAL CHEMISTRY LABORATORY

## Progress Report for FY 1996

### 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) 1996 (October 1995 through September 1996). This annual report is the thirteenth for the ACL. It describes effort on continuing and new projects and contributions of the ACL staff to various programs at ANL.

The ACL operates in the ANL system as a full-cost-recovery service center, but has a mission that includes a complementary research and development component:

*The Analytical Chemistry Laboratory will provide high-quality, cost-effective chemical analysis and related technical support to solve research problems of our clients -- Argonne National Laboratory, the Department of Energy, and others -- and will conduct world-class research and development in analytical chemistry and its applications.*

Because of the diversity of research and development work at ANL, the ACL handles a wide range of analytical chemistry problems. Some routine or standard analyses are done, but the ACL usually works with commercial laboratories if our clients require high-volume, production-type analyses. It is common for ANL programs to generate unique problems that require significant development of methods and adaption of techniques to obtain useful analytical data. Thus, much of the support work done by the ACL is very similar to our applied analytical chemistry research.

The ACL is administratively within the Chemical Technology Division (CMT), our principal ANL client, but provides support for many of the divisions and programs at ANL. The ACL has four technical groups -- Chemical Analysis, Instrumental Analysis, Organic Analysis, and Environmental Analysis -- which together include about 40 technical staff members (see

Fig. 1 for the organization chart at the end of FY 1996). Talents and interests of the ACL staff cross the group lines, as do many projects.

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. The Instrumental Analysis Group uses nuclear counting techniques to determine the radiological constituents in a wide range of sample types, from environmental samples with low radioactivity to samples with high radioactivity that require containment. The Organic Analysis Group uses a number of complementary techniques to separate organic compounds and measure them at trace levels and has development work in sensors, chemometrics, and detectors. The Environmental Analysis Group analyzes environmental, hazardous waste, and coal samples for inorganic constituents and provides quality assurance support to DOE and other clients for various kinds of analytical laboratory data. Together, the groups provide a full range of analytical capabilities, including inorganic, organic, and radiological analyses.

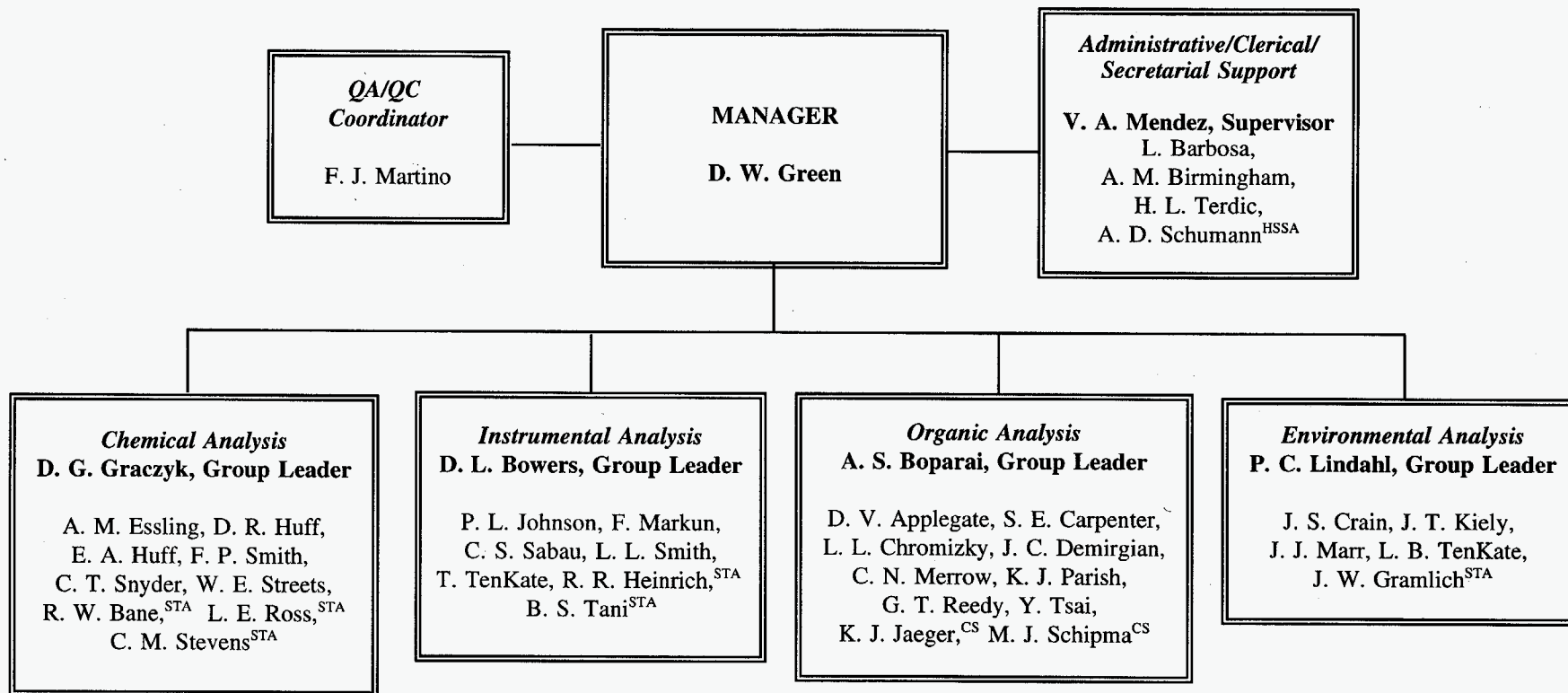
The ACL has a sample-receiving system to provide efficient processing of environmental, hazardous, and mixed-waste samples, including chain-of-custody procedures. The ACL also has a quality assurance (QA) program and quality control (QC) systems in place to produce data packages that meet the reporting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

More information is available through the ACL's Internet home page at <http://www.cmt.anl.gov/acl/acl.htm>. Details available through the home page include

- A list of all published ANL/ACL technical reports
- Major technical specialties of the ACL Staff
- Advice on how to submit samples
- The ACL Annual Report for FY 1995.

A replacement for our inductively coupled plasma atomic emission spectrometer was ordered in FY 1996 and should be operational in early FY 1997. This procurement was funded by the ANL General Purpose Equipment Fund, which continues to be the primary source of equipment funding for the ACL.

**Fig. 1. Analytical Chemistry Laboratory  
Organization Chart  
9/30/96**



HSSA = High School Student Aide; STA = Special Term Appointee; CS = Co-Op Student.

The ACL worked on two projects in FY 1996 with ANL Support Development Funds. In the first project, the ACL developed an analytical scheme for the determination of radium in environmental samples based on the Empore<sup>TM</sup> Radium Rad Disks. In the second project, the ACL installed and tested a DynaTrap purge-and-trap instrument for volatile organic analyses and determined its effectiveness for analyzing soil and water matrices. Progress on these projects is described in items 21 and 26 of this report.

The remainder of this report is a collection of abstracts of the analytical work performed by the ACL staff in FY 1996 on selected projects. The ACL receives about 1500 requests for laboratory analysis or technical support annually, some of which involve multiple samples. This report covers approximately 40 topics. Many 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 make an important contribution to the overall success of the project that receives the data. These contributions to projects can easily be overlooked, but collectively they form an important part of the ACL's mission.

## II. TECHNICAL HIGHLIGHTS

- (1) Support to Nuclear Technology Programs (C. S. Sabau, D. L. Bowers, J. S. Crain, A. M. Essling, D. G. Graczyk, D. R. Huff, E. A. Huff, P. L. Johnson, J. T. Kiely, F. P. Smith, and B. S. Tani)

The ACL analyzed samples from experiments conducted by the Waste Form Development, Electrorefining Development, and Pyrochemical Process Applications groups within the Nuclear Technology Department of CMT. Results from analysis of these samples supported efforts that included zeolite waste form development, treatment of spent oxide fuel and simulated Three Mile Island fuel debris, treatment of spent metallic fuels, and treatment of fuels from the Molten Salt Reactor Experiment (MSRE).

The samples required a variety of dissolution techniques prior to measurement of the requested analytes. Analytical tools applied to individual measurements included inductively coupled plasma atomic emission spectrometry (ICP/AES), inductively coupled plasma mass spectrometry (ICP/MS), X-ray diffraction (XRD), and thermal ionization mass spectrometry (TIMS). These instruments were used for obtaining elemental, isotopic, and compound information. Many of the samples that were submitted contained radioactive materials such as plutonium. These types of samples were analyzed with instruments that had been modified for this purpose (e.g., the "hot" ICP/AES). Dissolution schemes were developed for difficult-to-dissolve samples such as the fluoride salts used in the MSRE treatment process studies.

- (2) Continuous Emission Monitoring of Plasma Arc Furnace at ANL-West (J. C. Demirgian and E. Y. Hwang)

The continuous emission monitor (CEM) developed at ANL-East was used successfully to monitor the emissions from the first field test of the plasma arc furnace at ANL-West (K. Carney). The plasma system was developed with Z. Mao (Energy Systems Division) to treat radioactive waste by thermally destroying volatile materials and converting the radioactive waste to a nonleachable material. The initial test used nonradioactive material. The CEM was used to identify and quantify all volatile organic and selected inorganic species (CO, CO<sub>2</sub>, and HCl). The results, which were obtained on-line during combustion, were used to optimize combustion of the surrogate materials as the test proceeded. Further testing is planned through FY 1997.

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

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 stream from the automobile shredder industry. At present, shredder fluff is sent to landfills. Researchers in ANL's Energy Systems Division (E. Daniels et al.) have developed and are testing technologies to segregate and recover the fluff components for recycling. The ACL has contributed to this process development by analyzing various intermediates and products to determine their composition and to measure potential contaminants in recovered oils, foams, and residual materials classified as "fines."

In a FY 1996 pilot-scale study, the Argonne researchers tested an approach for cleaning polyurethane foam (e.g., from car seats or dashboards) so it can be recycled into value-added products such as carpet padding. For this study, the ACL analyzed foam samples, as well as distillates and still bottoms generated during recovery of the solvent used in cleaning the foam. The still bottoms from the solvent recovery system consisted of a mixture of contaminants removed from the foam and presented a particularly challenging matrix for analytical characterization. They ranged in texture from fine dry solids to thick, tar-like oils and contained particles of metal and glass, hydraulic fluid and motor oil, water, soil, solvent, and dirt of all kinds. In our analysis, we determined the water by Karl Fischer titration, ignited a portion of each sample to determine its ash content, measured chlorine and sulfur by oxygen bomb combustion and ion chromatography, measured mercury by cold vapor atomic absorption (CVAA) after a microwave-assisted acid digestion, and measured other metals by ICP/AES on a portion of ash dissolved in mineral acids. We developed special procedures to estimate the oil content of each sample by measuring the nonvolatile residue from a hexane extraction and measured polychlorinated biphenyls (PCBs) by using a modification of the EPA procedure for waste analysis. We measured residual cleaning solvent by gas chromatography/mass spectrometry (GC/MS). The data obtained from these analyses helped to establish the hazard classification of the still bottoms so they could be properly disposed of and, in some cases, helped to guide improvements to the solvent recovery process. If the foam recycling process is commercialized, the methods and procedures ACL developed for these pilot samples should also help the emerging industry meet its analysis needs.

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

The ACL continued to provide extensive analytical support to high-temperature superconductivity programs in ANL's Energy Technology and Chemical Technology Divisions. Much of this work involves analysis of starting materials, process samples, and products related to fabrication, as well as characterization studies on various ceramic compositions, including  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  ("1,2,3-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<sup>TM</sup> 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.,  $\text{Cu}^{3+}$ ), which is related to the oxygen stoichiometry of the compound.

The researchers in the Energy Technology Division (U. Balachandran et al.) have been developing methods for fabricating practical component parts for devices that might use the BSCCO materials for high-current applications. One approach involves preparing flexible silver tapes that contain a core of BSCCO ceramic. Measurements by ACL for this development effort have included the characterization of impurities in different lots of silver tubing used in the process, as well as determination of the BSCCO-to-silver ratio in some tapes. More recently, ACL analysts have devised approaches for separating the BSCCO ceramic core from individual tape specimens to perform analyses on the ceramic in the core. These efforts have permitted evaluation of composition changes that might have taken place when the ceramic was encapsulated in the silver sheath. Through these and related activities, the teamwork among chemists, ceramists, and analysts is helping to move the technology of superconducting ceramics toward practical applications.

- (5) Radon Remediation (F. Markun)

In collaboration with ANL's Reactor Analysis (RA) and Reactor Engineering (RE) Divisions, the ACL is developing and testing a low-cost radon mitigation apparatus to be used for reducing radon levels in homes.



Research personnel in RA had observed that vacuum pump oils concentrate noble gases. This observation led to the premise that oils may be useful for removing radon from air. The ACL was asked to test numerous oils for radon solubility. We determined that while most oils absorb radon to some degree, one of the better absorbers is corn oil. This finding led RA to apply for a patent on the application of this phenomena for the removal of radon from indoor air. The RE and RA groups designed and constructed the radon-removal apparatus, which consists of an absorber and a desorber. The apparatus is being tested by the ACL. Under ideal conditions, the absorber removed up to 98% of radon. The desorber, which removes radon from the oil, is more difficult to optimize than the absorber. Removing radon from oil is not difficult when time and a sufficient supply of radon-free air are available, but removing radon quickly with the smallest possible quantity of air poses a much more complicated problem. At present, the best result obtained while operating both absorber and desorber simultaneously is 61% radon removal. More work needs to be done to optimize the performance of the radon desorber.

(6) Development of "Smart" Chemical Sensors (S. E. Carpenter, Y. X. Noyes, and K. J. Jaeger)

Current ACL research efforts in chemometrics (the use of mathematics, statistics, and formal numerical methods for analysis of chemical data) are focused on the development of a software workbench for designing automated monitoring algorithms for spectroscopic, chromatographic, electrochemical, and radiochemical sensors of interest to DOE. The objective of this work is to produce "smart" chemical sensors. The term "smart" is used to imply that the sensor can (1) interpret multivariate data in real time; (2) perform data reduction and decision making; (3) communicate processed results to an end user, process control loop, or data fusion system; and (4) alter system operation on the basis of certain detection results. Smart sensors have obvious advantages for both invasive and noninvasive monitoring scenarios. We have designed unique signal processing algorithms that combine digital filtering, pattern recognition, and multivariate calibration methods to provide enhanced qualitative and quantitative data analysis. These algorithms are initially developed using software; however, final versions of the algorithms 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. Although the software workbench for designing these algorithms is still in the development phase (SAGE; see

item 15) novel algorithms have been successfully generated for several applications. One application is qualitative processing of data from a passive-remote Fourier transform infrared (FTIR) sensor used for remote monitoring of chemical emissions. Another is quantitative processing of data from an extractive FTIR system used for continuous monitoring of incinerator emissions. In FY 1997, we expect to develop processing algorithms for hyperspectral image data. Such data provide a complete spectrum for any point or object in an image. Smart sensor algorithms have also been designed for automated analysis of multispectral image data and recognition of naval ships.

- (7) Preparation of Simulated Solidified Waste Samples for the WIPP Performance Demonstration Program (K. J. Parish, A. S. Boparai, D. G. Graczyk, A. M. Essling, E. A. Huff, D. R. Huff, J. T. Kiely, M. J. Schipma, and L. B. TenKate)

The Waste Isolation Pilot Plant (WIPP) is a DOE installation consisting of large, interconnecting cavities hollowed out of deep geologic salt beds approximately 600 m underground. It is located approximately 26 mi (42 km) east of Carlsbad, NM. The facility is designed to demonstrate the safe handling, transportation, and disposal of transuranic (TRU) waste in the salt beds. The TRU waste destined for WIPP began to be generated during the 1940s from the nation's nuclear weapons programs at various DOE sites.

Wastes to be shipped to WIPP must first be characterized to identify the presence of hazardous materials in the waste, in addition to radioactive components. As part of the program to characterize TRU waste for WIPP, a Performance Demonstration Program (PDP) was established in 1995 for the analysis of Resource Conservation and Recovery Act (RCRA) constituents in Type I solidified waste. Type I waste is sludge from wastewater treatment that consists of inorganic salts that have been solidified by adding Microcell E adsorbent and cemented. As part of Cycle 1 of the RCRA PDP in FY 1995, the ACL prepared and characterized a simulated Type I solidified waste and developed methods for determination of metals and volatile and semivolatile organic compounds in this matrix.

In FY 1996, for Cycles 2 and 3 of the PDP, the ACL's task was to prepare and characterize unspiked samples of simulated Type I solidified waste (cemented and uncemented) for metals determinations. The samples were provided to the PDP coordinator for spiking and shipment to the laboratories participating in the PDP.

- (8) Orphan Waste Projects (D. G. Graczyk, A. M. Essling, D. R. Huff, E. A. Huff, F. P. Smith, C. T. Snyder, W. E. Streets, P. C. Lindahl, S. D. Kent, J. T. Kiely, J. J. Marr, D. L. Bowers, C. S. Sabau, L. L. Smith, T. TenKate, F. Markun, R. R. Heinrich, L. B. TenKate, J. S. Yaeger, B. S. Tani, P. L. Johnson, A. S. Boparai, D. V. Applegate, L. L. Chromizky, Y. Tsai, K. J. Parish, S. E. Carpenter, M. J. Schipma, P. D. Postlethwait, and J. S. Crain)

Many ANL facilities have "orphan" waste materials that have unknown origins or inadequate documentation or that remain from programs no longer funded. Funding made available through ANL's Environmental Management Operations (EMO) Division has provided many of these facilities with the opportunity to undertake projects directed at accumulating, characterizing, and documenting these orphan wastes so they can be disposed of properly. In support of these projects, the ACL is conducting chemical and radiological analyses to provide the composition and hazards-classification data needed by treatment/storage/disposal facilities that ultimately process the wastes. Working closely with EMO to ensure that information and procedural requirements of the appropriate treatment, storage, and disposal facility are satisfied, ACL staff have prepared detailed sampling and analysis plans for some projects, fielded sampling teams to collect appropriate representative samples, and collaborated with EMO staff in interpreting the analysis results for waste handling.

Wastes characterized by the ACL in FY 1996 included a substantial inventory of contaminated laboratory hoods, gloveboxes, equipment, and appliances (e.g., light fixtures). The management of the facility where the materials were stored determined that their future use was improbable. Chemical and radiological contamination on each item was assessed through smears and direct samples taken by EMO personnel, who performed size-reduction and packaging operations on the waste. Other orphan waste projects involved 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, 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 directly identify the material and then relied on a material safety data sheet to obtain pertinent information about hazards. 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 RCRA 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 projects, EMO has helped ANL divisions dispose of tens of thousands of kilograms of waste materials. The difficulty and expense of orphan waste disposal has brought home to many ANL divisions the need for keeping timely and complete records on stored materials and promptly disposing of materials that are no longer needed.

- (9) Characterization of Used HEPA Filters for Disposal (D. G. Graczyk, A. M. Essling, D. R. Huff, E. A. Huff, F. P. Smith, C. T. Snyder, W. E. Streets, P. C. Lindahl, S. D. Kent, J. T. Kiely, J. J. Marr, D. L. Bowers, C. S. Sabau, L. L. Smith, F. Markun, T. TenKate, L. B. TenKate, J. S. Yaeger, A. S. Boparai, D. V. Applegate, L. L. Chromizky, Y. Tsai, K. J. Parish, M. J. Schipma, and P. D. Postlethwait)

Used high-efficiency particulate air (HEPA) filters removed from ANL laboratory exhaust systems represent a sizable waste stream from site operations. A few years ago, ANL's Environmental Management Operations Division (EMO) established procedures for disposing of these wastes at DOE's Hanford Reservation. In the disposal process, EMO packages the used filters and takes composite samples, and the ACL characterizes the composite samples by chemical analysis to evaluate the waste against Hanford's waste acceptance criteria. In this characterization, each composite sample (consisting of pieces cut from 12 to 14 filters contained in a packed waste bin) is homogenized by manual cutting and then analyzed for metals, semivolatile organics, PCBs, and radionuclides. Samples shown to contain high concentrations of heavy metals are also processed by the Toxicity Characteristic Leaching Procedure to determine whether the metals they contain exceed regulatory standards for mobility under landfill conditions.

During FY 1996, the ACL characterized approximately 25 composite HEPA filter samples. Each composite consisted of portions of approximately 12 filters. Thus, all filters replaced at ANL-East in FY 1996 (approximately 300 filters) were tested by this process. By shipping these wastes to Hanford on a regular basis, EMO avoids accumulating large volumes of waste in its storage facilities. In addition to this current HEPA filter waste, the filter-characterization methodology was also applied this year to a historical accumulation of prefilter wastes. Prefilters are the relatively coarse filters that are installed in laboratory fume hoods to protect exhaust ducts from dirt and particulates. In a manner analogous to that used for the HEPA filters, EMO

repackaged and sampled a collection of more than 8000 prefilters that had been stored over the years because no disposal facility would accept them without hazards classification information, which was not available. These prefilters were sampled and compacted by EMO into six waste packages. Because a piece of every filter in each waste package was collected as part of the composite sample, the prefilter composites were exceptionally large, occupying 6 to 12 times the volume of a composite HEPA filter sample. The volume of material in each prefilter composite required special efforts to homogenize the material for representative subsampling. Characterization of the composite samples from these packages will help ANL make a substantial reduction in its historical waste inventory.

(10) Analysis of Process Liquors Used in Removing Zinc from Galvanized Steel (E. A. Huff, D. R. Huff, F. P. Smith, A. M. Essling, J. T. Kiely, S. D. Kent, and D. G. Graczyk)

Because the U.S. automobile industry is using more and more galvanized (i.e., zinc-coated) steel, an unprecedented volume of galvanized steel scrap is being generated. These low-carbon steels are ideal scrap feed for iron and steel furnaces, but only if the zinc coating is removed. Argonne's Energy Systems Division (F. Dudek et al.) is working with Metal Recovery Technologies, Inc., of East Chicago, IN, to develop a technology to remove the zinc coating from steel surfaces so that both the steel and zinc in galvanized scrap can be reused.

The ACL is contributing to this effort by performing chemical analyses on caustic process liquors and related materials. We have determined metal, chloride, and carbonate concentrations in strong sodium hydroxide solutions from the chemical de-zincing process, as well as process parameters described as "total" and "free" alkali. Occasionally, solids in the liquors were isolated and analyzed separately. In the course of this work, ACL analysts developed methods for determining the alkali parameters that are simpler than those recommended by the recycling industry. The ACL's measurements provided not only data for process characterization and control, but also information of environmental interest, including information about the behavior of toxic metal impurities such as Pb, Cd, and Sb and about the composition of waste streams associated with the zinc-removal technology.

In FY 1996, the Energy Systems team began studies to develop a zinc electrowinning process for recovering the zinc after it is removed from galvanized scrap by dissolution. The ACL is participating in these studies by analyzing process samples to track impurity elements such as

copper and by measuring physical and chemical properties of the metal product. The data from these measurements help the process engineers understand the effects of process variables and conditions on the electrowinning operations.

(11) Stand-Off Detection of Agent-Related Chemicals (J. C. Demirgian)

The U.S. Army's Edgewood Research and Development and Engineering Center has an ongoing program in stand-off detection of gaseous chemical plumes, such as dimethyl methyl phosphonate and methanol, using FTIR technology. 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 detect chemical plumes in the presence of interferences.

Effort in FY 1996 focused on testing the microFTIR from Designs & Prototypes. This instrument weighs less than a pound and can be transported easily. Initial testing consisted of releasing quantitative amounts of methanol and dimethyl methyl phosphonate at the low parts-per-million-meter (concentration times path length) level both in the laboratory and the field. In the laboratory, calibrated blackbody backgrounds were used. In the field, low-sky, grass, and brick wall backgrounds were tested. The results were compared with those obtained by using a conventional Midac spectrometer; a report giving details of these comparisons is being prepared. It was concluded that the microFTIR's performance was slightly less sensitive but comparable to the conventional spectrometer.

(12) Analysis of Environmental Samples for the U.S. Department of Agriculture (L. L. Chromizky)

Over the past eight years, the ACL has participated in a collaborative effort with ANL's Environmental Research Division (K. Spokas) that involves monitoring the removal of contaminants (carbon tetrachloride and chloroform) from groundwater under a former grain storage facility in Waverly, NE, that 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 remained fairly constant during the year, at approximately 20 ppb. The aquifer will continue to be sampled monthly in

FY 1997 because the concentration of carbon tetrachloride is still above the Federal health standard of 5 ppb. In FY 1996, the ACL analyzed more than 100 aqueous samples for this project. All samples were analyzed within the allowed holding time.

- (13) Transition Metal Speciation in Textile Mill Wastewater (J. S. Crain, A. M. Essling, D. G. Graczyk, D. R. Huff, E. A. Huff, J. T. Kiely, and P. C. Lindahl)

Under EPA Stream Quality Standards, discharges of certain metals present in industrial water effluents must be reduced. This reduction is especially significant to the U.S. textile industry because many of the important dyes used in textile processing are "premetallized"; that is, the dye molecules contain Cu, Cr, or Co as part of the chromophore. With support from the AMTEX™ partnership (an initiative based on a Cooperative Research and Development Agreement between DOE and the textile industry), ACL staff have developed a simple analytical procedure that will allow industrial facilities to determine the chemical lability of metals in their wastewater discharges. The procedure is based on well-established chromatographic techniques, whereby chemically labile cations (e.g., divalent copper) are separated from the sample matrix by using a sodium sulfonate ion-exchange resin, and nonpolar species (e.g., certain dyes) are separated from the cation exchange effluent by using a nonionic polymer resin. Anionic species (e.g., organic acid complexes) are not retained by the columns. When the two-column separation is combined with element-selective detection (e.g., ICP/AES), Co and Cu cations (labile species) are differentiated from their EDTA (ethylenediaminetetraacetic acid) complexes, and these species are further differentiated from Co and Cu dyes. Trivalent chromium, hexavalent chromium, and chromium dyes are also differentiated from one another.

Early work using metal-EDTA mixtures showed that the degree of Co(II) and Cu(II) retention on the cation column closely corresponded to that expected from the EDTA-to-metal ratios and the stability constant of the metal-EDTA complex. The procedure was tested more rigorously in FY 1996 by analyzing nearly 100 effluent samples taken from textile mills all over the southeastern United States. In general, the procedure performed as expected, with spike recoveries (for total metal) on the order of 95 to 100%, chemical recovery (for separated metal) on the order of 80 to 100%, method detection limits between 5 and 20 ng/mL, and excellent reproducibility. When proper precautions are taken to avoid false positives (that is, careful and

clean sample preparation), the procedure will facilitate realistic assessments of environmental impact due to metal discharges and will provide information needed to develop appropriate water treatment processes.

- (14) Characterization of Phosphate-Ceramic Stabilized Hazardous Wastes (L. B. TenKate, D. R. Huff, E. A. Huff, J. S. Crain, J. T. Kiely, T. TenKate, F. P. Smith, and D. G. Graczyk)

Argonne's Energy Technology Division (A. Wagh, D. Singh, et al.) is developing a stabilization process that converts solid waste materials into chemically bonded phosphate ceramics, which have physical properties that make them good candidates for use as structural products. The process is broadly applicable to a variety of wastes, including combustion-process ashes, low-level mixed radioactive and chemical waste, and hazardous sludges. The ceramics formed are not only inexpensive to fabricate but also dense, leach resistant, and stronger than concrete. During FY 1996, ACL staff assisted this development by performing tests to determine the leach-resistance of both simulated and actual wastes at several stages in the stabilization process through application of the Toxicity Characteristic Leaching Procedure (TCLP), as described in U.S. EPA Method 1311.

In applying the TCLP, waste samples were extracted with a specified acetic acid buffer solution to simulate leaching that might occur under landfill conditions. By leaching the wastes and analyzing the extracts by ICP/AES, ICP/MS, and CVAA, concentrations have been obtained for Pb, Cd, Cr, Ni, As, Ba, Se, Ag, Mo, Fe, Cs, Ru, Sr, La, Nd, Y, Hg, and Ce (a nonradioactive simulant for plutonium) and Re (a nonradioactive simulant for technetium). Concentrations of the radioactive isotopes  $^{137}\text{Cs}$ ,  $^{238}\text{U}$ ,  $^{235}\text{U}$ , and  $^{241}\text{Am}$  were determined in actual waste samples by gamma spectroscopy. Ion chromatography was used to determine the concentrations of phosphate, nitrate, sulfate, and chloride leached from the ceramic matrix. Interpretations of these data have allowed Energy Technology Division researchers to evaluate the effectiveness of their technology in imparting leach-resistance for the metals and radionuclides and, in some cases, to deduce the mechanism by which the stabilization takes place.



- (15) Automated Data Cataloging Procedure for Objects in AMPS Multispectral Imagery (Y. X. Noyes and S. E. Carpenter)

This work was conducted for DC Office of Nonproliferation and National Security to support its Airborne Multispectral Pod System (AMPS) program. The AMPS program collects a large number of images that contain many kinds of targets. Different research groups are interested in different types of targets, but searching through the large data set to find only those targets is time consuming. Therefore, a procedure is needed for automated retrieval of information on specified targets. The purpose of our project was to establish a database for cataloging the AMPS multispectral image data.

The ACL developed a procedure based on Microsoft Access to demonstrate the feasibility of establishing such a database. As the key element needed to uniquely identify data records, we chose Global Positioning System (GPS) information. Every target has a unique GPS value, and this GPS information is already collected by AMPS. The targets themselves are identified in the original image by the automated target recognition algorithm developed under another ACL project, the Sensor Algorithm Generation Environment (SAGE; see item 6). Data obtained were target location (the coordinates in the original image), target type, and the target image. A database was then established consisting of eight fields: GPS value, altitude at which the image was collected, original image file name, time at which the image was taken, target file name, target type, coordinates of the target in the total image, and target image (extracted from the total image). A C-language program was written to extract the GPS value, altitude, and time from the header file of the original image and to output the results into a file with a format that could be read into the database directly.

The establishment of this database makes it much easier for researchers to access the image data that contain targets of particular interest. In particular, the specific image of the target can be viewed without retrieving the whole image, which is usually at least 100 times as big as the target image itself. This feature is important because it allows researchers to pre-examine the targets quickly before they spend time retrieving the whole image for further research. We believe a similar type of database may be established for other sensors. Thus, a common framework may be created for performing multisensor data fusion.

(16) Support of Counternarcotics Efforts (J. C. Demirgian, B. S. Tani, and E. Y. Hwang)

The ACL continued to provide analytical support to a collaborative program between ANL's Technology Development Division (B. Micklich) and the Office of the National Drug Control Policy. In addition, another program was initiated with the Houston Advanced Research Center (HARC) to recommend methods for the destruction of large quantities of seized illicit substances.

The collaborative effort was divided into three support areas. Primary support was given to testing commercially available instrumentation and reporting the results to law enforcement agencies. Two reports have been issued and have provided important information on the applicability of a particular instrument. These tests now take place at HARC, and it is anticipated that they will continue at the rate of two a year. Instruments have also been field-tested on cargo containers at the Port of Miami. These tests demonstrate how viable each instrument is under harsh conditions.

The second support area has been in the study of cocaine contamination and transfer. A legal issue has arisen concerning correlating the level of contamination of a defendant with the likelihood that the contamination was obtained through handling cocaine. In FY 1995, ACL research determined that almost 80% of all paper money is contaminated with cocaine and that contamination was more prevalent in older and lower denomination bills. This year the study was expanded to determine under what conditions the contamination could be transferred to an innocent person (secondary contamination). It was found that it is virtually impossible to transfer the contamination unless the contaminated paper money is vigorously rubbed onto someone. Other tests showed that even though a person would become contaminated by touching cocaine, that contamination could only be transferred immediately after contact with the cocaine. The contaminated individual can stay contaminated for several days, but the cocaine could not be transferred to another individual or to objects.

The third support area was to understand *why* contamination does not occur when innocent people handle highly contaminated money. Scanning electron microscopy studies were performed on both contaminated and uncontaminated paper money. Both had identical surfaces, and the contamination was clearly not evident on the surface of the bill. Investigation of the subsurface showed that contaminated bills have cocaine adhering to the fibers trapped in cages under the

surface fibers. It appears that cocaine is not transferred because when someone handles contaminated currency, that individual touches only the surface of the bill, while the cocaine is below the surface and, hence, cannot contaminate the individual.

- (17) Analysis of Samples from Process to Recycle Aluminum Salt Cake (A. M. Essling, E. A. Huff, D. R. Huff, F. P. Smith, C. T. Snyder, L. B. TenKate, D. V. Applegate, S. D. Kent, P. L. Johnson, B. S. Tani, J. T. Kiely, and D. G. Graczyk)

When aluminum is recycled, it is melted in the presence of an alkali-metal-chloride flux, which serves to extract impurities from the molten scrap metal and to protect the metal from oxidation. The spent flux, after it solidifies, is referred to as salt cake. Composed of salt, aluminum metal, and oxide residue, the salt cake represents a substantial waste stream in the aluminum recycling industry, which is seeking ways to eliminate the environmental and economic disadvantages associated with disposing of salt cake waste. Researchers in ANL's Energy Systems Division (J. Hryn et al.) are working with the Aluminum Recycling Association to develop cost-effective processes for treating salt cake to recycle the aluminum and salt fractions and convert the oxide-residue fraction to high-value-added products.

The ACL assisted this research by analyzing samples from a pilot-scale test of a process that converts the oxide residues into fiber insulation products. Materials from various stages of the process were analyzed to determine elemental composition, leachable salt content, total halide content, free-metal content, and phase composition. The determinations of total halides and free metal presented challenging opportunities for the analysts involved in this project. Special procedures had to be devised to permit measurement of low concentrations of these analytes because they had a profound effect on the product obtained from the manufacturing process. By providing expertise in compositional analysis of refractory materials (dissolutions and analysis by atomic emission or ion chromatography), X-ray diffraction, and gas-generation methods (free metals by hydrogen evolution), the ACL helped the Energy Systems Division team resolve product-quality problems in the pilot-plant operations. This success helped advance program goals to enhance the viability of the aluminum recycling industry and the environmental and economic benefits associated with it.

(18) Sulfur Speciation in Methanol Contaminated with Diesel Fuel or Unleaded Gasoline  
(Y. Tsai and J. B Schilling)

The fuel transportation pipelines in the United States are used to transport various products, and some cross-contamination is expected. For example, if methanol is transported in the normal fuel pipelines, it may become contaminated with up to 1% diesel fuel or gasoline. This contamination presents a problem if the methanol is to be used in fuel cells, since sulfur compounds in the diesel fuel or gasoline poison catalysts in the fuel cells. In support of an ongoing project to develop sorbent traps to remove sulfur compounds from contaminated methanol (S. Lee, CMT), we first analyzed samples of No. 2 diesel fuel and unleaded gasoline to determine the organic sulfur species present. We then analyzed methanol samples that had been treated with various adsorbents to determine how much organic sulfur had been removed. These samples originally contained 1% diesel fuel or gasoline in methanol.

The sulfur speciation was done by using a GC/MS and a gas chromatograph with flame photometric detection (GC/FPD). The compounds were separated with a J&W Petro-100 column.

Because these samples are very complex mixtures, the sulfur species, in general, co-elute with hydrocarbons in the samples and are detected simultaneously. This problem is overcome by using GC/FPD, which responds only to sulfur species and allows the determination of retention times for the compounds of interest. Once the retention times are known, we can do the actual speciation by using GC/MS data.

The information obtained from the analyses provided a measure of the sulfur removal efficiency of the various adsorbents, which allowed the researcher to evaluate and optimize the treatment processes.

(19) Review of Analytical Data from Subcontractor Analytical Laboratories (J. J. Marr, L. B. TenKate, L. L. Smith, and P. C. Lindahl)

During FY 1996, the ACL assisted ANL's Environmental Management Operations (EMO) Division in validating data packages generated by subcontractor analytical laboratories. The data packages represented the soil and water samples collected for the Resource Conservation and Recovery Act Facility Investigation for the 317/319/East-Northeast Area at ANL-East. The ACL staff reviewed 20 data packages for the usability and quality of inorganic, organic, and radiological data. This work required reviewing data according to the guidelines of the U.S. EPA

Contract Laboratory Program (CLP) where applicable, providing detailed reports of problems encountered during analysis and data acquisition, and evaluating the usability of the data. For the inorganic target analyte analysis, the review included validation of sample preparation, ICP/AES data, and graphite furnace and cold vapor atomic absorption (GFAA and CVAA) spectrophotometry data. In the organic analysis area, sample preparation and instrument data were validated for the analysis of volatile and semivolatile organic compounds by GC/MS and analysis of PCBs by gas chromatography. Radiological analysis data for gross  $\alpha/\beta$ , tritium, and gamma spectroscopy measurements were also validated. The validation of radiological analysis data included a review of sample preparation and instrument analysis data. These efforts helped EMO assess the usability and the quality of the data gathered in this investigation.

(20) Battery Program (J. J. Marr)

In FY 1996, ACL staff continued to provide technical support to the CMT Battery Program (J. Smaga) in the area of post-test analysis of batteries. The objectives of the work are to obtain independent evaluation of failure modes and to identify critical, life-limiting problem areas of the batteries. The information obtained from the studies serves as input for more refined modeling studies, permits greater accuracy in assessing technical progress in battery research, and helps direct future battery development activities.

During FY 1996, ACL staff examined 23 nickel-metal hydride batteries and six lead-acid round batteries. The nickel-metal hydride batteries were obtained from Ovonic Battery Company. They were tested at the CMT Electrochemical Analysis and Diagnostics Laboratory under an electric vehicle testing regimen until they failed to meet performance standards. Results of post-test analysis of the metal hydride batteries indicated that the primary causes of failure were contamination of the nickel electrodes, electrolyte redistribution, excessive venting, and mechanical degradation of the separators. The lead-acid round batteries were developed by AT&T Bell laboratories and were used by nuclear power stations for backup power generation. After two years of service, five of the six batteries demonstrated a high-rated capacity loss. Results of the post-test studies indicated that degradation of the positive plate limited the capacity of these failed round lead-acid batteries. Severe positive grid corrosion, presence of lead-sulfate

crystals on the corrosion layer, poor adhesion of positive active material to the grid, and morphology change of the positive active material caused the degradation. Results of the studies have been documented and forwarded to the battery developers for review.

(21) Development of a Method for Radium Determination in Aqueous Samples Using Empore™ Radium Rad Disks (L. L. Smith and F. Markun)

Under a Cooperative Research and Development Agreement initiated in 1994, ANL and the 3M Company, in conjunction with IBC Advanced Technologies, Inc., have developed a new technology based on solid-phase extraction for radiochemical analysis. The work resulted in a new product, Empore™ Rad Disks. These are thin membranes that are loaded with element-selective particles embedded in stable, inert polytetrafluoroethylene fibrils. The Empore™ Rad Disks selectively separate target radioisotopes from aqueous samples, even in the presence of very large concentrations of other ions. When these membranes are used, sample preparation can be as easy as passing an aqueous sample through the disk, drying the disk, and placing it in an appropriate counting instrument. Empore™ Rad Disks obviate the time-consuming preconcentration, precipitation, filtration, purification, elution, and evaporation steps associated with conventional sample preparation methods. The ACL and ANL's Environmental Research Division (M. D. Erickson, J. S. Alvarado, and K. A. Orlandini) are collaborating to evaluate the performance of these disks and develop appropriate analytical procedures. Methods for  $^{99}\text{Tc}$  and  $^{89/90}\text{Sr}$  separation were developed in previous years.

In 1996, methods for  $^{226/228}\text{Ra}$  separation were developed and disseminated to the radiochemistry community. The radium-selective disks resulting from this work lend themselves to a variety of approaches for determining the pertinent radium isotopes. The major radium isotopes,  $^{226}\text{Ra}$  and  $^{228}\text{Ra}$ , may be determined simultaneously by isolating radium on the disk, placing the disk in a sealed container, and then gamma-counting the sample by means of the least-squares gamma spectrometric method. Alternatively, the radium itself or its daughter products may be eluted from the disk and further processed according to prescribed EPA methods.

The team of ANL, 3M, and IBC was awarded a 1996 R&D 100 Award by *Research and Development* magazine for the development of Empore™ Rad Disks.

- (22) Domestic Nuclear Smuggling Exercise (D. L. Bowers, J. S. Crain, A. M. Essling, D. G. Graczyk, D. W. Green, D. R. Huff, E. A. Huff, P. L. Johnson, J. T. Kiely, K. J. Parish, C. S. Sabau, F. P. Smith, L. L. Smith, T. TenKate, and B. S. Tani)

An exercise was carried out to demonstrate DOE's and other agencies' ability to respond to a nuclear smuggling scenario. "Intercepted" materials described in the scenario were sent to participating laboratories for forensic analysis. The ANL team was led by P. Persiani (Technology Development Division); the analyses were performed by the ACL and by L. Neimark, R. Strain, and others of ANL's Energy Technology (ET) Division.

The material consisted of radioactive metal, together with nonradioactive substances. Various tests were used to identify and quantify the material. They included extensive photographic and metallographic examination by ET personnel and analyses performed by the ACL using ICP/AES, ICP/MS, gamma spectrometry, alpha pulse, FTIR microscopy, XRD, and TIMS techniques. The results were then faxed to the organizer of the effort at Lawrence Livermore National Laboratory. The data were reported as the analysis proceeded, at intervals of 12, 24, 48, and 96 h. Interpretations of the data as to the origin and purpose of the material were also conveyed.

A post-exercise workshop was held at DOE Headquarters for the participants to share results, develop a consensus on measurements, and discuss source attribution issues. The workshop also addressed ways to improve the process and suggestions for future studies.

- (23) Separation of  $^{206}\text{Bi}$  from  $^{206}\text{Pb}$  for Experiment to Measure the Production Branch of an Internal Pair Conversion (D. L. Bowers and D. G. Graczyk)

Researchers in ANL's Physics Division (A. Wuosmaa and I. Ahmad) wanted to prepare a source containing the short-lived (6.2 days) nuclide  $^{206}\text{Bi}$  for use as a target in their study of internal pair conversion (IPC), the process by which a high-energy photon produced in the decay of an excited nuclear state converts to a positron-electron pair. They arranged for production of approximately 100  $\mu\text{Ci}$  of  $^{206}\text{Bi}$  by the  $^{206}\text{Pb}$  (p,n) reaction in an irradiation performed at the University of Notre Dame and asked the ACL to isolate the  $^{206}\text{Bi}$  product from the metallic  $^{206}\text{Pb}$  source material. Prior to the scheduled irradiation, the ACL devised and tested a separation scheme that used column chromatography with TRU-Spec Resin (Eichrom Industries, Darien, IL) to accomplish the separation.

When the irradiated  $^{206}\text{Pb}$  metal arrived, we dissolved it in nitric acid, adjusted the nitric acid concentration to 2 N, and put the solution through a prepacked column containing about 2 mL of resin. In this acid, lead shows no affinity for the resin, while bismuth is completely retained. After washing the lead through the column with 2 N  $\text{HNO}_3$ , we eluted the  $^{206}\text{Bi}$  with 6 N  $\text{HCl}$  and concentrated the eluate to a few microliters. This solution contained approximately 50  $\mu\text{Ci}$  of lead-free, carrier-free  $^{206}\text{Bi}$ . Both the  $^{206}\text{Bi}$  product and the  $^{206}\text{Pb}$  source material were thus recovered. The team of physicists prepared a 15- $\mu\text{Ci}$  source from the  $^{206}\text{Bi}$  and used the APEX positron-electron spectrometer in the ATLAS facility at ANL to obtain positron-electron pair energy spectra for the IPC transitions of the excited-state  $^{206}\text{Pb}$  nuclei produced during electron capture decay of the  $^{206}\text{Bi}$ . These spectra are currently being analyzed and are expected to provide better information than previously available on the IPC process and thereby allow an evaluation of existing theoretical models describing IPC transitions.

(24) Analysis of Polymethyl Methacrylate by Gel Permeation Chromatography (K. J. Parish)

Polymethyl methacrylate (PMMA) is a stiff, transparent plastic known as Lucite or Plexiglas. The PMMA polymer is being used for work at the Advanced Photon Source to micromachine parts using deep X-ray lithography. A plate of PMMA functions as a "photo resist." The photo resist breaks down in a predictable manner when exposed to the X-ray beamline.

The micromachining process involves placing a three-dimensional mask of a desired microstructure between the beamline and the photo resist. The exposed sections of the photo resist (sections not covered by the mask) will result in scissions of the polymer chains. After exposure, the photo resist is "developed" by preferential dissolution of the exposed material, resulting in a three-dimensional negative or reverse pattern of the mask. Finally, the microstructure is formed by placing the developed photo resist in an electroplating bath, where metal fills the trenches or pattern in the photo resist, thereby forming the desired metallic microstructure.

The success of the micromachining process depends on the control and monitoring of the breakdown of PMMA exposure to deep X-rays. The ACL determined the molecular weight distribution of unexposed and exposed PMMA by using gel permeation chromatography (GPC).



This information is used to monitor or troubleshoot the micromachining process. The GPC system is calibrated by using PMMA and polystyrene standards that have well-characterized molecular weight distributions.

(25) Field Sampling Assistance to the Environmental Management Operations Division  
(D. V. Applegate)

In work with ANL's Environmental Management Operations (EMO) Division, the ACL is assisting with field sampling at the several hazardous sites within the ANL-East boundaries. Some sites and matrices involved are landfills, leachate seeps, groundwater wells, sediments, and contaminated soils. The ACL provides support for sampling of soil borings, which EMO takes by using a rented truck-mounted hydraulic sampler called a "Geoprobe." Other sampling techniques involve using pumps and bailers to take water samples from groundwater wells and from surface water. With good knowledge of the sample requirements for proper laboratory analysis, the ACL is able to guide sampling efforts and, ultimately, insure high-quality samples. Help has also been provided through the use of a portable field screening instrument that detects volatile organic compounds. This instrument provides valuable and immediate sample information to direct sampling efforts and to alert the sampling team to possible safety concerns.

(26) Installation and Testing of a DynaTrap Purge-and-Trap Instrument for Volatile Organic Analysis (L. L. Chromizky and D. V. Applegate)

The goal of this Support Development Funds project was to install and test a DynaTrap® purge-and-trap system for analyzing water, soil, and other matrices for volatile organic compounds. The DynaTrap system (Baton Rouge, LA) consists of a 30-position autosampler carousel into which samples are placed in their as-received state. The minimal interaction between analyst and sample is especially an advantage in analyzing mixed-waste samples. The DynaTrap instrument was interfaced with the GC/MS system used for analysis of routine, mixed-waste, and orphan waste samples. We have completed testing to assess the effectiveness of the instrument for analyzing traditional soil and water matrices, as well as matrices requiring method development. Testing indicates that the instrument operates as expected; further testing is required to evaluate accuracy and precision.

- (27) Development of a No-Moving-Parts Fourier Transform Infrared Sensor (S. E. Carpenter, K. J. Parish, and G. T. Reedy)

Conventional FTIR sensors of the passive remote type are available from several commercial manufacturers. The term "passive" is used to indicate that these sensors, unlike laboratory FTIR spectrometers, do not contain an internal infrared (IR) radiation source. Instead, the sensors are equipped with a special set of input optics which collect ambient IR energy. The term "remote" is used to indicate that gas-phase samples to be measured are located some distance away from the instrument. Passive remote IR sensing is based on the fact that most organic vapors absorb or emit IR radiation at characteristic frequencies and, therefore, have unique IR spectral signatures. The amount of energy absorbed or emitted at these characteristic frequencies is a function of the temperature of the analyte relative to the thermal background.

The fragile component of existing FTIR sensors is the interferometer, which consists of a beam splitter, a stationary mirror, and a moving mirror. To obtain good data, a critical requirement is precise alignment of the two mirrors with respect to the beam splitter. Another requirement is that the moving mirror must maintain constant velocity during the scan. Anything that causes a departure from these criteria can introduce phase errors and other artifacts, which will degrade the data. Therefore, special efforts must be made to ensure that the sensors are not jarred or bounced during data collection.

The purpose of this ongoing project is to investigate the possibility of creating a novel FTIR sensor with *no moving parts* by creating an optical breadboard prototype. Unlike conventional FTIR sensors, which employ a moving mirror to temporally disperse interferometric information, the goal of this work is to produce an FTIR sensor design that spatially disperses interferometric patterns onto a two-dimensional focal-plane array detector. Since the new FTIR sensor design has no moving parts, the sensor is extremely rugged and immune to vibrations. To date, proof-of-principle has been demonstrated at visible wavelengths and infrared wavelengths of 3 to 5  $\mu\text{m}$ , and data have been collected for carbon dioxide and hexane. Future work will involve demonstrating the prototype at longer IR wavelengths.

(28) Mueller Matrix Spectral Imaging Sensor (C. N. Merrow and K. J. Parish)

The objectives of this proposed effort are (1) to design, develop, fabricate, and test a Mueller matrix spectral imaging sensor, which can be used by inspectors and laboratory analysts to rapidly scan, locate, and detect substances related to nuclear weapons production on surfaces; (2) to determine the potential for this sensor to remotely detect chemical tags applied to surfaces of nuclear materials for tracking purposes; and (3) to assess the potential "dual use" of this sensor for the detection of contraband substances or chemical and biological warfare agents on surfaces. This is a new ACL project, and the work to date has involved procuring the necessary equipment, setting up a laboratory, and obtaining initial data.

(29) Preparation and Distribution of Simulated Headspace Gas Performance Demonstration Samples for the Waste Isolation Pilot Plant Project (D. V. Applegate, A. S. Boparai, and C. T. Snyder)

The ACL is involved with the preparation and distribution of performance demonstration samples of simulated headspace gas samples for the WIPP. Every six months the ACL acquires standard gas mixtures from a specialty gas vendor; these mixtures contain known concentrations of various volatile organic compound (VOCs), hydrogen, and methane. These gases are metered through a heated multi-port manifold (constructed by ACL) into several SUMMA canisters at the same time, providing homogenous samples in each canister. The canisters are labeled, packaged, and shipped to laboratories that the WIPP Carlsbad Area Office wants to qualify for headspace gas analysis. The ACL then provides confirmation analysis on an aliquot of each of the gas mixtures for VOCs, methane, and hydrogen. This analysis provides quality assurance that the gas vendor's certified concentration values are correct and that the preparation manifold did not alter the standards in any way. Finally the ACL must inventory and clean any and all canisters used for the headspace gas performance demonstration program.

(30) Determination of Hydrogen Contamination in Deuterium for the Alternating Gradient Synchrotron at Brookhaven National Laboratory (C. T. Snyder and D. G. Graczyk)

Recent experiments performed at the Alternating Gradient Synchrotron (AGS) at Brookhaven National Laboratory (BNL) required prior knowledge of contaminants in deuterium. Liquefied deuterium was to be used as a target from which pions would be scattered. Hydrogen contamination was of particular interest because of possible side reactions.

Previous analysis of gas samples drawn from the headspace above the liquid deuterium had indicated approximately 2% hydrogen contamination. The reliability of the results was questioned since expected levels were less than 0.5%. The ACL was selected to analyze additional samples by gas mass spectrometry. Three gas samples were received from BNL, which included two deuterium samples from BNL sources and deuterium from a source outside BNL. Results from our analysis indicated that hydrogen concentrations were below the method detection limit of 0.015% in all three samples. This collaborative effort between ACL and BNL was successful and benefited AGS operations.

(31) Upgrade of Data Collection System for Thermal Ionization Mass Spectrometer (F. P. Smith and D. G. Graczyk)

The data collection system on ACL's TIMS has been upgraded to replace the nearly obsolete computer with a present-generation personal computer (PC) system. Availability of parts and service for the original computer were no longer guaranteed by the manufacturer. The VG Isomass TIMS system, Model 54R, was acquired in 1980. At that time, it represented the state-of-the-art TIMS instrumentation, with a 16-position-turret sample changer and fully automated operation controlled by a Hewlett-Packard 9845B Desktop Calculator. The hardware and most electronics on the system have remained extremely reliable and perform at a level consistent with currently available instruments.

The recent upgrade was procured with 1995 General Purpose Equipment funds and installed by FISIONS Instruments (Beverly, MA) in January 1996. The upgrade involved not only replacing the obsolete calculator but also enhancing the data acquisition electronics and software to take advantage of the better control and data storage capabilities of the new computer. The FISIONS software approach included conversion of all our existing operating programs to the new system, so any software features we had added in-house over the years were retained. The PC-based system is faster and more convenient to operate and has increased the data storage capacity by several orders of magnitude. We have used the upgraded instrument for isotopic analyses for uranium and plutonium for nuclear technology programs, strontium isotopes for geoscience studies, and lithium isotopes for fusion reactor studies.

- (32) Examination of Residues from Heat Transfer Experiments with a Liquid Metal Alloy (A. M. Essling, E. A. Huff, D. R. Huff, P. L. Johnson, B. S. Tani, and D. G. Graczyk)

Qmax, a small business that manufactures high-performance thermal management systems, encountered a problem while performing heat-transfer experiments with a liquid-metal-based cooling system that uses the alloy 61Ga-25In-13Sn-1Zn as the coolant. During their tests, a pasty residue appeared in the liquid alloy and adversely affected the performance of the system. Qmax came to Argonne for help in determining the nature of the residue and provided the ACL with two samples for examination and analysis. One sample contained a substantial quantity of free-flowing liquid metal with a relatively thin layer of sludgy residue; the other consisted almost completely of a thick, pasty material (the residue) with a darker appearance than the shiny liquid.

To characterize the residue, the ACL analyzed portions of the free liquid and the sludgy residue by (1) determining their elemental composition, (2) obtaining X-ray diffraction patterns, and (3) using scanning electron microscopy to look for particulate phases. Results of these examinations indicated that the residue contained substantial concentrations of elements (Fe, Cr, Ni, C, and Mo) that are common components of stainless steel and that are likely to affect the liquidus properties of the liquid metal alloy. These results suggested that the residues arose from interaction of the metal alloy with the stainless steel components of the Qmax coolant-handling system. Such interaction was not anticipated by Qmax when they designed their system. Results of the ACL effort in this study provided insight into the cause of the residues and also suggested that one way to avoid them would be to substitute more appropriate materials for the stainless steel components. Without these results, Qmax might have pursued remedies that would not have eliminated their problem.

- (33) JANUS Reactor Characterization Project (A. M. Essling, D. R. Huff, E. A. Huff, S. D. Kent, F. P. Smith, D. G. Graczyk, L. L. Smith, T. TenKate, J. S. Yaeger, D. L. Bowers, D. V. Applegate, L. L. Chromizky, K. J. Parish, P. D. Postlethwait, Y. Tsai, A. S. Boparai, D. A. Bass, J. T. Kiely, J. J. Marr, W. E. Streets, L. B. TenKate, F. J. Martino, and P. C. Lindahl)

The JANUS Biological Irradiation Facility, which operated at ANL-East from 1963 until 1992, is scheduled for decommissioning. The Technology Development (TD) Division of ANL is responsible for the JANUS Decommissioning Project. JANUS was a light water reactor that used highly enriched fuel to generate high neutron fluxes. The reactor, named for the mythical

two-faced Roman god of gates and doorways, had two faces that supplied neutrons of the same energy spectrum, but with different intensity. The high-dose side was used to provide acute exposures to specimens, while the low-dose side was used to provide chronic exposures. As part of the decommissioning process, the hazardous material and radiological characteristics of the facility had to be determined. The hazardous materials characterization provides data on the presence of inorganic and organic hazardous substances and their concentrations, as well as the potential regulatory status of materials to be disposed. The radiological characterization provides data to be used in assessing the impact of radioactivity on the facility and the surrounding area.

The ACL teamed with the Health Physics staff of ANL's Environment, Safety and Health Division (ESH-HP) (M. J. Robinet, C. M. Sholeen, M. O'Connor, D. C. Geraghty, et al.) in conducting the sampling and analysis needed for the facility characterization. The ESH-HP staff coordinated and conducted the sample collection. They also conducted on-site radioactivity surveys. The ACL was responsible for laboratory analysis of samples for inorganic, organic, and radiological constituents and selected parameters. The hazardous materials characterization included the analysis of soils, sludges, and waters for RCRA parameters, and target analytes and compounds such as pH, metals, volatile and semivolatile organic compounds, PCBs, and total cyanide and sulfide, as well as total organic carbon and phenols. The radiological characterization included the analysis of filters, smears, graphite powders and cores, concrete powders and cores, lead shavings and cores, hard board cores, and steel and other metal cores for thorium, uranium, plutonium, and gamma-emitting radionuclides. Instrumental analysis techniques such as ICP/AES, GFAA and CVAA spectrophotometry, and ultraviolet-visible spectrophotometry were used in the determinations of inorganic constituents. Gas chromatography and GC/MS were used to measure the organic compounds. Isotope-dilution alpha spectrometric and gamma spectrometric methods were used to determine the radionuclides. The ACL analyzed more than 150 samples in support of the characterization effort. These characterization data are included in the report prepared by ESH for TD.

- (34) Identification of Scale and Filter Residues in Cooling-Water Systems at the Advanced Photon Source (P. L. Johnson, B. S. Tani, S. D. Kent, K. J. Parish, P. C. Lindahl, and D. G. Graczyk)

The ACL has been providing occasional support to operations at the Advanced Photon Source (APS) by analyzing water from cooling systems to monitor the water quality or to determine the effectiveness of cleaning procedures applied to the systems. Recently, we were asked by members of the Accelerator Systems Division to help identify troublesome materials that appeared in individual water-handling systems. In one case, the material was a black deposit that had formed on a stainless steel flow-regulator component and interfered with its operation. We disassembled the component and found, in addition to the deposit on the outer surface, a heavy scale deposited on the inside of the component. This scale was only loosely adherent and readily flaked off the stainless steel surface. Using XRD and scanning electron microscopy/energy dispersive X-ray fluorescence (SEM/EDAX), we determined that both the internal and external deposits were made up of cuprite ( $\text{Cu}_2\text{O}$ ).

In a second case, the material of concern was a residue that had collected on a filter. The residue consisted of a brownish powder intermixed with what appeared to be a mat of small fibers. We examined the residue using XRD, SEM/EDAX, and FTIR microscopy. The brown powder was identified from its XRD pattern as a mixture of cuprite and copper metal. The fibers were identified as polypropylene by comparing their infrared spectrum to a computer-based reference library. Knowing the identities of materials contaminating these cooling-water systems will help the Accelerator Systems Division scientists determine where the contaminants come from and help them avoid problems in future operations.

- (35) Measurements to Track Electrolyte Segregation in Molten Carbonate Fuel Cells (E. A. Huff, A. M. Essling, and D. G. Graczyk)

Researchers in CMT (T. Kaun, S. Nied, S. Zelle, I. Bloom) are pursuing development of advanced electrolyte compositions for molten carbonate fuel cells (MCFCs). In a typical MCFC, a liquid electrolyte of lithium and sodium carbonate is retained by a porous ceramic matrix of lithium aluminate,  $\text{LiAlO}_2$ , which separates the cell anode and cathode. During operation of the cell, segregation of the electrolyte constituents and redistribution of the electrolyte in the  $\text{LiAlO}_2$  support can occur; these changes affect cell performance. To study the extent to which such

segregation takes place, the CMT researchers operated test cells fabricated with several different alkali carbonate compositions and known mass ratios of carbonate to  $\text{LiAlO}_2$ . After operating the cells, they cut sections of the  $\text{LiAlO}_2$  separator from specific locations and submitted the sections for chemical analysis to determine their carbonate composition and loading.

Analysts in the ACL used component elements in each compound of interest as markers for the carbonate and aluminate constituents. Thus, we measured aluminum as an indicator for  $\text{LiAlO}_2$ , lithium in molar excess of aluminum as an indicator for  $\text{Li}_2\text{CO}_3$ , and sodium as an indicator for  $\text{Na}_2\text{CO}_3$ . By completely dissolving each cell section and measuring the marker elements by ICP/AES, we obtained data that allowed us to calculate both the carbonate composition and the mass loading of carbonate relative to lithium aluminate in each section. Many samples showed substantial changes from the nominal composition of the cell; these results indicate that segregation had occurred and permitted an assessment of its extent. This information will help the CMT team to develop stable (i.e., nonsegregating) electrolyte compositions for future fuel cell designs and to evaluate performance of compositions they propose.

- (36) Support for Commercial Electric Utilities' Study of Dry-Storage Casks for Spent Nuclear Fuel (A. M. Essling, P. L. Johnson, D. L. Bowers, A. S. Boparai, K. J. Parish, Y. Tsai, B. S. Tani, E. A. Huff, D. R. Huff, L. B. TenKate, J. S. Crain, and D. G. Graczyk)

Because available space in spent fuel storage pools is nearly exhausted at some nuclear power plants, commercial electric utilities are implementing dry-storage options to increase their capacity for storing spent fuel. In the dry-storage operations, spent fuel assemblies are loaded into a special cask in the storage pool, water is removed from the cask, and the cask is sealed for removal to above-ground storage. During one such operation at a power plant operated by the Wisconsin Electric Power Company (WEPCo), gas inside the cask ignited while the lid was being welded in place. This ignition was attributed to a buildup of hydrogen caused by chemical interaction between a zinc-based coating used on internal surfaces of the cask and the somewhat acidic storage pool water.

In following up on this incident, WEPCo enlisted ACL support to chemically characterize samples of slightly radioactive solid residues collected from the cask lid and other solids filtered from the water removed from the cask. This characterization included XRD analysis to identify crystalline phases, comprehensive elemental analysis by ICP/AES and other methods, and GC/MS



analysis for possible organic constituents. We also analyzed samples of the storage pool water to determine pH, total organic carbon, and metal concentrations. At about the same time, another utility, Entergy Arkansas Nuclear One (EANO), initiated a laboratory study of the interaction between the zinc coating and the water in their spent fuel pool. The ACL provided chemical analysis data to characterize and identify solid residues formed during the EANO tests. The ACL's data helped these utilities better understand the chemistry of the interaction between the storage pool water and the zinc coating inside their dry-storage casks. This understanding, in turn, allowed them to better address engineering and safety issues related to dry-storage operations.

- (37) Chemical Analysis for Studies to Develop Treatment for Spent Oxide Fuel (A. M. Essling, F. P. Smith, C. T. Snyder, E. A. Huff, D. L. Bowers, C. S. Sabau, P. L. Johnson, B. S. Tani, and D. G. Graczyk)

Researchers in CMT (G. K. Johnson et al.) are investigating an electrometallurgical lithium-reduction process to convert some types of spent nuclear fuel from their oxide form to a metal that can be processed by electrorefining technology. The ACL has been providing a variety of chemical analyses to this program as they perform laboratory-scale experiments to better understand the electrometallurgical processes and prepare to demonstrate the process for treating fuel debris from the accident involving the Three Mile Island-2 (TMI-2) reactor.

In the process under consideration, the spent oxide fuel is reduced by reaction with lithium in the presence of a molten salt electrolyte, LiCl. The  $\text{LiO}_2$  formed during this reaction is soluble in the salt. For the process studies, ACL has performed several determinations. Hydroxide derived from  $\text{Li}_2\text{O}$  and lithium metal in the electrolyte was determined by acid titration. Small amounts of carbonate in  $\text{Li}_2\text{O}$  reagent were determined using manometric measurement of  $\text{CO}_2$  gas evolved during reaction of the carbonate with phosphoric acid in a special apparatus. The concentration of lithium metal present in LiCl electrolyte samples was determined by reacting the samples with aqueous acid and measuring the quantity of hydrogen gas evolved during the reaction. Results of these analyses provided information pertinent to starting and operating conditions, as well as information fundamental to engineering design of an optimized process. Some of the measurements, particularly the hydrogen evolution analysis for lithium metal, involved interesting analytical challenges because of the very low concentrations of metal in the samples.

In the technology demonstration for TMI-2 fuel debris, the ACL has performed XRD analysis and determined the chemical composition of the synthetic corium being used to simulate irradiated debris for testing. One aspect of the synthetic corium that complicates its analysis is the presence of a highly refractory chromite phase, which resists dissolution under normal analysis conditions. The ACL has overcome this difficulty by applying a high-temperature sealed-tube technique (Carius tube) to dissolve residues that remained after conventional dissolution methods were applied. As FY 1996 closed, ACL was beginning compositional analysis to characterize the products from early experiments involving reduction of the simulated oxide debris.

- (38) Participation in a Program to Establish the Center for Environmental Hazard Mitigation at Cairo University (F. P. Smith, L. L. Chromizky, K. J. Parish, Y. Tsai, E. A. Huff, J. S. Crain, J. T. Kiely, A. S. Boparai, and D. G. Graczyk)

Scientists from Argonne's Environmental Research Division (N. Sturchio and M. Sultan) are engaged in a collaborative effort with Egyptian scientists to establish the Center for Environmental Hazard Mitigation at Cairo University. This program, funded by the U.S. Department of Agriculture and the Egyptian Ministry of International Cooperation, involves assembling a state-of-the-art laboratory facility at Cairo University and training Egyptian scientists to use the equipment, acquire data, and interpret results related to soil, water, and air quality measurements.

The ACL has participated in this international collaboration by providing hands-on training workshops to visiting Egyptian scientists and by performing chemical analysis of environmental samples collected in Cairo and other Egyptian cities and villages. The workshops covered determination of anions in water by ion chromatography and determination of volatile organic pollutants by purge-and-trap GC/MS. They also provided overviews of high-performance liquid chromatography, liquid chromatography/mass spectrometry, and gel-permeation chromatographic preparation of sample extracts for analysis by gas-chromatographic methods. The analyses performed by the ACL included determination of trace metals and anions in water samples collected as part of a collaborative effort by the Argonne and Egyptian scientists to characterize the quality of water resources in urban and rural areas. The ACL also performed measurements for an investigation of the sources of suspended particulates in Cairo's air. Lead-isotope distributions in particulates collected on filters in Cairo were measured by ICP/MS; the results may help identify possible sources of lead contaminants. Total and organic carbon in the

filter deposits were also measured to permit differentiation of carbonaceous particles arising from natural and anthropogenic sources.

The support provided by the ACL to this program is expected to contribute to an understanding of Egyptian water and air quality and to advance Cairo University's capability to monitor and protect Egypt's environmental resources.

(39) X-ray Diffraction Analysis (P. L. Johnson and B. S. Tani)

The ACL continued to provide X-ray diffraction support to several programs in ANL's Chemical Technology, Chemistry, Energy Technology, Energy Systems, and Technology Development Divisions and the Advanced Photon Source. In addition, several samples from reactor operations in Arkansas and Wisconsin (see item 36) were analyzed.

In FY 1996, the Rigaku D/max-2400V diffractometer (12 kV rotating anode) purchased in FY 1995 was fully integrated into the ACL's operations. A sample changer was installed, which has increased our efficiency. We changed from the DOS software JADE to the Windows 3.1-based software JADE+ for Windows. The Windows software offers more flexibility in creating reports for clients, and it has some capabilities for presenting data graphically that lead to better visualization of results.

We have worked with both radioactive and nonradioactive samples, although most were nonradioactive. The ACL has purchased the current powder diffraction reference files from the International Centre for Diffraction Data, so our search and match software has a complete library.

(40) Cesium Separations in Tank Wastes (P. L. Johnson and B. S. Tani)

In FY 1996, the Tanks Focus Area (TFA) of the DOE Office of Environmental Management (EM-50) funded a project within CMT (J. Cunnane and T. Krause) to develop a process for separating and recovering cesium from crystalline silicotitanates (CSTs). X-ray powder diffraction studies provided data essential to defining the optimal processing conditions and explaining the observed separation efficiencies.

The CSTs are inorganic ion exchange materials that are being evaluated by the TFA for separating cesium from the soluble fraction of the tank wastes currently stored at the Hanford and

Oak Ridge sites. The primary concern regarding the use of CSTs in pretreating tank waste is that the high titanium content of the CSTs might require larger volumes of high-level waste borosilicate glass because of the current 1-wt%  $\text{TiO}_2$  loading limit in the glass. The objective of this CMT project was to develop a process that would optimize the separation and recovery of cesium from the CSTs, thus allowing cesium to be disposed of as high-level waste and the CSTs as low-level waste.

In the presence of nonreactive salts, thermal degradation of the CST framework is known to cause a phase change resulting in loss of crystallinity. In the presence of reactive salts, hydrolysis of the CST framework causes the phase change. The solid products resulting from the phase changes by both mechanisms were studied. Based on our X-ray powder diffraction studies of these solids, the CMT researchers were able to discern that the cesium recovery from the CSTs was maximized when CSTs had undergone the phase change. These results are consistent with previous research at Texas A&M University on the high selectivity of CSTs for cesium. From these results, processing conditions were identified under which cesium separation and recovery from CSTs are technically possible. Ongoing work in this area is dependent on TFA priorities concerning the need to develop a practical process for cesium separation from cesium-loaded CSTs.

### III. QUALITY ASSURANCE

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

Quality assessments continue to play an important role in uncovering deficiencies in the ACL quality system and identifying those areas in need of improvement. In 1996, an ACL assessment team consisting of the ACL Manager, Quality Assurance/Quality Control (QA/QC) Coordinator, and Group Leaders reviewed a dozen randomly selected ACL standard operating procedures (SOPs) and ACL document tracking practices. The assessment was conducted by means of document review, personal interviews, and laboratory tours. No deficiencies of any consequence were identified. This effort benefited from concurrent work to develop an ACL home page on the World Wide Web. One consequence of the assessment was the development of a "paperless" (electronic) system for distributing SOPs and other QA documents normally tracked as controlled documents. (A controlled document requires the use of signed document receipt forms acknowledging receipt of personal copies of the document.) The electronic system now provides access, through the ACL home page, to approximately 220 ACL SOPs and other important ACL QA documents (e.g., *ACL QA Plan*, *ACL QC Plan*, and *ACL Good Automated Laboratory Practices*). These documents are accessible to each member of ACL through use of the individual's computer password, a practice that effectively limits document access to ACL personnel and thereby ensures document security.

In other QA/QC efforts, the ACL continues to pursue relevant certifications. In 1994, the DOE Chicago Field Office asked the ACL to pursue State of Utah certification so that the ACL could provide ANL the capability for analyzing low-level mixed waste, which could then be shipped for disposal at the Envirocare of Utah, Inc., burial site. After an audit, certification was awarded in August 1994. The ACL has since taken the initiative to expand its certification to include additional methods for RCRA organics, inorganics, and radionuclides in support of ANL's mixed-waste disposal efforts. A second audit was conducted in June 1996, and certification has again been awarded. This new award of certification is effective until August 31, 1998.

In FY 1996, the ACL analyzed many samples to determine hazardous or radiological components. Most of these samples were from either the U.S. Department of Agriculture monitoring program (see item 12), or from ANL-East programs focused on orphan waste cleanup

and facilities decontamination and decommissioning (see item 8). A number of special analytical samples taken from U.S. EPA Region V sites were submitted for analysis as part of site remediation programs. In analyzing these samples, the ACL used methods prescribed in the *U.S. EPA CLP Statement of Work* or *Test Methods for Evaluating Solid Waste (SW-846 Method)*, or EPA-approved alternatives. Approximately 45 CLP-type data packages consisting of analytical results and raw data were prepared, reviewed, and forwarded to various clients for data evaluation.

As part of laboratory intercomparison studies carried out by DOE and EPA, sponsors often require that instrument and analyst performance be monitored by means of participation in the analysis of single-blind performance evaluation samples. ("Single-blind" means that the analyte values are not known by the analyst.) The U.S. EPA and DOE multilaboratory performance assessment programs in which the ACL successfully participated in FY 1996 include the following.

- The *U.S. EPA National Exposure Research Laboratory-Cincinnati (NERL-Ci) Water Supply (WS) and Water Pollution Study (WP)*, in which samples were analyzed for trace metals, cyanide, oil and grease, total dissolved and suspended solids, pH, volatile organic compounds, PCBs, semivolatiles (WS only), and a variety of anions. NERL-Ci reported that analytical results submitted for samples WP036 and WS037 showed excellent agreement with the EPA known values.
- The *U.S. DOE Environmental Measurements Laboratory (EML) Quality Assessment Program* and the *U.S. EPA National Exposure Research Laboratory-Las Vegas (NERL-LV) Performance Evaluation Studies Program* provided the ACL with single-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 the year. The comparison of ACL results to the accepted values showed that the ACL correctly determined all analytes.

- This year the *U.S. DOE Mixed Analyte Performance Evaluation Program (MAPEP)* was added to the performance evaluation programs in which the ACL participates. This program was established by DOE's Office of Environmental Management (EM) to help assure the quality and defensibility of analytical measurements being performed for EM. All laboratories that perform environmental analytical measurements on mixed analyte samples for EM must participate in the MAPEP. The ACL served as a pilot laboratory in 1994, helping to establish the MAPEP database. The ACL has since successfully analyzed its first sample as a participant in the program.

Significant progress was made toward completing the ACL sample bar code system. The system provides quick and efficient electronic logging, distribution, and tracking of analytical samples by means of bar code labels placed on all containers. Sample login and notification, chain-of-custody tracking, and the generation of sample status reports, all of which were previously done by hand, with records maintained in hard-copy files, may now be performed electronically. Satellite scanning stations have been installed in additional ACL laboratories located in Buildings 200, 203, and 205 and are scheduled to be on-line and operational by the end of calendar 1996.

## IV. PROFESSIONAL ACTIVITIES

A. Publications and Reports

Determination of Uranium by Flow Injection Inductively Coupled Plasma/Mass Spectrometry

J. H. Aldstadt, J. M. Kuo, L. L. Smith, and M. D. Erickson

Analytica Chimica Acta 319, 135-143 (1996)

Microwave Dissolution of Plant Tissue and the Subsequent Determination of Trace Lanthanide and Actinide Elements by Inductively Coupled Plasma-Mass Spectrometry

J. S. Alvarado, T. J. Neal, L. L. Smith, and M. D. Erickson

Analytica Chimica Acta 322, 11-20 (1996)

Containment Attachment for Mixed-Waste Analysis by Graphite Furnace AAS

D. A. Bass, L. B. TenKate, and A. M. Wroblewski

Atomic Spectroscopy 17(2), 92-97 (1996)

A Comparison of Continuous Pneumatic Nebulization and Flow Injection-Direct Injection Nebulization for Sample Introduction in Inductively Coupled Plasma-Mass Spectrometry

J. S. Crain and J. T. Kiely

Argonne National Laboratory Report No. ANL/ACL-95/4 (1995)

Applications of Inductively Coupled Plasma/Mass Spectrometry in Environmental Radiochemistry

J. S. Crain

Spectroscopy 11(2), 30-39 (1996)

Waste Reduction in Inductively Coupled Plasma Mass Spectrometry using Flow Injection and a Direct Injection Nebulizer

J. S. Crain and J. T. Kiely

J. of Analytical Atomic Spectrometry 11, 525-527 (1996)

Comparison of SW-846 Method 3051 and SW-846 Method 7471A for the Preparation of Solid Waste Samples for Mercury Determination

J. M. Giaquinto, A. M. Essling, and J. M. Keller

Oak Ridge National Laboratory Report No. ORNL/TM-13236 (1996)

Analytical Chemistry Laboratory Progress Report for FY 1995

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

Argonne National Laboratory Report No. ANL/ACL-95/5 (1995)

The Dreaded Lower Ten Percent

D. W. Green

Managing the Modern Laboratory 2(1), 2A-3A (1996)



## Forum on the Future of Chemists

D. W. GreenManaging the Modern Laboratory 2(1), 11A-12A (1996)

## Secondary Waste Minimization in Analytical Methods

D. W. Green, L. L. Smith, J. S. Crain, A. S. Boparai, J. T. Kiely, J. S. Yaeger, and J. B. SchillingAmerican Environmental Laboratory 8(3), 28-29 (1996)

## Scientists as Managers

D. W. GreenManaging the Modern Laboratory 1(4), 68A-69A (1995)

## Waste Minimization in Analytical Methods

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

Proceedings of the Pollution Prevention Conference XI, May 16-18, 1995, Knoxville, TN, 144-150 (1995)

## A Survey of Continuous Emission Monitoring Technologies for Volatile Organic Compounds, HCl, and Ammonia

W. Haas, N. French, J. C. Demirgian, and C. T. Snyder

Proceedings of the 1996 International Conference on Incineration and Thermal Treatment Technologies, Savannah, GA, May 6-10, 1996, 409-415 (1996)

Effect of Lead Loss and Sheath Structure on Phase Formation and Alignment in  $(\text{Bi,Pb})_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}/\text{Ag}$  Composite ConductorsJ. S. Luo, N. Merchant, V. A. Maroni, S. E. Dorris, M. T. Lanagan, and B. S. TaniJ. Am. Ceram. Soc. 78(10), 2785-2789 (1995)

## Applications of Inductively Coupled Plasma-Mass Spectrometry to Radionuclide Determinations

R. W. Morrow and J. S. Crain, Eds.

American Society for Testing and Materials, Special Technical Publication 1291 (1995)

## Evaluation of Three Analytical Techniques Used to Determine High Levels of Volatile Organic Compounds in Type IV Sludge from Rocky Flats Plant

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

Argonne National Laboratory Report No. ANL/ACL-96/3 (1996)

## Advanced Concept Proof-of-Principle Demonstration: Switchable Radioactive Neutron Source

E. A. Rhodes, D. L. Bowers, R. E. Boyar, and C. E. Dickerman

Argonne National Laboratory Report No. ANL/ACTV-95/2 (1995)

High-Pressure Microwave Digestion: A Waste-Minimization Tool for the Radiochemistry Laboratory

L. L. Smith and J. S. Yaeger

Radioactivity and Radiochemistry 7(10), 35-38 (1996)

B. Oral Presentations

Determination of Long-Lived Radioisotopes in Environmental Samples by Inductively Coupled Plasma-Mass Spectrometry

J. S. Alvarado, M. D. Erickson, J. S. Crain, T. J. Neal, and L. L. Smith

36th ORNL-DOE Conference on Analytical Chemistry in Energy Technology, Gatlinburg, TN, October 10-12, 1995

Determination of Water Soluble Volatile Organic Compounds (VOCs) in Solid Matrices

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

47th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 3-8, 1996

Arsenic Speciation Using HPLC-ICP-MS

D. A. Bass

24th ACL Technical Meeting, Argonne, IL, December 7, 1995

Detecting and Quantifying Lewisite Degradation Products in Environmental Samples Using Arsenic Speciation

D. A. Bass, J. S. Yaeger, J. T. Kiely, J. S. Crain, M. J. Gowdy, L. M. Shem, and G. B. Mohrman

Scientific Conference on Chemical and Biological Defense Research, Aberdeen Proving Ground, MD, November 14-17, 1995

High Performance Liquid Chromatography-Inductively Coupled Plasma-Mass Spectrometry (HPLC-ICP-MS) for Measurement of Arsenic Species in Soil

D. A. Bass, J. S. Yaeger, K. J. Parish, J. S. Crain, J. T. Kiely, M. J. Gowdy, L. M. Shem, and G. B. Mohrman

36th ORNL-DOE Conference on Analytical Chemistry in Energy Technology, Gatlinburg, TN, October 10-12, 1995

High Performance Liquid Chromatography-Inductively Coupled Plasma-Mass Spectrometry (HPLC-ICP-MS) for Measurement of Arsenic Species in Environmental Samples

D. A. Bass, J. S. Yaeger, J. T. Kiely, J. S. Crain, M. J. Gowdy, L. M. Shem, and G. B. Mohrman

47th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 3-8, 1996

Cleanup of Radioactive Mixed Waste Sample Extracts

A. S. Boparai, T. R. Henning, Y. Tsai, L. L. Smith, and E. L. Coleman

47th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 3-8, 1996

Analysis of FTIR Sensor Data Using Multiple Bandpass Digital Filtering Algorithms

S. E. Carpenter

47th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 3-8, 1996

Sensor Algorithm Generation Environment (SAGE)

S. E. Carpenter

University of Chicago Review Committee Meeting for the Chemical Technology Division, Argonne, IL, September 5-6, 1996

Actinides at the Crossroads: ICP-MS or Alpha Spectrometry?

J. S. Crain, L. L. Smith, F. P. Smith, J. T. Kiely, D. G. Graczyk, and J. S. Alvarado

Fourth Annual Meeting of The Council on Ionizing Radiation Measurements and Standards, Gaithersburg, MD, November 28-30, 1995

Actinides at the Crossroads: ICP-MS or Alpha Spectrometry?

J. S. Crain, J. T. Kiely, L. L. Smith, F. P. Smith, D. G. Graczyk, and J. S. Alvarado

Royal Society of Chemistry Atomic Spectroscopy Symposium, Howth, County Dublin, Ireland, March 28-29, 1996

Actinides at the Crossroads: ICP-MS or Alpha Spectrometry?

J. S. Crain

Poster Session for the University of Chicago Review Committee Meeting for the Chemical Technology Division, Argonne, IL, September 5-6, 1996

ICP-MS and the Determination of Environmental Uranium: A Match Made in Heaven?

J. S. Crain

U.S. Army Center for Health Promotion and Preventive Medicine, Edgewood Area, Aberdeen Proving Ground, MD, April 25, 1996

Cocaine Contamination Studies on Currency

J. C. Demirgian, E. Y. Hwang, B. S. Tani, C. T. Roche, S. Ulvick, T. Kunz, S. Rigdon, and C. Su

1995 Office of National Drug Control Policy International Technology Symposium Counter-Drug Law Enforcement: Applied Technology for Improved Operational Effectiveness, Nashua, NH, October 23-26, 1995

Cocaine Contamination Studies on Chicago Currency

J. C. Demirgian, E. Y. Hwang, J. M. Ewing, B. S. Tani, C. T. Roche, S. Ulvick,  
T. Kunz, C.-W. Su, and S. W. Rigdon

47th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied  
Spectroscopy, Chicago, IL, March 3-8, 1996

Examining Cash for Cocaine Using Scanning Electron Microscopy

J. C. Demirgian, B. S. Tani, E. Y. Hwang, C. T. Roche, and J. M. Ewing

Inter/Micro-96, Chicago, IL, July 25, 1996

Remote Detection of Environmental Pesticide Spray Using FTIR Spectroscopy

J. C. Demirgian, G. F. Busse, and J. Ditillo

47th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied  
Spectroscopy, Chicago, IL, March 3-8, 1996

Interlaboratory Study to Validate a Procedure for the Determination of Strontium-89/  
Strontium-90 in Water

M. A. Edgell, M. D. Erickson, L. L. Smith, P. C. Lindahl, W. E. Streets, A. E.  
Scandora, S. Goquin, and S. Hedayat

41st Annual Conference on Bioassay, Analytical, and Environmental  
Radiochemistry, Boston, MA, November 12-16, 1995

Analytical Methods for Environmental Radionuclides

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

47th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied  
Spectroscopy, Chicago, IL, March 3-8, 1996

Collaborative Study to Validate Procedure for the Determination of Strontium-89/  
Strontium-90 in Water

M. D. Erickson, M. A. Edgell, S. Hedayat, P. C. Lindahl, A. E. Scandora, L. L.  
Smith, W. E. Streets, and G. Su

36th ORNL-DOE Conference on Analytical Chemistry in Energy Technology,  
Gatlinburg, TN, October 10-12, 1995

Environmental Radioanalytical Methods Development

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

36th ORNL-DOE Conference on Analytical Chemistry in Energy Technology,  
Gatlinburg, TN, October 10-12, 1995

Faster, Cheaper, and Pollution-Preventing Analytical Methods for Environmental Radionuclides

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

41st Annual Conference on Bioassay, Analytical, and Environmental Radiochemistry, Boston, MA, November 12-16, 1995

A Comparison of SW846 Methods 7471A and 3051 for Mercury Preparation Using Standard Reference Materials

J. Giaquinto, T. Ross, and A. M. Essling

36th ORNL-DOE Conference on Analytical Chemistry in Energy Technology, Gatlinburg, TN, October 10-12, 1995

Challenging Projects in Analytical Support

D. G. Graczyk

University of Chicago Review Committee Meeting for the Chemical Technology Division, Argonne, IL, September 5-6, 1996

Overview of ACL

D. W. Green

University of Chicago Review Committee Meeting for the Chemical Technology Division, Argonne, IL, September 5-6, 1996

A Survey of Continuous Emission Monitoring Technologies for Volatile Organic Compounds, HCl, and Ammonia

W. Haas, N. French, J. C. Demirgian, and C. T. Snyder

1996 International Conference on Incineration and Thermal Treatment Technologies, Savannah, GA, May 6-10, 1996

Characteristics of Lithium Ceramics Irradiated in the FUBRIB/BEATRIX Experiment

C. E. Johnson, L. A. Neimark, and D. L. Bowers

The 4th Int. Workshop on Ceramic Breeder Blanket Interactions, Kyoto, Japan, October 9-11, 1995

Analysis of Polymer Plastics by Fourier Transform Infrared Microscopy

S. D. Kent, C. T. Snyder, and S. E. Carpenter

47th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 3-8, 1996

FTIR Sample Analysis: Microscopic and Macroscopic Techniques

S. D. Kent

24th ACL Technical Meeting, Argonne, IL, December 7, 1995

Flow Injection-Direct Injection Nebulization (FI-DIN) for High Efficiency Sample Introduction and Waste Minimization in ICP-MS

J. T. Kiely and J. S. Crain

36th ORNL-DOE Conference on Analytical Chemistry in Energy Technology, Gatlinburg, TN, October 10-12, 1995

Flow Injection-Direct Injection Nebulization (FI-DIN) for High Efficiency Sample Introduction and Waste Minimization in Inductively Coupled-Plasma-Mass Spectrometry

J. T. Kiely

25th ACL Technical Meeting, Argonne, IL, April 25, 1996

Radium Separation and Measurement using Empore™ Radium Rad Disks

R. M. Kinney, R. T. Shannon, L. L. Smith, K. M. Hoffman, and D. C. Seely

Workshop on "Innovative Radiochemical Analyses through the Use of Empore™ Rad Disks with Analig™" at the 41st Annual Conference on Bioassay, Analytical, and Environmental Radiochemistry, Boston, MA, November 11, 1995

The Department of Energy's Integrated Performance Evaluation Program (IPEP): Pilot Studies for Implementation

P. C. Lindahl, W. E. Streets, R. W. Newberry, M. H. Carter, C. Watkins, J. Connolly, J. Fisk, and E. Baugher

41st Annual Conference on Bioassay, Analytical, and Environmental Radiochemistry, Boston, MA, November 12-16, 1995

Pilot Implementation Studies of the DOE's Office of Environmental Management's Integrated Performance Evaluation Program (IPEP)

P. Lindahl, E. Streets, R. Newberry, M. Carter, C. Watkins, J. Connolly, J. Fisk, and E. Baugher

36th ORNL-DOE Conference on Analytical Chemistry in Energy Technology, Gatlinburg, TN, October 10-12, 1995

Pilot Implementation Studies of the DOE's Office of Environmental Management's Integrated Performance Evaluation Program (IPEP)

P. Lindahl, E. Streets, R. Newberry, M. Carter, J. Connolly, C. Watkins, J. Fisk, and E. Baugher

Field/National Sample Management Program, Working Group Meeting, Augusta, GA, October 23-26, 1995

Pollution Prevention/Waste Minimization in the Analytical Chemistry Laboratory: Efficiency Factors and Source Reduction

J. Lu, M. D. Erickson, D. P. Peterson, E. A. Huff, J. T. Kiely, and J. S. Crain

47th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 3-8, 1996

## Radon Remediation

F. Markun

24th ACL Technical Meeting, Argonne, IL, December 7, 1995

## Post-Test Analysis of Batteries

J. J. Marr

25th ACL Technical Meeting, Argonne, IL, April 25, 1996

## Analysis of Multispectral Image Data Utilizing a Neural Network Based on Adaptive Resonance Theory (ART2A)

Y. X. Noyes and S. E. Carpenter

47th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 3-8, 1996

## Identification and Quantitative Determination of Congeners in Unresolved Chromatographic Peaks Using Chromatography and Matrix Isolation Infrared Spectrometry

G. T. Reedy

47th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 3-8, 1996

## Surface Enhanced Infrared Absorbance (SEIRA)

G. T. Reedy

25th ACL Technical Meeting, Argonne, IL, April 25, 1996

## Comparison of Instrumental Methods Used in the Determination of Uranium and Plutonium

C. S. Sabau, D. L. Bowers, and F. P. Smith

Argonne National Laboratory Technical Women's Symposium, Argonne, IL, April 29-30, 1996

## Extraction of Semivolatile Organic Pollutants from HEPA Filters Using Supercritical Carbon Dioxide

J. B. Schilling

36th ORNL-DOE Conference on Analytical Chemistry in Energy Technology, Gatlinburg, TN, October 10-12, 1995

## Thermal Desorption of Agent-Simulant-Spiked Soil from Rocky Mountain Arsenal

L. Shem, H. J. O'Neill, D. A. Bass, D. O'Reilly, and M. Besmer

Federal Programs, 10th Annual Conference on Contaminated Soils, Analysis, Fate, Environmental and Public Health Effects and Remediation, Amherst, MA, October 23-26, 1995

## Thermal Desorption Screening of Agent-Simulant-Spiked Soil from Rocky Mountain Arsenal

L. M. Shem, H. J. O'Neill, D. A. Bass, D. O'Reilly, and M. Besmer

Scientific Conference on Chemical and Biological Defense Research, Aberdeen Proving Ground, MD, November 14-17, 1995

Application of Empore™ Disk Technology to Environmental Radiochemical Analysis

L. L. Smith, K. A. Orlandini, J. S. Alvarado, K. Hoffmann, D. Seely, and R. Shannon  
41st Annual Conference on Bioassay, Analytical, and Environmental  
Radiochemistry, Boston, MA, November 12-16, 1995

Innovative Radiochemical Analyses Through the Use of Empore™ Disk Technology

L. L. Smith, K. A. Orlandini, J. S. Alvarado, M. D. Erickson, K. Hoffmann, D. Seely,  
and R. Shannon  
36th ORNL-DOE Conference on Analytical Chemistry in Energy Technology,  
Gatlinburg, TN, October 10-12, 1995

An Evaluation of Empore™ Strontium Rad Disks

L. L. Smith, K. A. Orlandini, J. S. Alvarado, M. D. Erickson, K. M. Hoffmann, D. C.  
Seely, and R. T. Shannon  
Workshop on "Innovative Radiochemical Analyses through the Use of Empore™  
Rad Disks with Analig™" at the 41st Annual Conference on Bioassay, Analytical,  
and Environmental Radiochemistry, Boston, MA, November 11, 1995

Waste Minimization in Analytical Chemistry Operations

L. L. Smith  
Poster Session for the University of Chicago Review Committee Meeting for the  
Chemical Technology Division, Argonne, IL, September 5-6, 1996

Women in Chemistry

C. T. Snyder  
Invited Panelist, Chemistry Seminar Series, Elmhurst College, Elmhurst, IL,  
November 1, 1995

CO Fluxes in the Northern Hemisphere Temperature Zone in 1971

C. M. Stevens  
1996 Fall Meeting, American Geophysical Union, San Francisco, CA,  
September 11, 1996

Chromatographic Optimization of GC/MS Analysis of Semivolatile Organic Pollutants

Y. Tsai  
24th ACL Technical Meeting, Argonne, IL, December 7, 1995

Applications of High-Pressure Microwave Digestion to Radiochemical Analyses

J. S. Yaeger and L. L. Smith  
47th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied  
Spectroscopy, Chicago, IL, March 3-8, 1996



C. AwardsDelbert L. Bowers

American Society for Testing and Materials, C-26 on Nuclear Fuel Cycle, Achievement Award in recognition of many years of outstanding service in the work of this Committee.

D. Meetings AttendedAlice M. Essling

47th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 3-8, 1996

Doris R. Huff

47th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 3-8, 1996

Edmund A. Huff

47th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 3-8, 1996

Florence P. Smith

23rd Annual Meeting of the National Organization of Black Chemists and Chemical Engineers, Detroit, MI, April 8-13, 1996

Carmen S. Sabau

47th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 3-8, 1996

W. Elane Streets

Argonne National Laboratory Graduate School Mentoring Day (Mentor), Argonne, IL, October 17, 1995

Science Careers in Search of Women (Mentor), Argonne, IL, May 3, 1996

E. Professional Organizations and ActivitiesDean 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  
Royal Society of Chemistry  
Editorial Board, Atomic Spectrometry Updates

Jack C. Demirgian

American Chemical Society  
Air and Waste Management Association  
Technical Association of the Pulp and Paper Industry

Donald G. Graczyk

American Chemical Society  
Sigma Xi  
American Society for Mass Spectrometry

John W. Gramlich

American Chemical Society  
Chicago Section  
Institute of Nuclear Materials Management (Senior Member)  
International Union of Pure and Applied Chemistry  
Commission on Atomic Weights and Isotopic Abundances  
Commission on Isotopic Specific Measurements as References  
Subcommittee for Isotopic Abundance Measurements

David W. Green

American Chemical Society  
Division of Analytical Chemistry  
Analytical Laboratory Managers Association, Board of Directors  
Editor, 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

Stephen D. Kent

American Chemical Society  
Society for Applied Spectroscopy

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

Clifton N. Merrow

American Chemical Society  
American Physical Society

Ying X. Noyes

American Chemical Society

Gerald T. Reedy

American Chemical Society  
Society for Applied Spectroscopy

Laurids E. Ross

American Chemical Society  
Sigma Xi  
American Association for the Advancement of Science

Carmen S. Sabau

American Chemical Society  
 Division of Nuclear Chemistry and Technology  
 Chicago Section  
 American Nuclear Society  
 Fuel Cycle and Waste Management Division  
 Chicago Section  
 Environmental Sciences Division  
 American-Romanian Academy of Arts and Sciences  
 Association of Women in Science  
 Chicago Chapter  
 Humboldt Association of America  
 International Society for Intercommunication of New Ideas  
 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 TrainingLilia Barbosa

Using the Internet, Navigating the World Wide Web, Argonne, IL, October 5, 1995  
 Using the Internet, Authoring for the World Wide Web, Argonne, IL, October 12, 1995  
 Professional Development for Women, Countryside, IL, March 27, 1996

Alice M. Birmingham

The Indispensable Assistant, Westmont, IL, August 14, 1996

Alice M. Essling

CEM Corporation Fall Seminar, Complete Solutions for Sample Preparation, Naperville, IL, September 26, 1996

Stephen D. Kent

AP/PS Dedicated Mercury Analyzer Systems, Rolling Meadows, IL, October 11, 1995  
Practical Techniques for Development and Implementation of QC Methods, Naperville, IL, October 25, 1995

Vanessa A. Mendez

Microsoft Excel 5.0 for Windows, Argonne, IL, October 11, 1995

W. Elane Streets

University of Chicago Workshops in Technical and Scientific Writing, Argonne National Laboratory, January 29 and 31, 1996

Lynn B. TenKate

CEM Corporation Fall Seminar, Complete Solutions for Sample Preparation, Naperville, IL, September 26, 1996

ACL-Sponsored Internal Training Attended by Various Staff

Seven Habits of Highly Effective People Video Training, February 27, 1996  
Performance Appraisal Training, May 30, 1996

G. ACL Seminars

Automated Recognition of Targets in Multispectral Images: An Application to Naval Ships  
Scott E. Carpenter, Argonne National Laboratory, Argonne, IL  
December 11, 1995

Detecting Air Pollutants with Chemiluminescent Reactions  
Jeff Gaffney, Argonne National Laboratory, Argonne, IL  
May 24, 1996

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