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ANALYTICAL CHEMISTRY LABORATORY
Progress Report for FY 1997

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ANL/ACL-92/4 (December 1992)

ANL/ACL-93/2 (December 1993)

ANL/ACL-94/4 (December 1994)

ANL/ACL-95/5 (December 1995)

ANL/ACL-96/6 (December 1996)

TABLE OF CONTENTS

TABLE OF CONTENTS (Contd)

TABLE OF CONTENTS (Contd)

NOMENCLATURE

ACL	Analytical Chemistry Laboratory
AGHCF	Alpha Gamma Hot Cell Facility
AL	DOE's Albuquerque Operations Office
ANL	Argonne National Laboratory
APS	Advanced Photon Source
ART2A	Adaptive resonance theory
ASD	DOE/EM's Analytical Services Division
BSCCO	bismuth/strontium/calcium/copper oxides
CEM	continuous emissions monitor
CH	DOE's Chicago Operations Office
CLP	Contract Laboratory Program
CMT	Chemical Technology Division
CP-5	Chicago Pile-5
CVAA	cold vapor atomic absorption
DIMP	diisopropyl methyl phosphonate
DMMP	dimethyl methyl phosphonate
DOE	U.S. Department of Energy
EM	DOE Office of Environmental Management
EML	Environmental Measurements Laboratory
EMO	Environmental Management Operations
EPA	U.S. Environmental Protection Agency
ER	Environmental Research Division
ES	Energy Systems Division
FFT	fast Fourier transformation
FPXRF	field portable X-ray fluorescence spectrometer
FTIR	Fourier transform infrared [spectroscopy]
FT-Raman	Fourier transform Raman [spectroscopy]
FY	fiscal year
GC/MS	gas chromatography/mass spectroscopy
GFAA	graphite furnace atomic absorption
HARC	Houston Advanced Research Center
HEPA	high-efficiency particulate air [filters]
ICP/AES	inductively coupled plasma/atomic emission spectrometry
ICP/MS	inductively coupled plasma/mass spectrometry
ID	DOE's Idaho Operations Office
INEEL	Idaho National Engineering and Environmental Laboratory
IPEP	Integrated Performance Evaluation Program
KFT	Karl Fischer titration
LSDP	Large-Scale Demonstration Project
LTA	Lead Test Assembly
M&O	management and operations
MAG*SEP^{TM}	magnetic separation process patented by Bradtec Ltd.
MAPEP	Mixed Analyte Performance Evaluation Program

NOMENCLATURE (Contd)

MSRE	Molten Salt Reactor Experiment
NASP	DOE's National Analytical Services Program
NEET	nuclear excitation by electronic transition
NERL-Ci	National Exposure Research Laboratory, Cincinnati
NERL-LV	National Exposure Research Laboratory, Las Vegas
PCB	polychlorinated biphenyl
PDP	Performance Demonstration Program
PE	performance evaluation
PLDA	piecewise linear discriminate analysis
PNNL	Pacific Northwest National Laboratory
PUF	polyurethane foam
QA	quality assurance
QC	quality control
R&D	research and development
RESL	Radiological and Environmental Sciences Laboratory
RCRA	Resource Conservation and Recovery Act
RH-TRU	remote-handled transuranic [waste]
RRWAC	Reusable Property, Recyclable Materials, and Waste Acceptance Criteria
SAC	Synchrotron Advisory Committee
SAGE	Sensor Algorithm Generation Environment
SEM/EDS	scanning electron microscopy/energy dispersive spectroscopy
SMO	Sample Management Office
SOP	standard operating procedure
SVOC	semivolatile organic compound
TCE	tetrachloroethylene
TCLP	Toxicity Characteristic Leaching Procedure
TD	Technology Development Division
TIMS	thermal ionization mass spectrometry
TRU	transuranic
TSCA	Toxic Substances Control Act
TTQP	Tritium Target Qualification Project
VOC	volatile organic compound
WIPP	Waste Isolation Pilot Plant
WMCT	Waste Management Compliance Team
WP	Water Pollution Study Program
WS	Water Supply Study Program
XAFS	X-ray absorption extended fine structure
XRD	X-ray diffraction

ANALYTICAL CHEMISTRY LABORATORY
Progress Report for FY 1997

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) 1997 (October 1996 through September 1997). This annual progress report is the fourteenth in this series for the ACL, and it describes continuing effort on projects, work on new projects, and contributions of the ACL staff to various programs at ANL.

The ACL operates in the Argonne system as a full-cost-recovery service center, but it 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 (R&D) work at ANL, the ACL handles a wide range of analytical problems. Some routine or standard analyses are done, but the ACL usually works with commercial laboratories if high-volume, production analyses are required by our clients. It is common for the Argonne R&D 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 applied analytical chemistry research work.

The ACL is administratively within the Chemical Technology Division (CMT), its principal ANL client, but it provides technical support for many of the other technical divisions and programs at ANL. The ACL has four technical groups -- Chemical Analysis, Instrumental Analysis, Organic

Analysis, and Environmental Analysis -- which together include about 30 technical staff members (see Fig. 1 for the organization chart at the end of FY 1997). Talents and interests of staff members cross group lines, as do many projects within the ACL.

The Chemical Analysis Group uses wet-chemical and instrumental methods for elemental, compositional, and isotopic determinations in solid, liquid, and gaseous samples and provides specialized analytical services. The Instrumental Analysis Group uses nuclear counting techniques to determine the radiochemical constituents in a wide range of sample types, from environmental samples with low radioactivity to samples with high radioactivity that require containment. The Organic Analysis Group uses a number of complementary techniques to separate organic compounds and measure them at trace levels and has performed development work in sensors, chemometrics, and detectors. The Environmental Analysis Group analyzes environmental, hazardous-waste, and coal samples for the inorganic constituents and provides quality assurance support to the U.S. Department of Energy (DOE) and other clients for various kinds of analytical data. Together, the ACL groups have a full range of analytical capabilities for performing inorganic, organic, and radiological analyses.

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

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

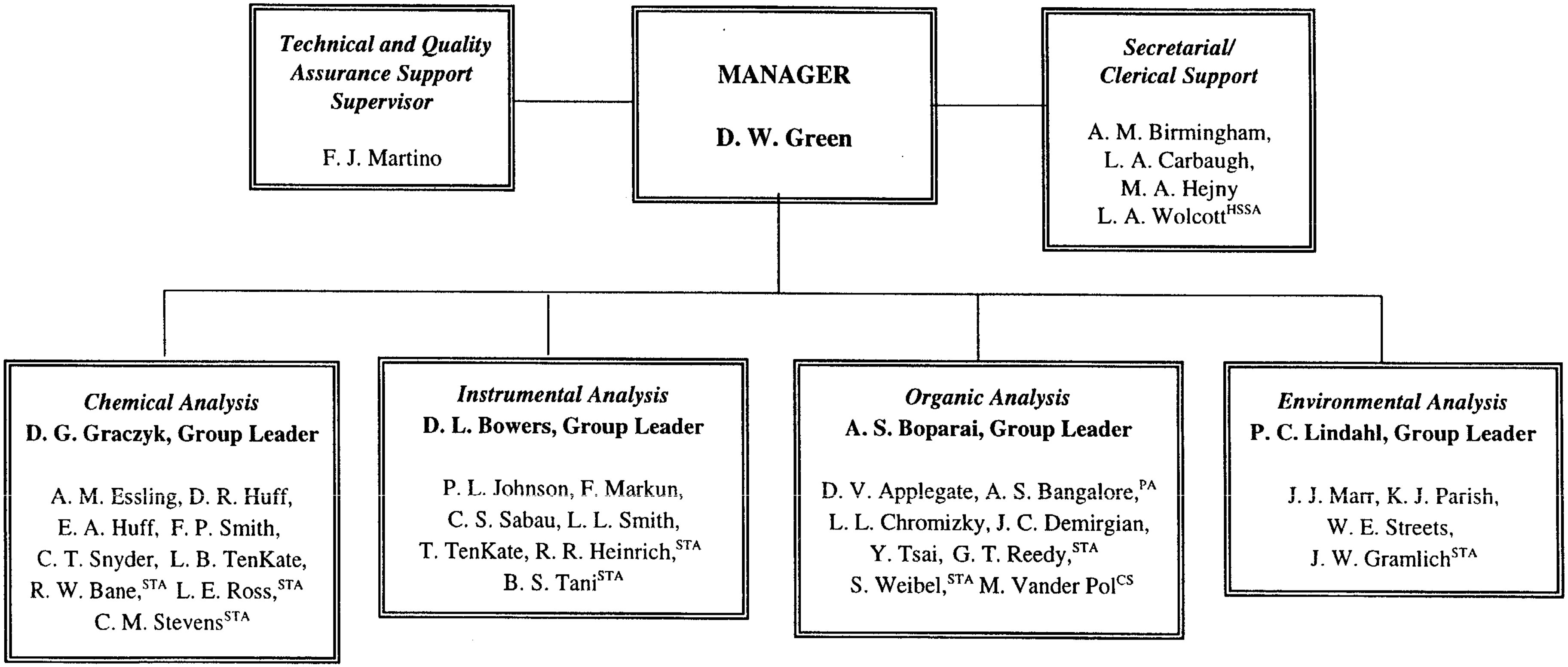
- List of all published ANL/ACL technical reports,
- Major technical specialties of the ACL Staff,
- Advice on how to submit samples, and
- The ACL Annual Reports for FY 1995 and 1996.

Installation of a replacement for our inductively coupled plasma/atomic emission spectrometer (ICP/AES) was completed in FY 1997. The ACL received new a carbon/sulfur determinator and a new oxygen/nitrogen determinator funded by the ANL General Purpose Equipment Fund, which continues to be the primary source of equipment funding for the ACL. Both instruments should be operational in early FY 1998. In addition, the ACL received and installed a new gamma spectrometry system to replace a 12-year-old instrument that was heavily used.

The ACL worked on two projects in FY 1997 with ANL Support Development Funds. In the first project, the ACL did performance assessment tests of the new ICP/AES system. In the second project, the ACL improved its ability to do spectral interference corrections for plasma source mass spectrometry. Progress on these projects is described later in this report.

The remainder of this report is a collection of short descriptions of the analytical work performed by the staff of the ACL in FY 1997 on selected projects. The ACL receives about 1,600 different jobs annually, many of which involve several 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 often make an important contribution to the overall success of the project that received the data. These contributions to projects can easily be overlooked, but collectively they form an important part of the mission of the ACL.

Fig. 1. Analytical Chemistry Laboratory Organization Chart 9/30/97



HSSA = High School Student Aide; STA = Special Term Appointee; PA = Postdoctoral Appointee; RA = Research Aide; SRP = Student Research Participant; CS = Co-op Student.

II. TECHNICAL HIGHLIGHTS

(1) Nuclear Technology (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, K. J. Parish, 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) spectrometry, and thermal ionization mass spectrometry (TIMS). These instruments were used for obtaining elemental, isotopic, and compound information. Many of the samples submitted contained radioactive materials, such as plutonium. These types of samples were analyzed with instruments, such as the ICP/AES, that had been modified for use with the radioactive samples.

(2) Continuous Emission Monitoring of Plasma Hearth Furnace (J. C. Demirgian)

A continuous emission monitor (CEM) based upon Fourier transform infrared (FTIR) technology that was developed at ANL has been used at the plasma hearth furnace, a unique waste-treatment facility at ANL-West, to monitor stack gases. The objective of the FY 1997 tests, which were conducted in collaboration with K. Carney (ANL-W), was to determine how operating conditions affect the combustion efficiency. Combustion parameters were changed for both the primary and the secondary combustion chambers, and the consequences were monitored by the CEM to determine the most efficient mode of operation for a variety of feeds.

The stack gas at the plasma hearth furnace differs significantly from that at the Toxic Substances Control Act (TSCA) incinerator in Oak Ridge, TN, which we had studied previously with the CEM. Several methods that we had used to monitor stack gases at the TSCA incinerator had to be modified to accommodate the different stack gas compositions. Using an older method to detect and quantify sulfur hexafluoride, carbon dioxide, water, tetrachloroethylene (TCE), and ethylene caused marginal matrix spike recoveries and false positive detection of TCE. A new method was developed for these constituents. Its calibration file contains an improved and expanded list of calibrated standards and analytical data from actual plasma hearth samples that were previously analyzed. This method is a significant improvement over the original one, which tended to misidentify TCE.

The older carbon monoxide (CO) detection method was also replaced with two new methods. The original method was limited by a 25-ppm maximum detection level and resulted in over-recoveries of the matrix spike samples. The first new method was developed to expand the detection range at lower CO levels. Some of the calibration standards were replaced, and a new 40-ppm standard was added to extend the range of the method. Plasma hearth sample data were also added to the method to make it more compatible with the plasma hearth stack gas. This new method has eliminated the over-recovery problem. A second method was developed to quantify high concentrations of CO. It is used when the CO concentration range is 70 to 1175 ppm. This method was developed and tested using actual plasma hearth samples in the training set.

Hardware upgrades were made, including the addition of a multiport sampler. The software was upgraded to provide automatic data backups, and a real-time monitor was added that transmits data to the control room so that the fuel and oxygen levels can be maintained in the proper combustion ratio. As part of these upgrades, an external transmission link was also set up, tested, and brought into operation. Our emission monitoring data can now be transmitted to any site in real time.

(3) Characterization of Products and Residues from Automobile Shredder Fluff Recycling

(A. S. Boparai, L. L. Chromizky, K. J. Parish, Y. Tsai, D. V. Applegate, A. M. Essling, E. A. Huff, D. R. Huff, and D. G. Graczyk)

About 225 kg (25%) of every scrapped automobile 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. Scientists in Argonne's Energy Systems division (B. Jody, J. Pomykala, et al.) are testing a potentially economical process to separate and recycle fluff recovered from scrapped automobiles. As part of this process, polyurethane foam (PUF) (e.g., from car seats or dashboards) is mechanically separated from other material produced when each scrapped automobile is shredded. In a pilot-scale study, the Argonne researchers are testing an approach to cleaning this PUF so that it can be recycled into value-added products, such as carpet padding.

The ACL has been supporting the recycling process development by analyzing various intermediates and products to determine their compositions and by measuring potential contaminants in recovered oils, foams, and residual materials classified as "fines".

The ACL has analyzed samples for metals, chlorine, sulfur, water, polychlorinated biphenyls (PCBs), and residual cleaning solvents. In our analysis, we determine the water content by the Karl Fischer titration method, ash content by igniting a portion of each sample and weighing, Cl and S by combustion in an oxygen bomb followed by ion chromatography, mercury by cold vapor atomic absorption following a microwave-assisted acid digestion, and other metals by inductively coupled plasma/atomic emission spectroscopy (ICP/AES) on a portion of ash dissolved in mineral acids. We developed special procedures to estimate the oil content of selected samples as the nonvolatile residue from a hexane extraction and to measure PCBs by using a modification of the EPA procedures for waste analysis. We measure residual cleaning solvent by purge-and-trap gas chromatography/mass spectrometry (GC/MS). The data obtained from these analyses help to evaluate the quality of recovered products and to establish the hazard classification of the waste streams from the process so they may be disposed of properly.

During FY 1997, the main emphasis has been on determination of organic compounds (PCBs and cleaning solvents) in foams and fines using the methods described above.

(4) Support for High-Temperature Superconductor Development (E. A. Huff, D. R. Huff, F. P. Smith, 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 involved analysis of starting materials, process samples, and products related to fabrication, as well as characterization studies on various ceramic compositions, including $YBa_2Cu_3O_{7-x}$ ("1,2,3-compound") and lead-doped bismuth/strontium/calcium/copper oxides ("BSCCO" ceramics). Our measurements included determining elemental composition by ICP/AES or classical wet chemical methods, anions by ion chromatography, carbon with a Leco™ analyzer, and phase composition by X-ray powder diffraction. We also used an iodometric titration method to determine excess valence of the metals in a given ceramic (e.g., Cu^{3+}), which is related to the oxygen stoichiometry of the compound.

The researchers in the Energy Technology Division (S. Dorris, M. Lanagan, 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. In addition, 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) Performance Demonstration Program for the WIPP Transuranic Solidified Waste Characterization Program (A. S. Boparai, Y. Tsai, K. J. Parish, L. B. TenKate, E. A. Huff, D. R. Huff, A. M. Essling, and D. G. Graczyk)

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; the WIPP is located approximately 26 mi 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 emplacement in the WIPP site stem from waste generated during the 1940s from the nation's nuclear weapons program at various DOE sites.

Wastes to be shipped to WIPP must first be characterized to identify any hazardous materials that might be present, in addition to the 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 (solidified sludges resulting from treatment of waste waters to recover TRU elements).

The purpose of the PDP for the RCRA Constituent Analysis of Solidified Wastes is to test laboratory performance in determining specified metals, semivolatile and volatile organic compounds (SVOCs and VOCs), and polychlorinated biphenyls (PCBs). Laboratory performance is demonstrated by the successful analysis of blind audit PDP samples.

For each testing cycle of the PDP, the ACL prepares approximately 1 kg each of unspiked cemented and uncemented simulated Type I solidified waste and determines the levels of metal analytes in this matrix. The waste matrix is then supplied to a contractor selected by the PDP coordinator for spiking with metal analytes and shipment to participating laboratories. The contractor also provides blind audit samples of SVOCs and VOCs to the laboratories participating in the PDP. During FY 1997, materials were supplied for two cycles of the PDP.

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

The ACL is involved with the preparation and distribution of performance demonstration samples of simulated headspace gas for the Waste Isolation Pilot Plant (WIPP). Every six months, the ACL acquires relevant gases from a specialty gas vendor and uses them to prepare standard gas mixtures containing known concentrations of various VOCs, hydrogen, and methane. These standard gas mixtures are metered through a heated, multiport manifold (constructed by ACL) into several SUMMA® canisters simultaneously, 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. The ACL is also responsible for the cleaning and inventory of all canisters used for the headspace gas performance demonstration program.

(7) Use of FT-Raman Spectroscopy for Screening of Semivolatile Organic Compounds in WIPP-Simulated Type I Solidified Waste Form (A. S. Boparai, D. Fimmen, K. J. Parish, and Y. Tsai)

Waste containing both radioactive and organic components (radioactive mixed waste), has been generated at DOE facilities during the last 50 years. The planned disposal site for this waste is the Waste Isolation Pilot Plant (WIPP) near Carlsbad, NM.

One major form of waste is Type I solidified sludge in the cemented and uncemented forms. Type I waste is sludge from wastewater treatment that consists of inorganic salts in a highly alkaline matrix which is potentially contaminated with acidic, basic, and neutral semivolatile organic compounds (SVOCs).

This year, the ACL initiated a project to evaluate the use of Fourier Transform Raman (FT-Raman) spectroscopy for rapid screening of radioactive samples of Type I solidified waste for the following WIPP semivolatile target analytes (following each analyte is the program-required quantitation limit for that analyte in ppm): o-cresol (40), m-cresol (40), p-cresol (40), 2,4-dinitrotoluene (2.6), hexachlorobenzene (2.6), nitrobenzene (40), pentachlorophenol (40), and pyridine (40). Our objective was to develop a method that required minimal sample preparation and identified samples containing high levels (parts per thousand, ppt) of WIPP-specified SVOCs. These samples could then be segregated and classified as containing high levels of SVOCs without extensive characterization.

Samples of simulated Type I sludge were prepared as the interfering matrix and sand was used as a noninterfering matrix. An FT-Raman analysis was performed using an In/Ga/As detector with a CaF_2 beamsplitter. Laser power was held constant at approximately 0.5 W. Without any preconcentration of analytes, a detection limit of 1 ppt was established in the noninterfering sand matrix for most of the WIPP SVOCs. In the simulated Type I sludge, however, detection limits were approximately 200 ppt. A report of this work will be prepared after verification of these preliminary results.

(8) 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, J. J. Marr, D. L. Bowers, C. S. Sabau, L. L. Smith, F. Markun, T. TenKate, L. B. TenKate, A. S. Boparai, D. V. Applegate, L. L. Chromizky, Y. Tsai, K. J. Parish, and M. J. Schipma)

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 (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. The ACL characterizes the composite samples by chemical analysis to permit evaluation of the waste relative to Hanford's waste acceptance criteria.

In this characterization, each composite sample (consisting of pieces from a collection of filters shredded and compacted into drums that are packed in a waste bin) is homogenized by manual cutting and then analyzed for metals, semivolatile organics, PCBs, and radionuclides. Samples found to contain high concentrations of heavy metals are also tested according to the Toxicity Characteristic Leaching Procedure to determine whether the metals they contain exceed regulatory limits for mobility under landfill conditions.

During FY 1997, the ACL characterized five composite HEPA filter samples. Each composite consisted of portions of approximately 48 filters. Thus, all filters replaced at ANL-East in FY 1997 (approximately 250 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 and lessens the waste storage necessary at ANL.

(9) Analysis of Process Liquors Used in Removing Zinc from Galvanized Steel (E. A. Huff, D. R. Huff, F. P. Smith, A. M. Essling, C. T. Snyder, 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 zinc-removal process, and also 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, as well as a method for determining the carbonate content of the caustic liquors and process sludges. The ACL's measurements provided not only data for process characterization and control but also information of environmental interest, including information about the chemical behavior of such toxic metal impurities as Pb, Cd, and Sb and about the composition of waste streams associated with the zinc-removal technology.

The Energy Systems team is also developing 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 such impurity elements 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.

(10) Analysis of Groundwater Samples to Monitor Chlorocarbon Removal (L. L. Chromizky)

For the past nine years, the ACL has participated in a collaborative effort with ANL's Environmental Research Division (K. Spokas, et al.) that involves monitoring the removal of contaminants (carbon tetrachloride and chloroform) from groundwater under a former grain storage facility operated by the U.S. Department of Agriculture in Waverly, NE. Contaminants in the Waverly aquifer are being removed by groundwater extraction and air stripping. Carbon tetrachloride contamination in the Waverly aquifer remained fairly constant through June 1997, at approximately 12 ppb, which is still above the Federal health standard for drinking water of 5 ppb. At that time the ACL's participation in the project ended. Future work may involve developing methods to determine VOCs in vegetation and special sampling for the Waverly project. In FY 1997 the ACL analyzed more than 80 samples for this project. All samples were analyzed within the allowed holding time.

(11) Characterization of Phosphate-Ceramic-Stabilized Hazardous Wastes (L. B. TenKate, D. R. Huff, E. A. Huff, J. S. Crain, T. TenKate, F. P. Smith, A. M. Essling, L. L. Smith, F. Markun, K. J. Parish, J. J. Marr, D. L. Bowers, and D. G. Graczyk)

Researchers in the ANL Energy Technology Division (A. Wagh, D. Singh, et al.) are developing a stabilization process that converts solid waste materials into chemically bonded phosphate ceramics. These ceramics 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 1997, 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. In this work, the Toxicity Characteristic Leaching Procedure (TCLP), as described in U.S. EPA Method 1311, and other standard leaching tests were applied.

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 cold vapor atomic absorption, concentrations have been obtained for Pb, Cd, Cr, Ni, As, Ba, Se, Ag, Mo, Fe, Cs, Ru, Sr, La, Nd, Y, Hg, Ce (a nonradioactive simulant for plutonium), and Re (a nonradioactive simulant for technetium). Concentrations of the radioactive isotopes ^{137}Cs, ^{238}U, ^{235}U, and ^{241}Am were determined in actual waste samples by gamma spectroscopy, and ^{99}Tc was measured by application of a solid-phase extraction technology (Empore™ Disks) that ACL analysts helped develop for commercialization. Ion chromatography was used to determine the concentrations of phosphate, nitrate, sulfate, and chloride leached from the ceramic matrix.

This year, TCLP tests were performed on samples from ceramic stabilization of mercury from fluorescent light bulbs removed from hot-cell fixtures, lead in soil from an abandoned firing range, and hazardous metals in solid and liquid radioactive waste from experimental processes. Interpretation of the leaching data has allowed Energy Technology Division researchers to evaluate the effectiveness of their technology in imparting resistance to leaching of metals and radionuclides and, in some cases, has also helped them to deduce the mechanism by which the stabilization takes place.

(12) Support to Counternarcotics Efforts - Analysis of British Money (J. C. Demirgian)

As part of collaborative work with the Houston Advanced Research Center, the ACL was asked to evaluate and to recommend methods for the destruction of large quantities of seized illicit substances. The major criteria used in evaluation and selection of the recommended technologies were safety of Drug Enforcement Agency personnel, security of materials, cost of acquisition and operation of destruction technology, and compliance with the Environmental Protection Agency's air emissions regulations. The ACL's evaluation indicated that medical waste incinerators had advantages over rotary kiln incinerators in meeting most of the evaluation criteria. This work will continue in FY 1998 with the permitting of a facility to thermally destroy illicit substances.

During FY 1997, our work on transfer of cocaine from U. S. currency to people was widely publicized in the U.S. and overseas press (print, radio, and television). We also briefly examined British currency and determined that the fibers used in U.S. and British currencies are different; consequently, they retain cocaine differently. The two samples of British currency that we studied were not cocaine-contaminated. Future work will expand on our efforts to characterize money samples from large cities. The question was often raised whether the same level of contamination exists in samples of currency from small towns. Therefore, two small towns will be studied. One will be near an interstate highway and one will be in an isolated area. The objective is to see if currency from rural areas is as contaminated as that found in large metropolitan areas.

(13) Development of a Rapid Water Sampling and Analysis System Incorporating Empore™ Rad Disks for Field Analyses (L. L. Smith, L. L. Chromizky, and L. B. TenKate)

Since 1994, ANL and the 3M Company have been collaborating to develop Empore™ Rad Disks, which are based on a new solid-phase extraction technology for radiochemical analysis. These disks are thin membranes that are loaded with element-selective particles embedded in stable, inert polytetrafluoroethylene fibrils. The Empore™ Rad Disks selectively target radioisotopes from aqueous samples, even in the presence of very large concentrations of competing ions. To date, disks have been developed for Tc, Sr, and Ra.

In 1997, the ACL, in collaboration with the Environmental Research Division (J. H. Aldstadt and K. A. Orlandini) and 3M, used the disks for Tc separation to evaluate the field performance of a rapid water sampling and analysis system developed by the ANL-3M team. The sampling system was designed to be easily portable and to work in remote locations. It was designed for large sample sizes (>4 L), for fast flow rates (>200 mL/min), and to allow for prefiltration as required. The system was successfully tested on a variety of natural and process waters at several DOE sites. Results from the membrane-based system agreed favorably with the analytical results for Tc obtained from traditional methods.

The rapid water sampling and analysis system using these solid-phase extraction disks offers several distinct advantages: (1) extremely large specimens (1-12 L) can easily be collected, so that very low levels of detection can be obtained; (2) the sample preparation normally performed by the chemists in fixed laboratories can now be accomplished at the time of sampling by the field personnel so that overall sample preparation time can be reduced by a factor of 10; (3) bulk shipping and storage can be reduced by a factor ranging from 300 to 4,000; by using the field sampler, one avoids the need to ship and store large quantities of raw water; only the small disks require shipment to the laboratory; and (4) the membranes lend themselves to field analysis. A portable beta spectrometer based upon solid scintillation and developed by K. A. Orlandini, was demonstrated in this project to allow counting of the radioactivity in these membranes in the field.

(14) Field Sampling Assistance to Environmental Management Operations (D. V. Applegate)

The ACL is assisting Environmental Management Operations (EMO) with field sampling at potentially hazardous sites within the ANL-East boundaries. These sites include landfills, leachate seeps, and groundwater wells. Sediments, water samples, and contaminated soils are being collected. The ACL provides support for sampling of soil borings, which EMO collects by using a 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, ensure high-quality samples. The ACL has also assisted in the operation of a portable field-screening instrument that detects volatile organic compounds (VOCs). This portable instrument provides valuable real-time sample information to guide sampling efforts and to alert the sampling team to elevated levels of VOCs, which are a possible safety concern.

(15) Analysis of Scale and Filter Residues in Cooling-Water Systems at the Advanced Photon Source (P. L. Johnson, B. S. Tani, A. M. Essling, D. G. Graczyk, Y. Tsai, R. W. Bane, E. A. Huff, D. R. Huff, J. J. Marr, and M. J. Schipma)

Operation of the synchrotron particle accelerators and storage ring at the Advanced Photon Source (APS) requires substantial volumes of high-quality cooling water to regulate the heat load from power supplies, magnets, and other components. The ACL has provided occasional support to the team of engineers responsible for maintaining the APS cooling water system by helping to identify deposits the engineers have found on some system components and by making measurements to assess the nature and extent of corrosion taking place in the system.

This year we examined several fittings associated with hoses on inlet and outlet lines from selected components in the storage ring. Two types of deposit were present on these fittings. One was a bright yellow film that was identified by FTIR spectroscopy as an epoxy resin. The other was a dark deposit identified as cuprite (Cu_2O) by XRD analysis and scanning electron microscopy/energy dispersive spectroscopy. We also examined a number of filters from various locations in the cooling water system and determined the nature and quantity of particulate matter they had collected. Optical and scanning-electron microscopy showed that many of the filters contained tiny particles of copper metal (roughly 10 µm diameter). By burning off the polypropylene filter medium, dissolving the ash in mineral acids, and analyzing the solution by ICP/AES, we determined that some filters contained as much as 0.25 g of copper. We were unable to pinpoint the source of the copper particles through measurements of the copper concentration in several sets of water samples and in residues obtained by filtering small volumes (1 L) of water because the concentrations were extremely low in all cases. Apparently, the copper loading on the filters resulted from accumulation of a small number of particles per unit volume as thousands of gallons of water passed through each filter.

The APS engineers have attributed the occurrence of these copper particles to corrosion of system components. Although the rate of corrosion is not particularly alarming, the engineers are taking steps to eliminate its occurrence, so it will not shorten the life of components in the APS systems.

(16) Chemical Analysis for Studies to Develop Treatment for Aluminum-Based Spent Fuels (A. M. Essling, F. P. Smith, E. A. Huff, D. R. Huff, C. S. Sabau, D. L. Bowers, P. L. Johnson, B. S. Tani, and D. G. Graczyk)

The U.S. Department of Energy expects that 128 metric tons of spent aluminum-matrix reactor fuel will be produced from U.S. and foreign research reactors over the next 40 years and has been evaluating alternative means of dealing with the fuel when processing in the canyons at the Savannah River Site is discontinued as planned in 2005. As a backup option to direct disposal of the fuel, DOE advisors recommended an electrometallurgical process for treating the aluminum-based fuels, which is being investigated by researchers in CMT (E. C. Gay, J. L. Willit, S. A. Slater, et al.). This process is especially attractive because it has the potential to separate and recover the aluminum and uranium components from the fuel and thereby greatly reduce the amount of high-level waste requiring disposal.

Work by the CMT team is currently focused on the process step involving separation and recovery of aluminum from the fuel by electrotransport in a molten-fluoride-salt electrolyte. The ACL has provided a variety of chemical analyses in support of these studies. Measurements of the composition of electrolyte samples taken over a period of time helped to demonstrate that the electrolyte mixture is stable. Analysis of product deposits from electrotransport tests helped demonstrate that aluminum recovery is viable. Finally, measurements of the component elements in simulated fuel materials and treatment residues helped confirm that the process can retain uranium in its desired location while the aluminum is removed and recovered.

The fluoride matrix in which the aluminum electrotransport process is carried out has provided interesting challenges to the analysts supporting this work. Some of the fluoride components are exceptionally difficult to dissolve and require decomposition by fusion techniques or other means. As more work is done, we anticipate that better methods will evolve for analyzing samples related to the process.

(17) Support for ANL Programs Using X-Ray Diffraction and SAC Activity (P. L. Johnson and B. S. Tani)

The ACL continued to provide X-ray diffraction (XRD) support to programs in ANL's Chemical Technology, Chemistry, Energy Technology, Energy Systems, Technology Development, Reactor Engineering, Accelerator Systems, and Experimental Facilities Divisions. Support was also provided to the Wisconsin Electric Power Company, Fernald Environmental Management Project, and Engineering Systems, Inc. Examples of systems studied by using XRD include

- Zeolite/sodalite waste forms which may contain compounds such as UO_2;
- Carbon deposited on silicon by researchers — XRD analyses can show that the carbon phase is diamond;
- Compounds containing La, Sr, Mn, Mg, Ga, and O -- XRD analyses can detect phases that are present;
- Compounds that are present in waste silos; and
- Compounds present in reactors that cause pumps to fail.

We have worked with radioactive samples as well as nonradioactive samples. The current powder diffraction files from the International Centre for Diffraction Data have been purchased by the ACL so our search and match software will include all available data. We can provide identification of phases readily using this software.

P. L. Johnson has served on the Chemical Technology Division's Synchrotron Advisory Committee. This committee has hired a postdoctoral research associate (Jeremy Kropf, University of Notre Dame) to assist CMT staff in conducting synchrotron experiments. Requirements for working at the Advanced Photon Source have been discussed and training for work on our sector has been taken by several CMT staff. Transmission X-ray absorption fine structure experiments will be carried out in early 1998. When the table for the Huber goniometer is received and installed, diffraction and scattering experiments will be conducted.

(18) Purification of Manganese-54 for Positron Decay Branch Measurement (D. L. Bowers and D. G. Graczyk)

The ACL was asked by researchers in the Physics Division (I. Ahmad and A. H. Wuosmaa) to remove trace amounts of sodium and zinc radioisotopes from a sample of ^{54}Mn intended for use in an experiment to measure the positron decay branch in ^{54}Mn. The sodium and zinc radionuclides emit particles that can interfere with the measurement of the ^{54}Mn positrons. This interference is particularly detrimental because the positron decay branch for ^{54}Mn is expected to be on the order of 1 x 10^{-9} and, consequently, effective removal of the zinc and sodium was mandatory.

The ACL devised a separation scheme in which the zinc was separated from manganese by anion exchange in 2 M HCl and the sodium was removed by cation exchange in 0.5 M HCl. Using this scheme, 2 mCi of ^{54}Mn were purified, and from it a 660 μCi source was prepared for placement in the APEX positron-electron spectrometer in the ATLAS linear accelerator facility. The spectrum of ^{54}Mn decay from the ACL-purified source was acquired over a period of two weeks. The physicists have reported that the experiment was successful, and their analysis of the data is under way.

(19) Preparation of Osmium Targets for Photon Excitation Studies (F. Markun)

In a collaboration with researchers in the Physics Division (I. Ahmad and D. S. Gemmell), the ACL prepared an osmium target for photon excitation studies by electroplating osmium metal onto a copper backing. The resulting metal planchet was then used as a target for photon excitation studies in which the high flux of photons from the APS would excite osmium nuclei by a process called NEET (Nuclear Excitation by Electronic Transition). Initially, the physicists requested that the osmium be plated on a copper block, but after some discussion, it was decided that the backing could be a 3/4-in., 1-mm-thick copper planchet compatible with ACL's existing electroplating setup. A procedure for electroplating osmium is available in the literature, but direct application of the procedure did not work. It was determined that successful electroplating is greatly dependent on osmium ion concentration in the plating solution. If the concentration of osmium ion was greater than 0.006 M, the osmium would precipitate rather than plate out. By modifying the procedure so that the concentration of osmium in the plating solution was <0.006 M, the ACL was able to produce a number of osmium targets ranging in thickness from 3 mg/cm^2 to 20 mg/cm^2 on both copper and gold planchets. After an irradiation of approximately 12 hours in the APS, an osmium target was counted by the physicists with a germanium detector to measure osmium X-rays. The use of these targets led to a successful experiment because evidence for the 5.7-hour half life excited-state isomer was observed.

(20) The Department of Energy's Integrated Performance Evaluation Program (P. C. Lindahl, W. E. Streets, J. J. Marr, and K. J. Parish)

In collaboration with the DOE's Radiological and Environmental Sciences Laboratory (RESL), the ACL is developing and piloting a comprehensive Integrated Performance Evaluation Program (IPEP) for the DOE's Office of Environmental Management (EM) as part of its National Analytical Services Program (NASP). The NASP is managed by EM's Analytical Services Division (ASD). The IPEP is designed to provide information on the quality of radiological and nonradiological analysis data being produced by all analytical chemistry laboratories that provide DOE and its contractors with data on environmental restoration and waste management samples. The ACL has been assisting in developing program requirements and implementation strategies, especially in the nonradiological portions of the program. The CMT's Computer, Applications, Network, and Security Group (A. E. Scandora, R. R. Krol, and J. M. Copple) is assisting the ACL in the development of strategies and systems for handling large data sets and for compiling and analyzing data from performance evaluation (PE) program studies.

The Interagency Agreement (IAG) established for the Program, between ANL and the Environmental Protection Agency's (EPA's) Region V, designating the IPEP as a pseudo-EPA Region, was renewed for FY 1997. This IAG allows the IPEP to nominate laboratories performing analyses for EM into the Water Supply (WS) and Water Pollution (WP) PE Program Studies sponsored by the EPA's National Exposure Research Laboratory, Cincinnati, OH (NERL-Ci), and to access relevant information about laboratories from these programs. Historical and current data from these PE programs are now routinely transferred electronically from the EPA's database at Research Triangle Park, NC, to the IPEP database at ANL. Changes in EPA's methods of data collection and dissemination have required that IPEP adapt its database formats. Historical and current data from the DOE's PE Program Studies, the Environmental Measurements Laboratory (EML) Quality Assessment Program (QAP), and the RESL Mixed Analyte Performance Evaluation Program (MAPEP), are also routinely transferred into the IPEP database.

Pilot studies for the production and evaluation of IPEP reports have been conducted at DOE's Idaho Operations Office and the Idaho National Engineering and Environmental Laboratory (INEEL) Sample Management Office (SMO), and at DOE's Albuquerque Operations Office (AL) and SMOs of several of the management and operations (M&O) contractors that report through AL. These pilot studies were designed to provide IPEP developers with an opportunity to test the feasibility of proposed operating mechanisms and to allow report users to have direct input on the report formats, technical content, and other needs during the development stage prior to implementation. Before the pilot studies began, information was collected from the SMO at INEEL and the SMOs of the M&O contractor for AL to determine which analytical laboratories were performing analyses for the EM programs at the various DOE sites. This information was collated with the IPEP data collected from the various PE programs to determine whether a laboratory was participating in the appropriate PE programs for the analytical work it was performing in support of EM programs and projects. Customized reports were generated for the SMOs at INEEL and AL. These reports contained information about contracted analytical laboratories in selected EPA and DOE PE program studies and used the proposed evaluation procedures previously developed for the IPEP. In addition to hard-copy reports, electronic versions of the IPEP reports are being developed to test various means of distributing the information to the users.

A world wide web server (http://www.cont.anl.gov/ipep/ipep.html) has been established with hyperlinks to ANL's Chemical Technology Division and Analytical Chemistry Laboratory, and also to EM ASD Internet servers. General information about DOE's IPEP, including contact names, PE program study distribution schedules, and hyperlinks to other servers of interest, is available by accessing the server; a password-protected section is used to post results for PE program studies. The server will accommodate both graphical and nongraphical browsers.

(21) Corrections for Spectral Interference in the Determination of Arsenic, Selenium, and Vanadium Using ICP/MS (K. J. Parish, J. S. Crain, A. Bangalore, J. T. Kiely, and S. E. Carpenter)

The ACL has procedures for the determination of heavy elements in a variety of solid and liquid media using inductively coupled plasma mass spectrometry (ICP/MS). However, for elements with atomic numbers less than 36, spectral interferences can be severe. Under a FY 1997 Support Development Funds Project, the ACL has developed and tested the validity of a computer program that will simultaneously correct for many interferences in a single mass spectrum. Current research involved the correction procedures for arsenic, selenium, and vanadium in a known interference matrix. These interference correction procedures will allow the accurate determination of interference-prone elements by ICP/MS, instead of by more mature analytical techniques that are either more labor-intensive (graphite furnace atomic absorption spectrophotometry) or have higher detection limits (inductively coupled plasma atomic emission spectroscopy). Measurement of these elements by ICP/MS will therefore result in lower detection limits and cost savings for ACL clients.

(22) Determination of Water in Solids by Karl Fischer Titration

(D. G. Graczyk and A. M. Essling)

The ACL uses a semiautomated apparatus based on the Karl Fischer Titration (KFT) method to determine the concentration of water in organic solvents and in materials that are readily dissolved in anhydrous methanol. On occasion, the need has also arisen to measure the water content of solid materials that are insoluble in methanol and do not give up their water when added to the solvent in the titration system. One such solid is the lithium chloride/potassium chloride eutectic salt used as the electrolyte in electrometallurgical processes under development in ANL's Chemical Technology Division. To measure the water content of LiCl/KCl samples, the ACL modified the Karl Fischer titration vessel; the vessel can now be interfaced with a pyrolysis furnace that allows the solid to be heated in a flowing stream of inert gas (nitrogen) which carries any water released by the sample into the KFT solvent for continuous measurement.

The pyrolysis attachment consists of a fused silica reaction tube positioned in an electrically heated cylindrical furnace capable of maintaining a temperature of up to 1000°C. Provisions were made to allow introduction of a platinum boat containing the sample to be analyzed and to control the carrier gas flow. Complete recovery of water present in LiCl/KCl salt was obtained for samples as large as 3 g. The system was operated at 700°C. This new capability will allow the ACL to determine water in many types of samples that could not be accommodated previously, including hydrated salts, moist solids, and zeolites. Uncertainty in the determination is estimated to be ±2% relative; as little as 0.3 mg of water in the subsample analyzed can be detected.

(23) Performance Testing of Alpha Gamma Hot Cell Facility Clay Absorbent

(D. G. Graczyk and P. C. Lindahl)

A process deficiency was identified by the Idaho National Engineering and Environmental Laboratory (INEEL) Waste Management Compliance Team (WMCT) during an audit of ANL's waste-handling procedures related to the characterization of remote-handled transuranic (RH-TRU) waste destined for disposal at the INEEL. This process deficiency was related to the lack of evidence demonstrating the satisfactory performance of a pelletized clay absorbent used at ANL's Alpha Gamma Hot Cell Facility (AGHCF). The INEEL Reusable Property, Recyclable Materials, and Waste Acceptance Criteria (RRWAC) requires that all absorbents undergo pressure and temperature tests to ensure that the specific liquid absorbed will not be released when subjected to temperature cycles ranging from -50°F to 110°F and pressure cycles ranging from 24 to 26 inches of mercury, as might be encountered during handling and storage of the waste.

To test performance of the clay absorbent used at the AGHCF, a sample of the absorbent containing a surrogate neutralized acid solution was submitted to the ACL for pressure and temperature tests. Because protocols and equipment are not specified for conducting the required performance demonstration, the ACL was asked to develop and conduct a performance testing experiment that would demonstrate that the clay absorbent meets the RRWAC's performance requirement. The test matrix sequences were designed to simulate actual handling and storage conditions; therefore the sequences tested were pressure cycles (24 to 26 inches of mercury) at the high temperature (110°F) and at the low temperature (-50°F). A special apparatus was designed and constructed for the performance testing.

The results from these tests were submitted by ANL's Environmental Management Operations to the INEEL WMCT for review and were subsequently accepted as a satisfactory demonstration that the absorbent material met the RRWAC requirement. Successful closure of this and all other findings related to this audit made ANL a fully approved waste generator for RH-TRU at the INEEL.

(24) Determination of Minerals and Metals in Milk (L. B. TenKate, A. M. Essling, E. A. Huff, D. R. Huff, J. S. Crain, and D. G. Graczyk)

In 1993, Argonne scientists successfully tested a magnetic separation process (MAG*SEP™) developed and patented by Bradtec Ltd., a British company, for removing radioactive ^{137}Cs from milk produced in the Ukraine. The milk is contaminated above safe drinking levels as a result of the 1986 accident at the Chernobyl nuclear power plant.

This year, as a continuation of this work, researchers in the Energy Systems Division (L. Shem et al.) investigated the effects of the cesium removal process on levels of beneficial nutrient minerals (calcium, potassium, iron, phosphorus, etc.) as well as on levels of heavy-metal contaminants in the milk. In support of this investigation, the ACL established a methodology to measure the mineral elements and contaminant metals in samples of raw and processed (pasteurized and homogenized) milk before and after cesium removal treatment. For these measurements, we implemented a microwave-assisted acid digestion procedure for preparing the milk samples based on U.S. EPA Method 3051. This procedure involved treating 5 g of milk with 10 mL of concentrated nitric acid in a closed-vessel system. This procedure produced a digestate suitable for ICP/AES measurement of Al, As, Ba, Be, Ca, Cd, Cr, Cu, Fe, K, Na, P, Mg, Mn, Pb, Sn, Sr, and Zn; ICP/mass spectrometer (ICP/MS) measurement of As, Cd, Sn, Cs, and Pb; and cold vapor atomic absorption (CVAA) measurement of mercury.

The preparation and analysis methods were demonstrated to visiting Ukrainian scientists who came to observe the treatment studies. The data from these measurements will help determine whether removing the radioactive cesium might lower the nutritional value of the milk or introduce undesirable contaminants into it. Early results showed no adverse effects.

(25) Chemical Analysis of Lithium Aluminate for the Tritium Target Qualification Project

(D. G. Graczyk, A. M. Essling, R. W. Bane, C. T. Snyder, and D. R. Huff)

In its Tritium Target Qualification Project (TTQP), Battelle Pacific Northwest National Laboratory (PNNL) was charged by the Department of Energy with coordinating the manufacture of ^{6}Li-enriched lithium aluminate powders and pellets for use as tritium production targets. In support of this effort, ANL's Energy Technology Division (R. Poeppel, J. Picciolo, H. Cramer) prepared batches of the $LiAlO_2$ powder, which were subsequently sintered into ceramic pellets by a commercial vendor. The pellets were ultimately to be incorporated into a test assembly (the Lead Test Assembly, LTA) that was scheduled for placement in a nuclear reactor in September 1997.

The ACL became involved in the TTQP when we were asked to provide chemical analysis of developmental batches of the powder and pellet materials to determine whether the products met program specifications. The ACL was in a unique position to provide these measurements because we had established capabilities for analyzing these extremely refractory materials with high precision and accuracy when we supported early development of the lithium aluminate target matrix (1983-

1990). By far the most challenging aspect of these analyses is dissolving the materials to prepare them for lithium and aluminum assay. A sealed-tube procedure using concentrated hydrochloric acid at 300°C is usually required, although we also had limited success with a high-pressure, microwave-assisted dissolution method. For the powders and pellets, we determined the isotopic composition of lithium in each material by thermal ionization mass spectrometry (TIMS), assayed lithium by isotope dilution and aluminum by 8-hydroxyquinolate gravimetry, measured carbonate impurity by carbon dioxide evolution, and measured leachable chloride by ion chromatography.

In addition to supporting the process-development activities for the TTQP, the ACL also participated in a qualification exercise conducted by the Idaho National Engineering and Environmental Laboratory (INEEL) as part of a project to identify commercial laboratories that could perform the needed measurements. This exercise required the rapid analysis, in duplicate, of seven prototype production pellets. Our results were pooled with similar data from other laboratories, including the National Institute of Standards and Technology, to evaluate method and laboratory performance in terms of precision and accuracy.

Based on ACL's historical experience with $LiAlO_2$ analysis, our performance in analyzing the developmental and prototype production materials, and our readiness to provide high-quality data with short turnaround time, the ACL was selected to provide chemical analysis data for certification of two production lots of pellets incorporated into the LTA. To meet the schedule needs of the TTQP, we undertook an intensive campaign to complete all measurements on the two pellet lots in less than four weeks. This successful effort made the data available in time for a planned review of the LTA by the Nuclear Regulatory Commission. The LTA was approved for use and shipped from PNNL on schedule during August 1997.

(26) Analysis of Respirator Cartridge Components for Waste Hazards Classification

(A. M. Essling, E. A. Huff, D. R. Huff, and D. G. Graczyk)

A type of respirator cartridge commonly used at ANL was designated a regulated waste by the disposal facility to which ANL sends much of its low-level radioactive waste. This designation arose from a hazards-assessment analysis that specified the nominal cartridge composition to include 24 wt% potassium carbonate, thereby exceeding the regulated-waste threshold value of 10 wt% potassium carbonate. As a regulated waste, the respirator cartridges were prohibited from being present in waste shipped to the disposal facility. Consequently, ANL was required to sort and repackage any waste that contained such cartridges and to consider finding alternative cartridges for respirator use.

Unable to confirm the nominal reference composition with the cartridge manufacturer, the Decontamination and Decommissioning Group of ANL's Technology Development Division submitted a cartridge to the ACL for a comprehensive determination of the physical and chemical characteristics of its components. The results of the ACL's measurements indicated that the cartridge contained no regulated materials other than potassium carbonate and that the potassium carbonate concentration in any individual component was less than 8 wt%. With this information, ANL asked the disposal facility to review their hazards assessment. The review resulted in a determination that the cartridges were, in fact, not regulated. Thus, ANL was able to avoid the cost and personnel exposure associated with sorting and repackaging waste that contained used cartridges and also retained the ability to use this type of cartridge in its operations.

(27) Characterization of Materials from Fernald Silos (P. L. Johnson, B. S. Tani, A. M. Essling, F. P. Smith, E. A. Huff, D. R. Huff, and D. G. Graczyk)

Fluor Daniel Fernald is currently evaluating options for the remediation of material stored in Silos 1 and 2 of Operable Unit 4 at the Fernald Environmental Management Project site in Ohio. The material in these silos consists predominantly of residues from pitchblende ore processed to extract uranium. To aid in selecting among available treatment options for remediating the silo contents, Fluor Daniel Fernald requested that the ACL analyze material from each of the two silos to identify the primary chemical compounds present.

In this effort, we determined the elemental composition of three subportions of each silo material and examined corresponding subsamples by X-ray diffraction (XRD) to provide information on specific compounds present. In the elemental analysis, each subsample was analyzed to determine 33 elements, including carbon, sulfur, phosphorus, and an assortment of metals. Of these, 31 elements were determined by ICP/AES after total dissolution of each sample using a lithium tetraborate fusion. Carbon and sulfur were measured with commercial combustion analyzers. We also determined the distribution of carbon between organic and inorganic (carbonate) forms in selected samples. Ultimately, the information from the elemental determinations and XRD patterns was combined and interpreted to allow assignment of the major components in each material to specific compounds. This analysis resulted in our assigning approximately 86% of the Silo 1 material's mass to the compounds SiO_2 (65%), $PbCO_3$ (11.8%), $BaSO_4$ (7.7%), $PbSO_4$ (1.2%), and $CaCO_3$ (0.5%). In the Silo 2 material, an average of 81% of the material's mass was assigned to $(Ca,Mg)CO_3$ (38%), SiO_2 (31%), Fe_2O_3 (6.4%), $PbCO_3$ (3.5%), and $BaSO_4$ (2.6%). Fluor Daniel Fernald is evaluating these results with regard to potential implications for various processes to stabilize the silo materials for disposal.

(28) Field Portable X-ray Fluorescence Spectrometer Demonstration for the CP-5 Large-Scale Demonstration Project (C. T. Snyder and D. G. Graczyk)

The Large-Scale Demonstration Project (LSDP) at ANL's Chicago Pile-5 (CP-5) reactor is testing innovative technologies that offer potential benefits to the decontamination and decommissioning of aging nuclear reactors and other facilities. In collaboration with researchers from the Technology Development, Environmental Research, and Energy Systems Divisions (R. Rose, TD; M. Erickson, ER; J. Schneider and M. Quinn, ES), ACL staff participated in a demonstration of two commercial field portable X-ray fluorescence (FPXRF) spectrometers for field screening of heavy metal contaminants on surfaces and on used high-efficiency particulate air (HEPA) filters removed from service at the demonstration facility. The FPXRF systems utilize sealed radioactive sources to provide the exciting radiation needed to induce emission of fluorescent X-rays. The instrument includes a device to expose a sample to the excitation source, a detector and energy-dispersive analyzer to acquire and record the fluorescent X-ray energy spectrum, and a data processor to convert the spectral data to elemental concentrations.

In the LSDP demonstration, measurements were made with two units from TN Spectrace (Fort Collins, CO). These units were the TN Lead Analyzer (designed to analyze for lead in matrices such as soil, paint, surface dust, or air filters) and the TN Spectrace 9000 (designed to determine the elemental composition of a broad range of materials for environmental applications, industrial quality control, or other uses). The demonstration activities showed that, although the FPXRF technology cannot completely displace the baseline technology of collecting samples for analysis by regulator-approved laboratory methods, these analyzers offer considerable advantages in cost and time when used as a screening tool to identify areas of potential concern, to define dimensions of a contaminated area, to track progress of remediation activities, or to guide waste management strategies. Results of the LSDP demonstration have been written up for publication as an Innovative Technology Summary Report to be issued by the U.S. Department of Energy, Office of Environmental Management.

(29) Performance Characterization of a New ICP/AES System (E. A. Huff, D. R. Huff, L. B. TenKate, and K. J. Parish)

The ACL, in collaboration with the CMT's Computer, Applications, Network, and Security Group (J. M. Copple), has installed and tested the performance of a new inductively coupled plasma atomic emission system (ICP/AES). This equipment incorporates a 47-channel polychromator for simultaneous analyses and a computer-controlled scanning monochromator for sequential measurements. Some of the performance-related improvements in the new instrument are the following:

- Axial observation of the plasma excitation zone, which increases the viewing volume of the analyte with resultant enhancement in sensitivity.
- A higher radio-frequency generator frequency (40 MHz) that provides lower excitation temperatures and thus decreases line intensities and emission continuum; despite the lower intensities, signal-to-noise ratios are improved and detection limits are lowered.
- Enhanced photomultiplier shielding, which minimizes spectral crosstalk between polychromator channels.

These improvements in optical, electronic, and plasma excitation components have provided significantly higher sensitivities without any sacrifice in analytical accuracy and precision. Data obtained so far indicate that, compared to our older ICP/AES system, detection limits have been lowered by about an order of magnitude. Results obtained with our new system showed good agreement with data generated by graphite-furnace atomic absorption (GFAA) and inductively coupled plasma mass spectrometry (ICP/MS) on several quality control sample sets. Work is currently in progress on the generation of client-friendly analytical reports that access data on-line.

(30) FTIR Analysis of Oils (Y. Tsai and K. J. Parish)

Oil samples from Fermi National Accelerator Laboratory (FNAL) were submitted to the ACL for identification by Fourier transform infrared (FTIR)-microscopy analysis. The FTIR spectra of the sample oils were collected, and they matched the library spectrum of mineral oil. Since FNAL was interested in whether silicone oil was present in the samples, another spectrum of each sample was collected, this time using a thicker film of the sample to increase the signal of any low-level components. The spectrum of each sample was scaled to emphasize the absorption bands in the 1300-800 cm^{-1} range, because mineral oil absorption bands do not occur in this region but major silicone oil absorption bands do occur. The spectrum in this region for each sample was carefully examined against the library spectrum of silicone oil. The presence of silicone oil in one of the Fermi sample spectra was noted. To determine the concentration, an FTIR spectrum was collected for a mixture of known concentration of silicone oil in mineral oil. By comparing the relative heights of the silicone oil absorption bands to the mineral oil absorption bands, the concentration of silicone oil in the Fermi sample was estimated to be low (~1%).

(31) Determination of Inorganic and Organic Compounds Formed in Simulated Arcing of Cables

(D. G. Graczyk, A. S. Boparai, C. T. Snyder. Y. Tsai, D. V. Applegate, and P. L. Johnson)

A set of eleven gas samples was submitted by an engineering company for determination of certain inorganic and organic compounds formed as a result of simulated arcing of electrical cables. Each of the gas samples was contained in a glass sample bulb. Analysis of each gas was performed in several stages. First, each gas was analyzed by gas mass spectrometry for determination of hydrogen, methane, ethane, oxygen, argon, carbon dioxide, and propane. Values were also obtained for nitrogen, but these values were subject to potential interference by carbon monoxide, which has ions that coincide with the ions from nitrogen at mass 28 and mass 14. Because of these coincident ions and interference on the carbon monoxide ion at mass 12 by hydrocarbons, we were unable to use the gas mass spectrometer to directly determine carbon monoxide. As described below, carbon monoxide was subsequently measured by a gas chromatographic method, and the information from that measurement was used to refine the nitrogen results from the gas mass spectrometer.

In the second step, low-molecular-weight organic compounds present in each gas sample were determined by gas chromatography/mass spectrometry (GC/MS) system fitted with a gas-handling injection valve to permit introduction of a measured amount of gas sample for analysis.

We determined the response factors (area counts/ng) for carbon monoxide, ethene, ethyne, and ethane by using commercial gas standards that were available in our laboratories. Response factors determined from our previous work with headspace gas analysis were utilized for quantitation of benzene and toluene. As for the other compounds detected in the submitted samples, the response factor for ethene was used for quantitation of all olefinic compounds, ethyne for all alkynes, and ethane for all alkanes. Therefore, quantification of organic compounds had to be considered semiquantitative, except for ethene, ethyne, and ethane, and also carbon monoxide.

The third step in analyzing each gas sample involved a gas chromatographic analysis to determine carbon monoxide. Under the conditions used (column oven temperature of -30°C), we determined that carbon monoxide and nitrogen coelute. Since both compounds have a base peak at mass 28, quantitation of carbon monoxide could not be performed in the presence of nitrogen using the m/e 28 ion. However, carbon monoxide has a peak at m/e 12 which is not present in the mass spectrum of nitrogen. It was determined that methane, which also has a peak at m/e 12, was separated from the carbon monoxide/nitrogen peaks. Therefore, carbon monoxide was determined using the peak at m/e 12.

In all, 56 different organic compounds were identified in one or more of the individual gas samples. The predominant species were light, unsaturated hydrocarbons, especially ethene and ethyne. Besides air components, inorganic gases present consisted of hydrogen, carbon monoxide (both at fairly high concentration in some samples), and carbon dioxide. Hydrogen cyanide was detected in four of the eleven gas samples.

(32) <u>Data Review Assistance</u> (D. V. Applegate, L. L. Chromizky and L. B. TenKate)

The ACL has assisted ANL's Environmental Management Operations in preparing a number of reports to the Illinois Environmental Protection Agency concerning potentially hazardous waste sites within the ANL-E boundary. Each of the reports included data from thousands of individual analyses. The ACL assisted in organizing the data and in checking the data for completeness. The

ACL also provided expertise in interpreting some of the data results and in evaluating the overall data quality. This information was used to assess the usefulness of the data and ultimately, to help summarize the large amount of information by omitting all nonessential data.

(33) Chemometrics - Sensor Algorithm Generation Environment (SAGE) (S. Carpenter, A. S. Bangalore, S. Crofts, M. Poulos, and A. S. Boparai)

The Sensor Algorithm Generation Environment (SAGE) is a graphical software "workbench" that consists of signal-processing algorithms coded, ported, or interfaced into a single environment. By using SAGE, the developer of any type of sensor (optical, chemical, electrochemical, nuclear, etc.) can selectively combine diverse procedures for digital filtering, pattern recognition, and multivariate calibration to produce an optimized algorithm for automated qualitative and/or quantitative analysis. This optimized SAGE algorithm can then be implemented onboard the sensor by using software or appropriate hardware to perform automated data analysis in real time.

Another goal in establishing the SAGE is to develop novel algorithms for creating smart chemical sensors. The term "smart" means that the sensor can (1) interpret multivariate data in real time; (2) perform data reduction and decision making; (3) communicate processed results to a user, multisensor data fusion system, or process control loop; and (4) alter its own operation based on the nature of these results. By developing automated numerical procedures that mimic the analytical reasoning and data analysis skills of a human expert, smart sensor algorithms can be produced.

The SAGE consists of three modules: digital filter, pattern recognition, and multivariate. A submodule, which will contain code for analysis of hyperspectral images, has also been added. During FY 1997, the digital filter and pattern recognition modules were essentially completed. In addition to standard chemometric techniques, neural network analysis has been applied for multispectral and hyperspectral image analysis. The original SAGE code, which began in a UNIX environment with modules written in C/C+++ and FORTRAN, was rewritten and implemented in the MathWorks MATLAB environment version 5. This environment is portable and operates in UNIX (SGI IRIX) and in MS Win95/NT. We are currently seeking data sets suitable for input to test the capabilities of SAGE. During FY 1997, M. Vogt and C. Klaus of Energy Systems Division also worked on this project and their contributions are acknowledged.

(34) Effect of Spectral Resolution on Pattern Recognition Results (A. Bangalore)

The Sensor Algorithm Generation Environment (SAGE) has been modified to incorporate the results of several research projects that were designed to identify more robust and efficient data analysis algorithms. Prior research studies included determining the advantages of using two bandpass filters as opposed to a single bandpass filter in preprocessing, using wavelet analysis, and using the Adaptive Resonance Theory - Version 2A (ART2A) neural network for automated target recognition.

We are currently investigating the effect of spectral resolution on pattern recognition. This study will establish the protocol for selecting the optimal resolution at which remote-sensing FTIR data need to be collected to obtain good pattern recognition without sacrificing the information content of the signal. With optimized resolution, disk space for storing high-resolution data that do not contribute to improved pattern recognition can be reduced. Collection of low-resolution data that give poor quality results can also be avoided.

The target analyte in this study is diisopropyl methyl phosphonate (DIMP). This compound has a strong absorption band centered around 995 cm^{-1} that is used for data analysis. Dimethyl methyl phosphonate (DMMP) is used as an interferant to DIMP. DMMP has four absorption bands of various intensities between 700 and 1150 cm^{-1}. The very strong absorption band centered around 1055 cm^{-1} overlaps the weaker 995 cm^{-1} absorption band of DIMP, making a strong test case for accurate identification. Initial results indicate that it is possible to establish a protocol for optimal spectral resolution that performs well even in the presence of interfering species and gives pattern recognition results comparable to those obtained with very high resolution data.

In the experimental design, short segments of a single-beam spectrum were identified that had maximum analyte information and were optimal in obtaining the best pattern recognition results. In the training procedure, the interferograms were converted to single-beam spectra through fast Fourier transformation and short segments of single-beam spectra that had analyte information were isolated. These short segments were subjected to piecewise discriminant analysis pattern recognition methodology to calculate and optimize a set of discriminants sequentially in order to separate maximum DIMP data from DMMP data and backgrounds. These discriminants were tested in the prediction procedure by using a separate data set of interferograms not used in the training procedure.

The results obtained in both training and prediction procedures were analyzed to establish the protocol for resolution to be used in data collection.

Analysis of the results indicates that it is sufficient to collect 4K remote-sensing FTIR interferograms that result in a nominal spectral resolution of 8 cm^{-1} in order to obtain good pattern recognition results.

III. QUALITY ASSURANCE

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

A 1996 management assessment performed by the ACL Manager and the Quality Assurance/Quality Control (QA/QC) Coordinator examined the potential benefits of a "paperless" (electronic) system for ACL standard operating procedures (SOPs) and other QA documents previously tracked as controlled documents (i.e., documents that typically require signed forms acknowledging receipt of personal copies). The ACL's Internet Home Page now provides ACL personnel with access to 226 SOPs and other QA documents, such as the *ACL QA Plan* and *ACL Good Automated Laboratory Practices.*

A successful audit of the ACL was performed in June 1996 by a representative of the State of Utah, Department of Laboratory Improvement. The audit confirmed Utah's previous certification award, which gave approval to the ACL to analyze low-level mixed waste intended for disposal at Envirocare of Utah, Inc. In 1997, the ACL took the initiative to expand its Utah certification in radiochemical methods and RCRA methods for the determination of organic compounds and inorganic analytes in support of ANL's mixed-waste disposal efforts. The ACL developed methods and procedures and subsequently obtained additional certification for the determination of reactive cyanide and sulfide and for free-liquids testing. This new certification, along with the previous certifications, is in effect through August 31, 1998.

In addition to the Utah certification process, three additional audits/assessments were performed in 1997 to verify ACL analytical capabilities and practices in support of programs for which the ACL provides analytical services. In March, the ACL was audited as a provider of services to the CMT's Nuclear Waste Management Section. Also in March, ACL facilities and technical services were assessed by a team from Pacific Northwest National Laboratory for the U.S. DOE-sponsored Tritium Target Qualification Project. In August, the ACL was audited for services provided in the Waste Isolation Pilot Plant Headspace and the Solid Waste Performance Demonstration Programs. The specific purpose of each assessment was to evaluate the degree to which ACL performance corresponds to stated performance requirements, to evaluate the adequacy of processes established to achieve quality, to verify the existence of documents and procedures, and to evaluate the performance of ACL scientific staff in meeting the requirements of the ACL QA Plan, program-related plans, and statements of work. No major findings were discovered in the three assessments.

The ACL typically analyzes samples submitted for the determination of hazardous or radiological components, many as a part of various site remediation programs. Samples are often received from outside programs focused on site cleanup and facilities decontamination and decommissioning. This year a large number of analytical samples (340) were received for the determination of specific radionuclides ($^{226/228}$Ra, ^{232}Th, $^{235/238}$U) as part of two major site remediation programs carried out by U.S. EPA Region 5: the Ottawa landfill site in Ottawa, IL, and the Belding Warehouse site in Belding, MI. Matrices included soil and water from Ottawa and shredded debris (paper/cardboard, plastic, rubber, wood, etc.) from Belding. The Ottawa samples involved the use of standard gamma spectroscopy and alpha spectroscopy methods; data were reported in 18 CLP-type data packages containing analytical results, raw data (e.g., quality control data, instrument output), and associated documentation (e.g., notebook entries, chain-of-custody forms). The Belding Warehouse samples required (1) development of a gamma spectroscopy counting technique appropriate for the sample container geometry (a one-gallon paint can) and (2) generation of a five-point self-absorption curve unique to the sample matrix and geometry. Documentation in support of each technique was prepared and forwarded to the U.S. EPA.

Laboratory intercomparison studies coordinated and monitored by the U.S. DOE and U.S. EPA provide the ACL with independent monitoring of its analytical performance and means to better assure the quality of its analytical results through participation in programs utilizing "blind" performance evaluation samples ("blind" refers to analyte values known only to the monitoring agency). Programs in which the ACL participated in FY 1997 include

- The U.S. EPA's *National Exposure Research Laboratory-Cincinnati Water Supply (WS)* and the *Water Pollution (WP) Study Performance Evaluation Programs*: samples from the WP study were analyzed for a variety of parameters (e.g., volatile organics, PCB/pesticides, metals, nutrients, pH, cyanide, oil and grease, total dissolved and suspended solids); samples from the WS study were analyzed for barium. The ACL's analytical results submitted for this year's samples, WP037 and WP038, and WS038 and WS039, showed excellent agreement with the EPA known values.

- The U.S. DOE *Environmental Measurements Laboratory Quality Assessment Program* and the U.S. EPA *National Exposure Research Laboratory-Las Vegas Performance Evaluation Studies Program:* Water, soil, and air filter samples were analyzed for a variety of radionuclides, using gross alpha-beta, gamma spectroscopy, and alpha spectroscopy techniques. The comparison of ACL results to the accepted values showed that the ACL correctly determined all analytes in each of the programs.
- Last year, the U.S. DOE Office of Environmental Management (EM) began requiring that, to help assure the quality and defensibility of analytical measurements being performed for EM, all laboratories reporting mixed waste analytical measurements for EM-sponsored work participate in the semiannual *Mixed Analyte Performance Evaluation Program (MAPEP).* The ACL served as a pilot laboratory in 1994 and assisted in the development of the initial MAPEP database. This year MAPEP was added to the performance evaluation programs in which the ACL participates. In 1997, as an active participant in the MAPEP, the ACL successfully analyzed a soil and a water sample.

The ACL sample bar code system was made operational and available to all staff in ACL this year through activation of four additional satellite scanning stations located in ACL laboratories in Buildings 200, 203, and 205. The system provides quick and efficient electronic logging and tracking of analytical samples by means of bar code labels affixed to sample containers. Sample log-in and notification, chain-of-custody tracking, and the generation of sample status reports are now performed and documented electronically.

IV. PROFESSIONAL ACTIVITIES

A. Publications and Reports

Atomic Spectrometry Update - Atomic Mass Spectrometry
J. R. Bacon, J. S. Crain, A. W. McMahon, and J. G. Williams
J. Analytical Atomic Spectrometry **11**, 335R-393R (1996)

Arsenic Speciation in Soil Using High Performance Liquid Chromatography/Inductively Coupled Plasma/Mass Spectrometry
D. A. Bass, J. S. Yaeger, K. J. Parish, J. S. Crain, J. T. Kiely, M. J. Gowdy, G. B. Mohrman, and M. G. Besmer
Argonne National Laboratory Report No. ANL/ACL-96/2 (1996)

Stability of Low-Concentration Calibration Standards for Graphite Furnace Atomic Absorption
D. A. Bass and L. B. TenKate
Atomic Spectroscopy **18**(1), 1-12 (1997)

Natural Isotopic Fractionation of Selected Elements
T. B. Coplen, J. W. Gramlich, et al.
International Union of Pure and Applied Chemistry, Commission of Atomic Weights and Isotopic Abundances (1997)

Determination of Labile Copper, Cobalt, and Chromium in Textile Mill Wastewater
J. S. Crain, A. M. Essling, J. T. Keily, E. A. Huff, D. R. Huff, and D. G. Graczyk
Argonne National Laboratory Report No. ANL/ACL-97/1 (1997)

Guidelines for Assigning Uncertainties to Published Isotope Abundance Measurements
P. DeBievre, J. W. Gramlich, K. R. Rosman, and P. D. P. Taylor
4th Ed., International Union of Pure and Applied Chemistry, Commission of Atomic Weights and Isotopic Abundances (1996)

Feedback/Criticism/Praise
D. W. Green
Managing the Modern Laboratory **2**(2), 14A-15A (1997)

Analytical Chemistry Laboratory Progress Report for FY 1996
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-96/6 (1996)

Kill Performance Evaluations
D. W. Green
Managing the Modern Laboratory **2**(3), 24A-25A (1997)

Forum on the Requirements for a Successful Performance Evaluation System
D. W. Green
Managing the Modern Laboratory **2**(3), 32A-36A (1997)

Analytical Chemistry of Aluminum Salt Cake
D. G. Graczyk, A. M. Essling, E. A. Huff, F. P. Smith, and C. T. Snyder
Proceedings of the technical sessions presented by the TMS Aluminum Committee at the 126th Annual Meeting of the Minerals, Metals, and Materials Society, Orlando, FL, February 9-13, 1997, pp. 1135-1140 (1997)

Reporting of Relative Sulfur Isotope-Ratio Data
K. G. Heumann, T. B. Coplen, J. K. Bohlke, H. D. Dietze, M. Ebihara, J. W. Gramlich, H. R. Krousew, R. D. Loss, G. I. Ramendik, D. E. Richarson, K. J. R. Rosman, L. Schultz, P. D. P. Taylor, L. Turpin, R. D. Vocke, Jr., P. DeBievre, Y. Xiao, M. Shima, A. Pires de Matos, and N. N. Greenwood
Pure and Applied Chemistry **69**(2), 293-295 (1997)

Atomic Weights of the Elements 1995
K. G. Heumann, T. B. Coplen, J. K. Bohlke, H. J. Dietze, M. Ebihara, J. W. Gramlich, H. R. Krouse, R. D. Loss, G. I. Ramendik, D. E. Richarson, K. J. R. Rosman, L.Schultz, P. D. P. Taylor, L. Turpin, R. D. Vocke, Jr., P. DeBievre, Y. Xiao, M. Shima, A. Pires de Matos, and N. F. Greenwood
Pure and Applied Chemistry **68**(12), 2339-2359 (1996)

Isotopic Compositions of the Elements 1997
K. J. R. Rosman, P. D. P. Taylor, J. W. Gramlich, et al.
International Union of Pure and Applied Chemistry, Subcommittee for Isotopic Abundance Measurements (1997)

Application of Empore^{TM} Strontium Rad Disk to the Analysis of Radiostrontium in Environmental Water Samples
L. L. Smith, K. A. Orlandini, J. S. Alvarado, K. M. Hoffmann, D. C. Seely, and R. T. Shannon
Radiochimica Acta **73**, 165-170 (1996)

Rapid Determination of Radiostrontium Using Empore™ Strontium Rad Disks
L. L. Smith, K. A. Orlandini, J. A. Alvarado, K. M. Hoffmann, D. C. Seely, and R. T. Shannon
DOE/EM-0089T, Method RP515, DOE Methods for Evaluating Environmental and Waste Management Samples (1996)

An Evaluation of Radium-Specific, Solid-Phase Extraction Membranes
L. L. Smith, F. Markun, J. A. Alvarado, K. M. Hoffmann, D. C. Seely, and
R. T. Shannon
Radioactivity & Radiochemistry **8**(1), 30-37 (1997)

Oxygen Stoichiometry Phase Stability and Thermodynamic Behavior of the Lead-Doped and Lead-Free Bi-2212 Systems
M. Tetenbaum, M. Hash, B. S. Tani, and V. A. Maroni
Physica C **270**, 114-128 (1996)

Reporting of Relative Lithium Isotope Abundance Ratio Data
R. D. Vocke, H. R. Krouse, J. W. Gramlich et al.
International Union of Pure and Applied Chemistry, Commission of Atomic Weights and Isotopic Abundances (1997)

B. Oral Presentations

Determination of Semivolatile Organic Compounds in Highly Alkaline Cemented and Uncemented Waste Water Sludges
A. S. Boparai, K. J. Parish, Y. Tsai, P. D. Postlewait, and M. J. Schipma
Poster Presentation, 48th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Atlanta, GA, March 16-21, 1997

Determination of Volatile and Semivolatile Organic Compounds in WIPP Sludges
A. S. Boparai
27th ACL Technical Talks, Argonne, IL, April 22, 1997

Switchable Radioactive Neutron Source: Proof-in-Principle
D. L. Bowers, E. A. Rhodes, and C. E. Dickerman
Poster Presentation, International Conference on Methods and Applications of Radioanalytical Chemistry - MARC IV, Kailua-Kona, HI, April 6-11, 1997

Radioanalytical ICP/MS: Principles and Applications
J. S. Crain, L. L. Smith, J. T. Keily, J. S. Yaeger, D. G. Graczyk, and J. S. Alvarado
U. S. DOE Environmental Measurements Laboratory, New York, NY
May 15, 1997

Determination of Labile Transition Metal Species in Treated Waste Water
J. S. Crain, A. M. Essling, J. T. Keily, E. A. Huff, D. R. Huff, and D. G. Graczyk
30th Great Lakes Regional ACS Meeting, Chicago, IL, May 28-30, 1997

Comparison of Partial and Classical Least Squares Algorithms for the Analysis of Stack Emissions Using FTIR Spectroscopy
J. C. Demirgian, Z. Mao, J. Ewing, and K. P. Carney
48th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Atlanta, GA, March 16-21, 1997

The Incorporation of Technetium into a Representative Low-Activity Waste Glass
W. L. Ebert, A. J. Bakel, D. L. Bowers, E. C. Buck, and J. W. Emery,
99th American Ceramics Soc. Meeting, Cincinnati, OH, May 4-7, 1997

Scientists as Managers
D. W. Green
Symposium on "Managing the Modern Analytical Laboratory", 48th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Atlanta, GA, March 16-21, 1997

Thermal Ionization Mass Spectrometry as Applied to Trace Element Isotopic Analysis
J. W. Gramlich
30th Great Lakes Regional ACS Meeting, Chicago, IL, May 28-30, 1997

Analytical Chemistry of Aluminum Salt Cake
D. G. Graczyk, A. M. Essling, E. A. Huff, F. P. Smith, and C. T. Snyder
126th Annual Meeting of the Minerals, Metals, and Materials Society, Orlando, FL, February 9-13, 1997

Analytical Chemistry of Aluminum Saltcake
D. G. Graczyk
27th ACL Technical Talks, Argonne, IL, April 22, 1997

Advanced Development in Radioanalytical Sample Preparation Using Empore™ Strontium, Technetium, and Radium Rad Disks
K. M. Hoffmann, D. C. Seely, M. D. Erickson, K. A. Orlandini, L. L. Smith, R. T. Shannon, R. Bruening, and S. Izatt
Presented at the 4th International Conference on Methods and Applications of Radioanalytical Chemistry, Kailua-Kona, HI, April 6-11, 1997

Analysis of Lead Cable Experiment Samples Using X-Ray Diffraction, Scanning Electron Microscope/Energy Dispersive Spectrometry, Inductively Coupled Plasma/Atomic Emission Spectrometry, Gas Mass Spectrometry, and Gas Chromatography/Mass Spectrometry
P. L. Johnson
27th ACL Technical Talks, Argonne, IL, April 22, 1997

The Department of Energy's Integrated Performance Evaluation Program (IPEP): Pilot Studies for Implementation
P. C. Lindahl, W. E. Streets, J. J. Marr, K. J. Parish, W. R. Newberry, J. R. Dahlgran, C. Watkins, J. Connolly, J. Fisk, and L. A. Souza
39th Rocky Mountain Conference on Analytical Chemistry, Denver, CO, August 3-7, 1997

Applications and Limitations of Surface Enhanced Infrared Spectroscopy
V. A. Maroni, S. A. Johnson, V. J. Novick, and Y. Tsai
48th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Atlanta, GA, March 16-21, 1997

Determination of Polychlorinated Biphenyls in Water Containing Citrikleen®
K. J. Parish, D. V. Applegate, A. S. Boparai, and M. J. Schipma
Poster Presentation, 48th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Atlanta, GA, March 16-21, 1997

Waste Minimization in Analytical Chemistry Methods
L. L. Smith, D. W. Green, J. S. Crain, J. B. Schilling, J. S. Yaeger, J. T. Kiely, and A. S. Boparai
IEPA Office of Pollution Prevention, 7th Annual Pollution Prevention Conference, Lisle, IL, October 7, 1996

Thermodynamic and Nonstoichoimetric Behavior of the Lead-Doped and Lead-Free Bi-2212 Systems
M. Tetenbaum, M. C. Hash, B. S. Tani, J. S. Luo, and V. A. Maroni
5th International Conference, Materials and Mechanisms of Superconductivity, High Temperature Superconductors, Beijing, China, February 28-March 4, 1997

C. Awards

D. G. Graczyk
The Kenneth J. Jensen Award for Excellence, presented by the Analytical Chemistry Laboratory, Chemical Technology Division, Argonne National Laboratory

L. L. Smith
1996 R&D 100 Award for the Development of Empore™ Rad Disks, presented by R&D Magazine

1997 Award for Excellence in Technology Transfer, presented by the Federal Laboratory Consortium

B. S. Tani
The Kenneth J. Jensen Award for Excellence, presented by the Analytical Chemistry Laboratory, Chemical Technology Division, Argonne National Laboratory

D. Meetings Attended

David W. Green

- 14th DOE Analytical Managers Meeting, Ames, IA, October 7-10, 1996
- Analytical Laboratory Managers Association, Morristown, NJ, October 16-18, 1996
- 15th DOE Analytical Managers Meeting, Buffalo, NY, September 16-18, 1997

Paul L. Johnson

- 46th Annual Denver X-Ray Conference, Steamboat Springs, CO, August 3-8, 1997

Florence P. Smith

- 24th Annual Meeting of National Organization of Black Chemists and Chemical Engineers, Orlando, FL, March 24-29, 1997

E. Professional Organizations and Activities

Amrit S. Boparai

- American Chemical Society
- Sigma Xi

Delbert L. Bowers

- American Chemical Society
- American Society for Testing and Materials

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
- DOE Analytical Managers Group, Board of Directors
- Managing the Modern Laboratory, Editor
- DOE Characterization Monitoring, and Sensor Technology (program reviewer), Gaithersburg, MD, April 16-18, 1997
- National Science Foundation Workshop on "Curricular Development in the Analytical Sciences," (participant), Atlanta, GA, March 14-15, 1997
- Chemical Measurement Task Group, Council on Chemical Research (member)
- Sigma Xi

Edmund A. Huff
- American Chemical Society
- Society for Applied Spectroscopy
- Sigma Xi

Paul L. Johnson
- American Crystallographic Association
- Chemical Technology Division's Synchroton Advisory Committee
- Sigma Xi

Peter C. Lindahl
- American Chemical Society
  - Division of Analytical Chemistry

Francis Markun
- Health Physics Society
  - Midwest Chapter

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

Florence P. Smith

- National Organization of Black Chemists and Chemical Engineers

Christine T. Snyder

- American Chemical Society

Charles M. Stevens

- American Geophysics Union
- American Association for the Advancement of Science

W. Elane Streets

- American Society for Testing and Materials
- American Society for Quality Control

Tony TenKate

- American Association of Physics Teachers
- Sigma Xi