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ANL/ACL-98/2

Analytical Chemistry Laboratory

Progress Report for FY 1998

Chemical Technology Division
Argonne National Laboratory, Argonne, Illinois 60439
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ANALYTICAL CHEMISTRY LABORATORY
Progress Report for FY 1998

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Analytical Chemistry Laboratory
Chemical Technology Division

December 1998

Reports for FY 1984 through FY 1997 are:

ANL/ACL-85-1 (March 1985)

ANL/ACL-85-4 (December 1985)

ANL/ACL-86-2 (November 1986)

ANL/ACL-87-2 (December 1987)

ANL/ACL-88-1 (December 1988)

ANL/ACL-89/6 (December 1989)

ANL/ACL-90/2 (December 1990)

ANL/ACL-91/1 (December 1991)

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)

ANL/ACL-97/5 (December 1997)

DISCLAIMER

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NOMENCLATURE

ACL	Analytical Chemistry Laboratory
ANL	Argonne National Laboratory
APS	Advanced Photon Source
BSCCO	bismuth/strontium/calcium/copper oxides
CHM	Chemistry Division
CLP	Contract Laboratory Program
CMT	Chemical Technology Division
ComEd	Commonwealth Edison
DIMP	diisopropyl methyl phosphonate
DMMP	dimethyl methyl phosphonate
DOD	Department of Defense
DOE	U.S. Department of Energy
EM	Environmental Management
EML	Environmental Measurements Laboratory
EMO	Environmental Management Operations
EPA	U.S. Environmental Protection Agency
ES	Energy Systems Division
ET	Energy Technology Division
FTIR	Fourier transform infrared
FUSRAP	Formerly Utilized Sites Remedial Action
FY	fiscal year
GC/MS	gas chromatography/mass spectrometry
GPE	General Purpose Equipment fund
GUI	graphical user interface
HPGe	high purity germanium
HTER	high throughput electrorefiner
HUP	hot uniaxial press
IAG	Interagency Agreement
ICP/AES	inductively coupled plasma atomic emission spectrometer
ICP/MS	inductively coupled plasma mass spectrometer
INEEL	Idaho National Engineering and Environmental Laboratory
IPCR	improved principal component regression
IPEP	Integrated Performance Evaluation Program
MACE	Melt Attack and Coolability Experiment
MAPEP	Mixed Analyte Performance Evaluation Program
MARLAP	Multiagency Radiochemistry Laboratory Analytical Procedures Manual
MSD	Materials Science Division
NAMP	National Analytical Management Program
NEET	Nuclear Excitation by Electronic Transition
NELAP	National Environmental Laboratory Accreditation Program
NERL-Ci	National Exposure Research Laboratory—Cincinnati
NERL-LV	National Exposure Research Laboratory—Las Vegas
NL	normalized mass loss

NOMENCLATURE (contd)

NRC	Nuclear Regulatory Commission
PCB	polychlorinated biphenyl
PDP	Performance Demonstration Program
PE	performance evaluation
PHY	Physics Division
PI	principal investigator
PLS	partial least squares
PNNL	Pacific Northwest National Laboratory
QA	quality assurance
QAP	Quality Assessment Program
QC	quality control
RA	Reactor Analysis Division
RCRA	Resource Conservation and Recovery Act
R&D	research and development
RE	Reactor Engineering Division
RH	remote-handled
SAS	special analytical services
SMO	Sample Management Organizations
SOP	standard operating procedure
SVOC	semivolatile organic compound
TCLP	Toxicity Characteristic Leaching Procedure
TD	Technology Development
TIMS	thermal ionization mass spectrometer
TRU	transuranic
TTQP	Tritium Target Qualification Project
USACE	U.S. Army Corps of Engineers
VOC	volatile organic compound
WIPP	Waste Isolation Pilot Plant
WP	Water Pollution Study
WS	Water Supply Study
WWW	world wide web
XFD	Experimental Studies Division
XRD	X-ray diffraction

ANALYTICAL CHEMISTRY LABORATORY
Progress Report for FY 1998

I. INTRODUCTION

This report summarizes the activities of the Analytical Chemistry Laboratory (ACL) at Argonne National Laboratory (ANL) for Fiscal Year (FY) 1998 (October 1997 through September 1998). This annual progress report, which is the fifteenth in this series for the ACL, describes effort on continuing projects, work on new projects, and contributions of the ACL staff to various programs at ANL.

The ACL operates within the ANL 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 ANL 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 had four technical groups in FY 1998 -- Chemical Analysis, Instrumental Analysis, Organic Analysis, and Environmental Analysis -- which together include about 30 technical staff members. (See Fig. 1 for the organization chart for September 1998.) 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), the U.S. Army Corps of Engineers (USACE), and other organizations. Project-specific quality assurance requirements can be handled within the ACL's QA structure.

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 the following:

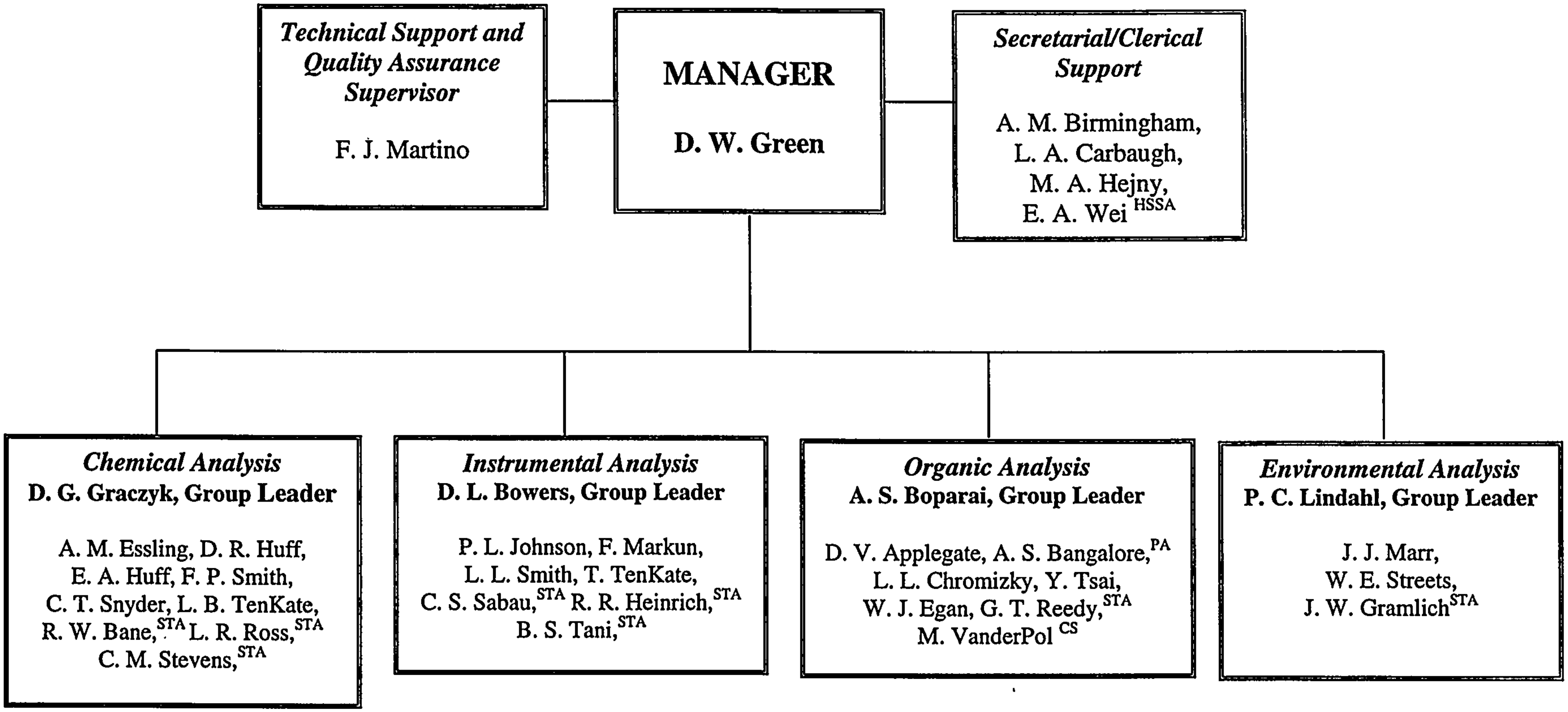
- List of all published ANL/ACL technical reports
- Major technical specialties of the ACL Staff
- Advice on how to submit samples
- The ACL Annual Reports for FY 1995, 1996, and 1997

Installations of a carbon/sulfur determinator and a oxygen/nitrogen determinator, which were funded by the ANL General Purpose Equipment Fund (GPE), were completed in FY 1998. A replacement was procured and installed for the ion chromatograph that is dedicated to radioactive samples. Both a low-level α/β proportional counter system and an accelerated solvent extractor for organic materials were procured using GPE funds in FY 1998 and will be installed in early FY 1999.

The ACL worked on one project in FY 1998 with ANL Support Development Funds. In this work, the ACL developed and implemented software to allow the direct electronic transmission of analytical data obtained with the new inductively coupled plasma/atomic emission spectrometer (ICP/AES) system. In addition to directly benefiting recipients of analytical reports from samples measured with the ICP/AES system, this project serves as a prototype for the possibilities of direct electronic transmission of analytical data to clients. Progress on this project is described later in this report.

The remainder of this report comprises short descriptions of the analytical work performed by the staff of the ACL in FY 1998 on selected projects. This report covers only a fraction of approximately 1,600 different jobs, many of which involve several samples, that the ACL receives annually. Many contributions by the ACL staff are not included because they involved generation of analytical data on only a relatively small number of samples. Despite their small scope, these 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
September 1998



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W. J. Egan, G. T. Reedy,$^{\text{STA}}$
M. VanderPol $^{\text{CS}}$

Environmental Analysis
P. C. Lindahl, Group Leader

J. J. Marr,
W. E. Streets,
J. W. Gramlich$^{\text{STA}}$

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

II. TECHNICAL HIGHLIGHTS

(1) Nuclear Technology (C. S. Sabau, D. L. Bowers, A. M. Essling, D. G. Graczyk, D. R. Huff, E. A. Huff, P. L. Johnson, 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 CMT's Nuclear Technology Department.

Results from the analysis of these samples supported efforts that included zeolite waste form development, treatment of spent oxide fuel and spent metallic fuels, and investigation into the use of non-lithium-metal material for reducing uranium oxide in liquid bismuth. The samples varied in the dissolution techniques required before measurement of the requested analytes. Analytical methods applied to obtain elemental, isotopic, and compound information 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). Many of the submitted samples contained radioactive materials, such as plutonium. These types of samples were analyzed with instruments, such as the ICP/AES system, that had been modified for use with the radioactive samples.

(2) Support for High-Temperature Superconductor Development (E. A. Huff, D. R. Huff, A. M. Essling, K. J. Parish, D. V. Applegate, P. L. Johnson, F. P. Smith, C. T. Snyder, 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 (ET), Materials Science (MSD), Chemistry (CHM), and Chemical Technology (CMT) Divisions. Much of this work involved the 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). Our measurements included determining elemental composition by ICP/AES or classical methods, anions by ion chromatography, carbon with a Leco Corporation analyzer, and phase composition by X-ray powder diffraction.

The researchers in the CHM and MSD have been developing methods for depositing thin films of specific compounds on semiconductor substrates such as silicon. Measurements by ACL for these development efforts included characterizing impurities in the films, as well as determining the elemental stoichiometry of the major constituents. For this work, ACL analysts devised ways to effectively dissolve the thin film coatings without attacking the underlying substrates. The solutions obtained have been analyzed by ICP/AES and ICP/MS to measure the very small amounts of individual elements present in the films. These efforts have permitted characterization of microgram quantities of material contained in films that are only a thousand angstroms thick. Through these and related activities, the teamwork among chemists, ceramists, and analysts is helping to move the technology of superconducting ceramics toward practical applications.

(3) Performance Demonstration Program for the WIPP Transuranic Solidified Waste Characterization Program (A. S. Boparai, L. L. Chromizky, and Y. Tsai)

The Waste Isolation Pilot Plant (WIPP) is a DOE installation designed to dispose of transuranic (TRU) waste left from the research and production of nuclear weapons. The WIPP site consists of large interconnecting cavities hollowed out of deep geologic salt beds approximately 600 m underground and is located approximately 26 miles east of Carlsbad, NM. The TRU waste destined for WIPP began to be generated during the 1940s from the nation's nuclear weapons programs at various DOE sites.

Wastes to be shipped to WIPP must first be characterized to identify the presence of any hazardous materials in addition to the radioactivity. As part of the WIPP TRU waste characterization program, a Performance Demonstration Program (PDP) for the Analysis of Resource Conservation and Recovery Act (RCRA) Constituents in Solidified Waste was established in 1995.

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

During FY 1998, the ACL prepared approximately two kilograms each of uncemented and cemented simulated Type 1 solidified waste form. The unspiked waste matrix was then supplied to a contractor selected by the PDP coordinator for spiking with metal analytes. The contractor also provided blind audit samples of SVOCs and VOCs to participating laboratories.

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

Researchers in the ANL Energy Technology Division (ET) (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 1998, 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 leachates 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, and other elements. Concentrations of the radioactive isotopes ^{137}Cs, ^{238}U, ^{235}U, ^{241}Am, ^{99}Tc, and ^{90}Sr were determined in waste samples by gamma spectrometry or radiochemical separation and alpha or beta counting methods. 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 hazardous metals in solid and liquid radioactive wastes from experimental processes, and a number of simulated waste forms that involved hard-to-stabilize chemical species such as anionic forms of hazardous metals like chromate, antimonate, selenate, and arsenate ions. Some TCLP test results evaluated the effectiveness of chemical treatment (e.g., with reducing agents) in helping stabilize such anionic species. With this information, the ET researchers are developing treatment alternatives and strategies that permit their process to handle a wide variety of waste compositions.

(5) Analytical Services for Site Remediation (D. V. Applegate and L. L. Chromizky)

The ACL, in cooperation with the Energy Systems Division (ES), set up an analytical system in a mobile laboratory. The mobile laboratory was set up to support an Environmental Management Operations (EMO) project to remediate soil contaminated with VOCs at a hazardous site within the ANL-East boundaries. The system comprised a static headspace sampler followed by gas chromatography/mass spectrometry (GC/MS) analysis. It was used to quantitate the levels of a wide range of VOCs in the experimentally treated soil. An analytical method was developed that allowed fast screening of samples that may be contaminated at a level from 10ppb to 500ppm. Several hundred samples were analyzed, with very fast turnaround time. Having data on hand helped the EMO to direct the remediation efforts instead of having to wait weeks to get results back from contract laboratories. The data that we were able to provide with quick turnaround time was compared to the full RCRA analysis of the same soils and showed good agreement. Since the work was done in a mobile on-site lab, shipping and other sample handling costs were greatly reduced.

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

The ACL was involved with the preparation and distribution of performance demonstration samples of simulated headspace gas for the WIPP project. Every six months, the ACL acquired relevant gases from a specialty gas vendor and used them to prepare standard gas mixtures containing known concentrations of various VOCs, hydrogen, and methane. These standard gas mixtures were metered through a heated, multiport manifold (constructed by ACL) into several SUMMA canisters simultaneously, which provided uniform samples in each canister. The canisters were labeled, packaged, and shipped to laboratories that the WIPP Carlsbad Area Office wants to qualify for headspace gas analysis. Because the WIPP Office increased the number of participating labs in 1998, the preparation of as many as 50 canisters per shipment was required. The ACL performed a confirmation analysis on an aliquot of each of the gas mixtures for VOCs, methane, and hydrogen. This analysis verified that the gas vendor's certified concentration values were correct and that the preparation manifold did not alter the standards in any way. The ACL was also responsible for the cleaning and inventory of all canisters used for the headspace gas performance demonstration program.

(7) Application of X-ray Diffraction to Waste Form Development (P. L. Johnson, B. S. Tani, and D. L. Bowers)

The ACL continued to provide XRD support to programs to the following ANL divisions: Chemical Technology (CMT), Chemistry (CHM), Energy Technology (ET), Energy Systems (ES), Technology Development (TD), Reactor Engineering (RE), Physics (PHY), Environmental, Safety, and Health (ESH), Accelerator Systems (ASD), and the Experimental Facilities (XFD). Data generated by these XRD measurements provided valuable information to each of these programs, but are not detailed in this report. Instead, one example is reported that is typical of the support in this analytical area.

One application of XRD was in CMT's Nuclear Technology Department. The Waste Form Development Group used XRD to examine zeolite and sodalite waste forms that may contain compounds such as PuO_2 or UO_2. A ceramic waste form will be prepared from zeolite that has been contacted with electrorefiner salt, which contains uranium, plutonium, and various other fission products. Theoretical calculations indicated that the plutonium present in this waste form will cause no harmful interactions. To verify this expectation, the Waste Form Development Group (M. K. Richmann) prepared ceramic waste forms for testing by mechanically blending a mixture of zeolite 4A and salt containing ^{239}Pu. The blended zeolite was mixed with a glass binder and the resulting mixture was pressed using a hot uniaxial press (HUP). Under these conditions, zeolite converted to sodalite. An important part of these studies was the determination of the structure of materials used and waste forms produced. Capillary XRD studies were performed to make these determinations.

Although rotating anode XRD would yield higher resolution, radiological constraints dictate the use of sealed capillaries. The XRD results helped determine the chemical form of the plutonium-loaded salt used to manufacture the ceramic waste forms, verify the quality of the zeolite used, and confirm that the zeolite/glass/salt mixture did, in fact, convert to sodalite as expected. Indications were also seen in the XRD data that various thermodynamic effects in the zeolite/salt exchange process affected the fate of the plutonium in the waste form.

(8) Multiagency Radiochemistry Laboratory Analytical Procedures (MARLAP) Manual

(L. L. Smith and P. C. Lindahl)

The Multiagency Radiochemistry Laboratory Analytical Procedures (MARLAP) Manual is a multiagency [EPA, DOE, the Department of Defense (DOD), and the Nuclear Regulatory Commission (NRC)] document that is currently being drafted to provide guidance in all relevant areas of radioanalytical laboratory work for laboratory personnel and project managers. The MARLAP manual discusses the theory and background of various analytical techniques as applied to samples containing radiological analytes. It is designed to be of use to developers of standard operating procedures (SOPs), so they can understand various options and choose the best analytical approach for a given situation.

Because of our radiological expertise, the ACL was asked to provide technical support to the MARLAP Manual. Members of the ACL authored the Sample Dissolution, Separation Techniques, and Evaluation of Radiological Laboratories chapters, and also provided technical comments on the Sample Preparation chapter.

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

The ACL is developing and implementing a comprehensive Integrated Performance Evaluation Program (IPEP) for DOE's Office of Environmental Management (EM) as part of its National Analytical Management Program (NAMP). The IPEP is designed to provide information on the quality of radiological and nonradiological analysis data generated 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 in implementing the program. The staff of the Computer, Applications, Network, and Security Group (A. E. Scandora, R. R. Krol, and J. M. Copple) in CMT assisted the ACL in developing strategies and systems for handling large data sets and in compilation and analysis of data from performance evaluation (PE) program studies.

The IPEP continued to function as a pseudo-EPA Region by nominating laboratories into the EPA's Water Supply (WS) and Water Pollution (WP) PE Program Studies and accessing the relevant PE information about those laboratories. All FY 1998 data from these PE programs and the two DOE PE programs [the Environmental Measurements Laboratory's (EML) Quality Assessment Program (QAP), and the Radiological and Environmental Sciences Laboratory's Mixed Analyte Performance Evaluation Program (MAPEP)] were transferred into the IPEP PE results database at ANL.

The EPA is privatizing the WS and WP Program Studies. The IPEP staff is working with the National Environmental Laboratory Accreditation Program (NELAP) to ensure a smooth transition and continued access to the relevant PE information needed by the IPEP.

Formats for the water and soil matrices in the MAPEP program were developed. The IPEP maintained routine production of WS, WP, QAP, and MAPEP reports for those Sample Management Organizations (SMOs) where the program was piloted in previous years (Idaho and Albuquerque). We also made a presentation of the IPEP to the Oak Ridge Operations Offices SMO personnel in February 1998 to initiate the program there, and we continued providing them with reports after that.

All reports produced in hard copy were also produced in .PDF format and posted on a password-secured section of the IPEP WWW site.

We collected laboratory information from all other DOE SMOs to determine which analytical laboratories were performing analyses for the EM programs at the various DOE sites; these data were entered into the IPEP laboratory identification database. This information must be collated with the IPEP data collected from the various PE programs to determine whether a laboratory is participating in the appropriate PE programs for the analytical work it is performing in support of EM programs and projects, as a prelude to preparing reports for these organizations.

We redesigned and updated the IPEP WWW server (http://www.cmt.anl.gov/ipep/ipep.html). The web site contains general information about the IPEP, including contact names, PE program study distribution schedules, and hyperlinks to other servers of interest. We also incorporated new password software, which allows external users to change passwords needed for access to protected information, such as PE reports for a given SMO.

We prepared reports of historic concentration ranges of all matrix-analytes for all studies and all PE programs in the IPEP database. The full compilation is being prepared for mounting on the IPEP WWW site, so it will be readily available as a valuable resource to producers of PE materials.

(10) Chemical Analysis of Lithium Aluminate for the Tritium Target Qualification Project (TTQP) (D. G. Graczyk, A. M. Essling, F. P. Smith, C. T. Snyder, D. R. Huff, E. A. Huff, P. L. Johnson, and B. S. Tani)

In its Tritium Target Qualification Project (TTQP), Battelle's Pacific Northwest National Laboratory (PNNL) is coordinating manufacture of ^{6}Li-enriched lithium aluminate ceramic pellets for use as tritium-production targets for DOE. The ceramic pellets are made by sintering lithium aluminate powders prepared with a specified lithium isotopic composition and lithium-to-aluminum stoichiometry. The ACL became involved in the TTQP during FY 1997, when we were asked to provide chemical analysis of developmental batches of the powder and pellet materials and to perform certification analysis of two production lots of pellets incorporated into a pilot-scale assembly that was fabricated for demonstration testing of the tritium-producing technology. 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). Our capabilities include a special sealed-tube procedure for dissolving the refractory samples using hydrochloric acid at 300°C, thermal-ionization mass spectrometry to measure lithium isotope ratios, specialized assays for lithium (using isotope dilution) and aluminum (by 8-hydroxyquinolate gravimetry), and an acid-reaction/gas-evolution procedure for measuring carbonate impurity.

During FY 1998, the TTQP pursued efforts with private-sector commercial vendors to develop production-scale processes for manufacturing the lithium aluminate powders and pellets needed for tritium production in coming years. In support of these efforts, the ACL provided chemical analysis of several developmental powder and pellet batches using our qualified procedures. Results of our measurements helped to evaluate alternative production methods and processes and to identify products that met program specifications.

Ultimately, the TTQP needs to move the chemical analysis of these lithium aluminate materials to private-sector commercial laboratories. The ACL has been working with the project to facilitate transfer of our established methodology to commercial providers and has begun investigating some alternative methods that might be more amenable to their operations. These methods take advantage of recent advances in technology related to sample preparation and analysis and promise better efficiency than the existing methods, which were developed some years ago. This methods-development work is discussed further in Section 26 of this report.

(11) Characterization of Silo Waste for Fluor Daniel Fernald (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 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 XRD to provide information on specific compounds present. For 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 (Leco Corporation). We also determined the distribution of carbon between organic and inorganic (carbonate) forms in selected samples. Ultimately, 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.

(12) Chemometrics: Sensor Algorithms and Medical Image Analysis (W. J. Egan, A. S. Bangalore, M. H. Brady, and A. S. Boparai)

Progress was made on two ongoing chemometric projects this year. The first project involves the development of algorithms and a data analysis system for multivariate sensor data. Advanced sensors provide voluminous quantities of data. The information contained within this enormous amount of sensor data, however, must be extracted in near real-time and converted into a form useful to decision makers. Goals are the following: (1) to extract information from sensor data, including hyperspectral imagers; (2) to use quantitative/qualitative algorithms to model the extracted information; (3) to conduct all operations in near real-time; and (4) to provide estimates of the reliability of the final answers.

Previous work coded algorithms in C/C++, Fortran, and Matlab to perform data preprocessing, linear discriminant analysis, digital signal processing, and neural network modeling. A multivariate calibration module has been developed, which includes the partial least squares (PLS) algorithm and an improved principal component regression (IPCR) algorithm that provides statistical estimates of the reliability of the model answers. A graphical user interface (GUI) in Matlab was completed to enable easy application of the preprocessing, pattern recognition, and multivariate calibration modules. The data analysis package is now available for Win95/WinNT, as well as UNIX.

The second chemometric project is a collaborative effort with the Rheumatology Clinic at the University of Chicago Center for Advanced Medicine. We are investigating the efficacy of thermal imaging for monitoring the extremities of rheumatoid arthritis patients. Rheumatoid arthritis randomly attacks the joints, causing pain, inflammation, and deformity. This clinical study has monitored over thirty rheumatoid arthritis patients over a four-month period. A ThermaCam infrared camera was used to acquire thermal images of the hands, feet, and knees of the patients. Blood tests and physical examinations were also conducted. Multivariate statistical analyses is being used to determine the correlation between the information provided by the thermal images and the traditional medical measurements.

(13) Chemical Analysis of Corrosion Products from Dresden 1 Spent Fuel Storage Racks

(D. L. Bowers, P. L. Johnson, F. P. Smith, L. B. TenKate, T. TenKate, and D. G. Graczyk)

The Commonwealth Edison (Com Ed) plan to remove nuclear fuel storage racks from the Dresden 1 fuel storage pool required that the oxide layer that was evident on the support racks be evaluated. Argonne's Technology Development Division (TD) was requested to perform this task. A. Purohit of TD designed a test plan to analyze samples from the fuel racks. These analyses included physical testing, such as shear-strength measurement, metallographic and microhardness analysis, and various chemical analyses. The ACL provided chemical analyses that included XRD, determination of metals, measurement of C and S, determination of anions, and radiochemistry. The results of these analyses were then used by Com Ed to ascertain the integrity of the fuel racks before removal.

(14) Preparation of Osmium-189 Targets for Photon Excitation Studies (F. Markun)

In collaboration with researchers in the Physics Division (D. S. Gemmell and I. Ahmad), the ACL prepared an osmium target for photon excitation studies by electroplating osmium metal onto a copper planchet. The resulting target was used for photon excitation studies in which the high flux of protons from the Advanced Photon Source (APS) would excite osmium nuclei by a process called NEET (Nuclear Excitation by Electronic Transition). The initial preparation of osmium targets was very successful; the starting point was osmium tetraoxide which was dissolved in a plating solution containing sodium hydroxide, sodium monohydrogen phosphate, sulfamic acid, and potassium hydroxide.

Subsequent to this initial work, the team of physicists wanted to produce ^{189}Os isotope targets by starting with metallic osmium rather than osmium oxide. Only 100 mg of metallic ^{189}Os powder was available. In order to insure the successful conversion of ^{189}Os metal into osmium tetraoxide, a separate experiment was carried out with isotopically "natural" metallic osmium, which turned out to be a success. To convert metallic osmium into osmium tetraoxide, the metal must be heated in the presence of oxygen, which produces gaseous osmium tetraoxide. A special apparatus was assembled to efficiently trap all of the gas generated, which was then dissolved in a plating solution. The ^{189}Os electroplated targets were used in APS and generated very useful data.

Based on these experimental results, additional experiments are being planned where ^{189}Os will be electroplated on both sides of a much thinner copper foil. This represents another challenge.

(15) Effect of Spectral Resolution on Pattern Recognition Analysis Using Passive Fourier Transform Infrared Sensor Data (A. S. Bangalore and A. S. Boparai)

In collabration with J. C. Demirgian (ER Division) and G. W. Small (Ohio University), the Fourier transform infrared (FTIR) spectral data of two nerve agent simulants, diisopropyl methyl phosphonate (DIMP) and dimethyl methyl phosphonate (DMMP), were used as test cases to determine the spectral resolution that gives optimal pattern recognition performance. The DIMP simulant was used as the target analyte for detection, while DMMP was used to test the ability of the automated pattern recognition methodology to detect the analyte selectively. A Midac passive FTIR instrument was used to collect interferogram data. The methodology was based on the application of pattern recognition techniques to short segments of single-beam spectra obtained by Fourier processing the collected interferogram data.

This work examined the effect of varying spectral resolution on the quality of pattern recognition results. The objective was to determine the optimal spectral resolution to be used for data collection. The results of this study indicated that the data with a nominal spectral resolution of 8 cm^{-1} provides sufficient selectivity to give pattern recognition results comparable to that obtained by using higher resolution data. We found that while higher resolution did not increase selectivity sufficiently to provide better pattern recognition results, lower resolution decreased selectivity and degraded the pattern recognition results. These results can be used as guidelines to maximize detection sensitivity, to minimize the time needed for data collection, and to reduce data storage requirements.

(16) Radiological Analysis Support for U.S. EPA Region V Sampling Sites

(F. J. Martino, F. Markun, L. L. Smith, and T. TenKate)

Each year the ACL analyzes a variety of waste samples for the determination of hazardous or radiological components, in addition to samples that contain both (i.e., mixed waste). Mixed waste samples are typically submitted as a part of remediation programs focused on site cleanup and/or facilities decontamination and decommissioning. In 1994, EPA Region V and the ACL established an Interagency Agreement (IAG) so that the ACL could provide special analytical services (SAS) for samples collected within Region V. The IAG requires that, to the extent possible, SAS samples be analyzed by methods found in the EPA compendium, *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, SW–846. Alternative methods may be used with prior EPA approval.

To date, the ACL has received samples for six different Region V projects. In 1998, for the second year in a row, a large number of samples were received for the determination of specific radionuclides. The samples were submitted as a part of three major site remediation programs.

- One site in Ottawa, was used in the past by a company that applied luminescent paint containing ^{226}Ra to clock dials. As a part of a followup of the integrity of landfills used to hold waste removed from this site after its demolition, two separate sampling phases resulted in 410 soil and water radiological samples being taken from a landfill, from the area around an original company building foundation, and from the surrounding grounds. The samples were submitted to the ACL for the determination of ^{226}Ra by gamma spectroscopy and $^{235/238}$U and ^{232}Th by alpha spectroscopy. Analytical results for all samples were reported in 18 CLP-type data packages, which contained all analytical results, quality control data, instrument output, and other documentation associated with the samples (e.g., notebook entries, chain-of-custody forms, etc.).
- Two sites in Michigan, the Belding Warehouse (Belding, MI) and the Benton Harbor Warehouse (Benton Harbor, MI), were privately owned facilities. Each site was used to store hundreds of WWII artifacts, including airplane instrument panel gauges, switches, and compasses that were marked with luminescent paint containing ^{226}Ra. Material stored at both facilities was later found to be contaminated with radium dust from deteriorating gauges. After a preliminary investigation, the site contractor determined that some materials could be collected and shredded to form a nearly homogenous sample. The materials included paper/cardboard, plastic, rubber, wood, and metal. About ten percent of the shredded material (180 samples) was packed and shipped to the ACL in one-gallon metal paint cans to be gamma counted, while the rest of the samples were kept and counted by the

contractor. Because the ACL uses a high purity germanium (HPGe) detector and the contractor a NaI detector, a self-absorption curve unique to the sample and the sample geometry, was developed by ACL analysts for the HPGe detector. Documentation in support of the curve was prepared and forwarded to the contractor, allowing the contractor to directly compare both sets of data. Analytical results for all samples were reported in eight CLP-type data packages, which contained all analytical results, quality control data, instrument output, chain-of-custody records, notebook entries, and other related documentation.

(17) Data Verification for Experiments Conducted on Corrosion Behavior of Ceramics

(L. L. Chromizky and J. J. Marr)

The purpose of the review was to examine spreadsheets generated by members of CMT's Waste Management group to determine if the data were traceable to the supporting analytical results and correctly transferred to the spreadsheets. The data were taken from experimental work that determined the corrosion behaviors of various ceramics. All data were examined, -- from simply checking that the pH values were correctly transcribed from experimental log sheets to spreadsheets, to checking that normalized mass loss (NL) calculations involving ICP/MS results were correct. These results were summarized in memoranda to the principal investigator (PI), A. Bakel, and other members of CMT.

The review process included comparing Initiation and Termination data sheets for various tests with data on the spreadsheets. Analytical results generated by ICP/MS analyses that were reported on spreadsheets for various elements were verified against original raw data. The NL calculations were checked for accuracy, as were calculations of averages, standard deviations, and molal concentrations. In addition, drafts of reports were reviewed to ensure agreement with spreadsheet data.

Errors and issues involving the spreadsheets were reviewed with the PI and changes were made accordingly to facilitate better understanding of the spreadsheets by other scientists.

(18) Headspace Gas Analysis of Remote Handled Transuranic Waste

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

Meaningful gas generation data are nonexistent for remote-handled (RH) transuranic (TRU) debris waste destined for the Waste Isolation Pilot Plant (WIPP). In order to provide knowledge of RH-TRU waste gas generation mechanisms, the Energy Technology Division (ET) (L. Neimark), in collaboration with INEEL personnel (K.Liekhus and associates), conducted experiments on generation of headspace gases (WIPP-specified volatile organic compounds and target gases) in RHTRU waste. The test waste was WIPP Content Code 104/107 RH-TRU, which originates primarily from non-retrievable, Argonne National Laboratory-West (ANL-W) test-fuel residue.

The ACL provided the ET division personnel with clean and certified SUMMA canisters (10- and 100-mL capacity) to collect headspace gas samples. The ACL determined VOCs and target gases in samples collected at the beginning and end of the test phase (approximately 9 months) and hydrogen and methane twice during the middle part of the test. The results of the measurements were provided to personnel at INEEL, who wrote the sampling and analysis plan, and after evaluating the data, will be providing a report on this project.

(19) Characterization of Particulate Debris from the Coolant Systems of a Commercial Nuclear Reactor (P. L. Johnson, A. M. Essling, E. A. Huff, D. R. Huff, F. P. Smith, and D. G. Graczyk)

A chemist at the Point Beach Nuclear Power Plant in Two Rivers, WI, requested that the ACL help provide base composition information on several samples of particulate debris that were obtained during scheduled maintenance operations on the cooling systems of this commercial nuclear reactor. The samples included particulate matter removed from a primary coolant pump seal, material obtained from a wipedown cleaning of a filter assembly, and material collected from a steam-generator-blowdown flow-throttle valve. Analysis of several of the samples was complicated by the manner the operations crews used to collect the debris -- by wiping it off mechanical components with rags or cleaning pads or by adhering the particles to tape or other sticky substrates. Many of the samples were slightly radioactive.

The ACL's approach to characterizing each particulate composition included examining the debris samples by XRD and determining their elemental composition by ICP/AES analysis of a solution obtained by dissolving a portion of each material. Some samples were also analyzed for leachable anions and carbon. For the flow-throttle valve debris sample, identification of the iron oxidation states by Mossbauer spectroscopy was requested. Although the ACL does not maintain a capability for this measurement, we were able to accommodate this request with the help of ANL colleagues (E. Alp and C. Johnson, Experimental Facilities Division) who use the technique in their research. The combination of data from the measurements made on these samples permitted assignment of the particulate debris to several types of ferrous and non-ferrous metal corrosion products. This information is being used by the Wisconsin Electric Power Co. to understand observations they make during maintenance operations at their Point Beach Plant and, thereby, to continually improve plant performance.

(20) New Capabilities for Measuring Carbon, Sulfur, Oxygen, and Nitrogen (F. P. Smith, C. T. Snyder, P. C. Lindahl, and D. G. Graczyk)

The ACL has upgraded its capabilities for the determination of carbon and sulfur in various inorganic materials and has added a capability for measuring oxygen and nitrogen. These capability improvements came about through the procurement of two new elemental analyzers with 1997 General Purpose Equipment funds.

One instrument replaced outdated carbon and sulfur determinators that have been in use in the ACL for about 30 years. The new system, a Leco Corporation Model C/S 300 Carbon/Sulfur Determinator, is a state-of-the-art elemental analyzer based on the ASTM-approved combustion technique. In the analysis cycle, the sample is heated with an induction furnace to a high temperature (2000°C) in a flowing oxygen stream to produce carbon dioxide and sulfur dioxide gases that are measured with solid state infrared absorption cells. The system can be applied to a wide variety of materials, including metals, ceramics, flyash, carbides, cements, and limestone. The useful measurement range (based on a 1-g sample) is 10 ppm to 3.5 % for carbon and 10 ppm to 0.35 % for sulfur; higher concentrations can be measured by analyzing a proportionately smaller sample. Precision of the measurements corresponds to a standard deviation of about 5 ppm or 2% of the measured value, whichever is greater.

The second instrument, a Leco Corporation Model TC-136 Oxygen/Nitrogen Determinator, provides a capability not recently available in the ACL. It uses the principle of inert-gas fusion to measure oxygen and nitrogen in the types of materials listed for the C/S system. Here, the sample is heated in a graphite crucible under flowing helium by means of an electrode impulse furnace to a temperature approaching 2000°C. Oxygen in the sample reacts with carbon in the crucible to form carbon monoxide, which is converted to CO_2 and measured by infrared absorption. Nitrogen in the sample is released as N_2 and is measured with a thermal conductivity cell after other gases (water and CO_2) are removed. The measurement range for a 1-g sample is 5 ppm to 0.2% for oxygen and 5 ppm to 0.5 % for nitrogen. Precision for these measurements corresponds to a standard deviation of 2 ppm or 1% of the measured value, whichever is greater.

These new instruments will help improve the dependability and efficiency of carbon/sulfur measurements that ACL has steadily provided for many years and will permit ACL clients to obtain measurements of oxygen and nitrogen when their programs need them.

(21) Chemical Analysis of Fuel Residues in Cladding Segments from Tests with the High Throughput Electrorefiner (A. M. Essling, E. A. Huff, D. R. Huff, F. P. Smith, and D. G. Graczyk)

The ACL analyzed a challenging set of samples related to a test carried out by engineers (E. C. Gay et al.) in CMT to evaluate how well their High Throughput Electrorefiner (HTER) will retain noble metals in the anode baskets when uranium is electrotransported from chopped metallic fuel. In this test, two baskets in the inner channel of the HTER were loaded with segments of unirradiated uranium/zirconium/fissium metal alloy clad in stainless steel. The HTER was operated to achieve anodic dissolution and separation of the uranium in the segments, and then segments were taken from top to bottom of each basket. These segments were submitted to the ACL for chemical analysis of the residue in the cladding. The remaining segments were subsequently processed again in the HTER to increase the fraction of uranium removed from them, and a second set of segment samples was also submitted for analysis.

The ACL analysts first weighed each segment and then treated each one with a mixture of acids to dissolve the fuel residue while only slightly attacking the stainless steel cladding hull. After the fuel residues were dissolved, each cladding hull was washed, dried, and individually weighed.

Solutions containing the dissolved residue from each segment were analyzed by inductively coupled plasma-atomic emission spectrometry to determine U, Zr, the fissium elements (Mo, Ru, Pd, and Rh), and electrorefiner salt constituents (Li, K, and Cd). The ICP/AES analysts also reported values for dissolved components of the stainless steel cladding (Fe, Ni, Cr, Mn).

By measuring all these components, the analysts were able to compile the data in a way that allowed the mass of each element to be calculated relative to the mass of cladding in each segment. This, in turn, permitted an evaluation of the extent to which each fuel component had moved out of the cladding hull. In the end, a combination of thorough chemical analysis and creative presentation of the analysis results helped the CMT engineers obtain the information they required from their tests.

(22) Testing to Determine Lithium Aluminate Solubility in Simulated Reactor Coolant Solutions

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

The ACL was asked by the TTQP, which is administered by Pacific Northwest National Laboratory (PNNL), to carry out specific tests that would provide insight into the solubility behavior of lithium aluminate ceramic pellets in aqueous solutions that simulate the environment of a nuclear reactor coolant system. Conditions for the tests involved sealing a test-pellet segment, together with a measured quantity of aqueous solution having a specified composition, in a fused-silica ("quartz") tube and heating the tube to 300°C for 72 hours under conditions that prevent the tube from rupturing under the pressure buildup. The ACL employed a similar technique in previous work with hydrochloric acid as the solution to completely dissolve the refractory ceramic for chemical analysis to characterize its constituent elements.

The solubility tests followed a detailed test plan and procedures, which covered operations involved in exposing the test pellets to the specified solutions, collecting solutions after exposure, measuring the pH of the solutions, and preparing samples of the solutions for analysis by ICP/AES to measure dissolved lithium and aluminum. Three solutions were tested in triplicate: (1) pure water; (2) borated water (2000 ppm boron as boric acid); and (3) borated water at pH 6.65 (borated water adjusted for pH by adding LiOH). The test results showed that all the solutions were made fairly basic by leaching of lithium from the ceramic pellets and that these basic solutions attacked the silica tubes under the test conditions.

The behavior observed for lithium in the test samples was consistent with release by an ion-exchange mechanism in which lithium ions in the $LiAlO_2$ matrix are displaced by hydrogen ions from solution. The release of aluminum into solution from the pellet specimens was considerably lower than the lithium release and was consistent with behavior expected from solubility considerations for Al_2O_3 and its hydrates. These results suggest that contact of lithium aluminate ceramic with reactor coolant could easily transfer percent fractions of the available lithium into solution but should only solubilize tiny fractions (on the order of 0.01%) of the aluminum in the ceramic. Engineers at PNNL will use this information to evaluate potential consequences of hypothetical cladding failures that could allow reactor coolant to contact the ceramic target material in tritium-production target assemblies.

(23) Liquid-Release Pressure Testing of Commercial Absorbent (L. B. TenKate and D. G. Graczyk)

Through the Technical Services Program administered by ANL's Industrial Technology Development Center, the ACL provided assistance to a small business (Phoenix International, Inc., Hinsdale, IL) that sought performance data for one of its products. The product, PetroFree Multipurpose Sorbent, is a specially formulated granular absorbent for oil, fuel, and other petroleum-based liquids. One potential application for this absorbent is to stabilize radioactive petroleum-based waste liquids for disposal at appropriate facilities. At least one such facility (the Hanford Site) requires that sorbents used for liquid wastes must pass a liquid-release pressure test to demonstrate that liquids will not separate from the absorbent when it is subjected to overburden pressures in storage or burial situations. For applications pertinent to the PetroFree sorbent, the loaded sorbent must pass a minimum 20 psig liquid release test as determined with the test procedure in U.S. EPA Method 9096.

To provide data for the PetroFree sorbent, the ACL carried out a series of pressure tests that determined the liquid loading at which release occurred under various pressures up to 40 psig, using petroleum-based liquids typical of low-level radioactive waste streams encountered at DOE facilities. The 40 psig tests were requested by Phoenix International to provide performance data at levels that exceeded the Hanford minimum requirement. The liquids tested in this study were vacuum pump oil, hydraulic fluid, and kerosene. In the test protocol that was applied, weighed amounts of test liquid were mixed with weighed portions of sorbent and the mixtures were tested at pressures of 20 and 40 psig in a Liquid Release Test Device manufactured by Associated Design and Manufacturing Co. The data from these tests are expected to allow Phoenix International to pursue designation of their product as an approved absorbent by the Hanford Site. Such designation, they believe, will permit new applications of the product and open new markets to them.

(24) Analysis of Complicated Oxides for Melt Attack and Coolability Experiments

(A.M. Essling, E. A. Huff, D. R. Huff, P. L. Johnson, B. S. Tani, and D. G. Graczyk)

The Reactor Engineering Division (RE) (M. Farmer et al.) at ANL is conducting Melt Attack and Coolability Experiments (MACEs) to investigate the interactions that will occur between molten core components and concrete when the fuel debris from a severe reactor accident comes in contact with the basemat of the reactor containment building under overlying water. The ACL has assisted these studies by providing comprehensive chemical analysis of samples retrieved from test assemblies where prototypic corium is heated to the molten state and allowed to contact a concrete base with a water overlayer. The ACL analyzed a set of about 50 oxide samples that included portions of debris, solidified melts, crusts, and corium from MACEs and earlier Advanced Containment Experiments.

Drawing on prior experience with similar samples, ACL analysts looked at several options for dissolving these highly refractory materials and settled on a lithium tetraborate fusion procedure that left only minor residues undissolved in most cases. The undissolved material was isolated and dissolved with a sealed-tube (Carius Tube) procedure and analyzed separately for inclusion in the sample composition. Solutions obtained for each sample were analyzed by ICP/AES to provide information on 19 component elements. Completeness of each analysis was evaluated by calculating a sum-of-oxides mass balance; results ranged from 88 to 107%, with an average near 100%. Selected samples were also examined by XRD to identify major chemical phases present in them. The data from all the samples are being used by the MACE program to help interpret information they recorded when individual frozen debris regions were formed during the tests. The data are also being used to develop and validate phase-segregation models employed in interpreting the MACE results.

(25) Alternative Methods for Analysis of Lithium Aluminate Ceramics (D. G. Graczyk, A. M. Essling, F. P. Smith, and C. T. Snyder)

The ACL has been providing data from chemical analysis of lithium aluminate ceramic materials to the Tritium Target Qualification Project (TTQP). This project has been administered by Pacific Northwest National Laboratory (PNNL) for nearly two years during which the program has worked to establish comparable capabilities in the private sector. Some of the procedures that we use in the ACL for specific analyses were developed years ago and are required for the current work because they are established and qualified, even though alternatives exist that might be more attractive in a higher volume production situation. Recently, PNNL asked analysts in the ACL to investigate two alternative methods that appeared to offer particular advantages.

One study looked at an instrumental combustion method for total carbon to replace the acid-reaction/carbon-dioxide-evolution method currently used to measure carbonate impurity in lithium aluminate. The total carbon determination is fast and efficient. The total carbon in specimens we tested correlated strongly with carbonate content but showed that the ceramics also contained carbon in forms other than carbonate. Based on these results, the TTQP is reviewing the lithium aluminate specifications and expects to replace the carbonate limit with a limit on total carbon. This change will better address the basis for the carbonate limit and, at the same time, permit use of the more efficient instrumental method by laboratories that test the ceramic materials.

The other alternative method involves replacing the current sealed-tube (Carius Tube) method we use for dissolving dense gamma-phase lithium aluminate for subsequent analysis operations with a dissolution that uses a newly available microwave-heated sample digestion system. The new microwave digestion apparatus has performance specifications that allow heating the Teflon digestion vessels to temperatures approaching the Carius Tube's 300°C. Work completed so far has demonstrated that the microwave system is capable of achieving complete dissolution. However, we have found that performance is not uniform when multiple vessels are heated at the same time. Because the microwave system promises convenient operation, enhanced efficiency, and ready transfer of operations to commercial laboratories, we plan to pursue resolution of this shortcoming in the next fiscal year.

(26) Performance Testing of Chemically Bonded-Phosphate-Ceramic Treatment of Fernald Silo 1 Waste (L. B. TenKate, D. L. Bowers, T. TenKate, and D. G. Graczyk)

Fluor Daniel Fernald is currently evaluating options for remediation of material stored in Silos 1 and 2 of Operable Unit 4 at the Fernald site in Ohio. The material in these silos consists predominantly of residues from pitchblende ore processed to extract uranium. They contain both heavy metal contaminants from the extraction processes and uranium daughter nuclides, such as radium, that the extraction processes separated out. Researchers (A. Wagh and S. Y. Jeong) in ANL's Energy Technology Division (ET) carried out a series of bench-scale tests with material from Silo 1 to determine whether it could be satisfactorily stabilized by treatment to form a chemically bonded phosphate ceramic.

The ACL supported this study by carrying out performance tests on samples of the treated waste using the U.S. EPA Toxicity Characteristic Leaching Procedure (TCLP). This testing required careful planning and handling of the test samples because of their relatively high radium content and concomitant emanation of radium's daughter, radon. The TCLP leachates were analyzed to determine regulated metals and radioactivity. Metal concentrations were found to be well below EPA's Universal Treatment Standards. Total alpha and beta activity in the leachates were also quite low. The results of this study indicated that the chemically bonded phosphate ceramic process may be a suitable way to package the Silo 1 waste for transportation and storage.

(27) Quality Assurance Laboratory Support to the U.S. Army Corps of Engineers

(D. W. Green, F. J. Martino, D. L. Bowers, A. S. Boparai, D. G. Graczyk, L. L. Smith, F. Markun, T. TenKate, A. M. Essling, E. A. Huff, D. R. Huff, L. B. TenKate, L. L. Chromizky, and Y. Tsai)

In October 1997, the U. S. Army Corps of Engineers (USACE) assumed responsibility for the Formerly Utilized Sites Remedial Action Program (FUSRAP), which had been the responsibility of the DOE since 1974. The objectives of this program are to study and remediate sites having radioactive materials from DOE and its predecessors' operations, dating back in some cases to the Manhattan Project. In late FY 1998, the Buffalo and Baltimore Districts of the USACE requested the ACL to serve as a Quality Assurance Laboratory for the analysis of ambient matrix samples (e.g., soil, sediment, ground or surface water) from various remediation sites. In this role, the ACL received and analyzed split samples for organic, inorganic, and radiological parameters to provide reference data that may be used to corroborate analysis results obtained by the projects' primary production laboratories on samples from the same locations. All organic and inorganic analytes have been determined using existing ACL facilities and procedures and methods described in the U.S. EPA laboratory manual *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, SW-846, in accordance with instructions from the USACE. Radiological measurements were carried out according to U.S. EPA methods and established ANL methods and procedures.

In FY 1998, the ACL received samples from two sites under the jurisdiction of the Buffalo District: the Luckey Site in Ohio, which was a magnesium and beryllium processing facility that received radiologically contaminated scrap metal from the Atomic Energy Commission, and the

Ashland 2 site in Tonawanda, NY, which was used from 1974 to 1982 to store soil containing low-level radioactive residues. Samples from the Colonie, NY site, which is under the jurisdiction of the Baltimore District, were also received. Analysis has begun on these samples and will be completed in early FY 1999.

(28) Top-of-Rail Lubricant By-Product Characterization (A. S. Boparai and Y. Tsai)

The Energy Technology Division (ET) (G. Fenske et al.) is collaborating with Tranergy Inc. and Texaco in testing several proprietary lubricants developed by Texaco to reduce friction between the rail tracks and locomotive wheels. The reduction in friction should lead to lower energy consumption by the locomotive engines and less wear on the rail hardware. When the engines are in front of the train, the lubricant is applied on the rail tracks behind the engines to allow normal traction between the tracks and the wheels of the engine while friction between the tracks and the rail cars is minimized. The automated system that applies the lubricant calculates the amount of lubricant needed based on weight of the rail cars and the length of the train. The lubricant is expected to significantly lose its lubrication power during use by a train before the next train uses the same tracks.

One of the tasks of the project is to identify volatile and semivolatile by-products produced during use of the lubricants. A device prepared by Tranergy Inc. that allows accurate measurement of friction between a simulated track and wheel system was used to apply lubricant and collect residue from the tracks after defined periods of use. The ACL analyzed samples of lubricant before and after use. The ACL utilized GC/MS to identify major components of the proprietary lubricant. In preliminary work, only minor semivolatile by-products have been detected in the lubricant after its use in the test apparatus. Identification of these compounds is in progress. A device is under construction to collect VOCs that may be produced during use of the lubricants.

(29) Development and Implementation of Software for Direct Electronic Transmission of ICP-AES Analytical Results (D. R. Huff and E. A. Huff)

In 1997, the ACL acquired a new Instruments S. A., Model JY180c, inductively coupled plasma-atomic emission spectrometer (ICP-AES) system. The software associated with this instrument transfers the results of real-time measurements to ASCII files. These data files are not amenable to direct manipulation or compilation of results for a given sample and, if used directly for generating reports of analysis, demand substantial analyst input activity. A project was undertaken to automate conversion of the ASCII file output to a more versatile format and to convert the data into a final analytical report. The objectives of this project were to improve efficiency in data handling and to minimize analyst involvement in processing data from the instrument. This project was carried out with the assistance of Argonne's Support Development Funds (SDF) program in FY 1998.

An Excel program was used to transfer the ASCII data into a spreadsheet. It was formatted to satisfy requirements of the standard ACL "Report of Analytical Results," was produced with a minimum of analyst input, and was in a form that could be electronically transferred directly to the client when requested. This choice was based on the premise that many clients put the data they receive from ACL into spreadsheets or spreadsheet-compatible tables for interpreting and reporting purposes. Thus, a spreadsheet report format would be convenient for them to use. This program was implemented by the CMT Computer Applications Networking and Security Group (J. Copple). It compiles data from several different runs, permits using different dilution factors for different elements, and includes features that allow analyst input of comments or other information. The program is now in use for all samples analyzed with the JY180c system and has proven beneficial to both ACL operations and ACL clients.

III. QUALITY ASSURANCE

Quality Assurance/Quality Control (QA/QC) (F. J. Martino)

The ACL's internet home page has been developed to provide ready access by ACL Staff to important ACL QA documents. Included are over 230 standard operating procedures (SOPs) covering ACL analytical and administrative operations, the *ACL Quality Assurance (QA) Plan*, and the *ACL Good Automated Laboratory Practices*. Links to other Argonne, DOE, and EPA Internet sites have been identified and made available to ACL personnel to provide additional QA/QC support. The general public may access the ACL Home Page at http://www.cmt.anl.gov/acl/acl.htm, but selected ACL QA documents are accessible by only ACL personnel through the use of their computer account password.

Audits/assessments play an important role in helping to ensure client satisfaction. Each audit or assessment is intended to evaluate the following: (1) the degree to which ACL performance corresponds to stated performance requirements; (2) the adequacy of processes established to achieve quality; (3) the performance of ACL scientific staff in meeting the requirements of the ACL QA Plan and program-related plans and statements of work; and (4) the existence of documents and procedures. Three audits/assessments of the ACL were performed in 1998 to verify analytical capabilities and practices in support of programs for which the ACL provides analytical services. In April, the Nuclear Waste Management Section of ANL audited the ACL as a provider of services. In September, the U.S. Army Corps of Engineers audited documentation of ACL analytical methods and facilities. In October, the State of Utah Department of Laboratory Improvement audited the ACL to renew its state certification, which permits the ACL to analyze low-level mixed waste intended for disposal at the facility Envirocare of Utah, Inc. All corrective action issues identified during these audits/assessments are being addressed, with resolutions expected by December 31.

Performance evaluation (PE) programs that provide intercomparison studies for participating laboratories are a means for independent evaluation of analytical performance for the ACL. Blind PE samples ("blind" refers to analyte values known only to the monitoring agency) are received and analyzed semiannually. The programs are usually a requirement for laboratories providing analytical results in support of EPA or DOE programs, and in some cases, are also a requirement for laboratories seeking state certification. Each program, coordinated and monitored by the EPA or DOE, has served to help ensure the quality of analytical results. Required programs in which the ACL participated in FY98 include the following:

- U.S. EPA

  National Exposure Research Laboratory (NERL-Ci)–Cincinnati, Water Pollution Study (WP): Water samples were analyzed to determine VOCs, PCB/pesticides, metals, nutrients, cyanide, oil and grease, total dissolved and suspended solids, and pH. Corrective actions are being addressed for the one WP039 analyte for which the concentration level was incorrectly reported. The EPA summary report for WP040 results reported by the ACL has not yet been received.

  National Exposure Research Laboratory–Cincinnati, Water Supply Study (WS): Water samples WS039 and WS040 were analyzed for barium. Analytical results for barium in WS039 agreed well with the EPA known values; results for WP040 have not yet been received.

National Exposure Research Laboratory–Las Vegas, Performance Evaluation Studies Program: Water samples were analyzed for uranium, tritium, and $^{226/228}$Ra. A comparison of ACL results to the known values showed that the ACL correctly measured all analytes.

- U.S. DOE

Environmental Measurements Laboratory Quality Assessment Program (QAP): Water and soil samples were analyzed to determine concentrations of a variety of radionuclides using gross alpha-beta, gamma spectroscopy, and alpha spectroscopy techniques. A comparison of ACL results to known values indicates that the ACL correctly identified and quantified all analytes except for a high concentration for ^{54}Mn in the most recent PE sample (QAP 48). Appropriate corrective actions are being determined to close out this item.

Mixed Analyte Performance Evaluation Program (MAPEP): All laboratories reporting mixed waste analytical measurements for EM-sponsored work are required to participate in this semiannual program. Water and soil PE samples, prepared as a mixture of inorganic, semivolatile organic, and radiological constituents, were analyzed to help ensure the quality and defensibility of analytical measurements generated for client mixed waste samples. The ACL correctly measured all but three analytes in MAPEP soil sample 97-S4 and a water sample 97-W5, which were received in 1998. Corrective actions have been completed for three analytes for which incorrect concentrations were reported. The EPA summary report for results recently reported for MAPEP sample 98-S5 has not yet been received.

IV. PROFESSIONAL ACTIVITIES

A. *Publications and Reports*

Evaluation of FT Raman and FTIR Techniques for Screening of Semivolatile Organic Compounds in an Inorganic Salt Matrix
A. S. Boparai, D. Fimmen, Y. Tsai, K. Parish, and M. Schipma
Proc. of Pittcon'98, New Orleans, LA, March 1-5, 1998

A Switchable Radioactive Neutron Source: Proof-of-Principle
D. L. Bowers, E. A. Rhodes, and C. E. Dickerman
J. Radioanal. Nucl. Chem. **233** (1-2), 161-165 (1998)

Chemistry and Management Education and Training
D. W. Green
Managing the Modern Laboratory **3**(2), 29A (1998)

Forum on Hiring the Right Person the First Time
D. W. Green
Managing the Modern Laboratory **3**(1), 11A (1998)

Missed Communication
D. W. Green
Managing the Modern Laboratory **3**(1), 4A (1998)

Uncommon Sense
D. W. Green
Managing the Modern Laboratory **2**(4), 44A-45A (1996)

Analytical Chemistry Laboratory Progress Report for FY 1997
D. W. Green, A. S. Boparai, D. L. Bowers, D. G. Graczyk, P. C. Lindahl, with contributions from ACL Staff
Argonne National Laboratory Report ANL/ACL-97/5 (1997)

Uranium Isotopic Composition and Uranium Concentration in Special Reference Material SRM A (Uranium in KCl/LiCl Salt Matrix)
D. G. Graczyk, A. M. Essling, C. S. Sabau, F. P. Smith, D. L. Bowers, and J. P. Ackerman
Argonne National Laboratory Report ANL/ACL-97/3 (1997)

An Experimental Analysis of the Feasibility of Fluid-Based Radon Removal Systems
R. E. Nietert, F. Markun, K. V. Liu, and J. D. Gabor
NT Technical Memorandum No. 43, Reactor Analysis Division (1998)

Extraction of Semivolatile Organic Compounds from High-Efficiency Particulate Air (HEPA) Filters by Supercritical Carbon Dioxide
J. B. Schilling
Argonne National Laboratory Report ANL/ACL-97/6 (1997)

History of the Recommended Atomic-Weight Values from 1882 to 1997: A Comparison of Differences from Current Values to the Estimated Uncertainties of Earlier Values
L. Schultz, R. D. Vocke, J. K. Böhlke, H.-J. Dietze, T. Ding, M. Ebihara, J. W. Gramlich, A. N. Halliday, J.-J. Kluge, H. R. Krouse, R. D. Loss, G. I. Ramendik, D. E. Richardson, M. Stiévenard, P. D. P. Taylor, J. R. deLaeter, P. DeBièvre, Y. Xiao, M. Shima, N. N. Greenwood, and H. S. Peiser
Pure Appl. Chem. **70** (1), 237-257 (1998)

Waste Minimization in Analytical Chemistry through Innovative Sample Preparation Techniques
L.L. Smith, J.H. Aldstadt, J.S. Alvarado, L.L. Chromizky, C.S. Sabau, B. Szostek, and J.A. Tinklenberg
Proc. of the U.S. DOE Pollution Prevention Conference XIV, Seattle, WA, June 1-4, 1998

Radiostrontium Analysis Using Sorbent Disks
L. L. Smith, J. S. Alvarado, K. A. Orlandini, K. M. Hoffmann, D. C. Seely, and R. T. Shannon
Current Protocols in Field Analytical Chemistry, John Wiley, New York (1998)

B. *Oral Presentations*

A System for Rapid Water Sampling and Analysis Based on Empore Membranes and Solid Scintillation Spectrometry
J. H. Aldstadt, K. A. Orlandini, L. L. Smith, D. C. Seely (presenter), and I. R. Shaw-Epperson
43rd Conference on Bioassay, Analytical, and Environmental Radiochemistry, Charleston, SC, November 9-13, 1997

Study of Effect of Spectral Resolution on Pattern Recognition Using Passive Fourier Transform Infrared Sensor Data
A. S. Bangalore
29th ACL Technical Talks, May 12, 1998

Evaluation of FT-Raman Spectroscopy for Determination of Semivolatile Organic Compounds in WIPP Sludge Samples
A. S. Boparai
29th ACL Technical Talks, May 12, 1998

Study of the Effect of Spectral Resolution on Pattern Recognition Results Using Passive FTIR Sensor Data

A. S. Bangalore, A. S. Boparai, and G. W. Small

49th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, New Orleans, LA, March 1-5, 1998

Technical Support for the Waste Isolation Pilot Plant (WIPP) Project

A. S. Boparai

Poster session, University of Chicago Review Committee, September 24, 1998

A Study of Isotopic Homogeneity in "Blended Uranium" Using ICP/MS

J. S. Crain, D. R. Huff, J. J. Marr, and P. Roach

49th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, New Orleans, LA, March 1-5, 1998

IPEP: The Department of Energy's Integrated Performance Evaluation Program; A Tool in a Total Quality System

J. J. Dahlgran, P. C. Lindahl, W. E. Streets, and W. R. Newberry

43rd Conference on Bioassay, Analytical, and Environmental Radiochemistry, Charleston, SC, November 9-13, 1997

Comparison of Infrared and Mass Spectroscopy as Methods of Continuous Emission Monitoring

J. C. Demirgian and C. T. Snyder

49th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, New Orleans, LA, March 1-5, 1998

Analysis of Lithium Aluminate for Tritium Production

A. M. Essling

29th ACL Technical Talks, May 12, 1998

Challenging Projects in Analytical Support--Two Examples

D. G. Graczyk

University of Chicago Review Committee, September 25, 1998

Chemical Analysis Support to the Tritium Target Qualification Project

D. G Graczyk

CMT Technical Seminar Series, June 19, 1998

Chemical Imaging

D. W. Green

49th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, New Orleans, LA, March 1-5, 1998

News from Argonne
D. W. Green
16th Annual DOE Analytical Managers Association Meeting, Cincinnati, OH, August 18-20, 1998

The Spectrum of Analytical Needs
D. W. Green
DOE Office of Environmental Management National Analytical Management Program (NAMP'98) Workshop, Bethesda, MD, June 2-5, 1998

Audit and Performance Evaluation Management–Overview of IPEP
P. C. Lindahl
DOE Office of Environmental Management National Analytical Management Program (NAMP'98) Workshop, Bethesda, MD, June 2-5, 1998

Results of Pilot Implementation Studies of DOE's Integrated Performance Evaluation Program (IPEP)
P. C. Lindahl, W. E. Streets, J. J. Marr, K. J. Parish, A. E. Scandora, Jr., G. A. Anast W. R. Newberry, J. R. Dahlgran, C. Watkins, J. Connolly, and J. Fisk
37th ORNL/DOE Conference on Analytical Chemistry in Energy Technology, Gatlinburg, TN, October 7-9, 1997

Performance Testing of Empore™ Rad Disks
L. L. Smith
Poster session, University of Chicago Review Committee, September 24, 1998

Waste Minimization in Analytical Chemistry through Innovative Sample Preparation Techniques
L. L. Smith, J. H. Aldstadt, J. S. Alvarado, L. L. Chromizky, C. S. Sabau, B. Szostek, and J. A. Tinklenberg
U.S. DOE Pollution Prevention Conference XIV, Seattle, WA, June 1-4, 1998

An Evaluation of Empore™ Radium Rad Disks
L. L. Smith, F. Markun, and J. S. Alvarado
43rd Conference on Bioassay, Analytical, and Environmental Radiochemistry, Charleston, SC, November 9-13, 1997

Comparison of Infrared and Mass Spectroscopy as Methods of Continuous Emission Monitoring
C. T. Snyder
29th ACL Technical Talks, May 12, 1998

The Integrated Performance Evaluation Program (IPEP): Technical Support for DOE's National Analytical Management Program

W. E. Streets

University of Chicago Review Committee, September 25, 1998

Integrated Performance Evaluation Program (IPEP)

W. E. Streets and P. C. Lindahl

Idaho National Engineering and Environmental Laboratory, Radiological and Environmental Sciences Laboratory, Idaho Falls, ID, August 11-12, 1998

Development of Evaluation Protocols for DOE's Integrated Performance Evaluation Program (IPEP)

W. E. Streets, P. C. Lindahl, J. J. Marr, K. J. Parish, A. E. Scandora, Jr., G. A. Anast, and W. R. Newberry

37th ORNL/DOE Conference on Analytical Chemistry in Energy Technology, Gatlinburg, TN, October 7-9, 1997

Status of Pilot Implementation of DOE's Integrated Performance Evaluation Program (IPEP)

W. E. Streets, P. C. Lindahl, J. J. Marr, K. J. Parish, A. E. Scandora, Jr., J. M. Copple, G. A. Anast, and J. R. Dahlgran

43rd Conference on Bioassay, Analytical, and Environmental Radiochemistry, Charleston, SC, November 9-13, 1997

C. *Awards*

Alice M. Essling

The Kenneth J. Jensen Award for Excellence, presented by the Analytical Chemistry Laboratory, Chemical Technology Division, Argonne National Laboratory, November 1997

Florence P. Smith

National Organization of Black Chemists and Chemical Engineers Meritorious Service Award, 1998

Alice M. Essling and Florence P. Smith

Pacesetter Award, for work on lithium aluminate samples for the Tritium Target Qualification Program, December 1997

D. *Meetings Attended*

Delbert L. Bowers

ASTM Meeting, Committee C-26, San Diego, CA, January 11-16, 1998

David W. Green
FY 1998 CMST Annual Review Meeting, Gaithersburg, MD, April 7-9, 1998

Paul L. Johnson
American Crystallographic Association, Arlington, VA, July 18-23, 1998

Peter C. Lindahl
11th Annual Workshop of the Interagency Steering Committee on Quality Assurance for Environmental Measurements, Tysons Corner, VA, June 2-4, 1998
4th Annual Meeting of the National Environmental Accreditation Conference, San Antonio, TX, June 29-July 2, 1998

W. Elane Streets
4th Annual Meeting of the National Environmental Accreditation Conference, San Antonio, TX, June 29-July 2, 1998

E. *Professional Organizations and Activities*

Amrit S. Boparai
    American Chemical Society

Delbert L. Bowers
    American Chemical Society
    American Society for Testing and Materials

Donald G. Graczyk
    American Chemical Society
    Sigma Xi
    American Society for Mass Spectrometry

David W. Green
    American Chemical Society
        Division of Analytical Chemistry
    Analytical Laboratory Managers Association, Board of Directors
        DOE Analytical Managers Group, Board of Directors
    DOE Characterization Monitoring, and Sensor Technology (program reviewer) Gaithersburg, MD, April 16-18, 1997
    Editor, Managing the Modern Laboratory
    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

Mary Ann Hejny
- International Association of Administrative Professionals, Argonne Chapter

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

Paul L. Johnson
- American Crystallographic Association
- Association for Computing Machinery
- 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, Chicago Section
  - Division of Nuclear Chemistry and Technology,
- 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
Lynn B. TenKate
Society for Applied Spectroscopy, Chicago Section
Sigma Xi

F. *Professional Training*

Daniel V. Applegate
Basic Training Course for PlasmaQuad 2 ICP/MS, June 15-16, 1998, Riverside, CA

Alice M. Birmingham
Managing Your Career for Success, March 19, 1998, Argonne, IL

Laurie Carbaugh
Managing Your Career for Success, March 19, 1998, Argonne, IL

Mary Ann Hejny
Managing Your Career for Success, March 20, 1998, Argonne, IL

Jane J. Marr
Advanced Excel, February 16, 1998;
Introduction to Access, April 16, 1998
Intermediate Access, April 23, 1998

Florence P. Smith
Carbon/Sulfur Analyzer C/S300 Training, June 23-24, 1998, St. Joseph, MI

Christine T. Snyder
Carbon/Sulfur Analyzer C/S300 Training, June 23-24, 1998, St. Joseph, MI

W. Elane Streets
Introduction to Access 7.0, April 16, 1998
Intermediate Access 7.0, April 23, 1998

Lisa Wolcott
Managing Your Career for Success, March 20, 1998, Argonne, IL

G. *ACL Seminars*

John F. Schneider and Hugh J. O'Neill, "Analytical Chemistry of Chemical Warfare Agents," April 16, 1998

John F. Suermann, "Overview of the Waste Isolation Pilot Plant Project," August 21, 1998

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