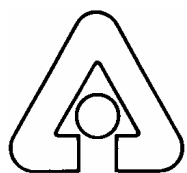


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## **Analytical Chemistry Laboratory**

### Progress Report for FY 2000



Chemical Technology Division  
Argonne National Laboratory, Argonne, Illinois 60439  
operated by The University of Chicago  
for the United States Department of Energy under Contract W-31-109-Eng-38

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**ANALYTICAL CHEMISTRY LABORATORY**  
**Progress Report for FY 2000**

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Analytical Chemistry Laboratory  
Chemical Technology Division

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## NOMENCLATURE

ACL	Analytical Chemistry Laboratory
ANL	Argonne National Laboratory
ATW	Accelerator Transmutation of Waste
CLP	Contract Laboratory Program
CMT	Chemical Technology Division
CVAA	cold vapor atomic absorption
D & D	decontamination and decommissioning
DEMSAR	Directory of EM Sampling and Analysis Resources
DOD	Department of Defense
DOE	Department of Energy
EM	Environmental Management
EML	Environmental Measurements Laboratory
EPA	Environmental Protection Agency
ET	Energy Technology Division
FBI	Federal Bureau of Investigation
FEMA	Federal Emergency Management Agency
FUSRAP	Formerly Utilized Sites Remedial Action Program
FY	fiscal year
GC/MS	gas chromatography/mass spectrometry
HP	Environmental, Safety, and Health-Health Physics
IAG	Interagency Agreement
IC	ion chromatography
ICP/AES	inductively coupled plasma/atomic emission spectrometry
ICP/MS	inductively coupled plasma/mass spectrometry
ICPT	Integrated Contractor Purchasing Team
IPEP	Integrated Performance Evaluation Program
MARLAP	Multiagency Radiochemistry Laboratory Analytical Procedures
MAPEP	Mixed Analyte Performance Evaluation Program
MS	mass spectrometry
MSDS	material safety data sheet
NAMP	National Analytical Management Program
NAMPnet	NAMP Laboratory Informational Network
NEMI	National Environmental Methods Index
NFSS	Niagara Falls Storage Site
NIST	National Institute of Science and Technology
NSTS	National Sample Tracking System
PCB	polychlorinated biphenyls
PDP	Performance Demonstration Program
PE	performance evaluation
PFS-WMO	Plant Facilities and Services-Waste Management Operations



## NOMENCLATURE (contd)

PMT	photomultiplier tube
PNNL	Pacific Northwest National Laboratory
PT	proficiency testing
QA	quality assurance
QAP	Quality Assessment Program
QC	quality control
RCRA	Resource Conservation and Recovery Act
R&D	research and development
RESL	Radiological and Environmental Sciences Laboratory
RSD	relative standard deviation
SAS	special analytical services
SCD	sequenced charge-coupled device
SEM	scanning electron microscopy
SOP	standard operating procedure
SPME	solid-phase microextraction
TOP	top-of-rail
TRU	transuranic
USACE	U.S. Army Corps of Engineers
VOC	volatile organic compound
WFO	Work-for-Others program
WIPP	Waste Isolation Pilot Plant
WWW	World Wide Web
XRD	X-ray diffraction

# **ANALYTICAL CHEMISTRY LABORATORY**

## **Progress Report for FY 2000**

### **I. INTRODUCTION**

This report summarizes the activities of the Analytical Chemistry Laboratory (ACL) at Argonne National Laboratory (ANL) for Fiscal Year (FY) 2000 (October 1999 through September 2000). This annual progress report, which is the seventeenth 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.*

The ACL handles a wide range of analytical problems that reflects the diversity of research and development (R&D) work at ANL. Some routine or standard analyses are done, but the ACL operates more typically in a problem-solving mode in which development of methods is required or adaptation of techniques is needed to obtain useful analytical data. The ACL works with clients and commercial laboratories if a large number of routine analyses are required. Much of the support work done by the ACL is very similar to applied analytical chemistry research work.

Although the Chemical Technology Division (CMT) is the major client of the ACL among ANL divisions, many divisions and programs require high-quality analytical chemistry support from the ACL to carry out their missions. The ACL had three technical groups in FY 2000 — Chemical Analysis, Instrumental Analysis, and Organic Analysis. Although staffing levels varied during the year, the ACL included about 20 technical staff members. (See Fig. 1.) Talents and interests of staff members cross group lines, as do many projects within the ACL.

The Chemical Analysis Group primarily measures the inorganic composition of non-radioactive samples. They use wet-chemical and instrumental methods for elemental, compositional, and isotopic determinations in solid, liquid, and gaseous samples and provide specialized analytical services. The Instrumental Analysis Group does most of the ACL's radiological analyses and inorganic analysis of radioactive materials. They use nuclear counting techniques to determine the radiochemical constituents in a wide range of sample types, from environmental samples with low levels of radioactivity to samples with high levels of radioactivity that require containment, and provide quality assurance support to the U.S. Department of Energy (DOE) and other clients for various kinds of analytical data. The Organic Analysis Group measures the organic composition of both radioactive and non-radioactive samples. They use a number of complementary techniques to separate organic compounds and measure them at trace levels. 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
- ACL Annual Reports for FY 1995 through FY 1999

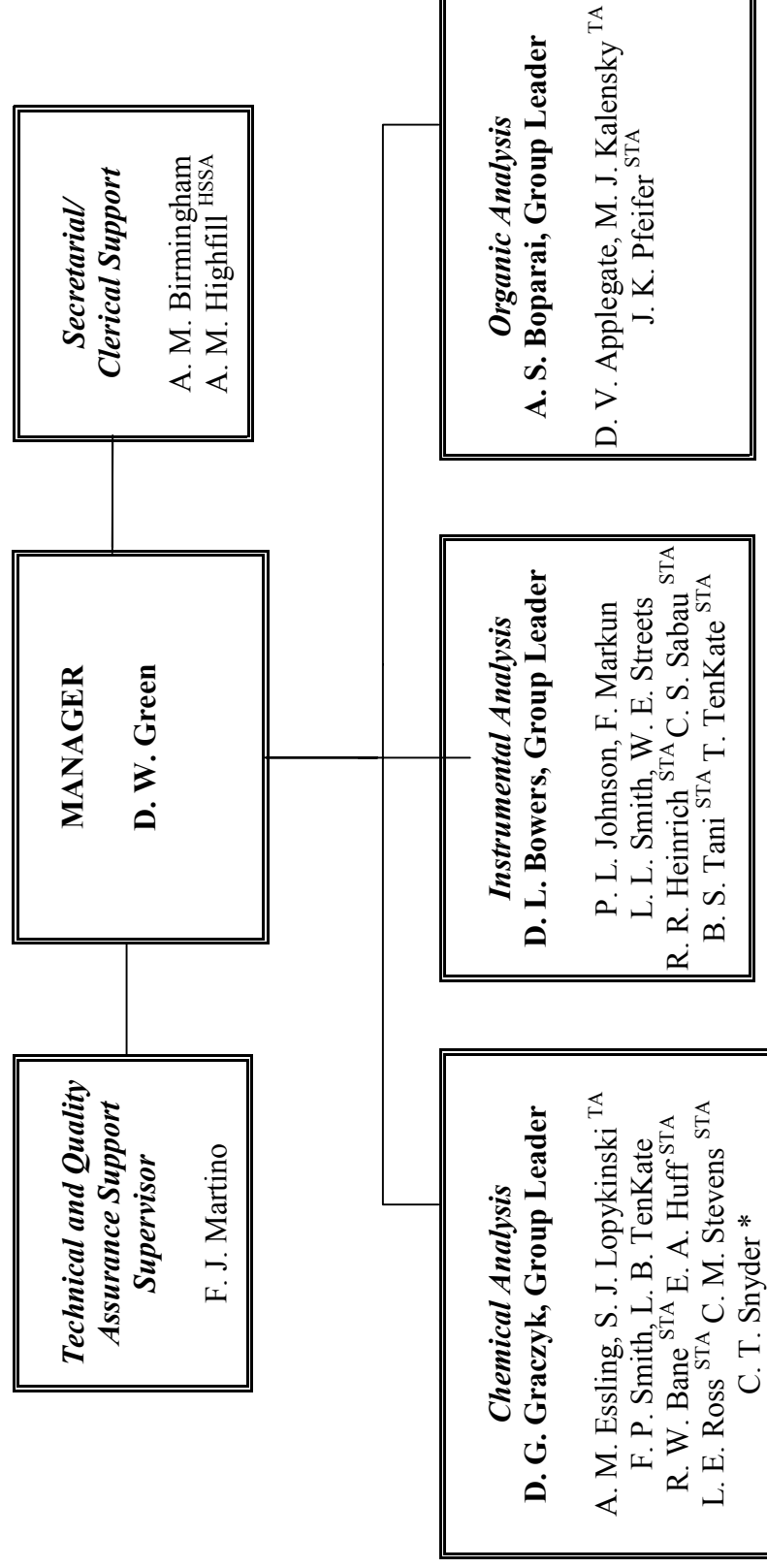
The ACL has continued the development of its Web site for internal use, including the siting of the current version of all ACL standard operating procedures (SOPs) and several quality assurance documents. With this mode of operation, we can readily access the most recent version of each SOP. A listing of analytical equipment and all current divisional safety reviews were added to the Web site this year.

A new inductively coupled plasma/atomic emission spectrometer (ICP/AES) system with an array detector was procured and installed. This system, which was funded by a project for Pacific Northwest National Laboratory (PNNL), allows more accurate measurements, in some cases, to eliminate certain bias errors. Installation was completed of a new alpha spectrometer and data handling system that was procured in FY 1999. No General Purpose Equipment Funds were received in FY 2000.

The remainder of this report comprises highlights and short descriptions of the analytical work performed by the staff of the ACL in FY 2000 on selected projects. Additional information about each of these projects can be obtained from the ACL Office and the ACL staff. Of the

approximately 1000 different jobs in FY 2000, many of which involve several samples, only a few are described in this report. This report includes descriptions of work by ACL staff on selected projects. Many contributions by the ACL staff to smaller projects are not included in this report. However, the analytical data in these cases provide important quantitative data for decisions made by the project generating the samples. These contributions to projects, which can easily be overlooked, collectively form an important part of the mission of the ACL.

**Fig. 1 Analytical Chemistry Laboratory  
Organization Chart  
September 2000**



HSSA = High School Student Aide; STA = Special Term Appointee; TA = Term Appointee.

\*Part-time assignment.

## II. TECHNICAL HIGHLIGHTS

### (1) Nuclear Technology (D.L. Bowers)

The Chemical Technology Division is participating in the Accelerator Transmutation of Waste (ATW) Program whose objective is to reduce the long-lived radioactivity in nuclear waste by transmuting selected nuclei. One of the key process steps in treating ATW fuel is the separation of zirconium from the transuranic elements (TRUs). In the proposed process, the zirconium will be chlorinated to form a volatile tetrachloride, while Pu and the TRUs will form stable chlorides and be soluble in a chloride salt. Experiments were conducted recently to demonstrate (1) the removal of Zr from a LiCl-KCl eutectic salt, (2) the removal of Zr from a LiCl salt, and (3) the removal of Zr from a LiCl salt in the presence of Pu. Measurements of the elemental compositions in these experiments were done using inductively coupled plasma/atomic emission spectrometry in a glovebox facility designed to analyze  $\alpha$ -containing materials. The analytical results indicated that zirconium could be removed as the tetrachloride from the LiCl salt, but not from the salt containing KCl. This behavior is due to formation of a strong K-Zr-Cl complex ( $K_2ZrCl_6$ ). Additionally, the analytical data indicated that Pu could be separated from Zr because it is converted to soluble  $PuCl_3$  with the chlorinating agent used ( $CdCl_2$ ).

### (2) WIPP Performance Demonstration Program for Resource Conservation and Recovery Act Constituent Analysis of Solidified Wastes (A. S. Boparai and M. J. Kalensky)

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 40 km east of Carlsbad, NM. The TRU waste destined for WIPP has been generated since the 1940s from the nation's nuclear

weapons programs at various DOE sites. The first shipment of waste for disposal was received at the WIPP site in March 1999.

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

The purpose of the RCRA PDP is to test the ability of various laboratories to analyze solidified TRU waste samples for specified metals, semivolatile and volatile organic compounds, and polychlorinated biphenyls (PCBs). The Carlsbad Area Office approves laboratories that successfully analyze blind audit samples for analyzing the TRU waste samples destined for WIPP.

During FY 2000 the ACL prepared approximately 2 kg each of uncemented and cemented simulated Type 1 solidified waste. These unspiked waste matrices were then supplied to a contractor selected by the PDP coordinator to be spiked with metal analytes. The spiked samples were checked by the contractor for homogeneity before shipment to the laboratories for analysis. The contractor also provided blind audit samples of semivolatile and volatile organic compounds to participating laboratories. These samples utilize a matrix that does not interfere with determination of the target analytes. In the future, RCRA PDP for metals will also utilize a non-interfering matrix.



(3) Long-Term Performance of Fuel Reforming Catalysts (D. V. Applegate, F. P. Smith, A. M. Essling, and S. J. Lopykinski)

The ACL is assisting CMT's Fuel Cell Group in testing the long-term performance of several of its fuel reforming catalysts. This task requires the operation of a new microreactor system that will run unattended safely for up to 1000 hours. The microreactor allows the catalyst to reform hydrocarbon fuel (pump gasoline) at approximately 700°C, breaking it down to hydrogen, carbon dioxide, and carbon monoxide. In FY 2000 the system was upgraded to include two complete microreactor systems, which allows the simultaneous testing of two catalysts. Several successful 1000-hour tests were completed in FY 2000. In order to evaluate the performance of the catalyst over time, an analytical system was set up to monitor the total gas-flow output, the apparatus's individual pressures and temperatures, and the gas composition. A backup gas chromatograph was also set up to analyze the output gas to look for unknown compounds and to confirm the primary analytical system. This gas chromatograph has a thermal conductivity detector and a mass selective detector with two different columns to look for a full range of permanent gases and light hydrocarbon gases. This gas chromatography/mass spectrometry system was upgraded in FY 2000 with new heated sample loops that enable the system to also analyze for condensable gases. The ACL also continues to assist the Fuel Cell Group by analyzing the inorganic composition of the catalysts. Analytical results for samples taken before and after the 1000-hour tests help shed light on the catalyst's operation and performance. The inorganic characterization tests include inductively coupled plasma/atomic emission spectrometry, inductively coupled plasma/mass spectrometry, and carbon, nitrogen, and sulfur analysis.

(4) Performance Demonstration Program for Analysis of Simulated Headspace Gases for the Waste Isolation Pilot Plant Project (A. S. Boparai, D. V. Applegate, and M. J. Kalensky)

The Waste Isolation Pilot Plant (WIPP) is expected to receive about 37,000 shipments of waste over the next 35 years. Among other characterizations of waste destined for WIPP, analysis of headspace gas for specified volatile organic compounds is required. The National TRU Program Office of the Carlsbad Area Office of DOE has established a Performance Demonstration Program (PDP) for laboratories that will participate in the analysis of headspace gas to be shipped to the WIPP site. The Carlsbad Area Office approves laboratories to analyze headspace gases after they are successful in analyzing blind audit samples of simulated headspace gases. The ACL was selected as the laboratory to prepare, verify, and distribute samples for the headspace gas PDP.

During FY 2000, the ACL prepared eleven sets of headspace gas PDP samples for Cycle 14A. Nine sets were sent to participating laboratories: TRUgas, NFT Inc., Idaho National Engineering and Environmental Laboratory (INEEL), (a set of samples at 6 psig and another at 30 psig), Rocky Flats Environmental Technology Site, Los Alamos National Laboratory, Savannah River Site, Environmental Protection Agency/Research Triangle Park, and Fluor Hanford. One set was used for verification analysis while another was archived for potential future needs. Each set of PDP samples consisted of 6-L SUMMA canisters containing low, high, special, and duplicate mixtures of analytes. A blank containing Ultra Zero grade air was also included in each set.

(5) The Department of Energy's National Analytical Management Program: Projects at Argonne National Laboratory (W. E. Streets)<sup>1</sup>

In response to a request from the Inspector General in 1990, the Department of Energy's Office of Environmental Management (DOE-EM) was charged with developing and implementing a program to assess the performance of all laboratories providing analytical services to EM programs; DOE-EM established the National Analytical Management Program (NAMP) to administer this initiative. The NAMP has developed related program elements designed to provide an integrated system for assessment of laboratory performance and improvement of data quality. These elements allow DOE Complex personnel to share information about EM contacts at the various sites, pertinent methodologies for environmental restoration and waste management, costs of analyses, and performance by contracted laboratories. They are supported by four centralized databases: the DOE Procedures Database, which was used to produce a World Wide Web (WWW) document entitled *DOE Methods for Evaluating Environmental and Waste Management Samples (DOE Methods Compendium)*; the National Sample Tracking System (NSTS); the Directory of EM Sampling and Analysis Resources (DEMSAR); and the Integrated Performance Evaluation Program (IPEP). The IPEP database is being developed by ACL and has always been located here. In FY 1999, at NAMP's request, we successfully transferred the other three databases from the Radiological and Environmental Sciences Laboratory (RESL) onto ANL servers to facilitate consolidation and streamlining of the gathering of information required for IPEP reports and other projects within the NAMP.

---

<sup>1</sup> Staff members from CMT's Computer, Applications, Network, and Security Group (J. M. Copple, J. E. Kulaga, and A. E. Scandora) assist the ACL in the development of strategies and systems for the NAMP projects.

The IPEP database is designed to provide information on the quality of radiological and non-radiological 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 IPEP collects data and supplies reports for four national performance evaluation (PE) program studies. Two programs managed by EPA are the Water Supply and Water Pollution PE Program Studies. Two programs managed by DOE are the Environmental Measurements Laboratory's Quality Assessment Program and RESL's Mixed Analyte Performance Evaluation Program.

The ACL completed several objectives for the IPEP portion of NAMP during FY 2000. We developed a production version of the common administrative database, which we had demonstrated at a meeting of the data users in October 1999. We updated the information in the administrative database to reflect current EM contacts and administrative structure for all DOE Operations Offices and Sample Management Offices. We linked this common administrative database to the IPEP Performance Evaluation Studies database to allow electronic generation of the IPEP reports. We now accumulate performance evaluation program data and produce reports on laboratory performance on a routine basis.

We also developed the NAMP Laboratory Informational Network (NAMPnet), based on the single centralized administrative database, to allow sharing of information about PE performance, audit information, contracts, and methods. The IPEP and DEMSAR databases and the *U. S. DOE Methods Compendium* are integrated into the NAMPnet, and the system is designed to either coordinate with, or integrate data on audits and laboratory capabilities. The NAMPnet accommodates data from DOE's Integrated Contractor Purchasing Team (ICPT) and regular contracts in various formats. The contract information must be collated with the IPEP

data collected from the various PE programs to determine if a laboratory is participating in appropriate PE programs for analytical work it performs for EM. The NAMPnet is installed on a password-secured WWW server (URL: <http://www.cmt.anl.gov/nampnet/>). The tiered database security system allows appropriate access to sets of information that can be tailored to deliver any reports DOE-EM requires, based on the NAMPnet content. Dynamic links from the NAMPnet Web site to the database allow immediate updates of contact and contract information and rapid publishing and dissemination of IPEP reports after receipt of data from parent programs.

The IPEP staff supported the interagency Methods and Data Comparability Board in the development of the National Environmental Methods Index (NEMI), which is designed to allow rapid communication and comparison of analytical methods and to help ensure that assessment and understanding of methods are more actively considered in all phases of environmental restoration projects. We assessed two NEMI prototypes and extracted summaries of ten methods from the *U. S. DOE Methods Compendium* for the current NEMI prototype database.

(6) Radiological Analysis Support for U.S. EPA Region V (F. J. Martino, T. TenKate, and F. Markun)

In FY 2000, the ANL Work-For-Others (WFO) Program generated a large number of samples requiring analytical effort by the ACL. Since 1994, one such WFO program is an Interagency Agreement (IAG) that provides the EPA with special analytical services (SAS) for field samples collected within Region V. One of the more important requirements of the IAG is that the ACL be readily available and capable of providing analytical services on short notice. The IAG requires that, to the extent possible, SAS samples be analyzed using stringent analytical methods found in the current version of the EPA compendium, "Test Methods for Evaluating

Solid Waste, Physical/Chemical Methods” (SW-846). Alternative methods may be used with prior EPA approval. Recent analytical effort has been focused on hazardous and mixed waste samples received as a part of EPA site remediation activities at the principal sites described below.

A number of analytical samples received in FY 2000 were submitted as a continuation of the Ottawa (IL) and Lindsay Light (IL) site remediation programs that were initiated in 1996. Continued monitoring for  $^{226}\text{Ra}$  and  $^{228}\text{Ra}$  contamination is anticipated through 2001.

- Ottawa (Ottawa, IL): This site originally belonged to a company that applied luminescent paint containing  $^{226}\text{Ra}$  to clock dials. Samples were taken from landfills used to hold waste removed from this site after its demolition, the area around the company building foundation, and from the surrounding grounds. Twenty-seven soil samples were submitted to the ACL for the determination of  $^{226}\text{Ra}$  and  $^{228}\text{Ra}$  by gamma spectroscopy. Analytical results were reported in a format required by EPA’s Contract Laboratory Program, which contained all analytical results, quality control data, instrument output, and other documentation (e.g., notebook entries, chain-of-custody forms) associated with the samples.
- Lindsay Light (Chicago, IL): This site in downtown Chicago originally belonged to a company that manufactured thorium lantern mantles. Current plans for development of that area require EPA sampling the grounds for hot spots of  $^{226}\text{Ra}$ . An extension of this remediation project developed as contaminated material excavated from the Chicago site was traced to a suburban landfill also undergoing commercial development. As part of the remediation activity associated with the Chicago site and the landfill, the ACL received approximately 140 soil samples for the determination of  $^{226}\text{Ra}$  by gamma spectroscopy. All samples have been analyzed and the results forwarded to the Region V Office.

Additional EPA remediation efforts involving the ACL during the past year involved samples from the Millennium (OH), Carey Electronics (OH), Taylor (MI), Morrison (IL), Twin City (MN), Hollander (OH), and St. Claire Oil (IL) sites. The level of analytical participation required for these sites, although critical for remediation decisions, was not as extensive as required for the relatively large Ottawa or Lindsay Light effort. The need instead has been for quick turnaround of results for unique samples resulting from unusual, usually one-time occurrences, such as spills of unknown solvents. Some of the characterizations required identification of isotopes in contaminated material.

Last year the EPA expanded the IAG to include analytical research, which was part of an EPA Counterterrorism program. This research also supports the FBI, FEMA, and other federal and state agencies for the consequence management phase of a response to a terrorist incident. One of the functions has been to develop the ANL Dilute Facility of the Center for Environmental Restorations Systems and make it available to the above agencies as an on-call provider of analytical services for the identification of suspected chemical agents. Additional information about this project can be found in Section 14 of this report.

- (7) High-Precision Assay of Lithium and Aluminum by ICP/AES (D. G. Graczyk, D. R. Huff, S. J. Lopykinski, and A. M. Essling)

Another new method for the chemical analysis of lithium aluminate (see Sections 8 and 16) that the ACL has been seeking to implement is a novel methodology developed at the National Institute of Science and Technology (NIST). It allows exceptionally precise measurements using ICP/AES by applying a drift-correction algorithm to compensate for long-term noise in a series of internal-standard-corrected measurements. The approach has been used by NIST to measure the lithium and aluminum content of lithium aluminate ceramics and offers

considerable advantages compared to the classical methods previously used (isotope dilution for determining lithium and gravimetric analysis for aluminum). The ACL has sought to demonstrate that the NIST approach can be established in non-NIST laboratories where reasonable skills exist. Ultimately, we expect to formulate the NIST approach into a standardized operating procedure that can be qualified and transferred to private sector laboratories for lithium and aluminum assay of production-lot ceramics.

Early work in the ACL showed that the method provided excellent precision [0.1% relative standard deviation (RSD)] analyzing standard solutions with our grating polychromator, which uses a slit/photomultiplier-tube (slit/PMT) detector. Later work with standards that contained lithium and aluminum in differing proportions and measurements with lithium having variable isotopic compositions, however, indicated that the data could be biased. Matrix effects that affected the aluminum measurements were overcome by selecting appropriate ICP/AES operating conditions. With lithium, a more daunting problem arose from isotopic differences between samples and standards. Because the grating polychromator achieves partial resolution of the isotopic emission wavelengths from  $^6\text{Li}$  and  $^7\text{Li}$  and the detector configuration collects light only from a small window on the emission peak, the slit/PMT system does not allow mathematical correction for isotopic differences. This effect ultimately precludes the use of a slit/PMT system for lithium assay with the NIST approach.

This year, we continued investigation of the ICP/AES method with an alternative instrument having a sequenced charge-coupled device (SCD) array detector, comparable to that used by NIST. By allowing the entire peak profile to be integrated for measurement, this system avoids problems associated with peak shifts for isotopic lithium. We discovered, however, that the  $^6\text{Li}$  and  $^7\text{Li}$  isotopes show slightly different sensitivity in their plasma emission and require



independent calibration for accurate analysis of samples with different isotopy. In a series of investigations, we established operating conditions and a calibration strategy that overcame the problems resulting from both matrix effects and lithium-isotope effects. We also worked with colleagues at Pacific Northwest National Laboratory to modify a software tool, which they had developed for data reduction, to include automated application of the formulas we derived to make corrections for isotopic differences in lithium among samples and standards. The combination of our detailed operating instructions and this modified software tool provides a methodology that is relatively easy to apply and that, we believe, will be readily transferable to any capable laboratory. It is especially noteworthy that, although the method was developed and established for lithium and aluminum assays in lithium aluminate ceramics, its implementation was purposely formulated in general terms so that it could be adapted almost effortlessly to other elements and applications.

A measure of the level of performance available from the methodology was obtained from a test series completed near the end of FY 2000. In these tests, a set of five simulated samples containing known amounts of lithium and aluminum were prepared by careful gravimetric mixing of stock solutions that included lithium enriched in the  $^6\text{Li}$  isotope. The simulated samples contained lithium with natural isotopic composition (7.6 at %  $^6\text{Li}$ ), 20 at %  $^6\text{Li}$ , 30 at %  $^6\text{Li}$ , 40 at %  $^6\text{Li}$ , and 95 at %  $^6\text{Li}$ . These five samples were analyzed nine separate times, with the torch assembly in the ICP system disassembled and replaced every third run. The pooled relative standard deviation of the nine runs of five samples was 0.06% for lithium and 0.02% for aluminum. Compared to the as-made concentrations of the simulated samples, lithium showed an average relative difference of -0.05% and aluminum of -0.06%, showing that the procedures seem to work very well indeed.

- (8) Method for Determining Iodide Impurity in Lithium Aluminate (F. P. Smith, A. M. Essling, and D. G. Graczyk)

Measuring small amounts of halides contained in refractory materials is difficult because methods generally used for decomposing the refractory can volatilize the sought-for elements or interfere with their determination. Last fiscal year, we developed a method for measuring fluoride, chloride, and bromide in such materials. That method uses a potassium hydroxide fusion to decompose alumina-based, siliceous, or aluminosilicate refractories, dissolution of the fusate in sulfuric acid, and distillation to isolate the halides for measurement by ion chromatography. Iodide added to individual samples was absent from the distillate, possibly as a result of oxidation to other iodine species such as  $I_2$  or iodate. This year, ACL analysts tested a modification of the procedure for use in determining iodide in lithium aluminate. To recover the iodide, the KOH fusate was simply leached with water and the basic leachate was analyzed directly by ion chromatography. We found that the KOH leachate had to be diluted to run well on the ion chromatograph, but with a pulsed amperometric detector system, good sensitivity could be readily obtained. Tests with ten lithium aluminate ceramic powders that were spiked with known amounts of iodide showed an average recovery of 93.5% at levels corresponding to 0.03 wt % iodide in the solid sample. The detection limit for the method was estimated to be about 36  $\mu\text{g}$  of iodide per gram of lithium aluminate (36 ppm). With this new approach, the ACL has achieved a goal of having methods in place for all impurity elements listed in specifications for lithium aluminate ceramics. It promises to be useful for analysis of glasses, zeolites, wasteform samples, and other materials as well.

- (9) Characterization Of Unidentified Waste Materials (D. G. Graczyk, A. M. Essling, D. R. Huff, S. J. Lopykinski, F. P. Smith, L. B. TenKate, D. L. Bowers, P. L. Johnson, F. Markun, L. L. Smith, B. S. Tani, T. TenKate, D.V. Applegate, A. S. Boparai, and M. J. Kalensky)

Many ANL facilities have unidentified waste materials that have unknown origins, inadequate documentation, or absence of ownership because programs are no longer active. These wastes must be characterized and documented so that they can be disposed of properly. In support of this effort, the ACL performs chemical and radiological analyses to provide the composition and hazards-classification data needed by the treatment/storage/disposal facilities that ultimately process the wastes. Working closely with the Plant Facilities and Services-Waste Management Operations (PFS-WMO) staff, the ACL helps assure that the informational and procedural requirements of the appropriate facility are satisfied.

Many different types of wastes were characterized by the ACL in FY 2000. Jars of wet vermiculite, unidentified radioactive rods, and multi-layered liquids were characterized, as were samples from processing equipment, sludges, and oils from several ANL-E divisions.

When the materials appeared to be pure chemical compounds or simple mixtures, the ACL identified the material and then supplied the submitter with a material safety data sheet (MSDS) listing the pertinent hazard information. Direct identification of solids was provided by X-ray diffraction (XRD) and/or scanning electron microscopy (SEM). Direct identification of organic liquids was provided by GC/MS. Those wastes that could not be identified or that proved to be complex mixtures were evaluated for the RCRA characteristics of ignitability, corrosivity, toxicity and reactivity. These evaluations involved measurement of pH, water miscibility and flash point determinations; mercury analysis by cold vapor atomic absorption (CVAA); and As, Ba, Cd, Cr, Pb, Se, and Ag determinations by ICP-AES. The ACL used GC/MS to identify RCRA target organic compounds, and ion chromatography (IC) to identify

any acids present. In some cases the amount of water in a waste was determined by a Karl-Fischer titration. Wastes from radiologically controlled areas or suspected of containing radioactivity were analyzed by gamma spectrometry, gas proportional counting, liquid scintillation counting, and/or alpha pulse height analysis.

In virtually every case, the characterization of an unidentified waste is a complex process. It involves constant re-evaluation of the analytical data each time a new component of the waste is identified, so that all the necessary analyses are performed and no components of the waste are left unidentified. Thus, the characterization of unidentified wastes is both costly and time-consuming. This fact highlights the need for all ANL-E operations to keep timely and complete records of stored materials and to promptly dispose of unneeded materials.

- (10) 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, S. J. Lopykinski, L. B. TenKate, D. R. Huff, P. L. Johnson, M. J. Kalensky, D. V. Applegate, and J. K. Pfeifer)

The Department of Energy in 1974 initiated a program to study and remediate sites having radioactive materials from DOE's and its predecessors' operations dating back, in some cases, to the Manhattan Project. Responsibility for this program, the Formerly Utilized Sites Remedial Action Program (FUSRAP), was transferred to the U.S. Army Corps of Engineers (USACE) in October 1997.

The Buffalo District of the USACE is responsible for several FUSRAP sites within its jurisdiction. The ACL serves the Buffalo District as a quality assurance laboratory for the analysis of samples of ambient matrices (e.g., soil, sediment, ground or surface water) from various remediation sites. In this role, the ACL helps the Buffalo District ensure the quality of the analytical data provided by the commercial laboratories that do the production analytical work. In FY 2000, the ACL received and analyzed samples from two FUSRAP sites.

In 1944 the Niagara Falls Storage Site (NFSS) was used by the Manhattan Engineering District to store radioactive residues and wastes from uranium ore processing. Radioactive wastes and residues continued to be brought to the site for storage until the late 1950s. In 1982 the DOE began cleanup and consolidation of the radioactive wastes and residues in an earthen containment structure constructed on the property, where they currently remain. The ACL completed 18 radiological measurements, 52 organic measurements, and 19 inorganic measurements in FY 2000 on waters and soils from the NFSS.

The ACL also analyzed eight samples in FY 2000 from the Luckey, Ohio, site. The Luckey site was a magnesium and beryllium processing facility that received radiologically contaminated scrap metal from the Atomic Energy Commission. The ACL had previously provided analytical data on radiological, inorganic, and organic constituents of samples from the Luckey site to help the Buffalo District understand better the data they had received from commercial laboratories. Fewer samples were analyzed in FY 2000 than in the previous year.

For both NFSS and the Luckey site, all organic and inorganic analytes were determined using existing 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 ACL methods and procedures.

(11) Top-of-Rail Lubricant (A. S. Boparai and M. J. Kalensky)

Personnel (G. Fenske, M. Alzoubi, et al.) from the Energy Technology Division (ET), in cooperation with Tranergy, Inc., and Texaco, are testing several proprietary lubricants developed by Texaco to reduce friction between rail tracks and rail car wheels. The reduction in friction

should lead to lower energy consumption by 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. Tranergy, Inc., has developed the SENTRAEN 2000<sup>TM</sup> automated lubricant application system. The system is fully computer-controlled and ensures that nearly all of the lubricant is consumed as the end of the train passes. Lubricant quantity dispensed is a function of grade, speed, curve, and axle load.

One of the tasks of the project is to identify volatile and semivolatile by-products produced during use of the lubricants. A laboratory-scale lubrication and traction testing device has been constructed by Tranergy, Inc., (LA4000<sup>TM</sup> wheel and rail simulator) that allows accurate measurement of friction between a simulated track and wheel system. This device was used to apply lubricant and collect residue from the tracks after defined periods of use. The ET personnel also built a shroud around the device to allow collection of volatile organic compounds (VOCs) on Carbotrap cartridges.

The ACL analyzed samples of a lubricant before and after use and also determined VOCs collected by Carbotrap cartridges. The ACL utilized gas chromatography/mass spectrometry to identify major components of the proprietary lubricant. The results indicated that in the volatile fraction, the only compounds on the Environmental Protection Agency's (EPA) Superfund List of Analytes detected were contaminants from either the room air or other potential contamination sources in the laboratory. Similarly, none of the detected compounds in the semivolatile fraction are on the EPA's Superfund List of Analytes. The major component in the semivolatile fraction was determined to be the major component of the top-of-rail (TOR) lubricant before testing.

Other compounds found in trace quantities either were present in the TOR lubricant or were small fragments from the polymeric component of the TOR lubricant.

(12) Tritium in Concrete Cores from the CP-5 Reactor (L. L. Smith)

The CP-5 nuclear reactor was a direct descendant of CP-1, the University of Chicago reactor that produced history's first controlled self-sustaining nuclear chain reaction. From 1954 to 1979, CP-5 provided neutrons for hundreds of experiments performed by both ANL and outside users, which probed the composition of solids and liquids. The facility is currently undergoing decontamination and decommissioning (D&D).

In support of the D&D program at CP-5, the ACL was asked by ESH-Health Physics (HP) personnel to determine tritium concentrations in concrete cores from the facility's floor. Owing to the volatile nature of the likely tritiated-compounds, careful sample preparation techniques had to be employed. The tritium was determined as tritiated-water using the conventional liquid scintillation technique. The data from the 65 core samples analyzed by the ACL helped HP personnel map the tritium contamination at the CP-5 facility.

(13) Data Package Review (A. S. Boparai)

During FY 2000, personnel from the ANL Environment, Safety and Health Division's Environmental Surveillance and Remediation group (P. Lynch and G. MacMillan) submitted eleven data packages for evaluation of data quality. Because full data packages were not available, summary data were reviewed for conformance with the case narratives given with each data package. The data packages contained data for determination in soil and water of the following: volatile organic compounds, semivolatile organic compounds, PCBs, pesticides, pH,

total metals, total organic carbon, alpha- and beta-containing materials, and tritium species. The ACL review of these data helped ensure compliance with quality assurance requirements.

(14) Determination of Chemical Agents at Low Levels in the Dilute Chemical Agent Laboratory (A. S. Boparai)

Argonne National Laboratory (ANL) has a U.S. Army-certified dilute chemical agent laboratory that is equipped with sample preparation and analytical instrumentation needed for characterizing chemical agents in various media. One of the projects that involves staff members of both the Chemical Technology Division and the Energy Systems Division (J. F. Schneider, L. L. Reed, and H. J. O'Neill) will utilize the capabilities of the dilute chemical agent laboratory to support U.S. EPA Region 5 in their emergency response and cleanup efforts after a potential terrorist incident that could involve chemical agents. Chemical agents most likely to be encountered during such scenarios include, but are not limited to, the following: sarin (Agent GB), soman (Agent GD), Agent VX (a phosphonothioic ester), mustard gas (Agent HD) and tear gas (Agent CS).

One of the EPA support functions will be to provide threat/hazard assessment, sampling, analysis, and subsequent identification of a suspected chemical agent and to advise the lead federal agency regarding actions necessary to protect human health and the environment. ANL will perform analyses for chemical agents on the samples submitted by the U.S. EPA as needed. Currently, ANL is developing gas chromatography/mass spectrometry (GC/MS) methods for samples containing chemical agents collected on solid-phase micro-extraction (SPME) adsorbents. This technique will satisfy the requirement that all material in the laboratory facility be dilute. Most of the work performed so far has been to optimize methods for determining the



presence of Agents GB, GD, HD, and VX using the recently installed Agilent 5973 GC/MS and Varian 2000 GC/ion-trap MS systems.

We are working to obtain calibration data for GB, GD, HD, and VX using direct liquid injections and using headspace gas samples obtained from the SPME technique.

(15) Technical Support for Hanford Immobilized Low-Activity Waste Product Acceptance Tests (P. L. Johnson and B. S. Tani)

The U.S. Department of Energy's Office of River Protection intends to immobilize Hanford's low-activity waste in borosilicate glass for storage and disposal. This borosilicate glass will be buried in a suitable facility, but tests are carried out first to show that the glasses will retain the radionuclides and not allow the spread of contamination.

Working together with CMT's Waste Materials Research Department, a series of glasses was examined. As part of the characterization, optical microscopy and X-ray diffraction studies were performed on these samples, as well as a limited amount of scanning electron microscopy with energy dispersive spectroscopy. This work contributed to the identification of the alteration products which are formed when glasses are subject to durability testing and is part of the Hanford Immobilized Low-Activity Product Acceptance Tests.

(16) Improvements to Microwave-Accelerated Acid Dissolution of Lithium Aluminate (A. M. Essling and D. G. Graczyk)

The ACL is continuing its investigation of alternative methods for the chemical analysis of lithium aluminate ceramic materials. The goal of this work is to determine whether certain established and qualified, but inefficient, methods can be replaced with modern alternatives that would be more attractive for production-lot analyses in private-sector laboratories. Evaluation of several methods this year culminated in the preparation of documented operating procedures and

successful demonstration tests. One demonstrated method is described in this section. Work carried out on other methods is described elsewhere in this report (see Sections 7 and 8).

In the previous ACL Annual Report, we described a method for dissolving lithium aluminate with a microwave-heated sample digestion system. The ACL had established conditions for dependable, complete dissolution of lithium aluminate ceramics with the microwave system and had demonstrated a standard operating procedure for the process. This procedure involved placing a 0.3-g sample in the closed-vessel system with 30 mL of 6 M HCl and 0.2 mL of HF and heating the solution at 240°C for 60 min. The amount of HF included in the mixture was found to be important: if too little was used, then some impurity elements did not dissolve; but if too much was used, insoluble aluminum fluoride formed. In addition, we discovered that the type of temperature sensor used for system control affected the ability to process multiple samples. Only a fiber-optic sensor allowed even heating between the control vessel and other vessels present in the microwave cavity at the same time.

During the current year, the manufacturer upgraded its microwave system and introduced a new vessel design for high-temperature, high-pressure operation. After converting our system to the new configuration, we began encountering problems with vessel failures that impaired sample throughput and reduced the attractiveness of the method. Through collaboration with the manufacturer and limited experimentation, the problem was overcome by reducing the operating temperature from 240 to 230°C. In addition, the manufacturer determined that overpressure protection was improved for our method if the relief system used different fittings and burst membranes. With the lower temperature and new fittings, vessel failures were virtually eliminated with no detriment to the effectiveness of the dissolution.

The microwave-accelerated acid dissolution procedure is as effective as the Carius tube method in dissolving lithium aluminate, is fairly convenient to apply, and should be easily transferable to private-sector laboratories for production-lot analyses. The solution obtained is suitable for use in lithium isotopic analysis, lithium and aluminum assay, and determination of cation impurities by spectroscopic methods.

### III. QUALITY ASSURANCE

#### Quality Assurance (F. J. Martino)

Audits and assessments continue to play an important role in the overall development and monitoring of ACL quality assurance systems, as well as helping to ensure client satisfaction. Each audit and assessment helps to evaluate (1) the degree to which ACL performance corresponds to stated performance goals and requirements, (2) the adequacy of processes established to achieve quality, (3) the performance of scientific staff in meeting the requirements of the ACL QA Plan, as well as program-related plans and statements of work, and (4) the existence of adequate procedures and QA documentation. One assessment and one readiness review were performed during the past year to verify ACL quality assurance systems and analytical capabilities in support of programs for which the ACL provides analytical services. In May 2000, the CMT Waste Materials Research Department performed a required annual assessment of the ACL. All QA systems were judged satisfactory, and corrective actions were not required as a result of this assessment. In July 1999, representatives of Pacific Northwest National Laboratory (PNNL) audited our technical capabilities and our ability to meet Tritium Target Qualification Program requirements that govern the development of analytical methods for the dissolution and analysis of  $\text{LiAlO}_2$ . Five findings and the appropriate corrective actions were subsequently addressed, and the issues were closed to PNNL's satisfaction. In September 2000, the same PNNL audit team returned to verify implementation of corrective action measures and to perform a program readiness review. Initial indications are that the review went well, with no findings or additional corrective actions required. Preparation of the summary report is underway.

Performance Evaluation (PE) and Proficiency Testing (PT) programs provide intercomparison studies for participating analytical laboratories and an independent evaluation of analytical proficiency. Participation in such programs is often a requirement for analytical laboratories that provide results in support of EPA and DOE programs. Participation in such programs may also be a requirement for laboratories seeking certification or validation by a federal or state agency. The DOE is the only government agency that currently provides and monitors such laboratory performance verification. Programs which provided blind (i.e., analyte values known only to the monitoring agency) PE samples to the ACL in FY 2000 are listed below. Each of the programs serves to verify the quality of ACL analytical results.

- DOE Environmental Measurements Laboratory Quality Assessment Program (EML QAP): Two PE samples (QAP 51 and QAP 52) were received and analyzed to identify and quantify a variety of radionuclides, using gross alpha/beta counting techniques and gamma and alpha spectroscopy methods. Soil, water, vegetation, and air filters were included in this study. Results indicate that the ACL correctly identified all radionuclides present.
- DOE Mixed Analyte Performance Evaluation Program (MAPEP): All laboratories reporting mixed waste analytical measurements for EM-sponsored work are required to participate in this semi-annual PE program. MAPEP samples are provided as two separate sample mixtures: (1) inorganic and radiological analytes and (2) semivolatile organic analytes. Analyses of these PE samples provide a way to evaluate the quality and defensibility of analytical measurements generated for client mixed waste samples. In FY 2000, the ACL participated in two such sample studies, MAPEP 99-W7 and MAPEP 00-S7.

The ACL correctly identified all inorganic and semivolatile organic analytes present in MAPEP 99-W7. Although all radiological target analytes were identified in this sample,

concentration levels reported for  $^{137}\text{Cs}$  and  $^{239/240}\text{Pu}$  were noted to be slightly lower than the supplier's reference value and judged "not acceptable." Followup revealed that neither of the two low analyte values was a consequence of the inability of the analysis to correctly identify that the analyte and corrective actions are complete.

A data report for MAPEP 00-S7 was prepared and submitted in October. The MAPEP report of results for this sample is pending and was not available to be included in this report.

The ACL also became a subscriber to three commercially available Proficiency Testing (PT) programs as substitutes for those previously provided by the EPA. Two commercial vendors have been enlisted to provide blind PT samples for inorganics (metals, mercury, pH, and minerals), organics (volatiles, semivolatiles, PCBs, and pesticides), and radiological (tritium, natural uranium, and  $^{226/228}\text{Ra}$ ) studies. The first of these studies was initiated with results submitted in October 2000; reports of performance are expected later.

#### IV. PROFESSIONAL ACTIVITIES

##### A. *Publications and Reports*

Chemical Analyses for GB (SARIN) in Media Generated by Munitions Disposal

K. L. Brubaker, J. F. Schneider, H. J. O'Neill, L. L. Reed, A. S. Boparai, C. T. Snyder,  
N. A. Tomczyk, and L. L. Jensen

Argonne National Laboratory Report ANL/ACL-99/1 (1999)

Analytical Chemistry Laboratory Progress Report for FY 1999

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

Argonne National Laboratory Report ANL/ACL-99/2 (1999)

Chemical Technology Division Annual Technical Report 1999

D. Lewis, J. F. Miller, R. E. Einziger, E. C. Gay, and D. W. Green

Argonne National Laboratory Report ANL-00/15, June 2000

Effect of Spectral Resolution on Pattern Recognition Analysis Using Passive Fourier Transform Infrared Sensor Data

A. S. Bangalore, J. C. Demigian, A. S. Boparai, and G. W. Small

*Applied Spectroscopy*, Vol. 53, No. 11, November 1999

Analysis for Chemical Agents to Support the U.S. EPA's Region 5 Emergency Response Branch

A. S. Boparai, J. F. Schneider, and L. L. Reed

Proc. of the Eighth International Conference On-Site Analysis, Las Vegas, NV,  
Jan. 23-26, 2000

The  $^{44}\text{Ti}$  ( $\alpha$ , p) Reaction and its Implication on the  $^{44}\text{Ti}$  Yield in Supernovae

A. Sonzogni, K. E. Rehm, I. Ahmad, F. Borasi, D. L. Bowers, F. Brumwell, J. Caggiano,  
C. N. Davids, J. P. Greene, B. Harss, A. Heinz, D. Henderson, R. V. F. Janssens,  
C. L. Jiang, G. McMichael, J. Nolen, R. C. Pardo, M. Paul, J. P. Schiffer, R. E. Segel,  
D. Seweryniak, R. H. Siemssen, J. W. Truran, J. Uusitalo, I. Wiedenhover, and  
B. Zabransky

*Physical Review Letters*, Volume 84, Number 8, pp. 1651–1654,  
February 21, 2000

Team Celebration

D. W. Green

*Managing the Modern Laboratory*, 4(2), 19A (1999).

Good Delegation = Good Management

D. W. Green

*Managing the Modern Laboratory* 4(3), 35A-36A (2000)

Questions Behind Successful Measurements

D. W. Green, D. G. Graczyk, D. L. Bowers, and A. S. Boparai  
*Managing the Modern Laboratory* **4**(3), 51-54 (2000)

Forum on Rewarding Productive People

D. W. Green  
*Managing the Modern Laboratory* **4**(3), 47A-52A (2000)

Balance is the Key

D. W. Green  
*Managing the Modern Laboratory* **4**(4), 56A-58A (2000)

Fluid-Based System for Radon Mitigation

K. C. Gross and F. Markun  
 Proc. of the 1999 International Radon Symposium, sponsored by the American Association of Radon Scientists and Technologists, Las Vegas, NV, November 9–12, 1999

B. *Oral Presentations*

Analysis for Chemical Agents to Support the U.S. EPA's Region 5 Emergency Response Branch

A. S. Boparai, J. F. Schneider, and L. L. Reed  
 Eighth International Conference On-Site Analysis, Las Vegas, NV, Jan. 23–26, 2000

Have Sample Will Travel—Analysis of Toxic Substances

A. S. Boparai  
 Illinois Institute of Technology, Chicago, IL, September 13, 2000.

Microwave-Accelerated Acid Dissolution of Lithium Aluminate

A. M. Essling and D. G. Graczyk  
 Pittcon 2000, Science for the 21st Century, New Orleans, LA, Mar. 12–17, 2000

Modernizing Methods for Chemical Analysis of Lithium Aluminate Ceramics

D. G. Graczyk  
 University of Chicago Review of the Chemical Technology Division, Argonne National Laboratory, Argonne, IL, September 13, 2000.

Practical High-Precision Assays by Inductively Coupled Plasma Atomic Emission Spectrometry

D. G. Graczyk  
 CMT Technical Seminar Series, September 29, 2000



# Analytical Chemistry Laboratory: Experience with Internal & External Interactions

D. W. Green

Partnerships Working Group Meeting, Argonne National Laboratory, May 8, 2000.

## Role of Analytical Laboratories within EM

D. W. Green

NAMP 2K, National Initiatives Towards Analytical Excellence, Gaithersburg, MD, June 6–8, 2000

## Role of Analytical Laboratories within EM

D. W. Green

18<sup>th</sup> Meeting of the DOE Analytical Managers Group, Denver, CO, August 14–17, 2000.

## High Precision Determination of Lithium and Aluminum by Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES)

D. R. Huff, D. G. Graczyk, and S. J. Lopykinski

Pittcon 2000, Science for the 21st Century, New Orleans, LA, Mar. 12–17, 2000

## Fluid-Based System for Radon Mitigation

K. C. Gross and F. Markun

1999 International Radon Symposium, sponsored by the American Association of Radon Scientists and Technologists, Las Vegas, NV, November 9–12, 1999

## Analytical Chemistry Laboratory Work-for-Others (WFO) Programs

F. J. Martino

University of Chicago Review of the Chemical Technology Division, Argonne National Laboratory, Argonne, IL, September 12, 2000.

## Status of Development of NAMP Informational Database Systems

W. E. Streets, A. E. Scandora, J. S. Morton and S. Woolf

NAMP SMO Workshop '99, Las Vegas, NV, October 28–29, 1999

## Status of Development of NAMP Information Systems

W. E. Streets, A. E. Scandora, J. S. Morton, and S. Woolf

NAMP Database Working Group Meeting, Salt Lake City, UT, Feb. 3, 2000

## The National Analytical Management Program's Laboratory Informational Network: NAMPnet

W. E. Streets and A. E. Scandora (CMT); J. S. Morton and S. Woolf

(U.S. DOE) NAMP 2K, National Initiatives Towards Analytical Excellence, Gaithersburg, MD, June 6–8, 2000.

## National Analytical Management Program

W. Elane Streets and D. W. Green

University of Chicago Review of the Chemical Technology Division, Argonne National Laboratory, Argonne, IL, September 12, 2000.

C. *Awards*Lesa L. Smith

The Kenneth J. Jensen Award for Excellence, presented by the Analytical Chemistry Laboratory, Chemical Technology Division, Argonne National Laboratory, November 1999

Donald G. Graczyk

Argonne National Laboratory Pacesetter Award, for Program Development of PNNL's Tritium Target Qualification Project into a major program for the Analytical Chemistry Laboratory

D. *Meetings Attended*David W. Green

Presided over Session "Infrared Spectroscopy: Food and Other Applications," Pittcon 2000, Science for the 21<sup>st</sup> Century, New Orleans, LA, March 12–17, 2000

Florence P. Smith

NOBCCChE Annual Conference, Miami, FL, April 17–22, 2000

D. *Professional Organizations and Activities*Amrit S. Boparai

American Chemical Society  
Sigma Xi

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

Edmund A. Huff

American Chemical Society  
Society for Applied Spectroscopy  
Sigma Xi

David W. Green

American Chemical Society  
Division of Analytical Chemistry  
Analytical Laboratory Managers Association, Board of Directors  
Chemical Measurement Task Group, Council on Chemical Research (member)  
DOE Analytical Managers Group, Board of Directors  
Editor, *Managing the Modern Laboratory*  
Sigma Xi

Paul L. Johnson

American Crystallographic Association  
Association for Computing Machinery  
Sigma Xi

Francis Markun

Health Physics Society  
Health Physics Society, Midwest Chapter

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

Tony TenKate

American Association of Physics Teachers  
Sigma Xi

Lynn B. TenKate

Society for Applied Spectroscopy, Chicago Section  
Sigma Xi

*Professional Training*

Susan J. Lopykinski

Perkin-Elmer Atomic Spectroscopy Workshop, February 2, 2000, Oak Brook, IL

Amrit S. Boparai

DOE Headspace Gas Workshop, March 22–23, 2000, Albuquerque, NM

Daniel V. Applegate

DOE Headspace Gas Workshop, March 22–23, 2000, Albuquerque, NM

Susan J. Lopykinski

Optima Instruments ICP WinLab Workshop, May 16–18, 2000, Oak Brook, IL

*ACL Seminars*

National Analytical Management Program (NAMP) Projects at Argonne National Laboratory

W. Elane Streets

CMT Technical Seminar Series, July 7, 2000

Practical High-Precision Assays by Inductively Coupled Plasma Atomic Emission Spectrometry

D. G. Graczyk

CMT Technical Seminar Series, September 29, 2000

**Distribution for ANL/ACL-00/1**Internal:

J. P. Ackerman	J. C. Demirgian	Y. Halpern
I. Ahmad	A. Demkovich	J. E. Harmon
I. Ambats	C. B. Dennis	H. M. Hartmann
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J. G. Asbury	D. R. Diercks	R. R. Heinrich
M. D. Atella	M. L. Dietz	J. E. Helt
A. J. Bakel	N. L. Dietz	W. F. Henning
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P. R. Betten	D. Duncan	E. A. Huff
M. H. Bhattacharya	A. J. Dvorak	A. B. Hull
S. K. Bhattacharya	C. Dwight	L. Iton
I. D. Bloom	W. L. Ebert	M. J. Janik
L. E. Boing	R. E. Einziger	D. J. Jankowski
A. S. Boparai	J. W. Emery	J. D. Jastrow
R. Bouie	A. Erdemir	B. J. Jody
D. L. Bowers	A. M. Essling	C. E. Johnson
T. Branch	F. Y. Fradin	D. O. Johnson
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