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ANL/ACL-92/4

Analytical Chemistry Laboratory

Progress Report
for FY 1992

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Chemical Technology Division
Argonne National Laboratory, Argonne, Illinois 60439
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Argonne National Laboratory
9700 South Cass Avenue
Argonne, Illinois 60439

ANALYTICAL CHEMISTRY LABORATORY
Progress Report for FY 1992

Prepared by:

D. W. Green, Manager
R. R. Heinrich, Group Leader
D. G. Graczyk, Group Leader
P. C. Lindahl, Group Leader
A. S. Boparai, Group Leader
D. A. Bass, Group Leader
with contributions from ACL Staff

December 1992

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I. INTRODUCTION

The purpose of this report is to summarize the activities of the Analytical Chemistry Laboratory (ACL) at Argonne National Laboratory (ANL) for Fiscal Year 1992 (October 1991 through September 1992). This is the ninth annual report for the ACL [the reports for FY 1984 through FY 1991 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), and ANL/ACL-91/1 (December 1991)].

The Analytical Chemistry Laboratory is a full-cost-recovery service center, with the primary mission of providing a broad range of analytical chemistry support services to the scientific and engineering programs at ANL. In addition, the ACL has a research program in analytical chemistry, conducts instrumental and methods development, and provides analytical services for governmental, educational, and industrial organizations. The ACL handles a wide range of analytical problems, from routine standard analyses to unique problems that require significant development of methods and techniques and analytical expertise.

The ACL is administratively within the Chemical Technology Division (CMT), its principal ANL client, but provides technical support for many of the technical divisions and programs at ANL. The ACL has four technical groups -- Chemical Analysis, Instrumental Analysis, Organic Analysis, and Environmental Analysis -- which together include about 45 technical staff members (see Fig. 1). Talents and interests of staff members cross the group lines, as do many of the projects within the ACL.

The Chemical Analysis Group uses wet-chemical and instrumental methods for elemental, compositional, and isotopic analyses of solid, liquid, and gaseous samples and provides specialized analytical services. Major instruments in this group include: an ion chromatograph (IC), an inductively coupled plasma/atomic emission spectrometer (ICP/AES), spectrophotometers, mass spectrometers (including two thermal-ionization mass spectrometers), emission spectrographs, autotitrators, sulfur and carbon determinators, and a kinetic phosphorescence uranium analyzer.

STA = Special Term Appointee; CS = Co-op Student.

Fig. 1. Analytical Chemistry Laboratory Organization Chart

The Instrumental Analysis Group uses nuclear counting techniques in radiochemical analyses over a wide range of sample types, from environmental samples with low radioactivity to samples with high radioactivity that require containment. Other types of analyses use X-ray diffraction and fluorescence of solids, and neutron activation of either liquids or solids. Major instrumentation includes: nuclear detectors, a variety of multichannel analyzers and associated counting equipment, X-ray diffractometers, energy dispersive X-ray analyzers, a scanning electron microscope, and a surface-area analyzer. Radioactive samples can be analyzed using ICP/AES or IC.

The Organic Analysis Group uses a number of complementary techniques to separate and to quantitatively and qualitatively analyze, at the trace level, complex organic mixtures and compounds, including toxic substances, fossil-fuel residues and emissions, environmental pollutants, biologically active compounds, pesticides, potentially hazardous wastes, and drugs. Major instrumentation in this group includes: gas chromatograph/mass spectrometer (GC/MS) systems, Fourier transform infrared (FTIR) spectrometers, a GC/matrix-isolation FTIR (GC/MI-IR) instrument, a GC/MI/FTIR/MS system, GCs, an FTIR microscope, high-performance liquid chromatographs, a supercritical fluid extraction (SFE) apparatus, liquid chromatography/mass spectrometry system (LC/MS), and a supercritical fluid chromatograph (SFC). Facilities exist for analysis of radioactive samples. Highly radioactive samples can be treated to separate the organic and radioactive constituents. Organic Analysis Group personnel also develop methods for such purposes as continuously monitoring incinerator emissions, detecting organic compounds remotely and performing rapid on-site analyses for organic constituents, and analyzing head space gases in DOE waste containers.

The Environmental Analysis Group performs analyses of environmental, hazardous waste, and coal samples for the inorganic constituents. Major instrumentation in this group includes: atomic absorption spectrophotometers (AAS) equipped with flame, graphite furnace, cold-vapor mercury, and hydride-generation capabilities; toxicity characteristic leaching procedure (TCLP) extraction facilities; an automated carbon/hydrogen/nitrogen (CHN) determinator; an inductively coupled plasma/mass spectrometer (ICP/MS); and a sulfur determinator.

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

II. ADMINISTRATIVE HIGHLIGHTS

The ACL is both a full-cost-recovery service center in support of ANL programs and an organization doing its own research and development work. Generally, funding for ACL research is included as part of CMT's total budget. In FY 1992, the total funding for the ACL increased to its highest level, as shown in Fig. 2.

The ACL recovered 90.5% of its total operating costs in FY 1992. Over the last eight years, the ACL has recovered 96.9% of its costs. Approximately half the income of the ACL comes as service work through ANL programs and the remainder comes directly to the ACL from DOE, other DOE sites, other federal agencies, and external users of ACL services. The ACL effort, including divisional administration, includes about one-half recoverable time (sales to users in Fig. 3). Figure 4 gives a breakdown of the total operating costs for FY 1992, which are largely effort related.

The ACL obtained valuable help from eight Special Term Appointees (see Fig. 1) in FY 1992 to assist with load leveling. The STAs provided about 1.5 full-time equivalents of assistance. In addition, nine students and co-ops, one high-school faculty member, and one college faculty member supplemented the regular ACL staff in FY 1992 (not all are included in Fig. 1).

New ACL capabilities established in FY 1992 include laboratory facilities for analysis of radioactive organic samples, a system for interfacing liquid chromatography with mass spectrometry for the determination of organic compounds, and an inductively coupled plasma/mass spectrometer system.

Three projects were accomplished in FY 1992 with Support Development Funds from ANL. In the first project, the capability was established to separate and identify contaminants in environmental samples using liquid chromatography/mass spectrometry. In a second project, software for automated data acquisition and generation of reporting forms was acquired and installed. The use of this system will result in an improved report of analytical results, more flexible reporting to meet client needs, fewer errors of transcription, and a more streamlined operation. In a third project, the capability to analyze mixed hazardous and radioactive wastes for organic constituents was established.

Fig. 2. Total ACL Funding History

FY 1992 Effort

Fig. 3. Analytical Chemistry Laboratory
FY 1992 Effort

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

FY 1992 Costs

Fig. 4. Analytical Chemistry Laboratory
FY 1992 Total Operating Costs

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

Analytical Development Funds supported four projects in FY 1992: the long-term stability was studied of low-concentration calibration standards for graphite-furnace AA standards; a method was developed to separate uranium from soils for measurement of uranium concentrations by alpha spectrometry; new software was added to the ACL powder diffraction system to improve efficiency; and a study was made employing supercritical extraction of high explosives to prepare them for infrared analysis. These projects, which were supported by internal ACL funding, are described in the following section.

III. TECHNICAL HIGHLIGHTS

Engineering Studies of Pyrochemical Processes for Integral Fast Reactor Fuels (D. L. Bowers, E. G. Rauh, C. S. Sabau, T. TenKate, and R. R. Heinrich)

In the electrorefining of spent fuels for the Integral Fast Reactor (IFR), metallic fuel pins (U, Pu, Zr) are chopped into small segments and placed into a basket that is introduced into molten salt (LiCl + KCl) at 500°C. The basket is connected to a dc power supply and made anodic; nearly pure uranium is removed from the spent fuel by electrotransport to solid cathodes, followed by electrotransport of the plutonium and any remaining uranium to liquid cadmium cathodes. Engineering-scale studies of this electrorefining process are being conducted in CMT to develop models that can predict the recovery of U and Pu as well as the decontamination of these elements from fission-product elements and process materials. The ACL has contributed to this effort by determining elements of interest in samples from the halide-salt electrolyte, cadmium anode, and the cathode product. Special dissolution procedures are followed for each type of sample matrix, and separation schemes based on solvent extraction and ion exchange are used to isolate the desired elements from matrix components and, subsequently, to separate these elements. Uranium and plutonium concentrations in IFR samples are measured with an ICP/AES instrument configured to analyze radioactive solutions. The mass spectrometric isotope dilution (MSID) technique is used whenever higher precision and accuracy than attainable with ICP/AES are required.

Experiments were conducted in an engineering-scale electrorefiner to simulate the end of a campaign run, when a heavy metal drawdown step is employed to remove most actinides from the salt and metal phases prior to removal of the salt for waste processing. These drawdown experiments were performed to understand the behavior of various constituents in the electrorefiner, including the substantial inventory of zirconium, rare earths, and residual uranium. The analytical data gave insight on electrochemical transport of these elements in solid or liquid cathodes under different electrorefiner conditions.

Chemical analyses of liquid-cathode samples from the engineering-scale electrorefiner were performed on the continuing experiments to study Pu-U transport from IFR fuel. Results indicated that a new design of liquid cathode, which incorporates an axial and rotating motion agitator, will achieve very high collection efficiencies for the heavy metals.

Competitive Exchange Experiments in IFR Chemical and Engineering Support Studies (E. A. Huff, D. R. Huff, F. P. Smith, I. M. Fox, A. M. Essling, A. G. Engelkemeir, and D. G. Graczyk)

Pyrochemical reprocessing of fuel in the IFR concept is based on the partitioning of chemical constituents from the spent metal fuel between molten cadmium and a molten LiCl-KCl eutectic. In one of the Chemical Engineering and Support Studies being conducted for the IFR, researchers (J. P. Ackerman and J. L. Settle, CMT) are using competitive exchange techniques to determine separation factors for elements of special interest, especially the lanthanides and alkaline-earth metals. These experiments start with two metals of interest, such as Y and Sm, dissolved in the cadmium phase or distributed between the salt and cadmium phases, depending on the relative activity of the metal pair. Then, successive additions of cadmium chloride or metal chloride are made to change the relative amounts of these metals in each phase. Both cadmium and salt phases are sampled in each step of the experiment, and the metal concentrations determined in the samples are used to compute the separation factor. Analytical support for these studies includes analysis of the salt and metal phases to determine not only the elements involved in the competitive exchange but also a variety of other parameters related to mass-balance checks and conditions prevailing during each experiment.

In most cases, each salt phase is analyzed by ICP/AES to determine Li, K, Cd, and the pertinent rare earths and by silver-chloride gravimetry to determine chloride. Each cadmium metal sample is similarly analyzed to determine Li, K, Cl, and the rare earths. Cadmium in the metal samples is measured by titration with ethylenediaminetetraacetic acid (EDTA). Many salt samples have been tested for the presence of species oxidizable by ferric ion according to a procedure

developed for measuring concentrations of divalent rare earths (Sm^{2+} , Eu^{2+}).

During FY 1992, approximately 70 salt-phase and cadmium-phase samples were analyzed to conclude the partitioning experiments. Competitive-exchange studies between rare earths were extended to include characterization of separation factors between the rare earths and selected other elements. In one set of experiments, measurements were made to obtain information on the Sm-Eu-Li-Ba-Sr system. Preliminary results from samples associated with these experiments suggested the presence of unanticipated species in the salt phase, possibly including monovalent alkaline earths. Methods for the qualitative and quantitative determination of these species were investigated in the ACL, using the probable reaction of these species with water as a basis for testing their existence. Methods were developed to measure products, such as hydroxide ion and hydrogen gas, that would be produced when salts containing the proposed species were dissolved in water. Preliminary interpretation of the data obtained with a series of test salts supported the hypothesis that uncommon species are produced during the competitive exchange redox reactions under some conditions.

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

The National Acid Precipitation Assessment Program (NAPAP) was initiated in the early 1980s with the goal of quantifying the extent to which air pollution, including acid deposition, accelerates the weathering of materials used in construction of cultural resources. Among the materials of high interest are marble and limestone because they are widely used in exterior structures and monuments and because the calcium carbonate stones are especially sensitive to acid attack. In one NAPAP subprogram at ANL (K. J. Reimann, Materials and Components Technology Division), limestone and marble briquettes are exposed to a variety of atmospheric conditions at a number of sites throughout the country and brought to ANL for sampling and analysis to determine the surface chemistry and mineralogical changes in the test stones. In support of this program over the past few years, the ACL has analyzed hundreds of specimens for anions (F^- , Cl^- , NO_3^- , SO_4^{2-} ,) by ion chromatography, and a smaller number by ICP/AES for metal cations. Little change in cation concentrations was found to

occur following exposure at the test sites. However, substantial elevations in sulfate and nitrate were found, and these results have provided a quantitative measure of attack by atmospheric sulfur and nitrogen oxides. Support by the ACL for this program continued this year with anion measurements performed on approximately 90 specimens.

Molten Corium Concrete Interaction Studies (A. M. Essling, E. A. Huff, F. P. Smith, I. M. Fox, D. R. Huff, L. E. Ross, K. J. Jensen, A. G. Engelkemeir, L. B. TenKate, B. S. Tani, and D. G. Graczyk)

In Molten Corium Concrete Interaction (MCCI) experiments at the Reactor Engineering Division (J. Fink et al.), mixtures of uranium oxide, zirconium, steel, fission product elements, and concrete are heated to temperatures simulating reactor meltdown conditions. The objectives are to study vaporization behavior of the elements present and to understand the release of refractory fission products during a degraded-core accident. Samples of solidified melt, aerosols collected by impaction or on filters, and gases are examined to study interaction and transport of the simulated reactor materials.

In FY 1992, we analyzed about 30 samples from the last scheduled large-scale MCCI test that involved a limestone/limestone concrete mixture. This test produced aerosols particularly rich in silver, which had been added to the corium to simulate control rod materials. Dissolving MCCI melts and aerosols for multielement determinations by ICP/AES is always challenging, and ACL has devised a repertoire of dissolution schemes for this purpose, including closed-vessel procedures using a Parr Decomposition Vessel or Carius tube, microwave procedures, fusion procedures with lithium tetraborate or sodium carbonate as flux, and other special techniques. For this last test, the aerosol dissolution procedures needed new modifications to accommodate the silver component, which was first identified in undissolved phases by X-ray diffraction (XRD) analysis.

In past MCCI tests, silicon compounds have accounted for most of the aerosols released. Evidence has accumulated that the silicon is present in certain aerosols as silicon metal and SiC, in addition to SiO₂. It is believed that most of the silicon is emitted from the MCCI melt as SiO gas,

which disproportionates during aerosol formation to Si plus SiO_2 . For carbonate concretes (i.e., those containing limestone), CO is present in the gas phase and reacts with SiO to form SiC. During FY 1992, we concluded a multiyear effort to determine the distribution of silicon among these various chemical species in representative aerosols. By means of a selective-dissolution procedure that sequentially dissolved elemental silicon in dilute sodium hydroxide solution and SiO_2 in hydrofluoric acid, the amount of silicon present in each form was determined. Solutions were analyzed by ICP/AES to measure the dissolved silicon. Silicon carbide was determined by measuring carbon in each aerosol and attributing the carbon to the presence of SiC. Results from the ACL measurements were compared to similar measurements performed with a different procedure by the AEA Technology Centre at Winfrith (United Kingdom). In general, results from the two laboratories are in excellent agreement; where differences exist, they are understandable in terms of the aerosol properties or differences in the samples that were analyzed.

Rare earth elements (simulated with Ce and La) are present in the aerosols at very low concentration but are of special interest because they represent abundant fission products with high specific activity. In previous analyses with ICP/AES, we had been unable to quantify reliably the rare earths at the levels at which they occur (one to a few hundred micrograms per gram of sample). Attempts to obtain values for Ce and La by neutron-activation analysis were unsuccessful because of interferences from other aerosol constituents (U, Ag, and Re). Recently, we submitted solutions prepared from selected aerosols to a commercial laboratory for ICP/MS determination of Ce, La, and Ru. The ICP/MS data demonstrated sufficient sensitivity to allow characterization of the rare earths in all samples. For those aerosols where the rare earth concentrations were high enough to have been measurable by ICP/AES, agreement between our earlier results and those from ICP/MS was good.

Geoscience Programs (E. A. Huff, D. R. Huff, A. M. Essling, F. P. Smith, I. M. Fox, A. G. Engelkemeir, and E. G. Rauh)

Analytical measurements were performed by the ACL in support of geoscience programs at

ANL (N. C. Sturchio, CMT) and at several universities in the Midwest. These measurements included characterization of groundwater samples by analysis for cations with ICP/AES or atomic absorption spectrophotometric (AAS) techniques; measurement of anion concentrations (F^- , Cl^- , NO_3^- , SO_4^{2-} , Br^- , I^-) by ion chromatography; determination of pH, alkalinity, or other properties by classical methods; and isotopic measurements by thermal ionization mass spectrometry. These techniques were applied to thermal waters from Yellowstone National park; borehole brines from the Illinois Basin; and thermal waters, gases, steam condensates, and gas-sampling absorber solutions from volcanoes in Colombia, Ecuador and Papua, New Guinea. The data generated by analysis of such samples (almost 200 samples were processed during FY 1992) have provided information about the age of geological formations, processes and mechanisms that produced the formations, and the movement of groundwater in the formations, both in geologic and modern times.

Environmental Monitoring Program (L. B. TenKate, J. J. Marr, F. P. Smith, and L. E. Ross)

During FY 1992, the ACL prepared approximately 400 environmental water samples in support of environmental monitoring programs at ANL-East. Determinations of metals in these digested samples by flame or graphite furnace AAS were subsequently performed by personnel from the Environment, Safety, and Health Division (ESH). During this same period, the ACL also analyzed wastewater samples collected by ESH personnel from the ANL-East site for the determinations of oil and grease, cyanide, total phenolics, or ammonia content or to measure conductivity. Most of these measurements were required by ANL's NPDES (National Pollutant Discharge Elimination System) permit, although data were also obtained in support of monitoring activities for the landfill located at ANL.

Analysis of Environmental and Waste Samples (D. G. Graczyk, A. G. Engelkemeir, A. M. Essling, I. M. Fox, E. A. Huff, F. P. Smith, L. E. Ross, P. C. Lindahl, J. J. Marr, L. B. Tenkate, R. W. Bane, D. A. Bass, K. J. Jensen, R. R. Heinrich, W. E. Streets, A. S. Boparai, P. M. Aznavoorian, G. A. Baudino, L. L. Lamoureux, G. T. Reedy, D. V. Applegate, Y. Tsai, D. R. Bierma, M. Clapper-Gowdy, C. T. Snyder, C. L. Hammer, R. L. Petty, C. Marquez, L. A. Vallow, F. J. Martino, L. L. Smith, F. Markun, R. B. Holtzman, D. R. Huff, T. TenKate, and S. D. Kent)

The ACL provided analytical services to a variety of environmental monitoring, characterization, and remediation projects administered by the Environmental Research (ER), Energy Systems (ES), and Environment/Waste Management (EWM) Divisions at ANL and programs at other DOE facilities (Rocky Flats Plant and Idaho National Engineering Laboratory). Samples processed during FY 1992 included waters, soils/sediments, and miscellaneous other samples (air, filters, and sludges). These samples were analyzed according to protocols described in the U.S. EPA Contract Laboratory Program (CLP) Statements of Work (SOW), the methods described in "Test Methods for Evaluating Solid Waste" (EPA Document SW-846, U.S. EPA Office of Solid Waste and Emergency Response, Washington, DC, November 1986), or other appropriate procedures. Approximately 500 analyses were completed in FY 1992.

Environmental analyses provided by the ACL include determination of inorganic constituents (metals, anions, cyanide, sulfide, and total dissolved and suspended solids), organic compounds (volatiles, semivolatiles, polychlorinated biphenyls/pesticides, phenolics, and oil and grease), and radionuclides (gamma emitters and actinides). One set of sludge samples was analyzed to provide a complete compositional characterization (anions, cations, moisture, etc.) in support of development work in CMT (G. Vandegrift et al.) on a waste treatment process. Mass and charge balance calculations applied to the data from this characterization demonstrated that all major cationic and anionic components had been properly taken into account.

Analytical Method Development in Support of Waste Characterization for WIPP (A. S. Boparai, G. T. Reedy, D. V. Applegate, K. J. Parish, and S. D. Kent)

The Waste Isolation Pilot Plant (WIPP) in Carlsbad, NM, has been selected as a possible permanent disposal site for transuranic radioactive wastes resulting from defense-related activities at

DOE sites. Over the next five years, DOE plans to evaluate the behavior of wastes designated for burial at the WIPP site. Analyses of these wastes will be done at Idaho National Engineering Laboratory, Rocky Flats Plant, and ANL.

Defense-generated wastes to be transported to, and stored at, the WIPP site must be characterized and shown to satisfy Resource Conservation and Recovery Act (RCRA) requirements, which limit the presence of hazardous compounds. One of the wastes to be shipped to the site is Type IV organic waste sludge. The sludge results from the "solidification" of organic waste liquids by the addition of calcium silicate absorbent to immobilize the spent liquids. Because polychlorinated biphenyls (PCBs) are suspected of being present in the liquids, and hence, the sludge, analyses for PCBs must be completed on each container of sludge before shipment.

The ACL is involved with the development of an analytical method for the determination of PCBs in the sludge. Batches of synthetic sludge have been prepared from liquids known to be present in existing waste sludges, such as lathe coolant, machining oils, and hydraulic fluids. The selected liquids are spiked with PCBs, and then blended with calcium silicate to form the sludge. The starting point for the proposed analytical procedure is EPA Method 8081 (in EPA document SW-846), which uses a solvent extraction step followed by gas chromatographic analysis with an electron capture detector.

Interferences from the waste that tend to obscure the presence of PCBs have been identified and can be eliminated before the gas chromatographic step. Measurements using the modified EPA Method 8081 have shown good recovery (70 to 100%) of PCBs from the sludge. Future work will involve the analysis of synthetic sludge containing radioactivity and of actual field samples.

Analysis of Volatile Organic Compounds in Headspace Gas for the WIPP Project (D. V. Applegate, Y. Tsai, G. T. Reedy, and A. S. Boparai)

The role of the ACL in this project is to serve as one of the participating laboratories in an effort to determine VOCs contained in the headspace of waste drums that will be shipped to the WIPP

site for burial. The VOC analyses being conducted are based on GC/MS methods developed by the ACL for analysis of 29 target compounds, that are of particular interest to the project. Target compounds include aromatic hydrocarbons, chlorinated aliphatic hydrocarbons, diethyl ether, alcohols, and ketones.

Gas samples are drawn from the waste drums into evacuated stainless steel SUMMA[®] canisters, which are then shipped to participating laboratories for analysis. Our analytical procedure involves pressurizing these canisters, then extracting aliquots into an intermediate laboratory canister, to which we add an internal standard. An aliquot of 200 mL is then drawn from the laboratory canister and passed through an analytical trap, which retains the target compounds. The target compounds are released from the trap by heating and swept into a GC/MS system set up for separation and quantitative analysis. Quantitation of each target compound is based on a relative response factor for the area count of a characteristic fragment ion and the area count of an internal standard characteristic ion. We are able, with this technique, to quantitate the target compounds over a concentration range from less than 1 ppmv in the headspace gas.

Canister Cleaning and Certification for WIPP (G. T. Reedy, K. J. Parish, Y. Tsai, S. D. Kent, G. McCarthy, and A. S. Boparai)

The Central Canister Cleaning Laboratory (CCCL) at ANL serves two main functions for WIPP. First, the laboratory cleans and certifies SUMMA canisters that are used in the Waste Characterization Performance Demonstration Program. In this program, synthetic headspace gas samples of known composition are placed in canisters, which are then shipped to participating WIPP analytical laboratories on a semiannual basis. Each laboratory analyzes the blind test samples and reports results back to the examiners for scoring. The CCCL has been cleaning and certifying canisters that are used in this program. Second, the CCCL provides cleaned and certified canisters for the Westinghouse Laboratory at the WIPP site. The WIPP site laboratory is not certifying waste for shipment, as do the laboratories at the generator sites, but requires the gas-sampling canisters for

two purposes: (1) the inspection of secondary containers and (2) assessment of changes in waste and waste containers stored at the site. During FY 1992, the CCCL provided about 200 total canisters for the WIPP site and the Performance Demonstration Program. The canisters have ranged in size from 250 mL to 6 L.

Analysis of Waste-Drum Headspace Gas for WIPP (A. G. Engelkemeir, C. T. Snyder, and D. G. Graczyk)

In addition to the analyses performed for volatile organic compounds, gas samples withdrawn from waste drums in the WIPP Experimental-Waste Characterization Program require compositional analysis to determine permanent gases and low-molecular-weight hydrocarbons (methane through propane). Volume-percent concentrations of each of ten target-analyte gases (H_2 , N_2 , O_2 , Ar, CO, CO_2 , NO_x , CH_4 , C_2H_6 , and C_3H_8) are to be measured. Our method for these determinations is based on mass spectrometry using a magnetic-sector, gas-analysis mass spectrometer having moderate resolution. Instrument stability and sensitivity have been demonstrated to meet program requirements regarding precision and detection limits for measuring the gases of interest. After some instrumental and procedural difficulties were resolved early in FY 1992, the mass spectrometric system has performed well, as indicated by passing scores for both performance demonstration program cycles (January and April, 1992).

Development of the Infrared Aerosol Analyzer (F. P. Smith)

The infrared aerosol analyzer (IAA) is a device for collecting ambient atmospheric aerosols by impaction and analyzing the material collected by infrared spectroscopy. Developed and patented at ANL (S. Johnson, CMT), the IAA has been used for several years for qualitative and semiquantitative studies involving aerosol species (ammonium, sulfate, and nitrate ions) related to acid precipitation. Use of the IAA for quantitative analysis of aerosols has been hindered because it is difficult to create and collect a synthetic aerosol of known composition for calibration. This problem was recently overcome by using ion chromatography (IC) to measure the ions of interest in actual ambient aerosols

after their infrared spectra were recorded with the IAA. Selected samples were nondestructively analyzed with the IAA and then dissolved, whereupon the amounts of nitrate, sulfate, and ammonium ions present were measured by IC. From these measurements, a consistent data set has been assembled to permit quantitative determination of individual ions from the infrared spectra of ambient aerosols.

Determinations of Rare Earth Elements and Calcium in Autopsy Tissues (E. A. Huff and D. R. Huff)

The Internal Emitter Project in the ER Division (A. Stehney) required measurement of rare earth element and calcium concentrations in digested autopsy tissues from (1) thorium refinery workers who had been exposed to dust particles from monazite ores and (2) a case study involving a victim of radium exposure. From quantities of rare earths present in the lungs and other tissues analyzed, the ER researchers will determine the individuals' exposure by inhalation and the distribution of rare earths to various body tissues by metabolism of the inhaled substances. Calcium data are needed to help define metabolism pathways for radium, which typically follows calcium in the body but can show discrimination effects in some tissues.

Both major and trace metal concentrations in the digested tissues were determined by ICP/AES. The spectral complexity and high concentrations of rare earths in some samples required multiple dilutions, spectral interference corrections, and careful interpretation of the ICP/AES data. In addition, several samples required separation of the components of interest from matrix components to achieve quantitation limits on the order of 1 to 12 $\mu\text{g/mL}$, which met the needs of the program. This was accomplished using extraction chromatography (with TRU•Spec resin) in HCl or HNO_3 media. For a few samples, thorium concentrations high enough to measure by ICP/AES ($>1 \mu\text{g/mL}$) were found; the ICP/AES thorium results were in good agreement with radiochemical analytical results on these same solutions.

Monitoring of Antimony Levels in Scrap Aluminum (I. M. Fox, A. M. Essling, D. R. Huff, and E. A. Huff)

In conjunction with the Aluminum Recyclers Association (ARA), researchers in the ES Division (E. Daniels and P. Bonsignore) have established a continuing monitoring program to follow the antimony level in secondary aluminum scrap. Of concern is the potential for intermixing in the secondary metal scrap of two widely used aluminum alloy modifying agents, Sb (from European and Asian sources) and Sr (mainly used in the United States). Work in the ES Division has shown that these two modifiers are antagonistic to one another, resulting in poorer mechanical properties for aluminum containing both modifiers. This work has also demonstrated an approach for removing the antimony if levels become troublesomely high. In support of these programs, the ACL has established procedures for measuring very low antimony concentrations in aluminum metal. Drillings or shavings of the metal are dissolved with a nitric/hydrofluoric acid mixture, and the solution is analyzed for antimony by graphite furnace AAS. The method detection limit is 5 µg antimony per gram of aluminum scrap.

Nuclear Waste Programs (E. A. Huff, D. L. Bowers, A. M. Essling, and D. R. Huff)

The production of stable nuclear waste forms and their long-term storage in suitable repositories are of great importance to nuclear power production. The Glass Testing Program (CMT) has the objective to evaluate factors that are likely to affect glass reactions in an unsaturated environment typical of the candidate repository site at Yucca Mountain, NV. Long-term leaching tests are being conducted on simulated nonradioactive analogues to provide elemental release data that could be used to assess performance of high-level nuclear waste (HLW) glasses. The Yucca Mountain Site Characterization Program is evaluating the suitability of Yucca Mountain as a site for a long-term HLW repository. The release and transport of actinides through the HLW barrier system, spent fuel stabilities, and radiation effects are some of the objectives for these studies.

The ACL has provided support for these programs by the analysis of leach-test solutions for

anions, cations, and uranium. Standard operating procedures and appropriate quality assurance measures have been implemented to support the validity of reported results. The following anions were determined by ion chromatography: Cl^- , F^- , NO_3^- , SO_4^{2-} , PO_4^{2-} , HCO_3^- , and $\text{C}_2\text{O}_4^{2-}$.

The following cations were measured by ICP/AES: Al, B, Ba, Ca, Cr, Cu, Fe, K, Li, Mg, Mn, Na, Ni, Si, Th, Ti, Zn, and Zr. The spectrometer system in Building 200 was used for samples with radiation levels less than 150 pCi/mL; samples exceeding this limit were analyzed in the Building 205 radioactive facility. Uranium in solution was determined by a kinetic phosphorimetry technique with a Chemchek phosphorescence analyzer.

Chemical Transformation of Isotopically Enriched Tin Oxide (F. P. Smith, K. J. Jensen, B. S. Tani, and D. G. Graczyk)

Materials with unusual atomic-energy level configurations are being investigated in the Advanced Photon Source (APS) Division (E. Alp et al.) for potential application in producing monochromatic X-ray beams from the X-ray continuum that will be available in the APS. In a series of experiments to evaluate the performance of isotopically pure ^{119}Sn at the Cornell High Energy Synchrotron Source (CHESS), use of several chemical forms of tin for presentation to the X-ray light was desirable. The ACL assisted in the chemical transformation of $^{119}\text{SnO}_2$ (the available isotopically pure compound) to ^{119}Sn metal and to ^{119}SnO . This work had to be done quickly to meet a previously scheduled session at the CHESS, and high yields were required to avoid losses of the expensive ^{119}Sn isotope. We prepared ^{119}Sn metal by hydrogen reduction of the $^{119}\text{SnO}_2$ starting material (yield >99.5% of theoretical); the tin metal was used as the starting material for the preparation of ^{119}SnO . The product from this preparation was confirmed, by XRD of identically prepared natural tin material, to be SnO. The yield for the ^{119}SnO preparation was 83% of theoretical. The ^{119}SnO and ^{119}Sn products were successfully used in the experiments at the CHESS.

In a related experiment, the APS researchers sought a stable ferrous iron salt for studies involving ^{57}Fe . In this case, ACL chemists recommended the use of Mohr's salt, $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$, and synthesized this compound from ^{57}Fe metal. The identity of the product

was again confirmed by XRD. Because it is uncommonly stable to air oxidation of ferrous iron, this compound proved to be excellent for the intended use.

Advanced Photon Source Component Materials (A. M. Essling, E. A. Huff, D. R. Huff, F. P. Smith, E. G. Rauh, and D. G. Graczyk)

In support of component-fabrication efforts for the APS, the ACL has verified specified compositions for a number of materials going into the hardware for the APS facility. Much of this work involves straightforward analysis to characterize alloy compositions or impurities in one or another high-purity metal product. However, analytical characterization of one material, a Zr-V-Fe alloy bonded to a substrate of constantan (a Cu-Ni alloy), was quite challenging. An APS researcher (D. Walters) required information on the major and minor components of the Zr-V-Fe alloy, which is designated for use in the APS as a getter to remove residual gases from the particle-beam path and thereby provide the high vacuum required for optimal APS operation. This analysis was difficult because the alloy of interest was inseparably bonded to the constantan substrate, and samples were not available for either the coating alloy or the uncoated substrate. To obtain the needed data, we sheared the strips of coated substrate to obtain fully coated samples from the center of each strip and nearly uncoated samples from along the strip edges. By dissolving and analyzing these coated and uncoated portions separately, we were able to mathematically resolve the compositions of the substrate and coating by assuming only that zirconium was not present in the substrate. Similar analyses of two different samples of the getter strip material showed remarkably uniform composition and coating thickness. These results permitted a determination to be made that the material met APS specifications.

Synchrotron Thrust Group (W. E. Streets and B. S. Tani)

The CMT Synchrotron Thrust Group is exploring possible interactions of CMT with APS for current and future research programs. The activities of this group include the following: (1) gather

and disseminate information on the application of synchrotron radiation (SR) in research areas of interest to CMT; (2) familiarize CMT staff with the capabilities, procedures, nuances, and costs associated with research at the APS; (3) arrange seminars on the use of synchrotrons in research areas germane to CMT; and (4) create a link with the other divisional synchrotron thrust groups at ANL. The eventual goal of the group is to develop a plan for utilization of the APS by CMT. The current members of the CMT Synchrotron Thrust Group are: D. Chaiko, R. Chiarello, P. Finn, C. Johnson, L. Leibowitz, M. Lewis, V. Maroni, E. Streets (Chairman), W. Swift, S.-W. Tam, and D. Zurawski.

As part of its support to CMT groups pursuing SR research and as a continuing educational resource to CMT, the Synchrotron Thrust Group identified a consultant (K. D'Amico) with broad expertise in SR to help CMT. This individual began in March 1992 to help in the assessment of CMT divisional needs for SR research, to assist CMT groups in various stages of development of research using SR, and to improve administrative and experimental protocols for use of existing SR facilities. Dr. D'Amico has presented two division-wide tutorials on the use of SR, both generally and specifically directed to identified CMT interests, has met with individual groups to advise and assist them however possible, and has reported his assessment of benefits and limitations of CMT involvement with existing APS Collaborative Access Teams. His affiliation with CMT will continue into FY 1993.

Grafenwöhr Soil Samples (D. A. Bass, D. G. Graczyk, A. M. Essling, E. A. Huff, J. J. Marr, L. B. TenKate, and A. G. Engelkemeir)

Soil samples from the Grafenwöhr firing range in Germany were analyzed for metals in the sample following a total digestion and a TCLP (toxicity characteristic leaching procedure) extraction. The work was in support of the ES Division (J. Schneider and S. Zelmer) in its efforts to characterize soil contamination by inorganic pollutants on the firing range. Approximately 50 soil samples were analyzed by ICP/AES and cold vapor AAS (for mercury) using EPA protocols for twenty-five metals of environmental concern. The elements of greatest interest were Sb, Ba, Cd, Cr, Cu, Pb, Mg, Sr,

Sn, and Zn. These measured data are also being used to develop X-ray fluorescence (XRF) as a field screening technique for lead and to compare the amount of metals extracted using TCLP with the total metals in the sample. We found fairly good agreement between the metal concentrations in the soils determined by wet chemical analysis and XRF. For a few metals, e.g., lead, metal particles in the soil made comparison difficult.

Laboratory Setup at Aberdeen Proving Grounds (D. A. Bass)

The ACL supported the ES Division (J. Schneider and K. Brubaker), which is setting up an analytical laboratory at Aberdeen Proving Grounds. We made recommendations for inorganic instrumentation, analytical methods, and laboratory configuration to screen for hazardous constituents and perform metals analysis according to CLP protocols. This laboratory will be used to do CLP and similar analyses on soils that are potentially contaminated with highly toxic materials from weapons testing and, therefore, may not be shipped to commercial laboratories.

Stability of Standards for Graphite-Furnace Atomic Absorption Spectrophotometry (D. A. Bass and L. B. TenKate)

A study was undertaken to determine the long-term stability of low-concentration standards used for graphite-furnace atomic absorption spectrophotometry (GFAAS). Currently, working GFAAS standards must be prepared daily, as required by the CLP guidelines. The ACL has been following these CLP guidelines because in-house criteria regarding GFAAS standards stability have not been established. This study examined the stability over a period of eight months of low-level concentrations of Sb, Pb, Tl, Se, and Ag in acids with respect to (1) variations in the acid concentration; (2) changes in analyte concentration; and (3) presence of other analytes. We concluded that all analytes were stable under all of the conditions studied over the entire eight months.

Report Generation Software for Graphite-Furnace Atomic Absorption Spectrophotometry (D. A. Bass and L. B. TenKate)

Numerous samples received by the ACL require reporting results on forms specified by the CLP. Because of the number and complexity of the forms, software packages that generate these forms are often used. In the past, the ACL used an inorganic forms generation program that was based on the EPA SOW 787 and required manually entering data from GFAAS measurements. During FY 1992 the ACL purchased a Ward Scientific Environmental Data Reporting System that allows GFAAS data collected on a personal computer to be directly input to the forms. Data can also be entered manually, and forms can be customized to specific reporting needs or any current EPA SOW. Computer disks containing completed forms can also be generated, as required in the current EPA SOW. In addition, quality control charts that allow tracking of the GFAAS system performance can be generated from this software package. We anticipate that this system will generate an improved report, allow more flexible reporting to meet individual client needs, result in fewer transcription errors, and give a much more streamlined operation.

Review of Quality Assurance Plans from Private-Sector Contractors (L. B. TenKate)

During FY 1992, the ACL assisted the CMT Special Projects Office (N. Beskid) in technically reviewing QA project plans prepared by private-sector contractors. These contractors are conducting applied R&D on new technologies needed for DOE to meet its 30-year compliance and cleanup goals for its facilities. The Special Projects Office provides technical and management oversight to assist the contractors in applying their technologies to DOE environmental restoration programs. The contractors conduct applied R&D to improve DOE's capabilities in such areas as groundwater and soil remediation, characterization of underground contamination and geologic features, and containment of contaminated sites. The ACL provided the Special Projects Office with written reviews, comments, and recommendations on a variety of project plans and QA plans involving chemical laboratory work and chemical analyses.

Radiochemical Method Evaluation and New Method Development (J. S. Crain and L. L. Smith)

In FY 1992, the Laboratory Management Division of DOE's Office of Environmental Restoration and Waste Management (DOE/EM) initiated a program to evaluate existing methods for environmental radiochemical analysis. The goal of this program is to identify and implement new techniques and procedures that will improve the time and cost efficiency of routine radiochemical analyses. Such improvements could help DOE meet its goals for environmental restoration and waste management. Argonne has been designated as the coordination center for this program.

In technical support for this program, we have reviewed DOE environmental surveillance reports and existing radiochemical methods to identify specific analyses that could be improved through integration of new technology. We have identified new technologies of potential interest to the program, e.g., ICP/MS and photon-electron-rejecting alpha liquid scintillation (PERALS) and have reported our overall findings to an expert working group convened by DOE/EM. Our findings, along with recommendations of the working group, will be used to formulate statements of work for technology development and integration into radiochemical methods.

Battery Program (J. J. Marr)

The ACL technical support to the battery-testing programs in CMT (J. A. Smaga) was continued during FY 1992. The ACL was involved in post-test examinations on both aqueous and high-temperature batteries that had completed testing at the ANL Battery Analysis and Diagnostic Laboratory and at other battery research centers. The objective of the post-test examination is to identify existing and potential failure mechanisms which limit the performance and life cycle of the batteries. The information obtained from these studies provides a measure of the technical progress made by battery developers and an indication of needed design or material changes. Individual reports were prepared for each of the batteries examined and were forwarded to the battery developers and to personnel associated with these programs for review.

Review of Analytical Data from Subcontractor Analytical Laboratories (J. J. Marr)

During FY 1992, the ACL again assisted the ER Division (ER) in reviewing inorganic and organic data packages generated by DOE subcontractor laboratories for environmental samples taken around the United States. This work included reviewing the data generated at subcontractor laboratories according to the guidelines of the CLP, providing detailed reports of problems encountered with the data, and evaluating the usability of the data. These efforts improved the usability of the data gathered in the ER investigations of the sites being characterized and promoted improvements in the quality of data supplied by subcontractor laboratories.

Characterization of Products from Automobile Shredder "Fluff" Recycling (C. T. Snyder, L. L. Lamoureux, A. M. Essling, E. A. Huff, I. M. Fox, and F. P. Smith)

About 225 kg (25%) of every junked car is made up of a mixture of plastics, glass, fibers, and foam; this mixture is referred to as "fluff." Researchers in the ES Division (E. Daniels et al.) have developed and are testing a procedure to dissolve and recover plastics from these wastes, which are left over after metals are recovered from scrapped automobiles. The ACL is providing chemical analyses to help characterize the various product streams from this process, including oils, recovered plastics, and residues from the dissolution and extraction steps. Residues and oils are analyzed for metals by ICP/AES, and oils are analyzed for their polychlorinated biphenyl content. An FTIR microscope is used to examine plastics recovered from solvent extraction of the shredder residues. Efficiency of the extraction and purity of the products are assessed by comparing spectra from polyvinylchloride and acrylonitrilebutadienestyrene standards to spectra obtained with reconstituted plastics from the process. Obtaining pure product fractions of known composition is important to efficient recycling of the materials recovered from treatment of the fluff waste. The data that ACL provides on the composition and purity of these fractions help to assess their suitability for recycling processes and to identify changes or improvements in the treatment process that will lead to improved products.

Stability of Calibration Standards for ICP/AES (D. R. Huff and E. A. Huff)

Knowledge of the useful lifetime of solution standards and an understanding of how to prepare standards with long shelf-life are increasingly important to operations in the analysis laboratory. Escalating costs for chemical waste disposal and the substantial effort involved in preparing multielement standard solutions make replacement of still-viable solutions wasteful and inefficient. To establish replacement schedules that avoid unnecessary cost but, at the same time, ensure that valid standards are available, the ICP/AES laboratory in the ACL undertook a project to study the stability of standard solutions as a function of time. In this project, the stability of a set of acidic solutions containing approximately 30 elements was monitored for a six-month period to provide data that related stability to variables such as analyte concentration, acidity, and type of acid used in preparing solutions. Results of this study, which were published (E. A. Huff and D. R. Huff, The Stability of Calibration Standards for ICP/AES Analysis: Six-Month Study, Argonne National Laboratory ANL/ACL-92/3, May 1992), demonstrate that, with properly chosen matrix characteristics, calibration standards can be stable over a period at least this long. The study is being extended for at least another additional six months to obtain a larger data base.

Determination of Platinum in Tissue (A. M. Essling and E. A. Huff)

Black staining of tissue surrounding the battery anode of bone-growth stimulators, which are used to increase fusion success in spine surgery, is being investigated by physicians at the Kalamazoo Center for Medical Studies, Michigan State University (MSU). In support of this work, the ACL was requested to determine the platinum content of blackened tissue from three patients who had undergone removal of the stimulator. For this determination, we ashed each tissue sample in a fused-silica beaker, dissolved the residue in aqua regia, and analyzed the resulting solution for platinum (and other metals) by ICP/AES. Microgram amounts of platinum were found in two of the three tissue samples, which weighed about 50 mg. These results are discussed by the MSU physicians in an article on the "Black-Pocket Syndrome," submitted to the Journal of Bone and Joint Surgery.

Isolation of Uranium and Thorium from Soils for Isotopic Analysis (A. M. Essling, E. A. Huff, F. Markun, and D. G. Graczyk)

Procedures developed in the ACL for isolating uranium from a soil matrix and purifying it for isotopic analysis by thermal-ionization mass spectrometry (TIMS) are being modified to permit isotopic analysis of the uranium by alpha pulse height analysis (APHA). Because APHA is the conventional radiochemical method for actinide isotopes, procedures designed for APHA measurements will find wider application than those designed for TIMS analysis, which is not as widely available in environmental laboratories. Moreover, the TIMS and APHA measurements are complementary: TIMS has an advantage for measuring ^{235}U (with reasonable mass abundance but low specific activity), while APHA has an advantage in measuring ^{234}U (with low mass abundance but high specific activity).

The major difficulty encountered in applying the sample preparation procedures for TIMS to APHA has been the incomplete removal of thorium isotopes, which have alpha energies in the range of interest for uranium measurements. This problem has been addressed by modifying the separation scheme that we previously used with U/TEVA•Spec chromatographic columns (EiChrom Industries, Darien, IL). By loading the column with 2 N nitric acid, U and Th are retained, while unwanted cations are eluted. After a crossover washing with 9 N HCl, thorium is eluted with 5 N HCl. Then, uranium is stripped from the column with 0.02 N HCl.

Using synthetic U-Th solutions and ICP/AES analysis of all column effluents, we demonstrated that this scheme will yield clean separation of these actinides, with >95% recovery of each in its respective eluent fraction. Application of this scheme to soil-matrix samples and APHA measurements on the separated actinides will be tested in the coming year.

Characterization of Nitrogen Species in Lime-Scrubber Waste (F. P. Smith, L. E. Ross, L. B. TenKate, A. G. Engelkemeir, and D. G. Graczyk)

In support of an investigation into the probable post-disposal behavior of sludge waste produced from NO_x/SO_2 scrubbing of flue gases with $\text{Fe(II)}\cdot\text{EDTA}$ in lime, ES Division

researchers (M. Mendelsohn et al.) sought to determine leachable nitrogen species and gaseous products evolved from representative wastes as a function of storage time. The ES researchers set up simulated storage of four lime-sludge wastes, with one portion of each waste open to the laboratory environment and another portion in a closed vessel with air removed from the headspace. Approximately every three months, a portion of each waste stored in the open was submitted to the ACL for leaching according to the TCLP and determination of ammonia, nitrate, nitrite, and total Kjeldahl nitrogen in the leachate. At each time interval, headspace gas over each closed-storage sample was also analyzed by gas mass spectrometry. Changes in both the quantities of leachable nitrogen species and the gas-phase composition were observed over the year-long test period. These changes were not uniform among the four wastes tested, reflecting differences in the wastes' compositions. An interesting feature of the gas-analysis results was a depletion of oxygen in the headspace with time. More interesting was the observation that the oxygen depletion was accompanied by ingrowth of CO_2 in three of the wastes but not the fourth. No release of nitrogen oxides from any of the wastes was detected. More detailed study of the chemistry involved in the aging of these wastes will be needed to fully understand these observations.

Properties of High-Temperature Superconductors (E. A. Huff, D. R. Huff, A. M. Essling, F. P. Smith, I. M. Fox, B. S. Tani, K. J. Jensen, and D. G. Graczyk)

The ACL continued providing extensive analytical support to high-temperature superconductivity programs in the Materials and Components Technology (MCT) and CMT Divisions. Much of this work involves analysis of starting materials, process samples, and products related to fabrication and characterization studies on various ceramic compositions, including $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ ("123-compound") and newer ceramics involving different rare earths, Bi, Pb, Sr, and other elements. Our measurements include determining elemental composition by ICP/AES or classical methods, anions by ion chromatography, carbon with a LECO Analyzer, oxygen coefficients (i.e., the value of x in the formula $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$) by an iodometric method, and phases by X-ray powder diffraction.

A major new initiative in the MCT Division was begun this year with the goal of producing

100-m lengths of high-temperature superconductor ribbon by means of a "powder-in-tube" process, wherein superconducting compounds containing Bi or Tl are loaded into a silver tube, which is then drawn and rolled into a thin, flat superconducting ribbon. For this work, ACL data have helped to eliminate some fabrication difficulties that were traced to materials problems (e.g., copper or other impurities in some batches of silver tubing) and to relate performance of individual products to their composition. Using chemical analysis results to guide materials and process selection has helped the MCT team make rapid progress toward their goal.

Applications of FTIR Microscope to Identification of Wastes, Spill Residues, and Contaminants
(C. T. Snyder)

The FTIR microscope (an FTIR spectrometer coupled to an infrared microscope equipped with a mercury-cadmium telluride detector) is a powerful tool for examination of samples from a variety of sources to provide rapid identification of unknown materials. Both reflectance and transmission techniques have generated spectra from solid samples as small as 10- μ m diameter, and liquid samples as small as 10 μ L. In many cases, the sample composition was determined by subjecting the spectra obtained to a computer-based library search. Examples of such successes achieved in FY 1992 include characterization of a white powder found on the outside of a polyethylene tube after disassembly of accelerator hardware (the powder was nylon); nonaqueous materials of unknown origin (e.g., pump oils stored in unlabeled containers); and spill residues encountered during emergency-response activities (a pool of liquid discovered in a parking lot was quickly identified as ethylene glycol). Even when positive identification through a library match was not achieved, the functional-group information provided by the infrared spectrum has helped in further characterization of unknown samples. Including analysis with the FTIR microscope as an early step in characterizing unknowns helps reduce the difficulty and cost of identifying and classifying certain undocumented liquid wastes and of solving contamination problems of various kinds.

Identification of Hazardous Wastes (A. M. Essling, D. R. Huff, E. A. Huff, J. J. Marr, F. P. Smith, L. B. TenKate, and L. E. Ross)

According to the Resource Conservation and Recovery Act (RCRA), a waste is classified as hazardous if it displays one or more of four characteristics: ignitability, corrosivity, reactivity, and toxicity. During FY 1992, the ACL strengthened its ability to identify characteristic wastes by obtaining a Pensky-Martins Closed Flash Point Tester (Fisher Scientific Co.) for measuring ignitability. Corrosivity is measured with pH meters. Reactivity and toxicity are determined by measurement of hazardous constituents (e.g., cyanide, sulfide) in the waste. For targeted inorganic constituents, the samples are leached by TCLP, then the extract is analyzed by ICP/AES for metals and cold-vapor AAS (for mercury). Samples analyzed in FY 1992 included sludges, soils, and photographic plates. Paint chips, sand blasting residues, soil and gravel from parking lot surfaces, and sludges from drain pipes have all required characteristic analysis to ensure that they were properly classified for treatment and disposal. Additional samples were screened by determining total metals. Whenever these measurements yield concentrations above RCRA definition of "hazardous," the TCLP can be used to determine if the hazardous constituents are in a leachable form. Wastes from many ANL programs, RCRA closure projects, decommissioning projects, and other sources were analyzed to provide hazard classification information.

DOE Environmental Audits (P. C. Lindahl and D. A. Bass)

The ACL assisted ANL's Environmental Assessment and Information Sciences Division in staffing DOE environmental audit teams for several DOE sites during FY 1992. The ACL staff also participated in the environmental audits of the Environmental Measurements Laboratory (New York) and the Alaska Power Administration. In both audits, the ACL staff were responsible for assessing the organization's environmental management and QA.

DOE Operational Readiness Reviews (P. C. Lindahl)

The ACL supported DOE/EM in conducting an operational readiness review (ORR) of the Defense Waste Processing Facility (DWPF) at the Savannah River Site. An ORR was performed for the cold chemical runs in which nonradioactive materials are to be tested with the process that will be employed in the DWPF. The ACL staff participated in the review of the Analytical Facilities Module.

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

In collaboration with the Radiological and Environmental Sciences Laboratory (RESL) in Idaho and the Environmental Measurements Laboratory (EML) in New York, ANL is developing and implementing a comprehensive Integrated Performance Evaluation Program (IPEP) for the DOE Laboratory Management Division (LMD, EM-563). The program is designed to provide information on the quality of radiological and nonradiological analytical data being produced by all laboratories on which DOE/EM is relying for analyses of environmental restoration and waste management samples. The ACL is assisting in developing the program requirements and implementation strategies, especially in the nonradiological portions of the program. The ACL is also developing strategies for compiling and analyzing the performance evaluation (PE) results and for monitoring to assure that needed corrective actions are taken. In addition, the ACL also participates in Future Analytical Support Team activities in support of LMD. This group, which consists largely of DOE contractor personnel doing analytical chemistry, provides advice and needed data that assist LMD in meeting its objective of ensuring that the required capacity and capabilities exist to provide the analytical support for DOE/EM programs.

The establishment of the IPEP computer system is essentially complete. All required data have been transferred to the IPEP VAX computer to allow for the necessary security. Paradox data base software and SAS (Statistical Analysis Software) are being installed to allow reports to be prepared

accurately and efficiently. Information on the identity of laboratories and data on their participation in PE programs has been transferred into the Paradox laboratory identification data base. A report formally recommending requirements for laboratory participation in existing PE programs was completed by ANL, RESL, and EML, and submitted to EM/LMD. These recommendations were incorporated into EM/LMD's Analytical Services Program QA guidance documents, which have recently been sent to the DOE Field Offices for final review.

Historical and current data from the Water Supply (WS) and Water Pollution (WP) Programs of the Environmental Monitoring Systems Laboratory in Cincinnati, OH (EMSL-Ci) can now be routinely transferred from the EPA data base at Research Triangle Park in North Carolina to the IPEP data base at ANL. In addition, data from the Contract Laboratory Program (CLP) and the EMSL-LV Radiological Intercomparison Studies (RIS) will be available electronically to IPEP from EMSL-LV. Currently, hardcopy information for DOE government-owned contractor-operated participants in the CLP programs is used; EML has supplied IPEP with the last nine years of data from the EML Quality Assessment Program (QAP).

To develop scoring schemes for PE results, a collaboration with Dr. S. Hedayat (University of Illinois, Chicago) was established. The IPEP data were subjected to a performance-based statistical assessment. A scoring scheme to address LMD criteria was developed and a report submitted to LMD and EML. An algorithm was developed to deconvolute the error in historical data, thus allowing all laboratories to identify those areas of individual performance which could most benefit from improvements. Draft reports of DOE laboratory performance were prepared and submitted to LMD for the CLP inorganic and organic PE programs for the four most recent quarters.

In support of LMD's effort to develop a pilot mixed-waste PE program, IPEP prepared draft letters requesting assistance, developed a draft reporting format, and prepared a list of candidate laboratories. A data base for receipt and analysis of the mixed waste PE data has been developed.

Determination of Americium in Environmental Samples (L. L. Smith)

Owing to the expanding DOE environmental restoration programs, vast numbers of radiochemical analyses are being required of analytical laboratories. The traditional analytical methods for the determination of americium are laborious and costly and are plagued by low and sporadic chemical yields. In a collaborative effort with the Chemistry Division (P. Horwitz, H. Diamond, M. Dietz, and R. Chiarizia) and the Environment, Safety, and Health Division (D. Nelson and T. Davis), we have developed a cost-effective, reliable method to determine americium in environmental samples by utilizing extraction chromatography. This method allows for the specificity of solvent extraction with the convenience of column chromatography. We have applied commercially available resins (EiChrome, Inc., Darien, IL) in the analyses of over 200 soil samples and have seen a dramatic savings in both time and cost (a factor of two in some cases). Equally important, we have reduced our generated waste by a factor of thirty.

Environmental Restoration Program of Rocky Flats Plant (L. L. Smith, R. W. Bane, A. M. Essling, D. G. Graczyk, R. R. Heinrich, R. B. Holtzman, F. Markun, E. C. Rauh, W. E. Streets, T. TenKate, and J. S. Yaeger)

Radiochemical analyses were completed in support of the Environmental Restoration Program at the Rocky Flats Plant (RFP). The ACL received over 200 samples and analyzed them for gamma emitters (i.e., Cs, Ra, Th, Am, Pu, and U). In addition, gross alpha/beta measurements were completed. Comprehensive data packages (both hard copy and electronic) were produced for this project. To improve cost effectiveness while maintaining accuracy and precision, new methods utilizing extraction chromatography were employed (see previous section). Additionally, various ACL staff members have served as technical advisors to the RFP program managers as they continue to develop the statement of work and the technical requirements for this expanding program.

Determination of ^{226}Ra in Human Tissues (F. Markun)

In the 1930s, some members of the medical community believed that radium possessed

therapeutic benefits. As a result, a number of patients were given known doses of ^{226}Ra for medical purposes. Subsequently, whole body counts and radon breath measurements were performed on these subjects to determine body burden levels.

It was general knowledge that ^{226}Ra is a bone-seeking radionuclide. However, little was known of its behavior in soft tissue. As a result, soft tissue samples such as internal organs, skin, and hair were obtained, posthumously, from one of these patients.

The standard technique of radon emanation was used to determine ^{226}Ra in these samples. Since the specific activity of ^{226}Ra in soft tissues was expected to be quite low, large samples were dissolved prior to analyses. Initially, radon-free air was bubbled through the samples to remove ^{222}Rn . At this point, the solutions were then sealed to allow ^{222}Rn to reach secular equilibrium with ^{226}Ra . Subsequently, the samples were once again sparged and the ^{222}Rn was determined.

The soft tissue specific activities for ^{226}Ra ranged from 0.03 to 5.00 pCi/g, whereas typical values for bone were on the order of 200 pCi/g. These radium results along with analytical data for calcium are being correlated by the ANL Biological and Medical Research Division to determine whether calculation of a radiological dose attributable to ^{226}Ra is feasible.

Rietveld Analysis of X-ray Powder Diffraction Data (P. L. Johnson and B. S. Tani)

A computer program for the Rietveld analysis of X-ray and neutron diffraction powder patterns, called DBW 4.0, has been installed on the VAX computer cluster at CMT. Test sets of data provided by D. Bish (Los Alamos National Laboratory) were successfully analyzed by this computer program.

X-ray powder diffraction data were also collected at ANL on alpha alumina and analyzed by DBW 4.0. For structural refinement by DBW 4.0, the intensity data measured at ANL with an automatic divergence slit were converted to intensities for fixed slits. A computer program was written for this data conversion. The data for alpha alumina were converted by this program and refined by DBW 4.0 to a reasonably good fit between the observed and calculated structure factors.

In addition to DBW 4.0, the computer programs XPAS and Lazy Pulverix-PC were added to

the software available in the ACL diffraction laboratory. The XPAS program analyzes powder X-ray diffraction data to obtain maximum information from a digital XRD pattern and includes automatic peak fitting, peak width determination, and zooming of a pattern into a window. The Lazy Pulverix-PC program calculates a powder pattern when the user specifies atomic positions, the space group, and the unit cell.

Radiochemical Analysis of the Experimental Boiling Water Reactor Vessel (D. L. Bowers, R. R. Heinrich, and W. E. Streets)

The Experimental Boiling Water Reactor (EBWR) facility was first put into operation at ANL in 1956 as a test reactor to demonstrate the feasibility of operating an integrated power plant with a direct-cycle, boiling-water reactor as its heat source. The EBWR was operated at the 20-MW (thermal) power level from its startup in December 1956 to December 1962, after which the output was increased to the 100-MW (thermal) level. After five years of successful operation at the higher power level, the EBWR was shut down in December 1967. All nuclear fuel was then removed, all liquids were drained from the process systems, and the facility was placed in a dry lay-up status. The facility was subsequently declared surplus and entered into the DOE Surplus Facilities Management Program. Decommissioning and decontamination (D&D) began in 1986. The current objectives are to remove all contaminated material from the containment building, clean up the facility to prescribed release levels, and eventually open the building to unrestricted use.

After 24 years of dry lay-up status, short-lived radionuclides could be ignored in the D&D operations; the remaining radionuclides of interest were ^{14}C , ^{55}Fe , ^{59}Ni , ^{63}Ni , ^{60}Co , ^{93}Mo , and ^{94}Nb . The radioactivity of the EBWR vessel was assessed using a combination of measured values from carbon and stainless steel samples and theoretical calculations based on the chemical composition of the alloy and the measured radioactivity levels.

During FY 1991, the ACL completed the determination of ^{60}Co , ^{55}Fe , and ^{63}Ni in the carbon and stainless steels. Other radionuclides of interest were present at levels too low to be measured.

During FY 1992, the ACL collaborated with personnel from the Engineering Physics (EP) Division (A. Purohit and D. Henley) in drafting the report of the radiological assessment of the EBWR vessel. We also assisted A. Olson (EP) in the comparison of his theoretically calculated activities for ^{60}Co , ^{55}Fe , and ^{63}Ni with experimental values; agreement was reasonably good, with best agreement for ^{60}Co , the most active radionuclide. A final report is being prepared.

Computer Generation of Reporting Forms for Environmental Analysis (Y. Tsai, L. L. Lamoureux, and E. Y. Hwang)

Numerous samples received by the ACL require reporting results on forms specified by the EPA CLP. In the past, calculations were done by the analyst, and several forms were filled out manually. We have installed ENVISION software (developed by THRU PUT System, Inc.) on a Hewlett-Packard (HP) UNIX Chemstation, which operates multiple instruments (HP gas chromatograph/mass selective detectors) for determination of volatile and semivolatile organic compounds. The ENVISION software is a report generation tool that automates the process of generating final reports and quality control reports. Analytical data for volatile and semivolatile organic compounds can be easily entered into a data base and processed by ENVISION to generate CLP reporting forms. In addition, ENVISION accepts data from other instruments that reside on a local area network (LAN). The PCB/pesticide analysis is performed on two HP gas chromatographs that are operated by an HP DOS system. Through the LAN we can transfer the PCB/pesticide data to the HP UNIX Chemstation and have these data processed by ENVISION for CLP forms generation. By automation, a significant time savings has been realized. A larger portion of the analyst's time can now be spent performing other analytical tasks.

Analysis of Environmental Samples for the U.S. Department of Agriculture (L. L. Lamoureux, G. A. Baudino, Y. Tsai, and A. S. Boparai)

Over the past four years, the ACL has participated in a collaborative effort with the ER Division (M. C. Hansen) that involves monitoring removal of contaminants (carbon tetrachloride and

chloroform) from groundwater under a former grain storage facility (Waverly, NE) operated by the U.S. Department of Agriculture. Contaminants from the Waverly aquifer are being removed by groundwater extraction and air stripping. Analyses are performed using CLP protocols. In FY 1992, carbon tetrachloride contamination in the Waverly aquifer was measured to have decreased over 40%. August 1992 data indicated carbon tetrachloride levels at 48 ppb in the Waverly aquifer. The aquifer will continue to be sampled monthly in FY 1993 because the decrease does not bring the level below the Federal health standard of 5 ppb. In FY 1992, the ACL analyzed over 100 groundwater and drinking water samples for this project.

Development of FTIR Analysis for Soils (J. C. Demirgian and M. Clapper-Gowdy)

The objective of the program is to develop a new method, utilizing FTIR spectroscopy, to analyze contaminated soil samples in the field for explosives and selected volatile and semivolatile analytes.

We have determined that soil contaminants can be thermally desorbed into a heated long-path infrared cell, and that explosives such as trinitrotoluene (TNT) could be detected at levels of 40 to 80 ppm in soil. Past experience has shown that results obtained with certified extraction methods have inaccuracies because of the bonding of the TNT to the soil. Analyses by our FTIR method eliminate this problem and can be done in minutes in the field.

Work is progressing on expanding the FTIR analysis to include 16 other volatile organic compounds. We have found that these volatiles can be detected at approximately 15 to 50 ppm in soils. Quantitative accuracy exceeds that obtained for TNT and the dinitrotoluene isomers due to better peak resolution. Prototype field equipment is being assembled, and a new method to thermally desorb the TNT from the soil is being explored.

This technology is applicable for site characterization and remediation and could play a significant role in monitoring clean up in areas contaminated by explosives.

Monitoring of Incinerator Effluent for Organic Analytes (J. C. Demirgian, E. Y. Hwang, P. L. Johnson, G. T. Reedy, and C. T. Snyder)

The objective of this collaborative effort with M. D. Erickson (CMT) and Z. Mao (ES) is to monitor incinerator effluent from different feeds and correlate the feed content with stack effluent by qualitatively and quantitatively analyzing the effluent using FTIR spectroscopy. A laboratory incinerator was constructed, tested, and interfaced to an FTIR spectrometer. The incinerator is now being used in experiments to identify and quantify the effluent from simulated PCB burns. To test the analysis equipment, the incinerator was deliberately run at lower temperatures than required for normal operation. Products of incomplete combustion were easily detectable by the FTIR method. Extensive QA/QC data for standards are currently being analyzed.

In the next year, we anticipate using the Toxic Substances Control Act incinerator at the DOE K-25 Plant in Oak Ridge, TN, for a field test program. The intention is to develop an EPA-certified method for using FTIR spectroscopy as a continuous emission monitor for incinerators.

Detection of Illegal Drug Laboratories (J. C. Demirgian)

The Drug Enforcement Administration is exploring new technologies for detecting illegal drug laboratories. The objective of this project is to perform chemical releases at field sites and then detect the release with advanced analytical instrumentation. The ACL is responsible for releasing known quantities of chemicals emitted by illegal drug laboratories and evaluating test results for all analytical equipment present at the release site. Equipment evaluated to date include spectrometers based on ion mobility, FTIR, forward-looking infrared, and fluorescence LIDAR (light detection and ranging).

The first chemical release has been completed, and the summary report is nearing completion. Both ion mobility spectrometry and FTIR spectroscopy were successful in detecting plumes emanating from simulated illegal drug laboratories, whereas the other techniques encountered problems. Plume models could not be used to track a plume because of slight variations in wind and mixing, as was demonstrated by the large differences between the concentrations detected by sorbent tubes and those

obtained at ground level with conventional infrared spectrometers.

Stand-off Detection (J. C. Demirgian and M. L. Tober)

The U.S. Army's Chemical Research, Development, and Engineering Center (CRDEC) has an ongoing program in "stand-off" or remote detection of chemical plumes. For this project, the ACL is determining the sensitivity of newly designed passive-remote FTIR spectrometers and enhancing the ability of existing spectrometers to properly detect plumes in the presence of interferences. Current work includes assisting in the development and testing of a hand-held FTIR spectrometer. The instrument does not yet have the sensitivity of conventional units, but its small size and weight offer potential for many specialized applications.

IV. QUALITY ASSURANCE

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

The ACL continued its participation in a variety of multi-laboratory programs sponsored by the EPA. These programs provided the ACL with sets of Quarterly or Semiannual Blind Performance Evaluation (PE) Samples for analysis. The active compounds in the blind samples were known only to the EPA. The ACL participation in PE programs is summarized below.

The ACL participated in two Organic and Inorganic Quarterly Blind PE studies (QB1FY92 and QB4FY92) from the U.S. EPA Environmental Monitoring Systems Laboratory-Las Vegas (EMSL-LV). Analytical results for the QB1FY92 study met EPA qualification criteria required for continued participation in the CLP. No results are yet available for QB4FY92.

As part of the ACL's continued participation in the EMSL-Cincinnati Water Pollution Study (WPS), semiannual blind samples (WP028) were analyzed for cyanide, oil/grease, total dissolved and suspended solids, and a variety of anions. During FY 1992, the EMSL-Ci (in Cincinnati, OH) reported that the analytical results reported by the ACL for the WP027 (analyzed in FY 1991) and WP028 studies were in excellent agreement with the EPA known values. Samples for the WP029 study were received in late September and will be analyzed in FY 1993.

The DOE Environmental Measurements Laboratory (EML) Quality Assessment Program (QAP) and the EMSL-LV coordinated multi-laboratory Radiological Interlaboratory Studies Program provide semiannual blind radiological PE samples for the ACL Instrumental Analysis Group. Water, soil, and air filter samples were received and analyzed for radiochemical parameters (e.g., gross alpha-beta, gamma, tritium, Ra, U, Pu, Np) throughout FY 1992. The ACL did well in these programs, with each of the radionuclides correctly determined.

In recent years, the ACL has analyzed thousands of non-environmental and environmental samples. Sample matrices include water, soil, sediment, sludge, air-filter paper, wood, and vegetation. Most analyses of environmental samples require methods prescribed by the EPA. These methods are described in statements of work written for the CLP or in the SW-846 methods developed for RCRA.

Sixty-one CLP-type sample data packages, consisting of analytical results and raw data, were prepared, reviewed for completeness and correctness, and forwarded to customer data-evaluation and management teams.

The total number of ACL standard operating procedures (SOPs) written to address analytical methods, records management, sample tracking, administrative protocol, etc., increased from 161 to 175 in FY 1992. All are implemented and available to ACL personnel in bound, multi-volume manuals strategically located throughout ACL offices and laboratory areas.

Because the ACL receives thousands of samples each year, many requiring multiple analyses, considerable time is spent issuing sample notification documents, tracking samples, monitoring analytical holding times, and managing sample-related documents in general. The ACL has thus developed a computer-assisted bar code reading system that provides quick and error-free logging, distribution, and tracking of analytical samples; and the associated documentation has been prepared. The system's capabilities include providing sample notification memos, chain-of-custody forms, and summaries of samples categorized by analyses requested.

As an analytical laboratory participating in the WIPP Experimental-Waste Characterization Program, the ACL was required to write and submit a Quality Assurance Project Plan (QAPjP) to the WIPP Program Office and the State of New Mexico Environment Department for approval. The ACL WIPP QAPjP (Rev. 05) has been approved for use in the Program.

Management (internal) and independent (external) assessments provide a means to determine the quality of work by the ACL and to identify areas needing improvement. Assessments of the CMT were conducted by the ANL-E Office of Quality Assurance and the DOE-Chicago Field Office. The ACL was selected for assessment by these organizations, which involved assessing the implementation of the ACL Quality Assurance Plan and determining the appropriateness of standard operating procedures. Data were gathered from personal interviews, a review of record systems, review of analytical documents, review of procurement documents, review of sample tracking documents, and visits to laboratories. No negative findings were identified by either assessment group.

V. PROFESSIONAL ACTIVITIES

A. Publications and Reports

Analytical Chemistry Laboratory Progress Report for FY 1991

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

Argonne National Laboratory Report No. ANL/ACL-91/1 (1991)

A Quantitative Method to Detect Explosives and Selected Semivolatiles in Soil Samples by Fourier Transform Infrared Spectroscopy

M. Clapper-Gowdy, J. C. Demirgian, K. Lang, and G. Robaillaite

Proceedings of the 1992 Joint Army Navy NASA Air Force (JANNAF) Safety and Environmental Subcommittee Meeting, Monterey, CA, August 10-14 (1992)

Comparison of Acid Leachate and Fusion Methods to Determine Plutonium and Americium in Environmental Samples

L. L. Smith, F. Markun, and T. TenKate

Argonne National Laboratory Report No. ANL/ACL-92/2 (1992)

Comparison of Passive-Remote and Conventional Fourier Transform Infrared Systems for Continuously Monitoring Incinerator Emissions

J. C. Demirgian and C. L. Hammer

Proceedings of the 11th Annual Incineration Conference, Albuquerque, NM, May 11-15, (1992)

Determination of Si Species

D. G. Graczyk, A. M. Essling, and J. K. Fink

Appendix F to ACE MCCI Test L8, Test Data report, Volume II-Aerosol Analysis, Argonne National Laboratory Report No. ACE-TR-C32, Volume II (1992)

Determination of Trace Impurities in Uranium Oxides by Laser Ablation Inductively Coupled Plasma Mass Spectrometry

J. S. Crain and D. L. Gallimore

J. of Analytical Atomic Spectrometry 7, 605-610 (1992)

Determining Regulated Metals in Soil: A Comparison of EPA Methods with Laser Ablation ICP-MS and XRF Spectroscopy

J. S. Crain, J. Hansel, and J. Troxel

Spectroscopy 7(5), 40-46 (1992)

Effects of Data Sampling Rate on Pattern Recognition Analysis of FTIR Interferograms

G. W. Small and S. E. Carpenter

Applied Spectroscopy 46, 49 (1992)

Growth of *c*-axis Oriented Films of $\text{YbBa}_2\text{Cu}_3\text{O}_{7.8}$ on Single and Polycrystalline MgO Substrates by Oxidation of a Liquid Alloy Precursor

J. S. Luo, N. Merchant, V. A. Maroni, D. M. Gruen, B. S. Tani, K. H. Sandhage, and C. A. Craven

Physica C 192, 356-361 (1992)

Innovative Methods for Inorganic Sample Preparation

A. M. Essling, E. A. Huff, and D. G. Graczyk

Argonne National Laboratory Report No. ANL/ACL-92/1 (1992)

Separation and Preconcentration of Uranium from Acidic Media by Extraction Chromatography

E. P. Horwitz, M. L. Dietz, R. Chiarizia, H. Diamond, A. M. Essling, and D. G. Graczyk

Analytica Chimica Acta **226**, 25-37 (1992)

Thermostability and Decomposition of the (Bi,Pb)₂Sr₂Ca₂Cu₃O₁₀ Phase in Silver-Clad Tapes

J. S. Luo, N. Merchant, V. A. Maroni, D. M. Gruen, B. S. Tani, W. L. Carter, G. N. Riley, Jr., and K. H. Sandhage

J. of Applied Physics **72**(6), 2385-2389 (1992)

Trace Elements in Pyrites of the Green River Formation Oil Shales, Wyoming, Utah, and Colorado

W. J. Harrison, D. R. Pevear, and P. C. Lindahl

Geochemical, Biogeochemical, and Sedimentological Studies of the Green River Formation, Wyoming, Utah, and Colorado, M. L. Tuttle, Ed., Chapter D in the U.S. Geological Survey Bulletin 1973-A-G, 23 pp. (1991)

Uranium Release and Secondary Phase Formation During Unsaturated Testing of UO₂ at 90°C

D. J. Wronkiewicz, J. K. Bates, T. J. Gerding, E. Veleckis, and B. S. Tani

J. of Nuclear Materials **190**, 107-127 (1992)

B. Oral Presentations

Advances in Passive-Remote Fourier Transform Infrared Detection and Quantitation of Plumes

J. C. Demirgian and C. L. Hammer

Workshop on Infrared Emission Measurements by FTIR, Quebec, Canada, February 12-14, 1992

An Interlaboratory Performance Evaluation Program for DOE's Environmental Restoration and Waste Management Programs

P. C. Lindahl, W. E. Streets, S. Morton, T. F. Gesell, D. C. Bogen, and D. Bottrell

32nd ORNL-DOE Conference on Analytical Chemistry in Energy Technology, Gatlinburg, TN, October 1-3, 1991

Computer Generation of EPA CLP Reporting Forms for Organic Analysis

L. L. Lamoureux

16th ACL Technical Meeting, Argonne National Laboratory, Argonne, IL, February 25, 1992

Determination of TRUs and Sr in Soil by Elution Chromatography

L. L. Smith

15th ACL Technical Meeting, Argonne National Laboratory, Argonne, IL, November 5, 1991

Determination of Uranium and Uranium Isotopes in High Silica Soils by Laser Kinetic Phosphorimetry and Thermal Ionization Mass Spectrometry

A. M. Essling, R. W. Bane, and D. G. Graczyk

37th Annual Conference on Bioassay, Analytical and Environmental Radiochemistry, Ottawa, Canada, October 7-11, 1991

Determination of Volatile Organics in Soils Utilizing Fourier Transform Infrared Spectroscopy

M. Clapper-Gowdy and J. C. Demirgian

Amoco/University Poster Session, Naperville, IL, October 4, 1991

Determination of Volatile Organics in Soils Utilizing Fourier Transform Infrared Spectroscopy

M. Clapper-Gowdy, J. C. Demirgian, K. Lang, and D. Scarborough

43rd Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, New Orleans, LA, March 9-13, 1992

Development of an FTIR Method to Measure Explosives and Selected Contaminants by Thermal Desorption FTIR

M. Clapper-Gowdy

15th ACL Technical Meeting, Argonne National Laboratory, Argonne, IL, November 5, 1991

Development of an Interlaboratory Performance Evaluation Program (IPEP) for the Department of Energy's Environmental Restoration and Waste Management

W. E. Streets, P. C. Lindahl, S. Morton, T. F. Gesell, D. C. Bogen, C. Sanderson, and D. Bottrell

37th Annual Conference on Bioassay, Analytical and Environmental Radiochemistry, Ottawa, Canada, October 7-11, 1991

Development of an Interlaboratory Performance Evaluation Program (IPEP) for the Department of Energy's Environmental Restoration and Waste Management Programs

W. E. Streets

16th ACL Technical Meeting, Argonne National Laboratory, Argonne, IL, February 25, 1992

DOE's Analytical Services Program

P. C. Lindahl

DOE-CH Waste Management/Waste Minimization Meeting, Brookhaven National Laboratory, Upton, NY, June 16-19, 1992

DOE's Environmental Sampling and Analysis QA Program - The Performance Evaluation Program

P. C. Lindahl

3rd International Environmental and Waste Management Conference (American Society for Quality Control - Energy Division), Las Vegas, NV, May 4-6, 1992

Ion Chromatography: A Modern Version of Ion Exchange Chromatography. A General Review

C. S. Sabau

17th Congress of the American Romanian Academy of Arts and Science, California State University, Northridge, CA, June 3-7, 1992

New Method for Isolating Strontium for Isotopic Analysis

A. M. Essling

37th Annual Conference on Bioassay, Analytical and Environmental Radiochemistry, Ottawa, Canada, October 7-11, 1992

Quality Assurance for Headspace Gas Analysis in the WIPP Experimental Waste Characterization Program

D. A. Bass, P. C. Lindahl, J. Arvizu, M. Connolly, J. Connolly, K. Knudtsen, and M. J. Duff

32nd ORNL-DOE Conference on Analytical Chemistry in Energy Technology, Gatlinburg, TN, October 1-3, 1991

Quality Assurance for Headspace Gas Analysis in the WIPP Experimental Waste Characterization Program

D. A. Bass

15th ACL Technical Meeting, Argonne National Laboratory, Argonne, IL, November 5, 1991

Quality Assurance Program Plan for the Waste Isolation Pilot Plant Experimental-Waste Characterization Program

K. Knudtsen, B. Marron, J. Arvizu, M. Connolly, J. Connolly, D. A. Bass, P. C. Lindahl, B. Draper, F. Dooley, and M. Duff

32nd ORNL-DOE Conference on Analytical Chemistry in Energy Technology, Gatlinburg, TN, October 1-3, 1991

Quantitation of Volatile Organic Compounds in the Headspace Over Transuranic Waste by Gas Chromatography/Mass Spectrometry

D. V. Applegate, G. A. Baudino, G. K. Gosztola, G. T. Reedy, Y. Tsai, and A. S. Boparai

32nd ORNL-DOE Conference on Analytical Chemistry in Energy Technology, Gatlinburg, TN, October 1-3, 1991

Quantitation of Volatile Organic Compounds in the Headspace Over Transuranic Waste by Gas Chromatography/Mass Spectrometry

D. V. Applegate

15th ACL Technical Meeting, Argonne National Laboratory, Argonne, IL, November 5, 1991

Radium Geochemistry and Isotopy of Thermal Waters and Travertines, Yellowstone National Park, Wyoming

N. C. Sturchio, J. K. Bohlke, and F. Markun

Geological Society of America Annual Meeting, San Diego, CA, October 21-24, 1991

The Potential of Remote Passive Fourier Transform Infrared Spectroscopy to Determine Compliance Under the Clean Air Act

J. C. Demirgian and C. L. Hammer

43rd Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, New Orleans, LA, March 9-13, 1992

The Stability of Calibration Standards for ICP/AES Analysis

D. R. Huff

16th ACL Technical Meeting, Argonne National Laboratory, Argonne, IL, February 25, 1992

TRU's and Strontium in Soils

L. L. Smith

37th Annual Conference on Bioassay, Analytical and Environmental Radiochemistry, Ottawa, Canada, October 7-11, 1991

Use of Supercritical Fluid Extraction/Gas Chromatography for Analysis of Organic Compounds

A. S. Boparai, R. L. Petty, A. M. Caison, and D. R. Bierma

43rd Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, New Orleans, LA, March 9-13, 1992

C. Meetings Attended

Dean A. Bass

43rd Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, New Orleans, LA, March 9-13, 1992

Amrit S. Boparai

43rd Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, New Orleans, LA, March 9-13, 1992

32nd ORNL-DOE Conference on Analytical Chemistry in Energy Technology, Gatlinburg, TN, October 1-3, 1991

Jeffrey S. Crain

Spectrum 92: Nuclear and Hazardous Waste Management Meeting, Boise, ID, August 24-27, 1992

EPA/ACS Waste Testing Symposium, Arlington, VA, July 16-17, 1992

Donald G. Graczyk

National Meeting on the DOE Moratorium on Off-Site Shipment of RCRA/TSCA Wastes to Unlicensed Commercial Treatment, Storage, and Disposal Facilities, Denver, CO, October 3-4, 1991

David W. Green

Analytical Laboratory Managers Association Meeting, Gaithersburg, MD, October 24-25, 1991

32nd ORNL-DOE Conference on Analytical Chemistry in Energy Technology, Gatlinburg, TN, October 1-3, 1991, Session Chairman

Paul L. Johnson

Argonne National Laboratory Symposium for Undergraduates in Science, Engineering and Mathematics, Argonne, IL, November 8-9, 1991, Session Chairman

CAT Scientific Programs Workshop, Argonne, IL, July 14-15, 1992

Peter C. Lindahl

Future Analytical Support Team (FAST) Meeting, Hanford, WA, February 2-6, 1992

SAFER (Streamlined Approach for Environmental Restoration) Workshop, Grand Junction, CO, March 25-26, 1992

Soil Quality Assurance Protocol Development Workshop, Las Vegas, NV, April 13-16, 1992

Carmen S. Sabau

17th Congress of the American Romanian Academy of Arts and Science, California State University, Northridge, CA, June 3-7, 1992, Science Session Chairman

Florence P. Smith

19th Annual Conference of the National Organization for the Professional Advancement of Black Chemists and Chemical Engineers, New Orleans, LA, April 20-24, 1992

W. Elaine Streets

Future Analytical Support Team (FAST) Meeting, Hanford, WA, February 2-6, 1992

SAFER (Streamlined Approach for Environmental Restoration) Workshop, Grand Junction, CO, March 25-26, 1992

Site-Specific Soil Quality Assurance Materials Interagency Workshop, Las Vegas, NV, April 13-16, 1992

Eighth Annual Waste Testing and Quality Assurance Symposium, Arlington, VA, July 13-17, 1992

Sixth Annual Interagency Workshop on Quality Assurance for Environmental Measurements, Las Vegas, NV, September 14-17, 1992

D. Professional Organization

Dean A. Bass

American Chemical Society

Sigma Xi

Society for Applied Spectroscopy

Amrit S. Boparai

American Chemical Society

Sigma Xi

Delbert L. Bowers

American Chemical Society

American Society for Testing and Materials

Scott E. Carpenter

American Chemical Society

North American Chapter of the International Chemometrics Society

Maureen Clapper-Gowdy

American Chemical Society

Society for Applied Spectroscopy

Jeffrey S. Crain

American Chemical Society

Division of Analytical Chemistry

Jack C. Demirgian

American Chemical Society

Air and Waste Management Association

Technical Association of the Pulp and Paper Industry

Irene M. Fox

Society for Applied Spectroscopy

Association for Women in Science

Sigma Xi

Donald G. Graczyk

American Chemical Society

Sigma Xi

American Society for Mass Spectrometry

David W. Green

American Chemical Society

Division of Analytical Chemistry

Analytical Laboratory Managers Association, Board of Directors

Sigma Xi

Doris R. Huff

Phi Beta Kappa

Edmund A. Huff

American Chemical Society

American Society for Testing and Materials

Society for Applied Spectroscopy

Sigma Xi

Paul L. Johnson

American Crystallographic Association

Association for Computing Machinery

Sigma Xi, ANL Chapter Assistant Secretary

Peter C. Lindahl

American Chemical Society
Division of Analytical Chemistry
Division of Fuel Chemistry

Society for Applied Spectroscopy

Francis Markun

Health Physics Society
Midwest Chapter of Health Physics Society

Gerald T. Reedy

American Chemical Society

Society for Applied Spectroscopy

Carmen S. Sabau

American Chemical Society
Nuclear Chemistry and Technology
Chicago Section

American Nuclear Society
Fuel Cycle and Waste Management Division
Chicago Section

American-Romanian Academy of Arts and Sciences

Association of Women in Science
Chicago Chapter

New York Academy of Sciences

Sigma Xi

Florence P. Smith

National Organization of Black Chemists and Chemical Engineers

Christine T. Snyder

American Chemical Society

Lynn B. TenKate

Society for Applied Spectroscopy

Sigma Xi

Tony TenKate

American Association of Physics Teachers

Sigma Xi

E. Professional Training

Dean A. Bass

SAS Statistical Software Program, Argonne, IL, January 23 and 28, 1992

Ward Scientific Environmental Data Reporting System, Argonne, IL, September 21-23, 1992

Maureen Clapper-Gowdy

Applications in Fourier Transform Infrared Spectrometry, Society for Applied Spectroscopy, New Orleans, LA, March 7-8, 1992

Explosives Basic Safety Training, United States Army Defense Ammunition Center and School, Savanna, IL, July 20-24, 1992

Supercritical Fluid Chromatography for the Nineties: Applications and Modifications, Skokie, IL, September 30, 1992

Antoinette G. Engelkemeir

Ward Scientific Environmental Data Reporting System, Argonne, IL, September 21-23, 1992

Alice M. Essling

Electronic Spreadsheet Using LOTUS 1-2-3, High School District #88, Addison, IL, November 13, 1991

Paul L. Johnson

SAS Statistical Software Program, Argonne, IL, January 23 and 28, 1992

Statistics, College of DuPage, Glen Ellyn, IL, June-August 1992

Ward Scientific Environmental Data Reporting System, Argonne, IL, September 21-23, 1992

Jane J. Marr

SAS Statistical Software Program, Argonne, IL, January 23 and 28, 1992

Ward Scientific Environmental Data Reporting System, Argonne, IL, September 21-23, 1992

Kathleen J. Parish

Troubleshooting HPLC Systems, Chicago LC Resources, Schiller Park, IL, April 8, 1992

Chromatography Data Handling, Chicago LC Resources, Schiller Park, IL, April 9, 1992

Carmen S. Sabau

1992 Varian Atomic Spectroscopy Workshop, South Chicago, IL, May 1, 1992

Fourth Annual Spring Atomic Spectroscopy Workshop -- AA and ICP, Varian Analytical, Alsip, IL, May 1, 1992

Christine T. Snyder

Partial Pressure Analyzers: Analysis and Applications, American Vacuum Society, October 1991

Lab Calc Software Training Course, Galactic Industries, Inc., Argonne, IL, May 19-21, 1992

Lynn B. TenKate

The Environmental Data Reporting System, Ward Scientific Ltd., Argonne, IL, September 21-23, 1992

Yifen Tsai

Introduction to Unix, Computing and Telecommunications Division, Argonne, IL, January 13-15, 1992

HP Target Training, Hewlett Packard Company, Argonne, IL, April 15-16, 1992

Envision Software Training, THRU-PUT Company, Argonne, IL, August 11-12, 1992

F. ACL Seminars

Radioactive Mixed Waste Characterization

Dr. Wayne H. Griest, Oak Ridge National Laboratory
February 20, 1992

Polyphosphazenes in Membrane Processes and Composite Materials

Dr. Eric S. Peterson, Idaho National Engineering Laboratory
April 23, 1992

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