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Analytical Chemistry Laboratory

Progress Report for FY 1991

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Chemical Technology Division
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ANALYTICAL CHEMISTRY LABORATORY
Progress Report for FY 1991

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December 1991

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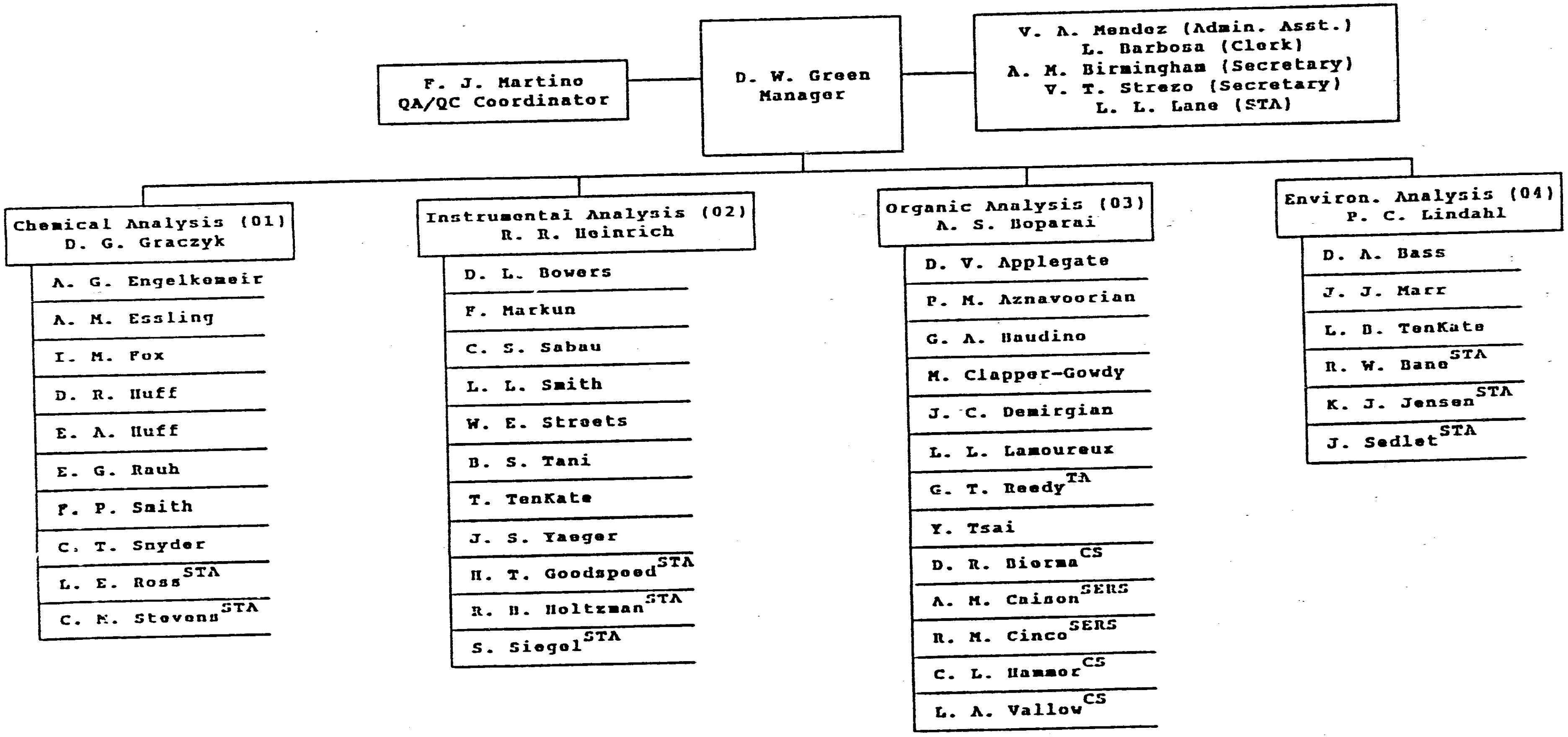
I. INTRODUCTION

The purpose of this report is to summarize the activities of the Analytical Chemistry Laboratory (ACL) at Argonne National Laboratory (ANL) for Fiscal Year 1991 (October 1990 through September 1991). This is the eighth annual report for the ACL [the reports for FY 1984 through FY 1990 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), and ANL/ACL-90/2 (December 1990)].

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 conducts a research program in analytical chemistry, works on 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.

The ACL is administratively within the Chemical Technology Division (CMT), its principal client, but provides technical support for most 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 50 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



CS - Co-op Student; STA - Special Term Appointee; TA - Term Appointee; SERS - Science and Engineering Research Semester.

Fig. 1. Analytical Chemistry Laboratory Organization Chart

(including two thermal-ionization mass spectrometers), emission spectrographs, autotitrators, sulfur and carbon determinators, and a kinetic phosphorescence uranium analyzer.

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, inert gas fusion of metals, 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, gas chromatographs, gas 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, 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, and a supercritical fluid chromatograph (SFC). Organic Analysis Group personnel also develop methods for such purposes as detecting organic compounds remotely with FTIR spectrometers and performing rapid on-site analyses for organic constituents.

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

II. ADMINISTRATIVE HIGHLIGHTS

The ACL acts both as a full-cost-recovery service center in support of ANL programs and as 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 1991, the total funding for the ACL increased to its highest level, as shown in Fig. 2.

The ACL recovered 98.4% of its total operating costs in FY 1991. Over the last seven years, the ACL has recovered 98.0% of its costs. 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 1991, which are largely effort related.

The ACL obtained valuable help from 11 Special Term Appointees (STAs) (see Fig. 1) in FY 1991 to assist with load leveling. The STAs provided about two full-time equivalents of assistance. Ten students and co-ops and two high-school faculty members supplemented the regular ACL staff.

New capabilities of the ACL established in FY 1991 include a laboratory to prepare radioactive environmental samples for analysis, new photoprocessing equipment, a microwave digestion system, an automated CHN analyzer, a new mass spectrometer for organic analysis, and the facilities and procedures to do TCLP analysis of radioactive samples.

Support Development Funds (SDF) allowed us to measure response factors and quantitation limits for the VG 30-01 gas mass spectrometer that was obtained in FY 1990. This instrument is capable of measuring common room-temperature gases, and has the resolving power to determine CO in a nitrogen matrix. Reporting forms for organic analytical data have been automated in a second project supported by SDF. Generation of forms by computer not only eliminates transcription errors, but reduces the time required of the analyst. The stability of calibration standards for the ICP/AES instrument was investigated in a third SDF project. Preliminary data indicate that most calibration standards are stable for at least six months.

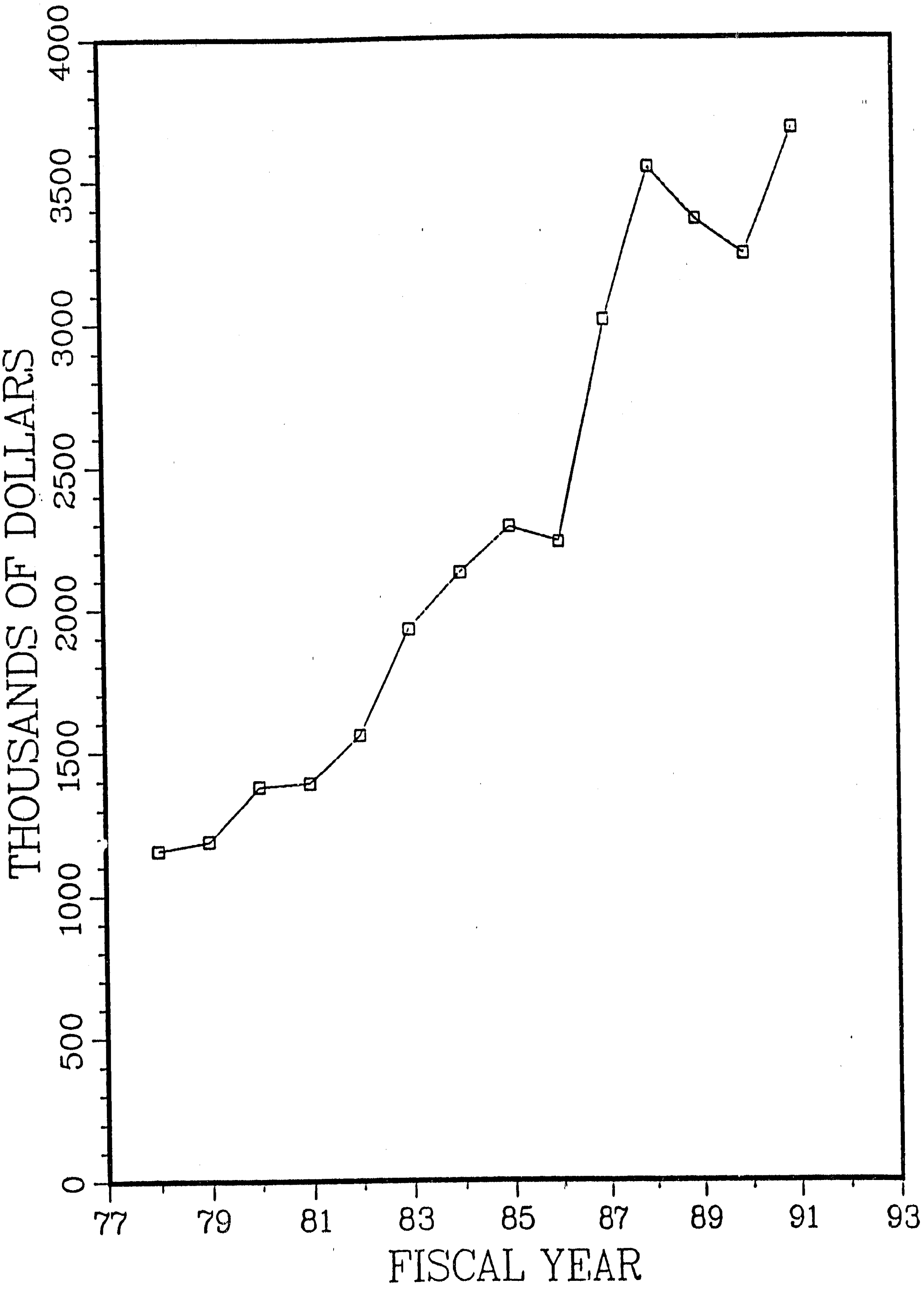


Fig. 2. Total ACL Funding History

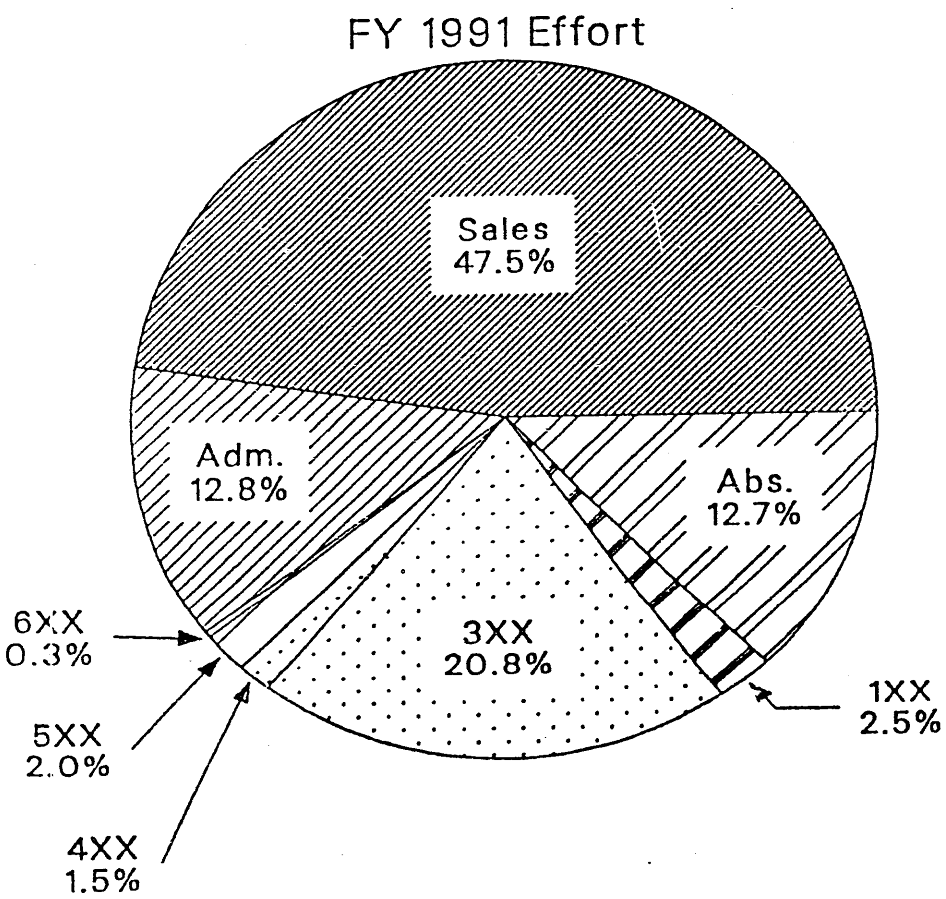


Fig. 3. Analytical Chemistry Laboratory
FY 1991 Effort

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

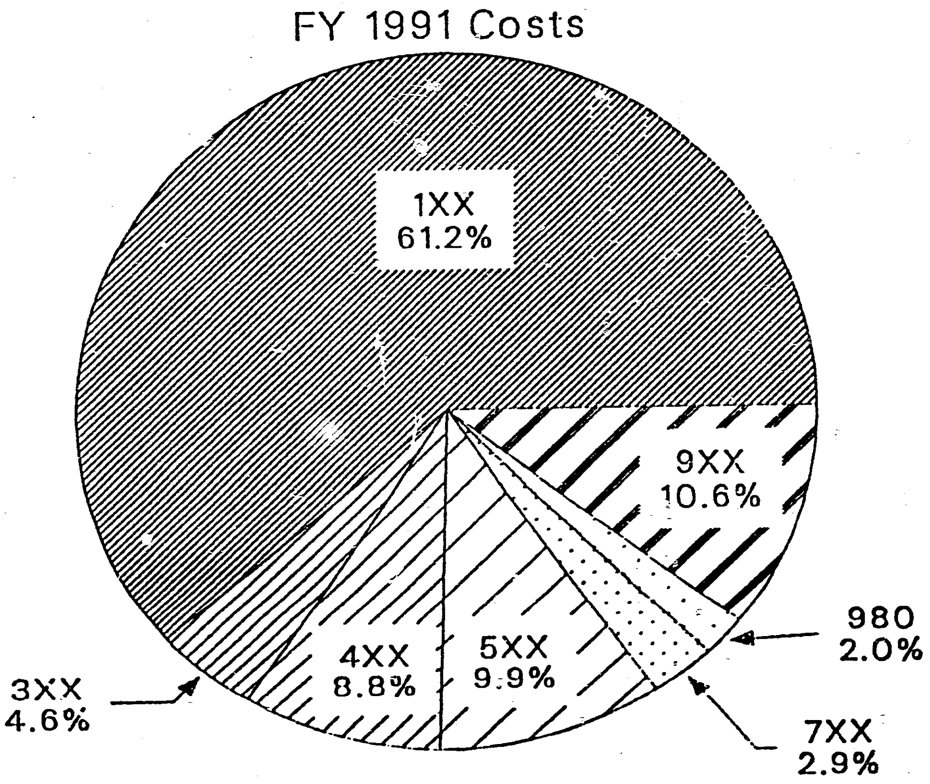


Fig. 4. Analytical Chemistry Laboratory
FY 1991 Total Operating Costs

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 three projects in FY 1991. The use of matrix modifiers was studied to improve accuracy in determinations by graphite furnace atomic absorption spectrophotometry. Analyte enhancement and suppression were observed as a function of modifier changes and were related to the concentrations of other ions in the various samples. Aluminum at a very low concentration (0.02%) was found to decrease the detection limit for lead and is now used in the modifier for samples in which either Pb or Ag is determined. In a second project, a novel method that utilizes octyl(phenyl)-N,N-diisobutylcarbamoyl-methylphosphine oxide (CMPO) in extraction chromatography was developed to determine transuranic elements in environmental samples. Chemical recoveries and turnaround times were improved by a factor of two to three, and chemical waste was reduced by a factor of 25 to 30. In a third project, separatory-funnel extraction was compared with continuous liquid-liquid extraction for the preparation of water samples for the analysis of semivolatile organic compounds. Liquid-liquid extraction proved to be the more efficient extraction method.

III. TECHNICAL HIGHLIGHTS

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

In the electrorefining of uranium and plutonium fuels for the IFR, metallic fuel pins (U, Pu, Zr) are dissolved in a molten cadmium anode. The actinide elements are electrochemically transported through a halide-salt electrolyte to the cell cathode, where they are collected as a metallic deposit. 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 cadmium anode, the halide-salt electrolyte, 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 a 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.

Samples were analyzed for zirconium by ICP/AES. Zirconium constitutes about 30 vol % of the IFR fuel and will be anodically dissolved in the electrorefiner during fuel reprocessing. Our analyses of components from a large-scale electrorefiner, after completion of uranium electrotransport experiments, provided insight to the fate of zirconium in the system. In addition, chemical analyses for various electrorefiner experiments helped demonstrate the concept of using a liquid cadmium cathode for obtaining a Pu-U mixture from spent IFR fuel.

Analyses in support of experiments dealing with IFR waste continued. Electrolytic salts containing small amounts of actinides (U, Pu, Am) were stripped of their heavy metals by reaction with Li in Cd-Li solutions at ~500°C. Analytical data from ICP/AES measurements showed sufficient reduction of actinides to allow disposal of these salts as a non-TRU waste.

Distribution studies of Nd and Pu in the cadmium metal-salt system were undertaken to understand their behavior in the pyrochemical process. The MSID method was used for the measurement of both Nd and Pu. From the data collected, distribution coefficients for these elements were determined over several orders of magnitude in metal and salt phases.

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, K. J. Jensen, 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 to determine Li, K, Cd, and the pertinent rare earths by ICP/AES and chloride by silver-chloride gravimetry. Each cadmium metal sample is similarly analyzed to determine Li, K, the rare earths, and Cl. Cadmium in the metal samples is measured by titration with ethylenediaminetetraacetic acid. Many salt samples are 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+}), which were present in some past experiments.

During FY 1991, approximately 130 salt-phase or cadmium-phase samples were analyzed for this project. Competitive-exchange studies between rare earths were extended to include characterization of separation factors between the rare earths and selected other elements. Experiments involved element pairs containing yttrium with cerium and yttrium with samarium. The Y-Sm experiment provided separation factors for the pair. Because lithium was transferred from the salt to the metal phase in these experiments, information was also obtained on the Li-Sm pair. Recently, measurements were begun to obtain similar information on the Sm-Eu-Li-Ba-Sr system. Preliminary results from samples associated with these experiments suggest the presence of unanticipated species in the salt phase. Methods for the qualitative and quantitative determination of these species are being investigated in the ACL.

X-ray Diffraction of Zeolite Materials (B. S. Tani and S. Siegel)

Zeolites are candidate materials for immobilizing IFR waste salt. X-ray diffraction (XRD) is a useful analytical technique for identifying phase changes resulting from the effects of dehydration procedures, equilibration temperatures, and salt compositions on the crystal structure of zeolites equilibrated with molten simulated IFR salt. This technique was used to identify non-zeolitic decomposition products that were formed during the equilibration of zeolite with molten salt under different experimental conditions. The XRD data were used for estimating the amount of non-zeolitic material in samples containing mixtures of "salt occluded" zeolite and non-zeolitic material. Relative compositional estimates were made from peak-height ratios determined for the major non-overlapped intensity peak of each phase. This is a reasonable approximation since the structures of the zeolites are not known completely. The XRD technique is being used to screen new starting materials and to determine phase stability by correlating changes in crystal structure with different experimental conditions.

Phase Composition/Solubility Studies on Equilibrated Mixtures of LiCl and KCl in Water (K. J. Jensen, E. G. Rauh, E. A. Huff, and A. M. Essling)

The LiCl-KCl eutectic electrolyte used in the electrorefining step of the IFR pyroprocess is inherently hygroscopic, and the possibility exists that this electrolyte could absorb water in the event of an earthquake and resulting destruction of the reprocessing facility. Conceivably under certain conditions, neutrons could be moderated by the diluted electrolyte and lead to criticality.

Researchers in CMT (J. P. Ackerman and R. A. Greene) are studying the equilibrium compositions of solid and liquid phases in mixtures of LiCl, KCl, and water at 8 to 10 temperatures ranging from 1 to 40°C above the dew point (in air of fixed absolute humidity) in each of four discrete experiments. The purposes of this work are to (1) understand the relationship among temperature, humidity, and the compositions of all solid and liquid salt-containing phases, (2) provide some of the data necessary to evaluate worst-case scenarios for the possibility of criticality, and (3) develop a facility design that precludes such criticality.

The ACL has thoroughly characterized 33 samples in support of this work. The samples included both LiCl/KCl solutions obtained from the equilibration studies and salts that separated from these solutions when equilibrium was reached under experimental conditions. The water and residue (salt) contents of each sample was determined, as well as the chemical composition of each residue. The amount of residue available for compositional analysis was generally small, ranging from 100 to 500 mg.

Water content was determined as weight loss on heating to a temperature of 350°C. The residue from the water determination was weighed and dissolved in water to provide a weighed solution from which weight aliquots were taken for determining Cl, K, and Li content. Chlorine, which is present as chloride, was determined gravimetrically as AgCl. After determination of the chloride content, the K and Li contents were calculated using an equation based on mass and charge balances and the knowledge that only K, Li, and Cl are present in the residue:

$$\text{Wt. \% K} = 121.5768 - 1.4537\ (\text{Wt. \% Cl})$$

Using these calculated values, we were able to take aliquots of an optimum size for the K and Li determinations. (For the reader who might wish to derive this equation, we note that a measured value for the atomic weight of lithium in these salts, 6.939, was used for our calculations.)

Potassium content was measured in the aliquots by a semi-micro gravimetric procedure as potassium tetraphenylborate. In a few cases where the amount of potassium available was insufficient for a gravimetric determination, the potassium content was measured by ICP/AES. Lithium content was measured by thermal ionization mass spectrometry (TIMS), using an isotope dilution technique with a ^{6}Li spike.

Results from these measurements have allowed an accurate accounting of the salt components in the liquid and solid phases that occur under various salt/water equilibrium conditions. The data have provided information on not only the extent of water absorption by the eutectic salt, but also the compositional changes that occur as the brine produced by water absorption becomes saturated, with re-separation of a solid phase. Empirical correlation and theoretical interpretation of the results are expected to benefit from the accuracy and completeness of the analytical measurements performed for this project.

Procedures for Determining Uranium and Uranium Isotopes in High Silica Soils

(A. M. Essling, R. W. Bane, E. G. Rauh, and D. G. Graczyk)

High silica soils present a special challenge to the analyst determining total uranium and uranium isotopes for environmental monitoring and remediation studies because quantitative recovery of the uranium requires complete dissolution of resistant siliceous mineral phases. This year, the ACL developed and implemented procedures which provide (1) complete dissolution of high silica soils without time-consuming fluoride/pyrosulfate fusions of the sample, (2) convenient measurement of total uranium by laser kinetic phosphorimetry, and (3) efficient isolation of uranium from the dissolved sample for isotopic analysis by TIMS. These procedures take advantage of ACL experience in dissolution chemistry, recent ANL advancements in actinides separation technology, and the capabilities of existing ACL instrumentation.

Complete dissolution of silica-bearing materials is accomplished by placing the sample (typically 0.25 g) in a Teflon-lined pressure vessel (Parr Decomposition Vessel) with a mixture of nitric and hydrofluoric acids and then heating overnight at moderate temperature (150°C). The solution from the Parr Decomposition Vessel is evaporated to dryness, and the residue is redissolved in dilute nitric acid for direct introduction to a Kinetic Phosphorescence Analyzer, which measures total uranium. The unused portion of this same solution is again taken to dryness and treated with sulfuric acid to remove fluoride, which inhibits separation of aluminum from uranium in subsequent operations (AlF_3 follows uranium in the column-extraction chromatographic process, although uncomplexed aluminum does not). The treated residue is dissolved in 2 N nitric acid and passed through a U/TEVA·Spec chromatographic column to isolate a pure uranium fraction for TIMS determination of the isotopic composition.

Performance of the procedures has been monitored under actual production conditions by analyzing a high silica matrix standard, NBS (currently NIST) Standard Reference Material (SRM) 4353 ("Rocky Flats Soil Number 1"). This laboratory control standard (LCS) was processed with each batch of field samples. Over a period of nine months, during which almost 200 soils and 18 laboratory control samples were analyzed, the LCS data show an average recovery for total uranium of 96.8%, and a standard deviation of 4.5%, relative to the 3.15 μg U per g soil reference value. Isotopic data for ^{234}U and ^{238}U agree with NBS reference values within the uncertainty of the TIMS measurements. Results for ^{235}U indicate a slight nonuniformity in the distribution of this isotope in SRM 4353 when 0.25 g samples are used but, on average, are consistent with the ^{235}U concentration (uncertified) shown on the NBS certificate.

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

The ACL assisted the Environmental Assessment and Information Sciences Division (EID) in staffing DOE Tiger Teams and Environmental Assessment Teams for several DOE sites during FY 1991. The ACL staff participated in the Tiger Team assessment of Lawrence Berkeley Laboratory (Environmental Subteam-Environmental QA), and the environmental audits of the Southwestern Power Administration (Environmental Management and Environmental QA), the Uranium Mill Tailings Remedial Action Project (Environmental QA), and the Components Demonstration and Integration Facility (Environmental Management and Environmental QA).

Battery Program (J. J. Marr)

The ACL technical support to battery-testing programs in CMT (J. A. Smaga) was continued during FY 1991. The ACL staff took part in the examination of 20 aqueous and high-temperature batteries tested at the Battery Analysis and Diagnostic Laboratory. These examinations served to identify existing and potential failure mechanisms of the hardware components, and to correlate changes in performance with changes in the morphology and composition of the electrodes. The information obtained from these studies permitted accurate assessment of the technical progress in battery research. It also provided a better definition of additional R&D needs. Individual reports were prepared for each of the batteries examined and were forwarded to the battery developers and to personnel associated with the program for review.

Analysis of a Novel Neutron Absorber (K. J. Jensen, E. G. Rauh, and E. A. Huff)

A new material to absorb neutrons has been developed at ANL to withstand high temperatures and is now in use at the Intense Pulsed Neutron Source (IPNS). The material was created by specialists in the Materials and Components Technology Division (T. Wiencek et al.) and consists of ^{10}B-enriched boron metal sandwiched between two copper plates.

Analysts in the ACL developed a procedure for accurately determining the total ^{10}B content in samples of known surface area. Results from this determination allow calculation of a density factor, i.e., atoms of ^{10}B per unit area of material, which provides a measure of the neutron absorption capacity of the material. This density factor is an important parameter for evaluating the suitability of the material for use in IPNS.

The ^{10}B was determined by an MSID technique using boric acid of natural isotopic composition (NBS SRM 951) as the boron spiking agent. Sample preparation prior to the MSID determination of ^{10}B required special care and use of a novel method for separating boron from copper in the nitric-acid matrix obtained from dissolving each sample. To prevent boron pickup from the dissolution apparatus and to avoid losses of boron by volatilization, each sample was dissolved in a quartz reflux apparatus. An insoluble silica residue (from an SiO_2 impurity in the boron metal used in fabricating the sample material) was isolated and separately analyzed to determine the small amount of boron that the residue contained. The separation of boron from copper nitrate and nitric acid was accomplished with a mixed-bed ion-exchange system, which yielded an eluate of boric acid in water. This solution was readily analyzed for boron by MSID. The results for all the materials analyzed demonstrated a ^{10}B density that met IPNS specifications.

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

During FY 1991, the ACL assisted the ANL Research and Development Program Coordination Office in providing technical reviews of quality assurance plans for private sector contractors. These contractors are conducting applied research and development to improve DOE's environmental restoration capabilities. This work included review of revisions of the QA plans. Standard Operating Procedures, which are step-by-step instructions for performing operations, were also reviewed for these contractors.

Review of Analytical Data from Subcontractor Analytical Laboratories (J. J. Marr and L. B. TenKate)

The ACL assisted the ANL Environmental Research Division (ER) in reviewing inorganic analytical data generated for environmental samples taken at ten sites around the United States. These sites are being studied to determine the extent of soil, surface water, and groundwater contamination. Analyses were performed by subcontractor analytical laboratories on samples collected by ER personnel for various government agencies. The ACL work included reviewing the laboratory data according to the guidelines of the U.S. EPA Contract Laboratory Program (CLP), providing detailed reports of problems encountered with the data, and evaluating the usability of the data.

Four private laboratories were also evaluated to determine their suitability to do work for ER programs. Inorganic data packages were evaluated to determine how well each laboratory could meet CLP analytical method and reporting criteria.

The ACL also worked with ER to establish a protocol for the data review process. This document explains the various types of data review that can be provided to ER project managers and sponsors. In addition, ACL and ER personnel collaborated on writing a summary of inorganic analytical methods to aid ER project managers.

Study of N_2O Emissions from Fluidized-Bed Combustor (L. B. TenKate and L. A. Vallow)

Nitrous oxide emissions from the ANL fluidized-bed combustor (FBC) were studied by a CMT researcher (W. F. Podolski). The objective of this study was to determine the effect of various parameters on the production of N_2O, which is considered to be one of the gases contributing to the greenhouse effect and the depletion of the ozone layer. The ACL work for this study included setting up a GC system to measure N_2O concentrations by direct on-line sampling of the FBC stack gas, periodically calibrating the GC, and calculating measured N_2O concentrations using the generated calibration curves. The level of N_2O emissions from the ANL FBC appeared consistent with other reported FBC measurements and is significant when compared with N_2O emissions from other types of combustors.

Environmental Monitoring Program (A. M. Essling, J. J. Marr, S. H. Newnam, L. E. Ross, F. P. Smith, and L. B. TenKate)

During FY 1991, the ACL prepared approximately 600 environmental water samples in support of the Environmental Monitoring Program at ANL-East. Metals determinations on these digested samples by flame or furnace atomic absorption spectrophotometry were subsequently performed by personnel from the Environment, Safety, and Health Division (ESH). During this same period, the ACL also analyzed 180 wastewater samples collected by ESH personnel from the ANL-East site for oil and grease.

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

Analytical support for the NAPAP (K. J. Reimann, Materials and Components Technology Division) continued this year. Limestone and marble briquettes, exposed to a variety of atmospheric conditions at a number of sites throughout the country, are brought to ANL for sampling and analysis. Over the past few years, hundreds of specimens have been analyzed for anions (fluoride, chloride, nitrate, and sulfate) by ion chromatography, and a smaller number have been analyzed 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 they have provided a quantitative measure of attack by atmospheric sulfur and nitrogen oxides. Anion measurements were performed on approximately 260 specimens during FY 1991.

Molten Corium Concrete Interaction (MCCI) 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, and B. S. Tani)

In 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. Analytical support for these studies involves application of a wide variety of measurement techniques and requires considerable flexibility in approaching the analytical problems that arise from changes in the materials that are brought together in going from one test to the next. The concretes that have been used in particular experiments have included common limestone/sand aggregate, mixtures of different sized limestone, highly siliceous concretes (common in Europe), and serpentine-aggregate concrete imported from Russia. In some tests, volatile fission product metals (e.g., In, Te, Ag) were added to the corium simulation and had to be accounted for in methods used to dissolve and analyze sample materials from each test.

Typically, the solidified melt and aerosol samples are analyzed for Ag, Al, Ba, Ca, Ce, Cr, Cu, Fe, K, La, Mg, Mn, Mo, Na, Ni, Ru, Si, Sn, Sr, U, W, Zn, and Zr by ICP/AES; for Si by AAS or gravimetry; for anions by ion chromatography (often following pyrohydrolytic decomposition of the sample to isolate halides); and for miscellaneous elements such as S or C by appropriate methods. X-ray diffraction (XRD) measurements are often made to identify particularly interesting phases in selected samples. Gas samples are analyzed by mass spectrometry.

In FY 1991, we analyzed samples from three large-scale MCCI tests, which produced approximately 150 samples. These tests involved a limestone/sand concrete, a mixed limestone concrete, and a combination serpentine-siliceous concrete. Dissolving MCCI melts and aerosols for multielement analysis by ICP/AES is always challenging, and ACL has devised a repertoire of dissolution schemes for these samples, including closed-vessel procedures in a Parr Decomposition Vessel or Carius tube, microwave procedures, fusion procedures with lithium tetraborate or sodium carbonate as flux, and other special techniques. For these recent tests, the dissolution procedures were often guided by XRD analyses which identified undissolved phases and helped select alternative or followup approaches to dissolving these phases.

Results from past MCCI experiments indicated that silicon is present in certain aerosols as silicon metal and SiC, in addition to SiO_2. It is believed that most of the silicon comes off 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. To test for these reactions, ACL began this fiscal year to devise a procedure for determining the distribution of silicon among the three forms (Si metal, SiO_2, and SiC) in selected aerosol specimens. Preliminary work has demonstrated that silicon metal can be selectively dissolved in dilute sodium hydroxide and measured by ICP/AES. Determinations of SiO_2 and SiC in synthetic mixtures of the compounds have been fairly successful whenever the SiO_2 had been selectively dissolved with hydrofluoric acid. However, with authentic aerosols, the phase structure of the SiC is sometimes of a nature that it dissolves in HF as well. Work is underway to develop a method for differentiating SiC from SiO_2 in these aerosols. One promising approach is based on the expectation that carbon in these aerosols is all present as SiC. If this is so, the SiC can be estimated from a total carbon determination and the SiO_2 calculated by difference.

ANL 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 techniques; measurement of anion concentrations (F^-, Cl^-, NO_2^-, SO_4^{2-}, Br^-, I^-) by ion chromatography; determination of pH, alkalinity, or other properties by classical methods; and isotopic measurements by TIMS. 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 300 were processed during FY 1991) 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.

Radium Measurements (F. Markun)

Radium isotopes were measured in a variety of geochemically significant matrices (groundwaters, brines, soils, and rocks) for N. C. Sturchio (CMT). Radium determination in groundwaters requires that a large volume of water be passed through ~200 cm^3 of special radium-selective resin. For convenience in handling and shipping, this operation was performed in the field, and only the resins were shipped to ANL. Upon arrival at ANL, each resin was sealed into a metal can and γ-counted with a NaI detector. If ^{224}Ra determination was requested, the sample was analyzed before a significant amount of the ^{224}Ra had decayed (half-life = 3.6 days). For samples requiring only ^{226}Ra and ^{228}Ra determinations, the canned resins were allowed to age until ^{226}Ra reached equilibrium with its daughter ^{222}Rn (half-life = 3.825 days), which was then measured. The time allowed before analysis was at least 30 days.

Some of the brine samples had activity levels that were sufficiently high to use direct canning of 200 cm^3 samples without use of the radium-selective resin. The lower limits of detection for ^{226}Ra and ^{228}Ra by the least-squares method of data analysis on canned samples counted with a NaI detector are ~6 and 8 pCi, respectively.

Rock samples were analyzed by the above direct canning method. Where rocks were too small to fill the can, they were introduced into the can with NaCl (table salt) as a filler to maintain the same counting geometry.

Several samples of minerals and volcanic rock from South America were dissolved and analyzed for ^{226}Ra using the radon emanation method. Due to very low levels, the radium could not be determined by NaI γ-counting followed by least squares analysis of the data.

These analytical results will be used to (1) determine the behavior of radium isotopes in groundwater and brine and (2) estimate the age of some of the minerals where the ratios of radium isotopes are determined.

Analysis of Environmental 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, 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. H. Newnam)

The ACL provided analytical services to a variety of environmental monitoring, characterization, and remediation projects administered by the Environmental Research (ER) and Energy Systems (ES) Divisions and programs at other DOE facilities (Rocky Flats Plant and Idaho National Engineering Laboratory). Samples processed during FY 1991 included 198 waters, 305 soils/sediments, and 335 miscellaneous matrix samples (air, filters). These samples were analyzed according to protocols described in the U.S. EPA Contract Laboratory Program Statements of Work, 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.

Environmental analyses provided by the ACL include determination of inorganic constituents (metals, anions, cyanide, sulfide, total dissolved and suspended solids), organic compounds (volatiles, semivolatiles, polychlorinated biphenyls/pesticides, phenolics, oil and grease), and radionuclides (gamma emitters, actinides).

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

The ACL was requested to perform radiochemical analyses in support of the Rocky Flats Plant (RFP) Environmental Restoration Program. To date, the laboratory has received over 200 samples requiring assays for gamma emitters (e.g., cesium, radium, thorium), plutonium, americium, uranium, and strontium. In addition, gross alpha/beta measurements are in progress. Many determinations have been completed, and some data have been reported. New methods have been developed that allow for increased efficiency (factor of two less time for Sr and Am preparation for counting), lower analytical costs, and minimization of waste (factors of two or more less volume). Additionally, various ACL staff members have served as technical advisors to the RFP program managers as they developed the Statement of Work and the technical requirements for this program.

Determination of Americium by Extraction Chromatography (L. L. Smith)

The determination of americium in environmental samples is crucial to those individuals responsible for effluent monitoring from facilities employing radioactive substances in their operations. However, the traditional analytical methods employed by most laboratories are extremely lengthy and manpower-intensive. More important, these traditional methods are often plagued by low and sporadic chemical yields.

In a collaborative study with the Chemical Separations Group of the Chemistry Division (P. Horwitz, H. Diamond, M. Dietz, and R. Chiarizia) and the Environmental Monitoring Group of the Environment, Safety, and Health Division

(D. Nelson and T. Sullivan), we have developed a method to analyze environmental samples for americium utilizing extraction chromatography. This type of chromatography employs an extractant coated onto an inert support material. Extraction chromatography allows for the specificity of solvent extraction with the convenience of column chromatography. Our method has dramatically increased chemical recoveries (factor of two in some cases) and sample throughput (two times as many samples), while significantly decreasing generated waste (a factor of thirty).

Development of an Interlaboratory Performance Evaluation Program (P. C. Lindahl, W. E. Streets, D. A. Bass, and J. J. Marr)

In collaboration with the DOE's Radiological and Environmental Sciences Laboratory (RESL) and Environmental Measurements Laboratory (EML), who serve as lead laboratories, ANL is developing and implementing a comprehensive Interlaboratory Performance Evaluation Program (IPEP) for the DOE EM-531 Laboratory Management Branch (LMB). The program is designed to provide information on the quality of radiological and non-radiological analytical data being produced by all laboratories on which DOE is relying for environmental restoration and waste management sample analysis. The ACL is assisting the two lead DOE laboratories in the development of the program requirements and strategies for implementation, especially in the non-radiological portions of the program. The ACL is also developing the strategies for compiling and analyzing the performance evaluation (PE) data and for monitoring to assure that needed corrective actions are taken.

The ACL is participating in the Future Analytical Support Team (FAST) activities in support of the LMB. This group, which is an outgrowth of a Task Group formed by EM, provides advice and needed data that assist the LMB in its objective of ensuring that the required capacity and capabilities exist to provide the analytical support needed for DOE environmental restoration and waste management programs.

The ACL has been active in this project since March 1991. We have attended two planning sessions with LMB, RESL, EML, and other participants (in April and in September 1991). The IPEP will use existing PE programs where available and appropriate; therefore, we have gathered and compiled information from those programs about pertinent analytes, matrices, concentration ranges, etc. The ACL personnel and personnel from the CMT Computer Applications Networking and Security Group (CANS) (R. R. Krol and A. E. Scandora) have visited probable EPA participants, Environmental Monitoring Systems Laboratories in Las Vegas (EMSL-LV) and in Cincinnati (EMSL-Ci), and EML to discuss technical requirements on the use of their PE programs and to learn about their computer facilities. An interagency agreement is being developed between ANL and the EPA-Region V to provide IPEP personnel access to EMSL-Ci PE data for laboratories supporting EM programs. The CANS personnel have analyzed IPEP computer requirements and recommended a system for this program.

The ACL has begun to identify the laboratories that will be affected by this program. In addition, we have analyzed the results of a survey by the FAST of environmental laboratory capacity to determine which laboratories should be participants. We have also established contacts with other groups in the broader program for assistance in this task.

We have identified a statistician to assist us with the development of assessment tools for the PE data and the development of a corrective action mechanism. Work on this task is expected to be initiated in early FY 1992.

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

This intradivisional group was founded in January 1989 to explore possible interactions of CMT with the Advanced Photon Source (APS) for current and future research programs. The group was directed to (1) gather and disseminate information on the application of synchrotron radiation 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 group consists of the following members: M. Blander, C. Bradley, D. Dees, M. Lewis, V. Maroni, Z. Nagy, E. Streets (Chairman), S. Siegel, S.-W. Tam, and B. Tani.

The major activity of the group in FY 1991 was to organize a workshop on Applications of Synchrotron Radiation to Chemical Engineering Science, jointly sponsored by the APS, the Division of Educational Programs, and CMT. The workshop was held on April 22-23, 1991, in CMT. A half-day roundtable discussion was also held on April 24, 1991, to make contact with other potential users and collaborators in developing a beam line for CMT. There were nearly 90 participants from across the nation and Canada. The first day of the workshop also served as a tutorial for ANL resident students. Contacts developed during the course of the workshop are being pursued. E. Streets attended the Users' Meeting at the Advanced Light Source in Berkeley, CA, in August 1991 to explore other options that might be of interest to CMT.

Radiochemical Analysis of the Experimental Boiling Water Reactor (EBWR) Vessel

(D. L. Bowers, R. R. Heinrich, and W. E. Streets)

The 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 (SFMP). Decommissioning and decontamination (D&D) were begun 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.

Since detailed operating records of the EBWR are no longer available, ANL could not calculate the activation or radiological inventory of the reactor components or the reactor vessel. Because this information was needed to perform the D&D, an acceptable alternative method of radiological assessment of the reactor vessel was developed, which involved experimentally characterizing samples extracted from the reactor vessel wall. These samples consisted of cores of carbon steel (A212, Grade B) with a thin layer of 304 stainless steel spot welded to the inner surface of the vessel.

After 24 years of dry lay-up status, short-lived radionuclides could be ignored in the assessment; 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 reactor vessel was assessed using a combination of measured values and theoretical calculations based on the chemical composition of the alloy and the measured radioactivity values. The ACL performed detailed chemical and radiological measurements to determine ^{60}Co, ^{55}Fe, and ^{63}Ni in seven reactor vessel core samples, and performed theoretical calculations to assess radioactivity values for ^{14}C, ^{59}Ni, ^{93}Mo, and ^{94}Nb based on the measured ^{63}Ni values. In all, 70 analytical measurements of the seven core-drilled samples were made for ^{60}Co assessments, along with 12 additional analytical measurements each for ^{55}Fe and ^{63}Ni. These analytical data enabled ANL to obtain the total radiological assessment of the EBWR reactor vessel.

Review of DOE Plant Laboratories (L. L. Smith and R. R. Heinrich)

The ACL staff have performed radiological reviews at DOE Plant Laboratories. The Mound Plant review, consisting of three tasks, was initiated in FY 1990 and was similar to that conducted for the Rocky Flats Plant in FY 1989. Task 1 was a laboratory review consisting of walk-throughs, records review, and employee interviews, which followed EPA CLP-type checklists. Task 2 was an extensive review of tritium analytical data to assess whether or not the data quality meets today's criteria. Task 3 was a correlation study of previous plutonium analytical data with those obtained at the Mound site during the DOE Environmental Survey. The final report is expected in early FY 1992.

FTIR Analysis of Semivolatiles in Soils (J. C. Demirgian, M. Clapper-Gowdy, and C. T. Snyder)

The objective of this program is to develop a method that utilizes FTIR spectroscopy to characterize, in the field, soil samples contaminated with explosives or selected volatile and semivolatile organic species. In the first year of the program, we determined that contaminants in soil samples could be thermally desorbed into a heated long-path infrared cell, and that explosives such as trinitrotoluene (TNT) could be detected at levels corresponding to 40 to 80 ppm in soil. The FTIR method will eliminate the problem that the certified extraction methods have with binding of the TNT to the soil. Field analyses by the FTIR method can be done in minutes.

The past year's effort was directed to field testing the FTIR method on TNT-containing samples obtained from the bioremediation of the Joliet Arsenal and to expanding the analytes to include volatile organic compounds. New equipment was ordered that will be able to contain and desorb 1 g of soil containing explosives. The new equipment is designed specifically for this type of sample. We anticipate analysis of Joliet Arsenal samples to begin in the spring of 1992. The acetone method used by Jenkins ("Development of

Simplified Field Method for the Determination of TNT in Soil," T. F. Jenkins, USATHAMA CRTHA-TS-CR-90125, Special Report 90-38, November 1990) to extract high explosives is also being investigated as a means to lower our current detection levels from 40 to 80 ppm to 4 to 8 ppm. Initial work shows the extraction to work well. However, the degree to which the detection level will be lowered cannot be determined until the arrival of the new equipment.

To apply the method to volatile organic compounds, eight organic compounds were selected for FTIR analysis. This number will be increased to 16 in FY 1992. Early work has shown that analytical conditions for the desorption of volatile compounds are different from those for semivolatile compounds and that the volatile compounds studied in these experiments can be detected at approximately 50 ppm in soils. Quantitative accuracy for measurement of these compounds exceeds that obtained previously for TNT and the dinitrotoluene isomers due to better peak resolution.

This technology is applicable for site characterization and site remediation and could play a significant role in the cleanup of the Joliet Arsenal and other areas contaminated by explosives.

Remote Detection of Agent-Related Chemicals Using FTIR (J. C. Demirgian and C. L. Hammer)

The objective of this project is to determine the feasibility of using passive-remote FTIR spectroscopy to detect chemical agents and their related precursors and degradation products. Prior work demonstrated that agent-related chemicals released at low parts per million levels could be detected when measured against low-sky and brick-wall backgrounds. Two-component mixtures were also analyzed by this method. Our results indicated no problem with false positives or negatives. In the past year, we extended our experimental work to include complex multicomponent mixtures (methanol, isopropanol, dimethylmethyl phosphonate, and diethyl malonate), all with overlapping infrared peak absorbances.

The experimental work was done in the laboratory using a Nicolet 6000 FTIR spectrometer modified to be a passive-remote instrument. The source and inlet optics were replaced with custom-designed optics, which focus with a beam width of 2.5 cm at a distance of 2 m from the target. Chemical releases of the multicomponent mixtures were performed using a special apparatus designed for this project. The FTIR data were analyzed using classical least squares (CLS) and partial least squares (PLS) algorithms. We found that quantitative analysis employing a well-characterized standard set was normally within 20% of the released concentration using PLS. The CLS algorithm outperformed the PLS one when a partial set of standards was used.

We concluded that FTIR spectroscopy shows much promise for remote detection of organics. This method has potential use for other environmental applications, such as compliance monitoring of air toxics released in the atmosphere.

Monitoring of Incinerator Effluent for Organic Analytes (J. C. Demirgian)

The objective is to monitor incinerator effluent from different feeds and to correlate the feed content with stack effluent by qualitatively and quantitatively analyzing the effluent. This work is a collaborative study with Energy Systems Division (Z. Mao and M. McIntosh). In FY 1990, an experimental incinerator was constructed, and an FTIR spectrometer was coupled to it. In addition, a library of the FTIR spectra of interest was collected.

In FY 1991, the analytical instrument was used to identify the effluent from methane, toluene, and chlorobenzene burns. To test the analytical equipment, the incinerator was deliberately run under non-optimum temperature and gas-flow conditions. The upset was easily detectable using the FTIR method. The primary products of incomplete combustion were methane and benzene, regardless of the feed.

The objective of future work is to use the analytical instrument at a DOE incinerator and ultimately develop an EPA-certified method for using FTIR spectroscopy as a continuous emission monitor.

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 of drug-related solvents at field sites so that analytical equipment can be tested. We are responsible for releasing known quantities of chemicals emitted by typical illegal drug laboratories and evaluating test results obtained at the release site by all participating organizations. Equipment evaluated to date includes spectrometers based on ion mobility, FTIR, forward-looking infrared (FLIR), and fluorescence LIDARs (LIght Detection and Ranging).

Computer Generation of Reporting Forms for Volatile Organic and PCB/Pesticide Environmental Analyses (G. A. Baudino, Y. Tsai, G. K. Gosztola, and L. L. Lamoureux)

Numerous samples received by the ACL require reporting results on forms specified by the EPA Contract Laboratory Program. In the past, calculations were done by the analyst, and several forms were filled out manually. We have developed the capability to electronically generate these forms for volatile organic compound determinations performed on a Hewlett-Packard gas chromatograph/mass selective detector (GC/MSD) and for PCB/pesticide determinations performed on a Hewlett-Packard GC. Work on this project was done in collaboration with R. Krol (Electronics Department). By automating, a significant time savings has been realized. A larger portion of the analyst's time can now be spent performing other tasks.

Quality Assurance for Headspace Gas Analysis in the Waste Isolation Pilot Plant (WIPP) Experimental Waste Characterization Program (P. C. Lindahl and D. A. Bass)

The objective of the Experimental Waste Characterization, which is a key component of DOE's WIPP program, is to characterize volatile organic gases and permanent and low-molecular weight hydrocarbon gases present in drums containing mixed (radioactive and hazardous) waste. These drums are to be placed in bins, which will go into DOE's long-term mixed waste storage facility (WIPP) in Carlsbad, New Mexico. The ACL participated in a multiorganizational effort (headed by EG&G Idaho) for writing DOE's Quality Assurance Program Plan (QAPP) and Guidance Manual for the WIPP Experimental Waste Characterization Program. The QAPP and WIPP Sampling and Analysis Guidance Manual were prepared to meet requirements of both EPA and DOE and were designed to assist laboratories in producing reliable and well-documented data. The QAPP establishes the sampling and analysis requirements, and the Guidance Manual is a compilation of recommended methods that ensure compliance with the QAPP. Both documents are designed to ensure that the Programs quality assurance objectives are met.

The ACL had lead responsibility for establishing analytical laboratory requirements for the analysis of headspace samples to determine permanent and low-molecular-weight hydrocarbon gases in waste bins and drums that are to be sent to the WIPP site. The recommended methods are mass spectrometry for all the analytes or gas chromatography for all the analytes except combined nitrogen oxides, for which spectrophotometry is recommended. Quality control checks are used both in the field and in the laboratory to assure acceptable performance. Field quality control samples include field reference standards, field duplicates, field blanks, and manifold blanks. Laboratory quality control samples consist of calibration verifications, laboratory control samples, laboratory blanks, and laboratory duplicates. Complete documentation is required, and each laboratory performing headspace gas analysis must participate in the WIPP-QAPP Performance Demonstration Program.

Analysis of WIPP Samples by the Solid Adsorbent Method (Y. Tsai, G. T. Reedy, G. A. Baudino, R. M. Cinco, and A. S. Boparai)

To determine the 29 target volatile organic compounds (VOCs) specified in the WIPP-Quality Assurance Program Plan, we have established an analytical system that consists of a Tekmar solid adsorbent inlet and the Hewlett-Packard GC/MSD. A constant sample flow, regulated by a mass flow controller, passes through the solid adsorbent trap at ambient temperature. The preconcentrated sample is then desorbed into the GC for separation and is detected by the MSD. This system can identify and quantitate all the target compounds, including alcohols and ketones, in a single run. Using this method, a sample can be analyzed in approximately 30 min. Since the installation of the new GC/MSD was completed, we have performed the analyses required for method validation with accuracy and precision required by WIPP; certified cleaned canisters as acceptable for use in the characterization program (all impurities below required limits); and analyzed WIPP VOC Performance Demonstration (PD) samples and obtained results on the PDs that qualify the ACL to do sample analyses in the characterization program. In addition, 35 sample canisters from the WIPP site laboratory in Carlsbad, NM, were analyzed.

We are working to extend the existing calibration range to eliminate the need for serial dilutions of field samples before analysis.

Canister Cleaning and Certification for the WIPP (G. T. Reedy, Y. Tsai, D. V. Applegate, R. M. Cinco, P. M. Aznavoorian, and A. S. Boparai)

An important concern in the analytical scheme for measuring the composition of headspace gases over radioactive wastes for the WIPP project is the maintenance of sample integrity. To avoid changes in the composition of gases from the time samples are collected until they are analyzed in the laboratory, the samples are contained in specially treated stainless steel canisters with electropolished interiors. These canisters, sold under the trade name "SUMMA," are used by all of the laboratories participating in the WIPP Project. To obtain uniformity in the condition of canisters used for sample collection, a decision was made to establish a Central Canister Cleaning Facility at ANL for cleaning and certifying the canisters between uses.

In this facility, up to 16 canisters can be heated simultaneously while they are flushed with humid air. They can then be baked out overnight while being pumped with an oil-free molecular drag pump to a pressure of ~1.3 mPa (~1 x 10^{-5} torr). The canisters are then leak tested on the same vacuum manifold. One canister from each batch is filled with clean humid air, and the contents are analyzed with a GC/MS. This analysis allows the batch to be certified based on the results of the test canister.

To date, ~250 canisters have been cleaned and certified for return to outside laboratories. An additional 250 have been cleaned for our own laboratory use.

Quantitation of Volatile Organic Compounds in Headspace Gas for the WIPP Project (D. V. Applegate and G. T. Reedy)

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 procedures given in the WIPP Sampling and Analysis Guidance Manual, and provide quantitative data on 24 target compounds, excluding alcohols and ketones, that are of particular interest to the project. Target compounds include aromatic hydrocarbons, chlorinated aliphatic hydrocarbons, and diethyl ether.

Gas samples are drawn from the waste drums into evacuated stainless steel 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 300 mL is then drawn from the laboratory canister and passed through a trap cooled to -100°C, which retains the target compounds. The target compounds are released from the trap and swept into a GC/MS 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 to 1000 ppmv in the headspace gas.

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

In addition to the analyses performed for other volatile organic compounds (see previous sections), 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. Problems with the mass spectrometer (new to ACL this fiscal year) impeded development of detailed procedures, but these problems were eventually resolved. Instrument stability and sensitivity have been demonstrated to meet program requirements regarding precision and detection limits for measuring the gases of interest. Results of a recent (October 1991) performance demonstration exercise have indicated that inaccuracies occur for a few of the target gases in certain mixtures. The causes of these inaccuracies and means for eliminating them are being investigated.

Supercritical Fluid Extraction/Gas Chromatography for Analysis of Organic Compounds (A. S. Boparai, R. L. Petty, A. M. Caison, and D. R. Bierma)

Supercritical fluid extraction (SFE), which employs a supercritical fluid such as carbon dioxide as the extractant, has been used for extraction for organic compounds from soil samples. As compared to the other methods of extraction of soil samples (Soxhlet and sonication), SFE can be performed rapidly and does not produce liquid waste.

To perform efficient analysis of organic compounds extracted from soil samples, we have interfaced an SFE apparatus to a GC. Supercritical carbon dioxide was used for most of the extractions; however, modifiers such as methanol when added to the supercritical carbon dioxide extractant improve the efficiency of the SFE process.

Evaluation has begun on the ability of an SFE/GC instrument to separate a mixture containing several high-molecular-weight aromatic hydrocarbons. Data on optimum extraction time and temperature and effectiveness of modifiers were obtained. In addition, preliminary data have been obtained on the performance of a flame ionization detector coupled to a SFE/GC.

Environmental Protection Agency Project (A. S. Boparai and G. A. Baudino)

The EPA supports a containment room at ANL for the handling and analysis of hazardous waste samples. The room is a unique facility that permits handling of potentially toxic samples in a safe manner and provides a means for testing and isolating any resulting waste. Samples are handled from receipt through analysis and disposal within a large glovebox. The final draft of protocols for operation and safety of this containment facility was prepared and is under review. The EPA also continues to support a storage facility on site for legally sensitive samples.

Analysis of Environmental Samples for the U.S. Department of Agriculture (G. A. Baudino and A. S. Boparai)

Contamination of both soil and groundwater with chloroform and carbon tetrachloride at a former grain storage facility (in Waverly, NE) operated by the U.S. Department of Agriculture (USDA) has led to a collaborative effort between the Environmental Research (ER) Division (M. C. Hansen) and the ACL. Chloroform and carbon tetrachloride, classified as possible carcinogens, were commonly used as grain fumigants in the 1950s and 1960s by the USDA and companies that operated grain storage facilities in Nebraska. A groundwater extraction and air-stripping system, operated at the Waverly site by a team from the ER Division, is used to remove contaminants from the aquifer and contain the spread of contaminated groundwater that once supplied drinking water to Waverly residents. For the past three years, soil gas and groundwater samples have been taken monthly and sent to the volatile organics laboratory of the ACL for analysis to monitor the progress of the site cleanup. Soil gas samples, collected on charcoal tubes and carbotraps, and groundwater samples are analyzed according to protocols from the U.S. EPA and Standard Operating Procedures written by the ACL. Tests show that the level of carbon tetrachloride in the aquifer has been reduced from 4000 parts per billion (ppb) to 100 ppb. Although these results indicate some success, more work needs to be done to get the contaminant level for carbon tetrachloride below the Federal health standard of 5 ppb.

Environmental Assessment Study for Contaminants at Hohenfel Training Area Site, Germany (C. T. Snyder, P. M. Aznavoorian, C. Marquez, D. R. Bierma, C. L. Hammer, A. S. Boparai, L. L. Lamoureux, and R. L. Petty)

An exploratory study was conducted at the Hohenfel Training Area site in Germany to assess environmental effects of the troop training exercises by U.S. Armed Forces. Plants, leaves, grass, and soil samples were collected from areas within the site and submitted for determination of two components that have the potential to be contaminants in the training field. The major component of interest was fog oil, a petroleum product used to create an artificial battlefield fog. The second component of interest was 2-chlorobenzaldehyde, a compound found in tear gas used in training exercises. Thirty-three soil samples and 27 plant samples were extracted with organic solvent by sonication. The concentrated extract was analyzed by GC with flame ionization detection. Selected samples were also analyzed by GC/MS. Because there is no certified method for the determination of these compounds, procedures were developed following the protocol of U.S. EPA certified methods. Because very complex chromatograms were obtained from both soil and plant samples, ratio and subtraction techniques were applied to the data. No fog oil or 2-chlorobenzaldehyde was detected in the soil or plant samples.

FTIR Microscopy (C. T. Snyder)

Fourier transform infrared microscopy has contributed to the organic analytical support offered by the ACL. Recent samples analyzed include unknown residues on gold wire, stainless steel, and ceramic surfaces. Individual crystals and inclusions in crystals have been studied. Powders and liquids were analyzed on salt disks after appropriate sample preparation. Selected solid samples have been analyzed by dissolving the solid and analyzing a thin film of residue prepared from the solution. Polymers and other samples containing organic components have been successfully analyzed and functional groups identified.

IV. QUALITY ASSURANCE

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

The ACL continued its participation in four multilaboratory programs sponsored by the U.S. EPA. Each program provided the ACL with sets of either Quarterly or Semiannual Blind Performance Evaluation (PE) Samples for analysis. The compounds present in the blind samples and their concentrations were known only to the EPA. The PE programs in which the ACL participated are summarized below.

The ACL analyzed Quarterly Blind PE samples from the U.S. EPA Environmental Monitoring Systems Laboratory-Las Vegas (EMSL-LV): three inorganic samples (QB1FY91, QB3FY91, and QB4FY91) and one organic (QB2FY91). Results on all four sets of samples met EPA criteria for participation in the Contract Laboratory Program (CLP).

The ACL continued participation in the EMSL-CI Water Pollution Study (WPS). Three inorganic semiannual blind PE samples (WP025, WP026, and WP027) were analyzed for cyanide, oil/grease, total dissolved and suspended solids, and a variety of anions. The EMSL-Ci reported that the ACL's analytical results for samples WP025 and WP026 were in excellent agreement with EPA's known values. Sample WP027 was submitted for scoring just prior to this report, and no score is available at this time.

The DOE Environmental Measurements Laboratory (EML) Quality Assurance Program and EMSL-LV coordinated multilaboratory Cross-Check PE Program both provide blind semiannual radiological PE samples to the ACL. Water, soil, and air filter samples were received and analyzed for radionuclide determinations (e.g., gross alpha-beta, gamma, tritium, Ra, U, Pu, and Np) throughout FY 1991. The ACL did well in these programs, with each of the radionuclides being correctly determined.

Over 800 environmental samples, made up of a number of matrices including water, soil, sediment, sludge, air, filter paper, wood, and vegetation, were submitted to the ACL for analysis during FY 1991. Most analyses were performed according to inorganic or organic methodologies described in the Statements of Work written for the U.S. EPA CLP, or the Resource Conservation and Recovery Act (SW-846). Approximately 71 CLP-type sample data packages, consisting of analytical results and raw data generated for these samples, were prepared and reviewed for completeness and correctness, and forwarded to customer data management teams.

The total number of ACL standard operating procedures written to address analytical and administrative protocol increased from 146 to 161 in FY 1991. All are in place and available to ACL personnel in bound, two-volume manuals strategically located throughout the ACL.

An inventory of ACL laboratory chemicals was completed. Each laboratory chemical was tabulated and matched against Material Safety Data Sheets (MSDS) on hand. The MSDSs were then obtained for those chemicals having none in file through the vendor or through the Sigma-Aldrich MSDS Computer Database. A system for maintaining the MSDSs within the ACL was developed, such that each ACL laboratory now has its own manual in which are contained MSDSs for the chemicals found in that laboratory, as well as a library index of MSDSs on file with the ANL Industrial Hygiene Section of ESH. One occupant of each ACL laboratory has been designated to monitor and coordinate the updating of the MSDS binder in their laboratory area.

Because of the large number of samples received by the ACL, many requiring multiple analyses, a great deal of time is spent on issuing sample notification documents, tracking samples and monitoring analytical holding times, and managing documents in general. Support Development Funds were used for the development of a computer-assisted bar code reading system designed to provide quick and error-free logging, distribution, and tracking of analytical samples and associated documentation. The system's capabilities include providing sample notification memos, chain-of-custody forms, and summaries of samples categorized by analyses requested. The system is currently being debugged and tested.

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 for approval. The ACL WIPP QAPjP (Rev. 01) was submitted, and technical and editorial comments have been received. Revisions to the draft copy are near completion. Nine additional standard operating procedures, written by ACL personnel and approved specifically for use in the WIPP Program, are currently in use in the analysis of WIPP headspace organic sample canisters.

As a part of a June audit of the CMT conducted by the ANL-E Office of Quality Assurance, the ACL was one of the organizations within CMT to be audited. Implementation of the ACL quality assurance program and the Yucca Mountain Project requirements were reviewed. During the ACL on-site review, which consisted of interviews, record reviews, and laboratory visits, no findings were identified.

V. PROFESSIONAL ACTIVITIES

A. Publications and Reports

A Simultaneous Comparison of Four Se Hollow Cathode Lamps Used in Graphite Furnace Atomic Absorption Spectroscopy
G. D. Rayson and D. A. Bass
Applied Spectroscopy, 45(6), 1049-1050 (1991)

Alpha Particle-Induced Formation of Nitrate in the Cm-Sulfate Aqueous System
D. T. Reed and D. L. Bowers
Radiochimica Acta 51, 119-125 (1990)

Analytical Chemistry Laboratory Progress Report for FY 1990
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-90/2 (1990)

Determination of Cadmium in Blood, Plasma, and Urine by Electrothermal Atomic Absorption Spectrophotometry after Isolation by Anion-Exchange Chromatography
D. P. Peterson, E. A. Huff, and M. H. Bhattacharyya
Analytical Biochemistry 192, 434-440 (1991)

Determination of On-Stream Destruction Removal Efficiency Using Fourier Transform Infrared Spectroscopy
J. C. Demirgian, Z. Mao, M. McIntosh, and C. A. Wentz
Proceedings of the 1991 Incineration Conference on Thermal Treatment of Radioactive, Hazardous Chemical, Mixed and Medical Wastes, 563-570, Knoxville, TN, May 13-17 (1991)

Evaluation of Gas Chromatography/Matrix Isolation-Infrared Spectroscopy for the Quantitative Analysis of Environmental Samples
J. F. Schneider, K. R. Schneider, S. E. Spiro, D. R. Bierma, and L. F. Sytsma
Applied Spectroscopy 45(4), 566-571 (1991)

Leaching Action of EJ-13 Water on Unirradiated UO_2 Surfaces Under Unsaturated Conditions at 90°C: Interim Report
D. J. Wronkiewicz, J. K. Bates, T. J. Gerding, E. Veleckis, and B. S. Tani
Argonne National Laboratory Report No. ANL-91/11 (1991)

Mechanisms of Selenium Vaporization with Palladium Modifiers Using Electrothermal Atomization and Mass Spectrometric Detection
D. L. Styris, L. J. Prell, D. A. Redfield, J. A. Holcombe, D. A. Bass, and V. Majidi
Analytical Chemistry 63, 508-517 (1991)

Remote Detection of Chemical Agents by Infrared Spectroscopy, Progress Report for FY 1989
J. C. Demirgian and S. M. Spurgash
Argonne National Laboratory Report No. ANL/ACL-90/1 (1990)

Speciation of Pu(VI) in Near-Neutral Solutions via Laser Photoacoustic Spectroscopy
S. Okajima, D. T. Reed, J. V. Beitz, C. S. Sabau, and D. L. Bowers
Radiochimica Acta, 52/53, 111-117 (1991)

B. Oral Presentations

Analysis of Canister Air Samples Involving Cryogenic Trapping Prior to GC/MS
D. V. Applegate
Thirteenth ACL Technical Meeting, Argonne National Laboratory, December 6, 1990

Analysis of Complex Mixtures by Gas Chromatography/Matrix Isolation Infrared Spectroscopy
A. S. Boparai and J. F. Schneider
Eighth Triangle Chromatography Symposium and Instrument Exhibit, Raleigh, NC, May 16, 1991

Analysis of Potential Contamination by Fog Oil in Soils and Plants
C. T. Snyder
Fourteenth ACL Technical Meeting, Argonne National Laboratory, April 9, 1991

Analytical Laboratory Management - Interlaboratory Performance Evaluation Program (IPEP)
D. W. Green
Ninth DOE Analytical Managers Meeting, Kansas City, MO, September 10-12, 1991

Computerized Calculation of Environmental Levels of Radioactivity Determined by Gamma Spectrometry
W. E. Streets, B. S. Tani, and L. R. Greenwood
36th Annual Conference on Bioassay, Analytical, and Environmental Radiochemistry, Oak Ridge, TN, October 22-26, 1990

Computerized Calculation of Environmental Levels of Radioactivity Determined by Gamma Spectrometry
W. E. Streets
Thirteenth ACL Technical Meeting, Argonne National Laboratory, December 6, 1990

Determination of On-Stream Destruction Removal Efficiency Using Fourier Transform Infrared Spectroscopy
J. C. Demirgian, Z. Mao, M. McIntosh, and C. A. Wentz
1991 Incineration Conference, Knoxville, TN, May 13-17, 1991

Evaluation of Supercritical Fluid Extraction/Gas Chromatography/Matrix Isolation-Infrared Spectrometry for Analysis of Organic Compounds
A. S. Boparai, D. R. Bierma, and D. V. Applegate
42nd Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 4-8, 1991

In Search of a Universal Matrix Modifier
L. B. TenKate
Fourteenth ACL Technical Meeting, Argonne National Laboratory, April 9, 1991

Matrix Isolation as an Interface for GC/IR: How Does it Fit in with the Other GC/IR Interfaces?
J. F. Schneider
17th Annual Meeting of the Federation of Analytical Chemistry and Spectroscopy Societies, Cleveland, OH, October 7-12, 1990

Monitoring Incinerator Emissions from Remote Sites Using Fourier Transform Infrared Spectroscopy (FTIR)
J. C. Demirgian, S. M. Spurgash, and C. T. Snyder
2nd Annual Pacific Northwest International Section/Air and Waste Management Association Conference, Portland, OR, November 14-16, 1990

New Method for Isolating Strontium for Isotopic Analysis
A. M. Essling
Thirteenth ACL Technical Meeting, Argonne National Laboratory, December 6, 1990

Rapid Field Characterization of Soil Samples for Hazardous Components by Fourier Transform Infrared Spectroscopy
J. C. Demirgian and M. Clapper
42nd Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 4-8, 1991

Remote Detection of Organics Using Fourier Transform Infrared Spectroscopy
J. C. Demirgian and S. M. Spurgash
Second International Symposium on Field Screening Methods for Hazardous Wastes and Toxic Chemicals, Las Vegas, NV, February 12-14, 1991

TRUEX Processing of Plutonium Analytical Waste Solutions at Argonne National Laboratory
J. C. Hutter, D. B. Chamberlain, E. H. Van Deventer, G. F. Vandegrift, D. R. Fredrickson, R. A. Leonard, J. A. How, M. A. Tranovich, and D. L. Bowers
Summer AIChE National Meeting, Pittsburgh, PA, August 18-21, 1991

C. Awards

Donald G. Graczyk
ANL Pacesetter Award for his leadership as chairman of Argonne's Water Quality Management Task Force.

Vanessa A. Mendez
Employer of the Year, Bolingbrook High School Cooperative Education Program

D. Meetings Attended

Peter M. Aznavoorian
42nd Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 4-8, 1991

Dean A. Bass
ACS Northwest Regional Meeting, Symposium on Chemistry of Mixed Wastes, LaGrande, OR, June 10-12, 1991

EPA, DOE Workshop, Characterizing Heterogeneous Hazardous Wastes, Las Vegas, NV, March 26-28, 1991

42nd Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 4-8, 1991

Gerald A. Baudino
42nd Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 4-8, 1991

Amrit S. Boparai
42nd Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 4-8, 1991

Delbert L. Bowers
42nd Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 4-8, 1991

Plasma Spectroscopy Task Group Meeting, American Society for Testing and Materials, San Diego, CA, January 27-31, 1991

Plasma Spectroscopy Task Group Meeting, American Society for Testing and Materials, Colorado Springs, CO, July 29-August 1, 1991

Jack C. Demirgian
DOE Systems and Technology Optics Workshop, Washington, DC, July 15-17, 1991

Alice M. Essling
42nd Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 4-8, 1991

Irene M. Fox
42nd Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 4-8, 1991

Donald G. Graczyk
42nd Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 4-8, 1991

David W. Green
42nd Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 4-8, 1991

Geralyn K. Gosztola
42nd Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 4-8, 1991

Robert R. Heinrich
Applied Research and Technology Colloquium, Phoenix, AZ, April 2-5, 1991

42nd Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 4-8, 1991

Edmund A. Huff
42nd Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 4-8, 1991

Plasma Spectroscopy Task Group Meeting, American Society for Testing and Materials, San Diego, CA, January 27-31, 1991

Laura L. Lamoureux

42nd Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 4-8, 1991

Peter C. Lindahl

Fifth Annual Interagency Meeting on Quality Assurance for Environmental Measurements, Richmond, VA, September 25-27, 1991

DOE Environmental Management D&D Workshop, Knoxville, TN, August 12-16, 1991

Francis Markun

42nd Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 4-8, 1991

Jane J. Marr

42nd Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 4-8, 1991

Fredric J. Martino

Waste Testing and Quality Assurance Symposium, Washington, DC, June 16-20, 1990

The Second International Waste Management Conference, American Society for Quality Control, Las Vegas, NV, March 17-20, 1991

Steven H. Newnam

42nd Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 4-8, 1991

Gerald T. Reedy

42nd Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 4-8, 1991

Carmen S. Sabau

42nd Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 4-8, 1991

Florence P. Smith

18th Annual Conference of the National Organization for the Professional Advancement of Black Chemists and Chemical Engineers, Washington, DC, April 1-5, 1991

Lesa L. Smith

42nd Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 4-8, 1991

Christine T. Snyder

42nd Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 4-8, 1991

W. Elane Streets

42nd Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 4-8, 1991

Interagency Workshop on Characterizing Heterogeneous Hazardous Waste, Las Vegas, NV, March 26-28, 1991

CMT/APS Workshop, Argonne, IL, April 22-23, 1991

Fourth APS Users Meeting, Argonne, IL, May 7-8, 1991

Advanced Light Source Users' Meeting, Berkeley, CA, August 15-16, 1991

Fifth Annual Interagency Meeting on Quality Assurance for Environmental Measurements, Richmond, VA, September 25-27, 1991

Benjamin S. Tani

42nd Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 4-8, 1991

CMT/APS Workshop, Argonne, IL, April 22-23, 1991

Lynn B. TenKate

42nd Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 4-8, 1991

Tony TenKate

42nd Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 4-8, 1991

Yifen Tsai

42nd Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Chicago, IL, March 4-8, 1991

E. Professional Organization Affiliations

Dean A. Bass

American Chemical Society

Sigma Xi

Amrit S. Boparai

American Chemical Society

Delbert L. Bowers

American Chemical Society

American Society for Testing and Materials

Maureen Clapper-Gowdy

American Chemical Society

Jack C. Demirgian

American Chemical Society

Technical Association of the Pulp and Paper Industry

Air and Waste Management Association

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 of the American Chemical Society

Analytical Laboratory Managers Association, Treasurer

Sigma Xi

Edmund A. Huff

American Chemical Society

American Society for Testing and Materials

Society for Applied Spectroscopy

Paul L. Johnson

Association for Computing Machinery

American Crystallographic Association

Sigma Xi

Peter C. Lindahl

American Chemical Society

Division of Fuel Chemistry of the American Chemical Society

Division of Analytical Chemistry of the American Chemical Society

Society for Applied Spectroscopy

Francis Markun

Health Physics Society

Midwest Chapter of Health Physics Society

Carmen S. Sabau

American Chemical Society

American Nuclear Society

Sigma Xi

American Women in Science

American-Romanian Academy for Science and Arts

New York Academy of Sciences

Florence P. Smith

National Organization of Black Chemists and Chemical Engineers (NOBCChE), Chairperson, Science Bowl Science Fair

National Organization of Black Chemists and Chemical Engineers (NOBCChE), Executive Board Member

Lynn B. TenKate

Society for Applied Spectroscopy

Sigma Xi

F. Professional Training

Dean A. Bass

Environmental Law, Argonne, IL, November 8, 1990

Environmental Quality Assurance, Richland, WA, February 25, 1991

Delbert L. Bowers

Environmental Law, Argonne, IL, November 8, 1990

Alice M. Essling

Microwave Users Meeting, Hinsdale, IL, August 30, 1991

Irene M. Fox

Environmental Law, Argonne, IL, November 8, 1990

Perkin-Elmer Workshop, Development and Advances in Atomic Absorption and Electrothermal Atomization Atomic Absorption, Lisle, IL, March 18, 1991

Perkin-Elmer Workshop, Discussion of Software and Introduction to Ver. 6.0, Oak Brook, IL, July 24, 1991

David W. Green

Environmental Law, Argonne, IL, November 8, 1990

Robert R. Heinrich

Environmental Law, Argonne, IL, November 8, 1990

Paul L. Johnson

Introduction to UNIX, College of DuPage, Glen Ellyn, IL, January-March 1991

Laura Lamoureux

An Introductory Course in Gas Chromatography, University of Illinois College of Pharmacy, Chicago, IL, March 19-22, 1991

Mattson Instruments, Getting More From Your FTIR Spectrometer, Oak Brook, IL, August 8, 1991

Carmen S. Sabau

Environmental Law, Argonne, IL, November 8, 1990

Christine T. Snyder

Nicolet Instrument Corporation 700 Series FTIR Operations Course, October 29-November 2, 1990

W. Elane Streets

Environmental Law, Argonne, IL, November 8, 1990

Benjamin S. Tani

Environmental Law, Argonne, IL, November 8, 1990

Lynn B. TenKate

American Chemical Society Short Course on Environmental Laboratory QA/QC Data Validation, Cleveland, OH, October 11-13, 1990

Environmental Law, Argonne, IL, November 8, 1990

Perkin-Elmer Workshop, Development and Advances in Atomic Absorption and Electrothermal Atomization Atomic Absorption, Lisle, IL, March 18, 1991

Tony TenKate

Environmental Law, Argonne, IL, November 8, 1990

F. ACL Seminars

Alpha Spectrometric Determination of Plutonium Isotopic Composition and Concentration--Application to Nuclear Safeguards
Dr. Robert P. Larsen, Argonne National Laboratory
October 18, 1990

High-Throughput Liquid Absorption Air and Aerosol Sampler
Dr. Sol Zaromb, Argonne National Laboratory
April 16, 1991

Analytical Director: A Computer Based Artificial Intelligence/Robotic System for the Analytical Laboratory
Dr. Thomas L. Isenhour, Department of Chemistry, Kansas State University
July 15, 1991

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C.-Y. Yuen
S. K. Zussman
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ANL Contract File
TIS Files (3)

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