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Argonne National Laboratory
9700 South Cass Avenue
Argonne, Illinois 60439

ANALYTICAL CHEMISTRY LABORATORY
Progress Report for FY 1989

Prepared by:

D. W. Green, Manager
R. R. Heinrich, Group Leader
D. G. Graczyk, Group Leader
P. C. Lindahl, Group Leader
M. D. Erickson, Group Leader
with contributions from ACL Staff

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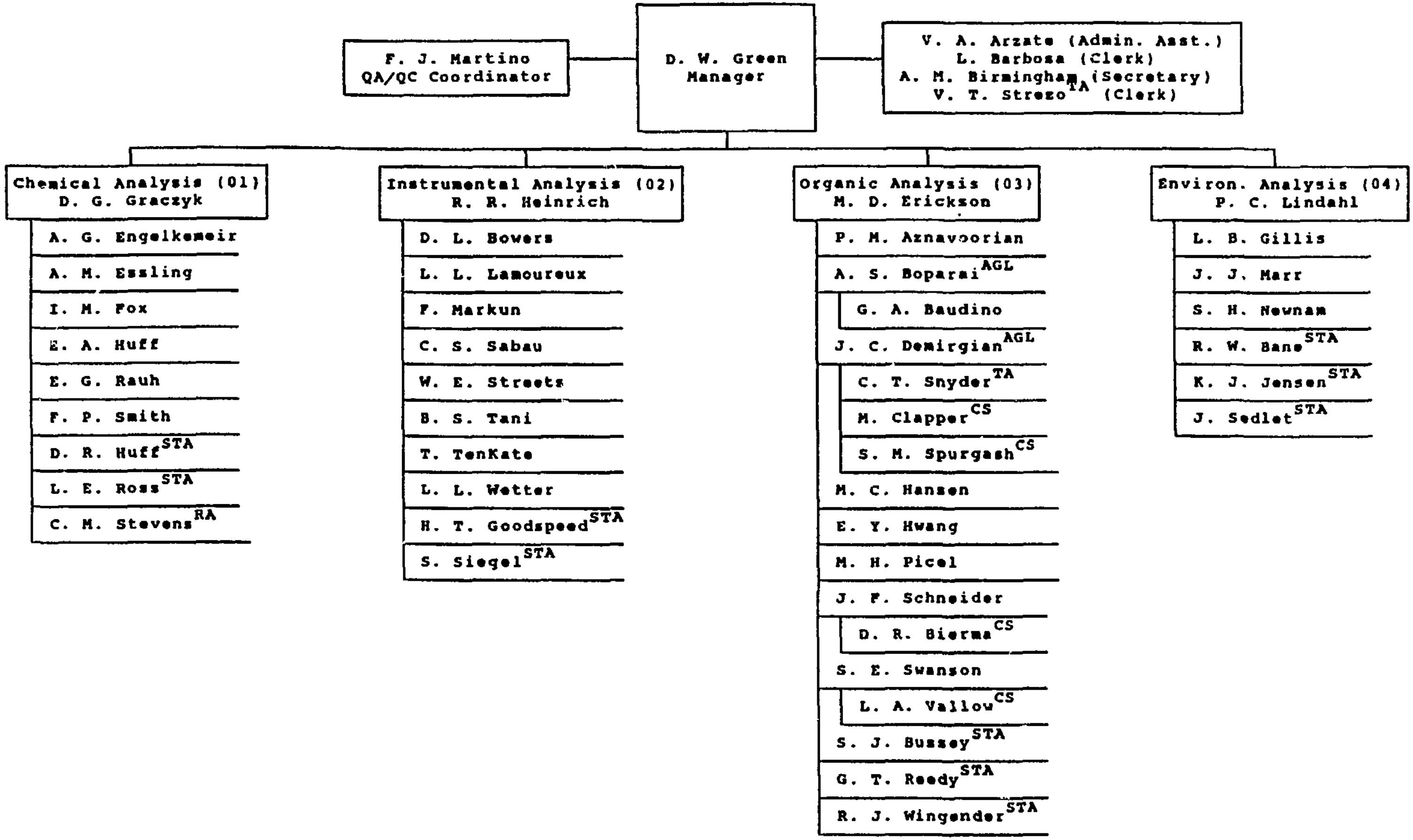
I. INTRODUCTION

The purpose of this report is to summarize the activities of the Analytical Chemistry Laboratory (ACL) at Argonne National Laboratory (ANL) for Fiscal Year 1989 (October 1988 through September 1989). This is the sixth annual report for the ACL [the reports for FY 1984 through FY 1988 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), and ANL/ACL-88-1 (December 1988)].

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), the principal user, but provides technical support for all 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, an inductively coupled plasma/atomic emission spectrometer (ICP/AES), spectrophotometers, mass spectrometers (including two thermal-ionization mass spectrometers), emission spectrographs, 9/30/89



TA - Term Appointee; AGL - Associate Group Leader; CS - Co-op Student; STA - Special Term Appointee; RA - Resident Associate.

Fig. 1. Analytical Chemistry Laboratory Organization Chart

autotitrators, sulfur and carbon determinators, and a fluorimetric uranium analyzer.

The Instrumental Analysis Group uses nuclear counting techniques in radiochemical analyses over a wide range of sample types from low-level environmental samples to samples of high radioactivity. Other types of analyses use gas chromatography, 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.

The Organic Analysis Group uses a number of complementary techniques to separate and to quantitatively and qualitatively analyze complex organic mixtures and compounds at the trace level, including synthetic fuels, toxic substances, fossil-fuel residues and emissions, pollutants, biologically active compounds, pesticides, and drugs. Major instrumentation in this group includes: gas chromatograph/mass spectrometers (GC/MS), Fourier transform infrared (FTIR) spectrometers, a GC/matrix-isolation FTIR (GC/MI-IR) instrument, a GC/matrix-isolation/FTIR/MS system, GCs, high-performance liquid chromatographs, and a supercritical fluid chromatograph.

The Environmental Analysis Group performs analyses of inorganic environmental and 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 analyzer; and a sulfur determinator.

II. ADMINISTRATIVE HIGHLIGHTS

The major users of ACL services among ANL organizations for FY 1989 are listed in Table 1.

Table 1. Distribution of ACL Work Load for FY 1989 among the Major Divisional Users

ANL Organization/Source	% ACL Effort
Chemical Technology (CMT)	53.5
Energy and Environmental Systems (EES)	16.2
ACKs (outside procurements)	6.0
ANL Work Projects	5.1
Materials and Components Technology (MCT)	3.5
Chemistry (CHM)	2.9
Environment, Safety, and Health (ESH)	1.3

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; thus, in Table 1, the work by the ACL on CMT programs includes ACL research. Compared with last year, both the funding from CMT and the total funding for the ACL have decreased, as shown in Fig. 2, but remain higher than years before FY 1988. In addition to the work identified in Table 1, the ACL continued to provide analytical support for programs in other ANL divisions, as well as over $200,000 of technical services for non-ANL clients.

The ACL recovered 94.6% of its total operating costs in FY 1989. Over the last four years, the ACL has recovered 98.2% 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 1989, which are largely effort related.

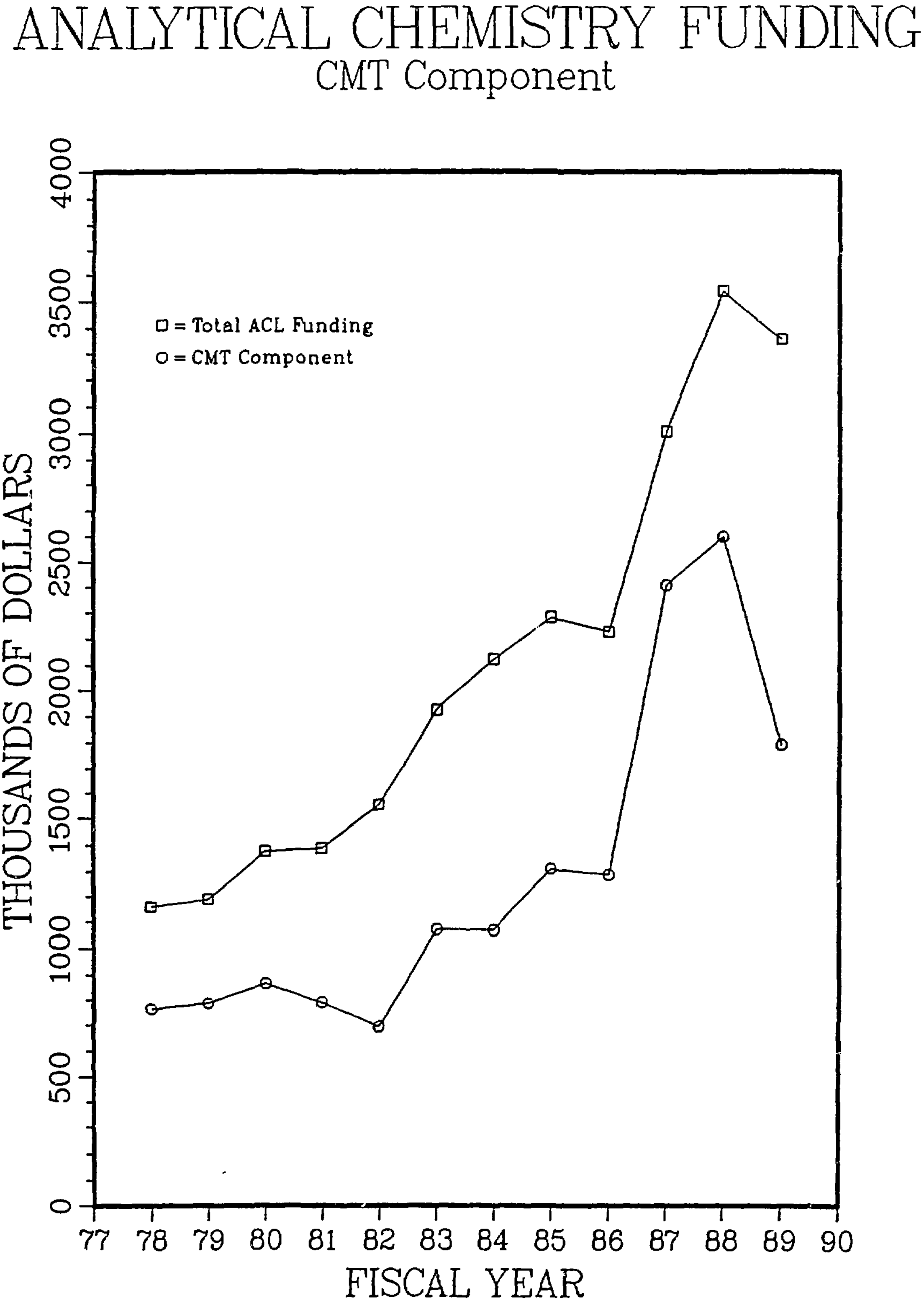


Fig. 2. ACL Funding History, Total and CMT

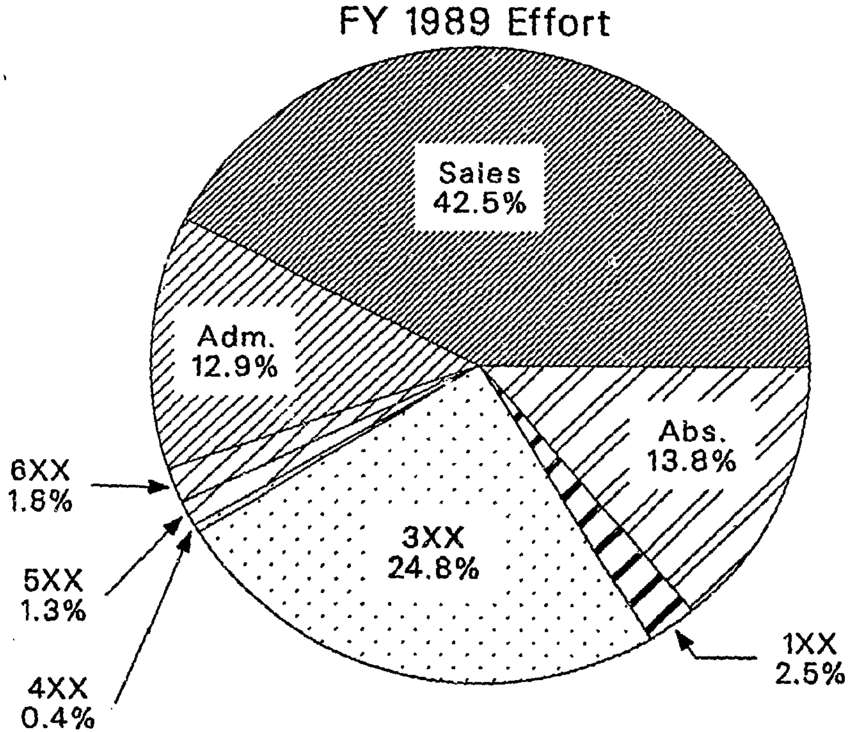


Fig. 3. Analytical Chemistry Laboratory
FY 1989 Effort

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

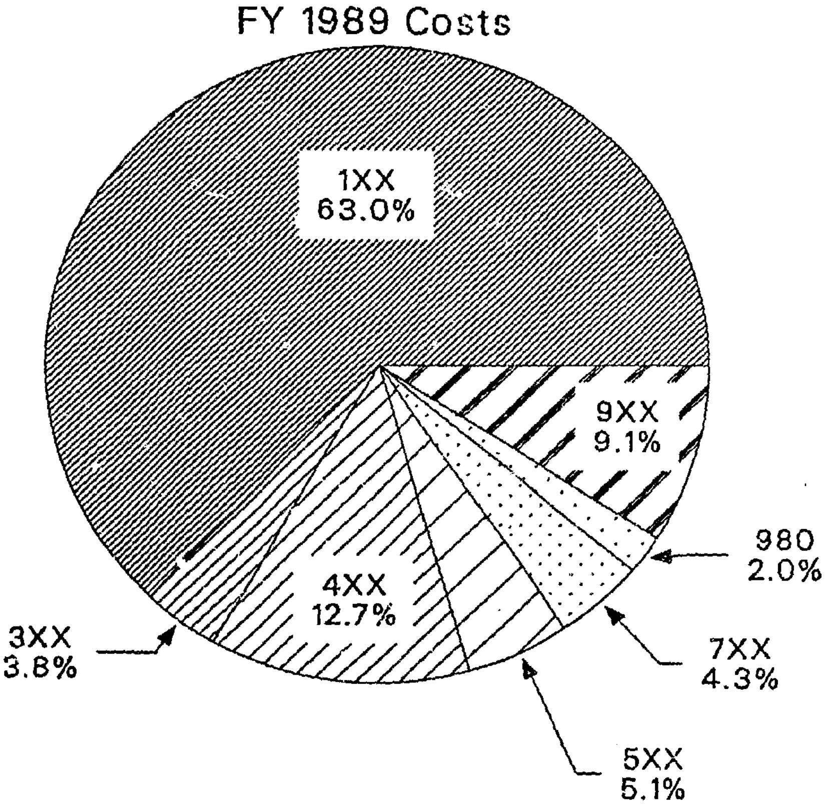


Fig. 4. Analytical Chemistry Laboratory
FY 1989 Total Operating Costs

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

The ACL continued to obtain valuable help from ten Special Term Appointees (STAs) (see Fig. 1) in FY 1989 to assist with load leveling. About 1.5 full-time equivalents were used, and continued help from STAs is anticipated in FY 1990. Ten students and co-ops, one Resident Associate, one high school teacher, and two college Faculty Research Leave Appointees supplemented the regular ACL staff.

New capabilities of the ACL established in FY 1989 include an automated uranium analyzer, a micro ion chromatograph for analysis of small samples, a gel permeation chromatograph for cleanup of environmental samples, two gas chromatographs for determinations of pesticides and PCBs, and a high-efficiency gamma-ray detector for counting environmental samples.

The 1989 Cryolect Users Meeting and Symposium on GC/MI-IR was hosted by the ACL. The three-day meeting was held on May 15-17, 1989. The purpose of this meeting was to provide those using the technique with a forum for exchanging ideas and experiences related to the operation and application of the instrument. Invited speakers included Lester Andrews of the University of Virginia, who presented a paper on matrix FTIR spectroscopy of transient species, and Gerald Reedy of ANL, who presented a paper on the history of the development of GC/MI-IR here at Argonne in the ACL. Nine papers were presented during the symposium.

III. TECHNICAL HIGHLIGHTS

Engineering Studies of Pyrochemical Processes for Integral Fast Reactor (IFR) Fuels (D. L. Bowers, A. G. Engelkemeir, A. M. Essling, E. A. Huff, S. H. Newnam, E. G. Rauh, C. S. Sabau, and F. P. Smith)

In the electrorefining of uranium and plutonium fuels for the IFR, metallic fuel pins (U, Pu, Zr) are dissolved in a molten cadmium anode and 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 in order 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 Cd anode, the halide-salt electrolyte, and the cathode product. Special dissolution procedures were developed for each type of sample matrix, and separation schemes based on solvent extraction and ion exchange were established to isolate the desired elements from matrix components and, subsequently, to separate these elements from each other. Additional analyses by high resolution gamma-ray spectrometry with a high purity Ge detector were performed on samples to determine ^{241}Am and ^{244}Cm. The purpose was to better understand the behavior of these elements in the electrorefining process.

The "hot" ICP/AES instrument continues to be the workhorse for the determination of cations in these types of samples. This instrument is used to determine U and Pu concentrations in samples where high accuracy is not needed. Whenever high precision and accuracy are required, the method of choice is mass spectrometric isotope dilution (MSID). Samples containing no plutonium, which are from studies of fission product distribution coefficients in the Cd anode and the electrolyte, are analyzed by the "cold" ICP/AES.

Additional analytical support was provided for studies being conducted in CMT to determine the best waste form for the products of these electrorefining processes. In these studies. the gamma irradiation behavior of various waste forms was determined. Mass spectrometry was used for the determination of hydrogen, nitrogen, and oxygen content of the off-gas as a function of radiation dose rate.

The waste electrorefining salts generated by IFR fuel reprocessing will be contacted with Cd-Li-K alloys to remove essentially all of the actinides. The treated salt will be immobilized in mortar for disposal as an intermediate-level waste. Formulations of mortar are being developed to prepare radioactive waste with high compressive strength and low leachability. The leach tests are performed by the standard ANSI6.1 procedure. Ion chromatography is used to measure the amount of Cl^- present, which determines the leach rate from the grout.

During FY 1989, approximately 320 samples were analyzed for all the CMT engineering groups associated with this program, with almost 210 samples from electrorefining studies, 75 from fission product distribution studies, and 35 from waste studies. Each of these samples required determination of up to ten elements.

DOE Environmental Survey (P. C. Lindahl, D. V. Applegate, P. M. Aznavoorian, R. A. Baldino, R. W. Bane, G. A. Baudino, A. S. Boparai, S. J. Bussey, J. C. Demirgian, A. G. Engelkemeir, M. D. Erickson, A. M. Essling, I. M. Fox, I. W. Frank, L. B. Gillis, D. G. Graczyk, M. C. Hansen, R. R. Heinrich, E. A. Huff, E. Y. Hwang, K. J. Jensen, L. L. Lamoureux, J. J. Marr, F. Markun, F. J. Martino, S. H. Newnam, M. H. Picel, E. G. Rauh, L. E. Ross, J. F. Schneider, C. Seils, F. P. Smith, C. T. Snyder, W. E. Streets, L. F. Sytsma, T. TenKate, and L. L. Wetter)

In early 1986 the DOE initiated a program designed to identify current and/or potential environmental problems and areas of environmental risk at DOE facilities, about 40 sites nationwide. Argonne initially staffed one of the six field sampling teams in the program, and for the last three years the ACL has provided environmental analytical chemistry support in the form of organic, inorganic, and radiological analyses for the Argonne sampling team and for sampling teams from the other participating laboratories. The Idaho National Engineering Laboratory (INEL), Oak Ridge National Laboratory (ORNL), and Battelle-Columbus Division/Pacific Northwest Laboratories (BCD/PNL) have been involved in the program in roles similar to ANL in field sampling and analysis. The Oak Ridge Gaseous Diffusion Plant (ORGDP) has provided additional analytical support. The U.S. Environmental Protection Agency Environmental Monitoring Systems Laboratory-Las Vegas (U.S. EPA EMSL-LV) has been providing an independent quality assurance overview; Environmental Monitoring Services, Inc. (EMSI), a consulting firm, has conducted third-party audits; and Nuclear Utilities Services (NUS), a consulting firm, has been providing DOE with technical advice in developing site-specific sampling and analysis plans.

The ACL role includes consulting with and advising the field sampling teams on analysis problems, providing analyses and associated quality assurance/quality control (QA/QC) by methods selected for the program, and developing an analytical methodology suitable for the program in cases where the sample matrix is atypical and/or the types of required data are unique. The methodologies for the organic and inorganic analyses [many of which follow the EPA Contract Laboratory Program Statement of Work (CLP SOW)] and the radiological analyses are coordinated with ORNL, ORGDP, INEL, and BCD/PNL and are published in the Environmental Survey Manual to ensure a consistent and uniform application of methods and treatment of data.

In FY 1989, the ACL provided analytical support for the Survey and technical analytical support to other participating laboratories, including:

(1) For Idaho National Engineering Laboratory (INEL)

  a. 180 environmental samples from the Bryan Mound Strategic Petroleum Reserve (Bryan Mound, TX) for inorganic and organic analyses.

b. 85 environmental samples from the Stanford Linear Accelerator Center (Palo Alto, CA) for inorganic and organic analyses.

c. 75 environmental samples from the Lawrence Berkeley Laboratory (Berkeley, CA) for inorganic and organic analyses.

(2) For Oak Ridge National Laboratory

a. 9 solidified waste samples from the Lawrence Livermore National Laboratory (Livermore, CA) for inorganic and organic analyses.

Additional Survey activities have required ACL to participate in several external QC programs: in the inorganic and organic analysis areas, the EPA's Superfund Contract Laboratory Program (CLP) Quarterly Performance Evaluation Studies; in the inorganic analysis area, the EPA's semiannual Water Pollution Performance Evaluation Studies. Acceptable performance has been demonstrated in each area as detailed in Section IV of this report.

The ACL has also provided technical support to Science Applications International Corporation (Oak Ridge, TN) in the preparation of Survey draft data documents for the Y-12 Plant (Oak Ridge, TN), the Kansas City Plant (Kansas City, MO), and the Portsmouth Uranium Enrichment Center (Pikeston, OH).

Gas Analysis for Argonne Premium Coal Sample Program (A. G. Engelkemeir)

The Argonne Premium Coal Sample Program provides high-quality coal samples from eight U.S. mines for distribution to the basic coal research community. The program involves processing, packaging, characterization, storage, and distribution of the various coals. It also includes gas mass spectrometric analyses of the cover gas (N_2) in randomly selected sealed ampoules of each coal to determine composition changes over time. The presence of O_2 in the cover gas is of particular concern, but also of interest are changes in the amounts of CH_4, CO, and CO_2, since they may signal ongoing chemical or biological degradation of the coal material. To date, the ACL has analyzed the gas from more than 140 such ampoules, with 35 ampoules having been analyzed during FY 1989. Although trends are apparent in the trace-gas concentrations data from some coals, no significant coal degradation has been detected.

Coupling of SFC and MI-IR and MS (A. S. Boparai and D. V. Applegate)

The ability of GC/MI-IR to aid in characterization of isomeric organic compounds has been demonstrated previously in our laboratory. Other chromatographic techniques are needed to separate those compounds that cannot be separated by normal gas chromatography. It has been demonstrated that supercritical fluid chromatography (SFC) is well suited for the separation of high molecular weight, polar, or thermally labile compounds. A project was initiated to develop an interface between the ACL's supercritical fluid chromatography unit and the matrix isolation-infrared spectroscopy/mass spectrometer system.

After development, we propose to apply SFC/MI-IR/MS to the characterization of toxins and biomolecules present in complex mixtures. The ability to obtain infrared spectra of separated matrix-isolated biomolecules present in complex mixtures and the mass spectra of these same molecules will be useful to identify the molecules and to confirm their structure. Because MI-IR yields very narrow IR bands, and band-broadening due to intermolecular hydrogen bonding is absent, any intramolecular hydrogen bonding in a complex biomolecule can be probed.

Deuterium/Hydrogen Measurements for "Cold Fusion" Studies (A. G. Engelkemeir, I. M. Fox, and D. L. Bowers)

Support was provided to one ANL experiment (L. Redey, CMT) investigating the notorious "cold fusion" phenomenon by performing measurements to characterize the H/D composition of the heavy-water electrolyte and of gases dissolved in the palladium electrode. The electrolyte was analyzed by reacting a small aliquot of the heavy water with hot (450°C) uranium-metal chips in a closed container and measuring the isotopic composition of the resulting (H_2, HD, D_2) gas by mass spectrometry. The Pd electrode was degassed, under vacuum, in a specially assembled apparatus that permitted measurement of the rate of gas release from the Pd rod and periodic acquisition of gas samples for mass spectrometric analysis. Outgassing by the Pd was followed at room temperature and at several higher temperatures (up to 600°C) until the dissolved gases were virtually exhausted. The Pd rod was weighed prior to each temperature change to determine the weight of gases lost. The data obtained permitted characterization of the behavior of these two hydrogen isotopes during outgassing, as well as a measure of the quantity of gas dissolved in the electrode. The electrolyte of this experiment was also analyzed for tritium using liquid scintillation counting. A baseline analysis was performed (tritium present in the initial solution), and several samples of the electrolyte throughout the experiment were analyzed to determine if any tritium was produced.

Purification and Electrodeposition of Ni for Metal Clusters Source

(R. W. Bane and K. J. Jensen)

Researchers in the CHM Division (S. Riley and E. Parks) studying the reactions and properties of gas-phase metal clusters have found that the best target for producing nickel clusters is one prepared by electrodepositing purified nickel metal on a copper mandrel. The ACL was instrumental in fabricating one such target several years ago by purifying the Ni (using precipitation with dimethylglyoxime followed by ignition of the precipitate to NiO) and electrodepositing a 10-mil (~250-μm) layer of the purified nickel on the Cu mandrel. This target was recently exhausted and had to be replaced. Since the Ni involved was a relatively expensive separated isotope (^{58}Ni), the residual Ni on the target was reclaimed by machining the mandrel, which introduced copper as an additional contaminant in the reclaimed Ni. We removed this copper by ion exchange and combined the reclaimed ^{58}Ni with new material prior to purifying it according to the previously used procedures. To give the new target a longer expected lifetime, we were requested to attempt electrodepositing 15 mil (380 μm) of Ni rather than the 10 mil (250 μm) applied to the first target. The first 10 mil (250 μm) of Ni deposited satisfactorily, but difficulties were encountered in adding more thickness. The thicker Ni layers developed cracks, produced Ni nodules that grew on the electroplated surface, and peeled away when the target was machined to produce a uniform finish. Better, but still not ideal, deposits were obtained when a platinum rod rather than a graphite rod was used as counterelectrode. The target produced this time, though thicker than the earlier one, was of poorer quality. Based on lessons learned in this effort, future targets will be restricted to the 10-mil (250 μm) thickness where smooth, uniform, and adherent deposits can be readily achieved.

Characterization/Calibration of Daly Detector on Thermal Ionization Mass Spectrometer (E. G. Rauh, P. A. G. O'Hare, D. G. Graczyk, and G. F. Bokowy)

During FY 1988, we added a Daly scintillation detector to our VG Isotopes Model 54R thermal ionization mass spectrometer (TIMS) to complement the Faraday cup detection system on the original instrument. The Daly detector is about 100 times more sensitive than the Faraday cup and permits isotope-ratio measurements on samples where only tiny quantities (<1 μg) of the element of interest are available or on elements whose ionization characteristics limit the intensity of ion beams achievable with thermal ionization.

In FY 1989, we carried out experiments to characterize and calibrate the Daly detector for use in measuring uranium isotope ratios. Measurements were performed on a series of NIST uranium isotopic standards to characterize the detector's response over a range of isotope ratios from 1:200 to 200:1. Early measurements showed the presence of an unexpected bias contribution that we ultimately traced to two sources: (1) the peak flat for the Daly detector was not optimized; and (2) trace uranium impurities in the mass spectrometer filaments, which are negligible for operation with the Faraday cup, gave ion currents that were measurable with the more sensitive Daly detector. We subsequently optimized the peak flat and eliminated the filament impurities by heating each filament under vacuum at high temperature (5.5 A current) for several hours prior to its use for sample analysis. Linearity of the Daly detector was characterized through a series of measurements on the NIST standards wherein we used a special internal-standard procedure to compensate mass discrimination during each analysis. After corrections for the observed nonlinearity were incorporated into the TIMS operating programs and the corrections were verified by additional internal-standard runs, we ran another series of the NIST standards to determine the magnitude of mass-discrimination corrections required for external-standard isotope ratio determinations. These data were accumulated in a special data file, which is accessed by the TIMS operating programs during each analysis run for the purpose of automatically computing and applying a correction based on the most-recently analyzed standards. The Daly detector is presently operable in a fully automatic mode with computer-applied corrections. Uranium isotope ratios measured by the external-standard procedure show a between-runs standard deviation of 0.10%, relative; residual bias (after nonlinearity and mass discrimination corrections) is less than 0.01% of the values measured. Future work with the Daly detector will include optimizing analysis conditions (heating sequence, sample loading, etc.) to reduce the run-to-run variations.

Carbon Tetrachloride and Chloroform Monitoring at a USDA Facility (A. S. Boparai, G. A. Baudino, and S. J. Bussey)

Carbon tetrachloride and chloroform have been extensively used at U.S. Department of Argriculture's grain storage site at Waverly, Nebraska, for many years. Environmental concerns have resulted in a regular monitoring of these two compounds in air and ground water. The ACL has performed analyses on all of the monthly (approximately 10 samples) and quarterly (approximately 60 samples) samples. The water samples are analyzed using the CLP Volatile Organic Analysis protocol while the air samples are analyzed using the NIOSH Method 1003.

Analytical Support for Studies of Alkali in Hot Off-Gas from Pressurized Fluidized-Bed Combustors (PFBC) (I. M. Fox and F. P. Smith)

Alkali metal compounds present in the hot off-gas from PFBC, such as chlorides and sulfates of sodium and potassium, may cause corrosion of the gas turbine used to recover energy released during combustion. Researchers in CMT (S. Lee) are developing an analytical alkali sorber bed technique for reliable quantification of the level of vapor-phase alkali in PFBC off-gas on a time-averaged basis so that the effectiveness of different alkali control strategies might be evaluated. In this technique, analytical alkali sorber beds containing activated bauxite or diatomaceous earth are inserted in the primary (activated bauxite) sorber bed that is used to condition the PFBC process stream. After a given test, the analytical sorber beds are contacted with an appropriate leaching solution, and the leachates are analyzed for alkali metals by atomic absorption spectroscopy in the ACL. During the past year, nearly 250 samples of bauxite leachate and other samples (auxiliary absorber solutions, PFBC feed materials, gases) were analyzed in support of this project.

Infrared Aerosol Analyzer (G. T. Reedy)

Concern is increasing about the possible adverse health effects of acidic particles and gases in the air, including the gaseous oxides of sulfur and nitrogen and the corresponding sulfuric and nitric acids in the fine particles suspended in the air. Instruments for real-time analysis of ambient gases are commercially available and have been used routinely for many years. For the particulate matter, however, typical procedures involve collection of samples of the aerosol over long periods of time (many hours or even weeks), with the analysis being conducted afterward in the laboratory, sometimes many days or weeks later.

In a collaborative effort, CMT staff (S. A. Johnson and R. Kumar) have developed a highly sensitive Infrared Aerosol Analyzer (IAA), which can provide real-time chemical analysis of ambient aerosol particles. This portable instrument divides the particulate matter into three different size fractions (coarse, fine, and ultrafine) and analyzes each size fraction separately. Size fractionation and separation of the ambient aerosol are especially important to prevent interaction among particles after collection. The coarse fraction contains basic crustal materials; the fine fraction contains the bulk of the acidic materials, if any are present; the ultrafine particles are the very fresh, newly formed aerosol which, under certain conditions, can contain a high concentration of sulfuric acid. (Mixing of the sizes during sampling or analysis could destroy these differences.) The analysis by infrared spectroscopy simultaneously detects and measures all acidic components of the aerosol, as well as any other constituents that have an infrared fingerprint. This instrument also has the potential for use in a variety of aerosol research projects as well as use as a monitor for industrial and environmental concerns. In 1989 the IAA received one of Research and Development Magazines R&D-100 Awards.

Analytical Support for Rare-Earth Characterization Studies (E. A. Huff, S. H. Newnam, A. M. Essling, I. M. Fox, and F. P. Smith)

In one of the Chemical and Engineering Support Studies being conducted in CMT (J. Ackerman and J. Settle) for the IFR, the distributions of the more abundant rare earths between the salt phase (as the rare-earth chlorides) and the cadmium solution (as metals) are being compared. In the experiments performed for this work, lanthanum and one other rare earth are dissolved in a pool of cadmium, which is covered with a layer of molten LiCl-KCl eutectic. Samples of the metal and salt phases are withdrawn from the system following stepwise changes in the redox potential (varied by addition of cadmium chloride) or temperature and are submitted to the ACL for chemical analysis.

Each salt-phase sample is analyzed to determine Li, K, Cd, and the pertinent rare earths by ICP/AES and chloride by argentometric titration. Required ICP/AES detection limits for the rare earths are met by carefully selecting analysis wavelengths and generating profiles of individual emission lines. Each cadmium metal sample is similarly analyzed to determine Li, K, and the rare earths by ICP/AES. Cadmium in the metals is measured by titration with EDTA. Our initial approach to measuring chloride in the metal samples entailed dissolving each metal under reflux conditions in dilute nitric acid and measuring chloride in the resulting solution by ion chromatography. Inconsistent data from measurements made in this way were subsequently understood as arising from incomplete dissociation of cadmium/chloride species in the sample solutions; complex species do not behave on the chromatographic column in the same way as free anions, and the chloride they contain is not measured by the ion chromatograph. We presently measure chloride in the cadmium metal samples by silver chloride gravimetry.

During FY 1989, experiments were completed on two rare-earth pairs (La-Nd and La-Eu) and were begun on a third (La-Sm). For europium and samarium, the divalent state of the rare earth is relatively stable, and additional measurements (besides those listed above) were made to determine divalent rare-earth concentrations in selected salt-phase samples. Special care was required for these measurements to avoid air oxidation of the europous or samarous ions during sample preparation or measurement. We developed a procedure wherein the salt sample is dissolved in a nitrogen-sparged solution containing excess ferric [Fe(III)] ion, which is reduced to ferrous [Fe(II)] by the divalent rare earth. The resulting ferrous ion, which is considerably more stable than the divalent rare earth ion, is accurately determined by titration with Ce(IV) using a Ferroin indicator. By performing this titration with a digital autoburet and locating the endpoint with a Dipping-Probe Colorimeter, uncertainties of 1%, relative, are easily achieved. Comparison of the Eu(II) and Sm(II) results from this method with total Eu or Sm concentrations measured by ICP/AES has shown that virtually all Eu in the salt phase exists in the divalent state while only about 20% of Sm is divalent.

Transuranic Extraction (TRUEX) Process Flowsheet Development (E. A. Huff, A. M. Essling, and I. M. Fox)

Technology under development in the TRUEX Program involves a solvent extraction process to separate and recover actinides from nitric acid solutions without a need for valence state adjustments. Extraction, scrub, and strip sections in a multistage centrifugal contactor are used in the process and require characterization for elemental composition. Performance of the multistage centrifugal contactor is being tested utilizing simulated nuclear waste streams. These streams typically consist of fission products (Ag, Ba, Cd, Cs, Sr, Pd, Rh, Ru, Ce, Eu, La, Nd, Pr, Sm, Y) and structural or fuel components (Al, Be, Ca, Cr, Cu, Fe, Mg, Mn, Mo, Na, Ni, Sn, Ti, Zr). Data describing element concentrations in various process stages are being acquired to generate and validate computer models of the TRUEX flowsheet. The ACL has analyzed hundreds of samples (almost 500 in FY 1989) by ICP/AES or other techniques (e.g., laser fluorimetry for U or AAS for Cs) for this effort. The multielement capability of the ICP/AES system, combined with its wide dynamic range for elemental concentrations, is well suited to this type of measurement, although the complicated mixtures involved often present challenging problems pertaining to wavelength selections and handling of spectral interferences. Measurements provided by the ACL have contributed to the accumulation of an extensive TRUEX flowsheet data base in a relatively short time. In turn, this data base has permitted verification of theoretical models and improvements in process performance.

National Acid Precipitation Assessment Program (NAPAP) (F. P. Smith and E. A. Huff)

Analytical support for the NAPAP Program continued this year. Marble and limestone test briquettes, exposed to a variety of atmospheric conditions at a number of sites throughout the country, are brought to ANL for sampling and analysis. Hundreds of specimens (approximately 350 during FY 1989) have been analyzed for anions (fluoride, chloride, nitrate, and sulfate) by ion chromatography, and a smaller number have been analyzed for metals by ICP/AES. Substantial elevations in sulfate and nitrate concentrations have been found to occur in the limestones and have provided a quantitative measure of attack by atmospheric nitrogen and sulfur oxides. Little change in cation concentrations occurs following exposure at the test sites.

Molten Corium Concrete Interaction (MCCI) Studies (A. M. Essling, E. A. Huff, F. P. Smith, I. M. Fox, S. H. Newnam, K. J. Jensen, L. B. Gillis, A. G. Engelkemeir, and B. S. Tani)

In MCCI experiments, mixtures of uranium oxide, zirconium, steel, fission product elements, and concrete are heated to temperatures simulating reactor meltdown conditions with the objective of studying the vaporization behavior of the elements present and understanding 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. Typically, the solidified melt and aerosol samples are analyzed for Ba, Sr, La, Ca, Al, Mg, Na, K, Fe, Cr, Ni, Zr, Ag, and Ce by ICP/AES, for U by laser fluorimetry or ICP/AES, for silicon by AAS or ICP/AES, for soluble anions by ion chromatography, and for miscellaneous elements such as sulfur or carbon by appropriate techniques. Often X-ray diffraction measurements are made to identify particularly interesting phases in selected samples. Gas samples are analyzed by mass spectrometry.

This fiscal year, samples from two large-scale MCCI tests involving high-silica concretes were analyzed. 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 high-pressure sealed-tube procedures, microwave dissolution procedures, and other special techniques. Nevertheless, we had considerable difficulty dissolving the high-silicon samples from the second of these tests, which involved temperatures approaching 2600 K. Ultimately, we put the samples in solution by fusing each one with lithium tetraborate at 1000 to 1100°C and dissolving the resulting material in dilute HCl. Standards for the ICP/AES measurements were prepared in the $Li_2B_4O_7$ flux matrix, and fusion blanks were analyzed with the samples.

Among the results obtained for these tests was evidence that silicon metal was present in addition to SiO_2 in many aerosol samples (an observation explained by disproportionation of gas-phase SiO into Si and SiO_2), and that silicon carbide had formed in some samples. Formation of SiC was unexpected. However, postulating that the SiC arose from gas-phase reaction between SiO and CO_2 helped explain lower-than-anticipated CO_2 concentrations in the gases that were analyzed.

Analytical Support for ANL Geosciences Programs (E. A. Huff, A. M. Essling, F. P. Smith, I. M. Fox, A. G. Engelkemeir, E. G. Rauh, and F. Markun)

A wide variety of analytical measurements are performed by the ACL in support of geoscience programs at ANL. These include characterization of groundwater samples by analysis for cations with ICP/AES or atomic absorption techniques, measurement of anion concentrations (F^-, Cl^-, NO_3^-, SO_4^{2-}, Br^-, I^-)

by ion chromatography, and determination of pH, alkalinity, or other properties by classical methods. This year, we applied these and other, more-specialized, techniques to measurements on thermal waters and minerals from Yellowstone National Park and to measurements on thermal waters, gases, steam condensates, and gas-sampling absorber solutions from volcanoes in Columbia and Ecuador (N. Sturchio, CMT).

As part of the Yellowstone studies, the ACL was requested to implement procedures to isolate and purify Sr from geological samples for isotopic analysis by methods already in place in our laboratory. We evaluated the effectiveness of several column configurations for the critical cation-exchange separation of strontium and calcium in typical geological compositions. Elution behavior of the two elements was characterized by ICP/AES analysis of individual eluant fractions from each column. By going to longer, narrower columns than those used by others, we were successful in isolating as little as 10 μg Sr in high yield from as much as 50 mg Ca, while needing only about half the volume of high-purity acid solutions that short, wide columns require. Our procedure is now being used for preparing Sr isotopic samples from thermal waters and dissolved travertine ($CaCO_3$) deposits from Yellowstone hot springs.

A recently developed ^{226}Ra and ^{228}Ra method was used to analyze these Yellowstone samples. Large volumes of water were passed through the special Dowex radium selective resin upon collection, and mailed to Argonne where the resins were processed and counted. Short holding times were required to determine ^{224}Ra, which has a half life of 3.66 days. Other radium isotopes were determined on subsequent countings. Additionally, a radon emanation method was applied to dissolved $CaCO_3$ deposits from Yellowstone hot springs to determine ^{226}Ra more precisely.

One-Step Separation of Uranium from Mixtures of Cations (A. M. Essling, E. A. Huff, and D. G. Graczyk)

Separation of uranium from mixtures containing a variety of other metals is frequently required for effective application of analytical measurement techniques, either for the determination/characterization of the uranium itself or for measurement of the other metals. Most methods for isolating uranium from complicated matrices or for isolating common metals from a uranium matrix involve several steps and, consequently, are time-consuming, prone to contamination, and likely to give poor recovery for the metals of interest. In collaboration with ANL separations chemists (E. P. Horwitz, CHM), we developed a simple, one-step uranium separation based on supported-liquid column-extraction chromatography, which uses a packing prepared from 100- to 120-mesh XAD-7 ion exchange resin coated with 40 wt. % diamyl amylphosphonate (DAAP). Uranium is selectively retained on the column from 3 $\underline{N}$ HNO_3 and eluted with high recovery by 0.01 $\underline{N}$ HNO_3. Data obtained in our studies include uranium recovery at the microgram level, column capacity, and retention behavior of a number of common metals and radionuclides.

We have successfully applied this separation to the simultaneous isolation and purification of uranium for mass spectrometric measurement of the U isotopes in a variety of mixtures, including digested environmental materials (e.g., soil, sludge, sediment, tar sand, rock, or bone). In this application, microgram quantities of U may be separated from gram quantities of matrix constituents to give a product sufficiently pure for direct loading on the mass spectrometer filament.

The substantial uranium capacity of the DAAP packing (130 mg U per mL resin) and its virtual nonretention of most other metals make it attractive also for removing uranium interference when determining the other metals in a given mixture. For example, we have used this approach to isolate metal impurities from a uranium matrix for multielement determination by ICP/AES.

Determination of Organic Fluid Fouling in Heat Exchange Systems (C. T. Snyder and J. C. Demirgian)

The fouling of heat exchange systems is currently being studied using an organic fluid model. The goal is to elucidate the mechanism by which fouling occurs by characterizing soluble precursors in the organic fluid. Samples of heptane spiked with 6% styrene were extracted at varying time intervals and analyzed using GC with flame ionization detection and GC/MS. System temperatures ranged from 30 to 180°C. Results indicated that heptane stability declined after 24 h of loop circulation, and the resulting alkane fragments combined with styrene to form propyl benzene and other C_3-benzenes. Styrene formed dimers and trimers by 60 h at the higher temperature. Based on these findings, a mechanism for organic fluid fouling was proposed. Methodology has also been developed to evaluate fouling deposition on the system pipe using FTIR microscopy, SEM, and XRD.

Analytical Support for Research on High-Temperature Superconductors (E. A. Huff, A. M. Essling, F. P. Smith, and B. S. Tani)

The ACL continued providing extensive analytical support to high-temperature superconductivity programs in the MCT and CMT Divisions. Much of this work involves analysis of starting materials, process samples, and products related to fabrication and characterization studies on $YBa_2Cu_3O_{7-x}$ ("123" compound) ceramics or other ceramic compositions. Measurements we perform include determining elemental compositions by ICP/AES, anions by ion chromatography, oxygen coefficients (i.e., the value of 7-x in the formula $YBa_2Cu_3O_{7-x}$) in selected ceramics by an iodometric method, and phases by X-ray powder diffraction.

Determination of the oxygen coefficient for copper-containing ceramics was established as a new capability within the ACL during FY 1988. It is based on our modification of a method by Appelman et al. [Inorg. Chem. 26, 3237 (1987)] and employs an autoburet and special colorimeter to provide optimal precision in the critical titration of iodine with thiosulfate. From the results of replicate measurements performed on the almost 60 samples analyzed during FY 1989, we have determined that reproducibility of better than ±0.005, absolute, is routinely achieved for samples weighing from 15 to 40 mg. We are considering modification of the method to accommodate smaller samples so that measurements may be extended to single-crystal superconductors where only 1 mg or less might be available for analysis.

Crystalline preferred orientation is usually not desirable in phase identification, but is desired in some superconductivity preparations and is monitored by observing anomalous intensities for a certain class of reflections in the "123" compound. Lattice constant measurements have also become important with the level of various dopants that are added to superconductive and composite materials in order to influence their physical properties. A change in lattice constants with dopant level indicates incorporation of the dopant into the structure; no change in lattice constants could still indicate incorporation into vacancies of the structure.

In addition to supporting research on superconducting ceramics, the ACL also contributes to ongoing work in MCT toward characterizing thin-film superconductors. Elemental compositions of thin films, usually containing only a few micrograms of material, are measured by ICP/AES. Through a special arrangement with the MCT researchers, data on the thin films are often reported within a few hours after the samples are submitted so that deposition conditions may be adjusted to give a desired film composition.

Analytical Support Using X-ray Powder Diffraction (XRD) and Scanning Electron Microscopy (SEM) (B. S. Tani)

During this period, 381 XRD and SEM analyses were completed. The SEM is one of the ACL instruments that is available to users at ANL. The SEM facility has a JEOL 35U scanning electron microscope with EEDS II Ortec EDX analyzer and

Technics metallic sample coater. Training in use of this instrument is provided, if necessary, by ACL staff.

The ACL has provided XRD services to a number of programs in CMT, MCT, and CHM, such as the IFR, fuel cell, composite materials, advanced batteries, high level waste repository interactions, and superconductivity studies.

X-ray diffraction is useful for identifying crystalline phases and the presence of amorphous phases and for monitoring the relative purity of samples.

Computerized Calculation and Reporting of Environmental Levels of Radioactivity Determined by Gamma Spectroscopy (W. E. Streets and B. S. Tani)

For an analyst, the most time-consuming aspect of gamma spectrometry of samples containing environmental levels of radioactivity is the calculation of the data. The main reason is the low sample activity, which requires that each energy used for nuclide identification be corrected for room background. A typical sample may contain from 20 to 40 separate energies to be calculated. Using ANL Support Development Funds, we have investigated with L. R. Greenwood and S. D. Gabelnick (CMT) ways of streamlining and simplifying this calculation to eliminate unnecessary data transfer and possible transcription errors and to reduce time spent in calculating and reporting sample data.

We first investigated the use of software on our three gamma spectrometer systems for background correction prior to transfer of the sample data to the CMT VAX computer for analysis by the GAMANAL [Gunnick, R. and Niday, J. B., UCRL Report 76699, 1975] radionuclide identification program. This approach was quickly abandoned for several reasons, including (a) separate background stripping procedures would be required for each of the three systems; and (b) documentation of the background correction was insufficient to meet the QA requirements of some of the ACL work.

Since all of the gamma spectrometry data are analyzed by GAMANAL on the CMT VAX, we concentrated our efforts on developing the calculation on the VAX.

Starting with an Excel spreadsheet on an IBM personal computer, we developed a manual entry approach to making the necessary calculations, which we subsequently transferred into the C-CALC Plus spreadsheet on the VAX computer. Since we now had both the nuclide identification file generated by GAMANAL and the spreadsheet on the central VAX computer, we investigated ways of automatically transferring data from the GAMANAL output to the spreadsheet for calculation. Fortran programming was done to generate GAMANAL output in a format compatible with the spreadsheet. Thus, we are now able to automatically transfer data from the GAMANAL output of sample and background files into the spreadsheet and calculate results very quickly. We are currently investigating the use of VAX macros to further reduce the number of steps in the process and to generate finished reports from the individual sample analysis sheets.

Lansdowne Pennsylvania Project (L. L. Wetter, F. Markun, W. E. Streets, L. L. Lamoureux, A. M. Essling, and R. R. Heinrich)

In the early 1900s, Professor Kabakjian, from the University of Pennsylvania, supplied radium for medical applications on a world-wide basis. Professor Kabakjian accomplished this task by refining uranium ore to extract radium in the basement of his home over a period of several years. Consequently, his work led to widespread contamination of the house, its household contents, and the surrounding property.

During this past year, the Kabakjian property in Lansdowne, PA, has been part of a U.S. EPA Superfund cleanup project. A group of ESH staff coordinated the demolition and decontamination of this site. In support of this project, the ACL analyzed approximately 120 samples for a number of analytes (Th, Ac, Pa, U, and gamma emitters). Because the project included demolition of the home itself, many of the samples analyzed (e.g., insulation, whole bricks, floor carpeting) were unique and, therefore, challenging.

Determination of ^{226}Ra and ^{228}Ra in Water and Soil (F. Markun)

The radium in soil is the source of radon that is found in homes. The EPA has established 4.0 pCi/L of radon in air as the "level of concern." Radium in soil and rocks contributes to elevated levels of radium in water. Radium in drinking water is regulated by the EPA, where the maximum allowable total radium is 5.0 pCi/L.

Using ANL Support Development Funds, we have demonstrated the effectiveness of a newly developed ion exchange method utilizing a radium selective Dowex resin. This method is much faster and less involved than other methods available but requires a large sample size, preferably, 20 L.

In this method, a water sample is acidified with hydrochloric acid to approximately pH 3 and then processed through the special Dowex ion exchange resin bed. The resin containing the radium is subsequently sealed in a metal can and aged before counting on a NaI detector. The counting data are computer processed by a spectrum analysis program that uses the least squares method.

Soil and crushed rock samples are processed in a similar manner. The samples are sealed in a metal can and allowed to age, during which time ^{226}Ra comes to an equilibrium with ^{222}Rn and its daughters. Counting and data processing are the same as described for water samples. The sensitivity of this method is 0.5 pCi/L for water and 0.1 pCi/g for soil samples when sufficient sample size is available (i.e., 20 L for water and ~200 g for soil). This method for determining the ^{226}Ra and ^{228}Ra is highly desirable since it is fast and accurate and, therefore, cost effective.

At present, this is the method being used for the determination of radium in the EMSL Performance Evaluation samples and in municipal waste samples. In addition, soil and rock samples have been analyzed in an environmental survey of suspected storage sites for uranium ore tailings.

Evaluation of Gas Chromatography/Matrix Isolation-Infrared Spectroscopy for Quantitative Analysis (J. F. Schneider, S. E. Spiro, and M. D. Erickson)

The purpose of this project, which is funded by the U.S. EPA, is to evaluate the utility of the commercial gas chromatograph/matrix-isolation infrared spectrometer (GC/MI-IR) for quantitative analysis. This instrument, which was developed at Argonne in the ACL, has found several applications as a qualitative tool for identifying compounds that are difficult to identify by other methods such as GC/MS. We ran a series of standard mixtures to determine instrument reproducibility, linearity, and limit of detection. The reproducibility of the response was 40 to 60%, which is poorer than expected. An ANL/ACL report is being written on this project. The U.S. EPA has funded a second phase to this project to determine the cause of the poor reproducibility in the commercial instrument and find ways to correct it.

Preliminary Environmental Assessments for the Army and National Guard (J. F. Schneider, M. C. Hansen, E. Y. Hwang, L. B. Gillis, and L. E. Sytsma)

The ACL is assisting the Environmental Research Division (ERD) in performing preliminary environmental assessments on various Army and National Guard sites around the country. These assessments are the first step in identifying environmentally significant operations that may require remediation. Work has included several site visits, report writing and editing, and interacting with the program sponsor--the U.S. Army Toxic and Hazardous Materials Agency (USATHAMA).

Plutonium Residue Recovery (PuRR) Program (D. L. Bowers, C. S. Sabau, R. R. Heinrich, and W. E. Streets)

Experimental work for determining the most economical process for the recovery of Pu from various types of scrap and residues continued in CMT during FY 1989. Several different chemical processes have been explored to determine which is the most efficient. These processes utilize a number of different molten salt mixtures along with varying reductant alloys composed of Al, Cu, Ca, Mg, and/or Zn. The search for a very efficient process for the removal of TRU elements is important in the disposal of the waste effluents, which ideally will be rendered non-TRU waste.

Consistent with this goal, material balances have to be accurate, and reliable analyses of the feed material, salt, and reductants have to be performed. The primary analysis was quantification of elements by ICP/AES and by gamma spectrometry. In addition to the reductants and Pu, elements of interest were Am, B, Cd, Fe, K, Li, Mo, Na, Si, Ta, and Ti. Approximately 130 samples were analyzed in FY 1989.

Environmental Radiochemistry (A. M. Essling, R. R. Heinrich, L. L. Lamoureux, F. Markun, W. E. Streets, T. TenKate, and L. L. Vetter)

The principal effort in this category for FY 1989 has been the application of methodologies developed over the last two years for analyzing environmental samples containing radionuclides. Methodologies were developed for determination of gamma emitters, as well as strontium, technetium, plutonium, americium, uranium, thorium, actinium, protactinium, and tritium. Numerous samples of soil, vegetation, and water have been analyzed for ANL-East (ESH), ANL-West, and other sites already mentioned (Lansdowne). Approximately 200 samples collected from the two ANL sites were analyzed for most of the radionuclides listed.

Rocky Flats Plutonium Metal Exchange Program (D. L. Bowers)

The ACL has participated in this exchange program for approximately two years since the successful operation of the "hot" ICP/AES instrument in our plutonium laboratory. The primary objective of the program is to provide an independent evaluation for plutonium analysis at participating laboratories and to provide a data base and data review to the DOE on the measurement capabilities of the participating laboratories. The program provides sample materials to participating laboratories for analysis. The plutonium metal is dissolved and chemically separated by anion exchange, and subsequently, the metal impurities are determined by ICP/AES. The impurity measurements from the individual laboratories are statistically evaluated for differences. The ACL has participated in four of these intercomparison studies, and each effort has resulted in metal impurity values that have been consistent with the stated target values being supplied by selected laboratories.

Environmental Protection Agency (EPA) Project (J. F. Schneider, S. E. Swanson, L. A. Vallow, and R. J. Wingender)

Region V of the U.S. EPA has asked the ACL to assist in the initiation of sampling and analysis procedures, described in U.S. EPA Method TO-14, for ambient air monitoring of toxic organics. This project will require: (1) adapting the TO-14 procedures to the specific Region V requirements; (2) assembling, operating, and validating sampling and analysis equipment; and (3) providing standard operating procedures and training for the validated procedures. The task of assembly and validation is being accomplished with the assistance of P. Doskey (ERD).

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 glove box. Several analytical instruments (GC, GC/MS, and ICP/AES) are located in this area for use with these toxic samples. The EPA also continues to support a storage facility on site for legally sensitive samples.

Determination of Herbicides in Water Treatment Systems (P. M. Aznavoorian, I. W. Frank, E. Y. Hwang, S. E. Swanson, and R. J. Wingender)

The West Coast Regional Water Authority (Clearwater, FL) has asked the ACL for assistance in the determination of herbicides that may be concentrated in this region's water treatment systems. In this project water samples and resin extracts have been prepared for analysis by the ACL using U.S. EPA Method 8150. These extracts were then analyzed by GC/MS.

Remote Detection of Agent-Related Chemicals (J. C. Demirgian and S. M. Spurgash)

Fourier transform infrared (FTIR) spectroscopy capability has been established for the remote detection of chemical agents and their related precursors and degradation products. Agent-related chemicals have been detected at concentrations of 1.6 ppm for a 1-m pathlength when released in front of brick walls and other environments. The objectives of this ACL effort are to develop FTIR technology for two scenarios: (1) monitor facilities to determine whether they are producing chemical agents and (2) remotely detect agent from undeclared facilities. Remote FTIR spectroscopy uses ambient energy as the infrared source. The instrumentation consists of an interferometer, detector, associated optics, and computerized data system. A vaporizer was developed to allow us to release small quantities of chemicals at low ppm concentrations reproducibly. Future work will include measurements at greater distances for more chemicals.

Analysis of Soil Sample for Explosives and Selected Contaminants (J. C. Demirgian)

The objective of this work is to validate an FTIR-based method that uses thermal desorption for the qualitative and quantitative analysis of soils for selected pesticides, semivolatile and volatile organic compounds, and high explosives. This work will combine ANL's capabilities in FTIR spectroscopy of gas-phase molecules and trace organic analysis. Initial work in FY 1989 consisted of redesigning a variable pathlength, heatable cell and thermal desorption unit. Work will continue throughout FY 1990.

Review of Rocky Flats Plant Laboratories (M. D. Erickson, A. S. Boparai, R. R. Heinrich, P. C. Lindahl, and L. L. Wetter)

The ACL conducted an extensive review of Rocky Flats Plant's (RFP) chemical analysis activities in support of the RFP Environmental Restoration

Program for Albuquerque Operations' Technical Support Office at Los Alamos National Laboratory. The capabilities were assessed of four laboratories (two on-site at RFP and two subcontractors) to perform Contract Laboratory Program (CLP)-type analyses. Sample analysis data from three of these four laboratories were evaluated. The laboratory assessments consisted of walk-throughs, records inspection, and employee interviews using checklists derived from the CLP methods. Data were assessed using data acceptability criteria derived from the CLP and were categorized as "valid," "acceptable with qualifications," or "rejected." Five reports were prepared and submitted to both the sponsor and the reviewed laboratories. The findings of this review have aided RFP in making changes in the chemical analyses in support of their Environmental Restoration Program.

<u>Chemistry of Bottom Sediments from the Chicago River and North Branch Canal, Lake Street to North Avenue</u> (P. M. Aznavoorian, M. D. Erickson, I. M. Fox, A. G. Engelkemeir, E. A. Huff, S. H. Newnam, J. A. Ruskamp, and J. F. Schneider)

The ACL, together with the EES Division, participated in a joint venture that involved the analysis of bottom sediments of the North Branch Canal and Chicago River. This study was requested by the United States Army Corps of Engineers (USACE) and resulted from the fact that this organization is in charge of directing and maintaining the commercial navigation that passes through the Chicago River. To facilitate this navigation, the USACE deemed it necessary that this section of the river be dredged. Before these dredgings could be transferred to a landfill, the EPA required that they be analyzed for priority pollutants, including PCBs, volatile and semivolatile organics, metals, and cyanide. Thus, the present study was undertaken to comply with EPA requirements. Strict sampling and chain-of-custody protocols were maintained.

Preparation and analysis of these samples were performed using the CLP protocols. The procedures involved extraction of the PCB and semivolatile samples, determination of PCBs using GC with an electron capture detector, and analysis for the semivolatile compounds using GC/MS. Volatiles were determined by a gaseous purge and trap technique, followed by on-line GC/MS analysis. Graphite furnace atomic absorption spectrometry was used to determine for the presence of As, Se, and Tl; the samples were digested in concentrated nitric acid solution, vaporized, and then analyzed by AA spectroscopy. Inductively coupled plasma/atomic emission spectroscopy was used for determination of the remaining metals. The samples underwent acid digestion, excitation by means of a high-energy RF field, and quantitation by emission spectroscopy. Cyanide was measured colorimetrically following distillation of the previously acidified samples.

Total PCB contents for all composited samples ranged from below detection limits to a high of 96 mg/kg (dry weight). Mean PCB values for the composited samples taken at each sampling location (an overall core depth of 7 m) ranged from 5.2 to 45.6 mg/kg (dry weight). The PCB concentrations at most of the sampling stations generally increased with the depth from which the sample was taken.

Samples were analyzed for volatile organic compounds, semivolatile organic compounds, and metal priority pollutants. Volatiles and semivolatiles showed traces of toluene, ethylbenzene, xylene, naphthalene, acenaphthalene, phenanthrene, fluoranthene, pyrene, benzo(a)anthracene, and chrysene.

A Systems Approach to Development of Technology for Verification of Treaties Limiting Chemical Weapons

(A. S. Boparai, J. C. Demirgian, M. D. Erickson, C. T. Snyder, J. F. Schneider, and S. E. Swanson)

Progress has been made on bilateral and multilateral treaties to limit chemical weapons (CW). The ACL and other Argonne staff conducted a preliminary project to assess the chemical measurement technologies available for verification of treaty provisions relative to CW agents and related compounds. Verification is needed to ensure compliance with several provisions of the treaties, including destruction of stockpiles or prohibitions against manufacture. In this preliminary study, we found a large amount of primary literature on the available technologies but no critical reviews, which should be available for U.S. and international negotiators. We also determined, using assumptions on production technology, that there should be detectable quantities of "signature" chemicals emitted from a production facility; therefore, "fenceline monitoring" appears feasible. In addition, we assessed the current and potential utility of advanced sample preparation, gas chromatography, ion mobility spectrometry, infrared spectroscopy, mass spectrometry, multi-instrument arrays, multivariate signal processing, near infrared spectroscopy, preconcentrators, and electrochemical sensors.

Fourier Transform Infrared (FTIR) Microscopy (C. T. Snyder and J. C. Demirgian)

The FTIR microscope user facility consists of a Spectra-Tech IRPLAN-II and a Nicolet 60SX FTIR. Applications of this microscope include characterizing changes in plastic pipe as a function of aging and identifying deposits on tubing. The FTIR microscope has also been used to analyze fouling deposition in heat exchangers and to identify organics in coal and water in minerals.

Walter Reed Army Institute of Research (WRAIR) Site Characterization (A. S. Boparai, G. A. Baudino, P. M. Aznavoorian, E. Y. Hwang, R. R. Heinrich, D. G. Graczyk, F. J. Martino, A. M. Essling, S. H. Newnam, R. W. Bane, I. M. Fox, L. B. Gillis, E. A. Huff, and A. G. Engelkemeir)

The WRAIR building is proposed to be built on an uncontrolled landfill site in Forest Glenn, Maryland. Prior to construction of the building, hydrogeological and chemical characterization of this site is required. The ACL has provided assistance to the U.S. Army Corps of Engineers in writing of the sampling and analysis and quality assurance plans for the site. The ACL is managing the total analytical work for this project and will be performing most of the analyses.

Advanced Instrumental Methods for Determining Organics in Solid Waste (A. S. Boparai, L. A. Raphaelian, M. D. Erickson, I. W. Frank, and D. V. Applegate)

The main objective of this project, which is funded through the Morgantown Energy and Technology Center (METC), is to apply advanced instrumentation [e.g., GC/MI-IR, supercritical fluid extraction (SFE), and supercritical fluid chromatography (SFC)] to the characterization of organic compounds present in coal waste samples.

Some results of the GC/MI-IR work have been published [J. F. Schneider, L. A. Raphaelian, A. S. Boparai, M. C. Hansen, and M. D. Erickson, J. Chromatogr. Sci. 27, 592-595 (1989)]. The SFE/SFC analyses of coal waste samples demonstrated that SFE using carbon dioxide for a short time (5 to 7 min) extracted compounds of at least as high molecular weight as were extracted using the labor-intensive and time-consuming Soxhlet solvent extraction. The results of SFE/SFC have been submitted for publication.

Environmental Analyses for DOE Sites (P. C. Lindahl, D. V. Applegate, P. M. Aznavoorian, R. A. Baldino, R. W. Bane, G. A. Baudino, A. S. Boparai, S. J. Bussey, J. C. Demirgian, A. G. Engelkemeir, M. D. Erickson, A. M. Essling, I. M. Fox, I. W. Frank, L. B. Gillis, D. G. Graczyk, M. C. Hansen, R. R. Heinrich, E. A. Huff, E. Y. Hwang, K. J. Jensen, L. L. Lamoureux, J. J. Marr, F. Markun, F. J. Martino, S. H. Newnam, M. H. Picel, E. G. Rauh, L. E. Ross, J. F. Schneider, C. Seils, F. P. Smith, C. T. Snyder, W. E. Streets, L. F. Sytsma, T. TenKate, and L. L. Wetter)

The ACL did environmental analyses for DOE sites in addition to those done as part of the DOE Environmental Survey (see page 9).

(1) Rocky Flats Plant - The ACL received approximately 60 environmental samples from the Rocky Flats Plant. These samples collected by the INEL field sampling team consisted of waters, soils, and sludges to be analyzed for target analyte list (TAL); inorganics, anions, and target compound list (TCL); and volatile and semivolatile organics by CLP protocols. This work is being coordinated through the DOE Office of Environmental Audit and will be completed in FY 1990.

(2) Argonne National Laboratory-West - The ACL received environmental samples from ANL-W for the determination of RCRA analyses (CFR Appendix VIII and IX inorganic and organic constituents). This work has been initiated with anticipated completion in early FY 1990.

Preparation of Environmental Compliance Documents (M. H. Picel)

Environmental compliance documents (e.g., those required by CERCLA, NEPA) were prepared and reviewed. These documents are necessary to proceed with remedial action on DOE sites under FUSRAP (Formerly Utilized Sites Remedial Action Program) and SFMP (Surplus Facilities Management Program).

IV. QUALITY ASSURANCE

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

Much of the ACL analytical effort in FY 1989 was directed toward completion of the U.S. DOE Environmental Survey (DES). Because the quality assurance/quality control (QA/QC) associated with this program is significant, much of the ACL's QA/QC activities for FY 1989 were related to DES.

To help assure high-quality analytical performance, the ACL participated in four multilaboratory programs sponsored by the U.S. EPA and DOE. Each program provided the ACL with sets of either Quarterly or Semiannual Blind Performance Evaluation (PE) Samples for analysis.

The ACL has received organic and inorganic PE samples quarterly from the EPA Environmental Monitoring Systems Laboratory-Las Vegas (EMSL-LV). The scores received for FY 1989 Inorganic PE samples (QB1 through QB4 for FY 1989) were 94.8, 88.4, 75.2, and 95.1%. The organic PE scores are given as a combined score for the volatile, semivolatile, and pesticide quarterly organic fractions; the combined FY 1989 Organic PE sample scores (QB1 through QB4 for FY 1989) were 71.6, 97.0, 90.4, and 100%, respectively. All scores were within the acceptable range as determined by the EPA.

In addition, the ACL participated in the EPA Water Pollution Study (WPS). Semiannual Inorganic PE Samples were analyzed for analytes such as cyanide, oil/grease, total dissolved and suspended solids, and a variety of anions. The EMSL-LV does not report scores for these samples but did report that ACL's analytical results were in excellent agreement with EPA's known values.

The DOE Environmental Measurement Laboratory (EML) and EMSL-LV coordinated multilaboratory Cross-Check PE Programs and provided radiological PE samples for analysis by the ACL. Water, soil, and air filter samples for radionuclide determinations, e.g., gross alpha-beta, gamma, tritium, radium, uranium, plutonium, neptunium, were received alternately throughout FY 1989, on a semiannual basis. The ACL did well in these programs, with each of the radionuclides analyzed being in agreement with reference values.

An in-house quality control (QC) program was initiated in 1987 to provide the ACL with a means of assuring that analytical systems were performing satisfactorily prior to the analysis of each Organic Quarterly Blind (QB) PE sample. The commercially obtained QC samples provide the ACL with volatile, semivolatile, pesticide, and PCB (Aroclor) organic standards of known value to be analyzed with the QB samples. Analytical results obtained using in-house QC standards were compared to the known values supplied by the vendor. Satisfactory performance on this internal self-check sample is an ACL requirement prior to submitting organic PE results to the EPA for scoring.

ACL participation in the DOE Environmental Survey during FY 1989 included the analysis of samples from the following sites: Idaho National Engineering Laboratory, Stanford Linear Accelerator Center, Lawrence Livermore Laboratory, Lawrence Berkeley Laboratory, Morgantown Energy and Technology Center, Nevada Test Site, Bryan Mound Petroleum Reserve, and the Oak Ridge Gaseous Diffusion Plant (X-10). Approximately 125 sample data packages were reviewed for completeness and correctness according to CLP protocol prior to shipping to data management teams for data entry. A representative group of inorganic and organic CLP sample data packages for five of the sites was submitted to EMSL-LV for evaluation by the Lockheed Engineering and Sciences Company. Information gathered from these packages and forwarded by EMSL-LV indicated that some areas of data reporting needed attention. Corrective actions were formulated in those areas and our responses forwarded to EMSL-LV. Corrective actions were implemented by the ACL where required.

The ACL QA Plan is reviewed internally each year by the ACL QA/QC Coordinator to ensure that it is up-to-date and in agreement with current quality assurance policies of CMT and ANL. The revision resulting from this review was completed and distributed in March 1989. An August 1989 audit of the ACL resulted in no findings, but observations were made that led to suggested revisions of the ACL QA Plan.

An ACL QC Plan was written to provide information and direction relative to an ongoing, program-independent, in-house quality control program. The Plan was written to provide guidelines that will aid in achieving that goal. The QC Plan addresses the procurement, distribution, and analysis of samples from the EPA as well as those received from commercial vendors.

We prepared and distributed a set of written guidelines that are intended to aid in providing consistency and clarity in the reporting of analytical results by ACL analysts. The guidelines provide information about (1) the quality of analytical measurements, (2) the use of blanks, replicates, detection limits, spike recoveries, standard measurements, statistical quantities, etc., to support the validity of analytical results, and (3) common terms used in the reporting of analytical results.

The total number of ACL standard operating procedures written and now in place rose to 140 in FY 1989. All are available in two-volume manuals located in each laboratory area.

V. PROFESSIONAL ACTIVITIES

A. Publications and Reports

Analytical Chemistry Laboratory Progress Report for FY 1988
D. W. Green, R. R. Heinrich, D. G. Graczyk, P. C. Lindahl, M. D. Erickson, with contributions from ACL Staff
Argonne National Laboratory Report No. ANL/ACL-88-1 (1988)

Application of High Sensitivity Laser Techniques to the Study of TRU Speciation
J. V. Beitz, D. T. Reed, D. L. Bowers, and J. K. Bates
Proceedings of the Department of Energy In Situ Characterization and Monitoring Technologies Workshop, Department of Energy Report DOE/HWP-62 (1988)

Assessment of the Impact of the Chernobyl Nuclear Accident in Europe
M. N. Sabau and C. S. Sabau
Journal of American Romanian Academy 12, 118-132 (1989)

Chemistry of Bottom Sediments from the North Branch of the Chicago River and North Branch Canal, Lake Street to North Avenue, Chicago, Illinois
W. Harrison, P. M. Aznavoorian, D. Kullen, M. D. Erickson, J. F. Schneider, D. G. Graczyk, and C. Tome
Argonne National Laboratory Report No. ANL/EES-TM-372 (1989)

Detection and Speciation of Transuranium Elements in Synthetic Groundwater via Pulsed-Laser Excitation
J. V. Beitz, D. L. Bowers, M. M. Doxtader, V. A. Maroni, and D. T. Reed
Radiochimica Acta 44/45, 87-93 (1988)

The Determination of Impurities in Plutonium Metal by Anion Exchange and ICP/AES
E. A. Huff and D. L. Bowers
Applied Spectroscopy 43(2), 223-226 (1988)

Effect of Heat Treatment Time and Temperature on the Properties of $YBa_2Cu_3O_{7-x}$
I. D. Bloom, B. S. Tani, M. C. Hash, D. Shi, M. A. Patel, K. C. Goretta, N. Chen, and D. W. Capone, II
Journal of Materials Research 4(5), 1093-1098 (1989)

Experimental Evidence of Miscibility Gap in the $YBa_2Cu_3O_x$ System
M. Tetenbaum, B. S. Tani, B. Czech, and M. Blander
Physica C 158, 377-380 (1989)

Influence of Chlorine Ratio and Oxygen Bleaching on the Formation of PCDFs and PCDDs in Pulp Bleaching. Part 1: A Laboratory Study
F. de Sousa, M.-C. Kolar, K. P. Kringstad, S. E. Swanson, C. Rappe, and B. Glas
Tappi Journal 72(4), 147-153 (1989)

The Influence of Chlorine Ratio and Oxygen Bleaching on the Formation of PCDFs and PCDDs in Pulp Bleaching. Part 2: A Full Mill Study
K. P. Kringstad, L. Johansson, M.-C. Kolar, F. de Sousa, S. E. Swanson, B. Glas, and C. Rappe
Tappi Journal 72(6), 163-170 (1989)

In Situ Soil Gas Monitoring by FTIR
M. D. Erickson and J. C. Demirgian
Proceedings of the Department of Energy In Situ Characterization and Monitoring Technologies Workshop, Department of Energy Report DOE/HWP-62 (1988)

Isotopic Anomalies in High Z Elements: Uranium?
S. Jovanovic, G. W. Reed, Jr., A. M. Essling, E. G. Rauh, and D. G. Graczyk
Argonne National Laboratory Report No. ANL-89/9 (1989)

The Isotopic Composition of Methane in Polar Ice Cores
H. Craig, C. C. Chou, J. A. Welhan, C. M. Stevens, and A. G. Engelkemeir
Science 242, 1535-1539 (1988)

Measurement of Long-Lived Radionuclides in Fusion Materials
L. R. Greenwood and D. L. Bowers
Reactor Dosimetry: Methods, Applications, and Standardization, ASTM STP 1001, Harry Farrar IV and E. P. Lippincott, Eds., American Society for Testing and Materials, Philadelphia, pp. 508-514 (1989)

Methyl-, Polymethyl-, and Alkylpolychlorodibenzofurans Identified in Pulp Mill Sludge and Sediments
L. -O. Kjeller, S. E. Swanson, and C. Rappe
Environmental Science and Technology 23, 1130-1137 (1989)

A New Technique for Enhancing Helium Production in Ferritic Materials
L. R. Greenwood, D. G. Graczyk, and D. W. Kneff
Journal of Nuclear Materials 155-157, 1335-1339 (1988)

Polychlorinated Dibenzofurans and Other Thermal Combustion Products from Dielectric Fluids Containing Polychlorinated Biphenyls
M. D. Erickson, S. E. Swanson, J. D. Flora, Jr., and G. D. Hinshaw
Environmental Science and Technology 23, 462-470 (1989)

The Potential of Continuous Emission Monitoring of Hazardous Waste Incinerators Using Fourier Transform Infrared Spectroscopy
J. C. Demirgian and M. D. Erickson
Proceedings of the 1989 Incineration Conference on Incineration of Hazardous, Infectious, Radioactive, and Mixed Wastes, (1989)

Quality of Environmental Analyses: Field Analysis, Laboratory Analysis, and Monitoring
D. W. Green and P. C. Lindahl
Proceedings of the Department of Energy In Situ Characterization and Monitoring Technologies Workshop, Department of Energy Report DOE/HWP-62 (1988)

The Standard Molar Enthalpy of Formation at 298.15 K of $S_2N^+AsF_6^-$ by Fluorine Combustion Calorimetry
P. A. G. O'Hare, E. G. Awere, S. Parsons, and J. Passmore
Journal of Chemical Thermodynamics 21, 153-158 (1989)

Standard Molar Enthalpy of Formation at 298.15 K of the β-Modification of Molybdenum Ditelluride
P. A. G. O'Hare and G. A. Hope
Journal of Chemical Thermodynamics 21, 701-707 (1989)

Thermochemistry of Uranium Compounds XVII. Standard Molar Enthalpy of Formation at 298.15 K of Dehydrated Schoepite $UO_3 \cdot 0.9H_2O$. Thermodynamics of (Schoepite + Dehydrated Schoepite + Water)
P. A. G. O'Hare, B. M. Lewis, and S. N. Nguyen
Journal Chemical Thermodynamics 20, 1287-1296 (1988)

B. Oral Presentations

Analyses for Argonne Premium Coal Sample Program
A. G. Engelkemeir
Tenth ACL Technical Meeting, Argonne National Laboratory, April 18, 1989

ASTM Methods for Trace Element Analysis
P. C. Lindahl
International Conference on Trace Elements in Coal, Bowling Green, KY, September 11-13, 1989

Comparison of Supercritical Fluid and Solvent Extracted Coal Using Supercritical Fluid Chromatography
A. S. Boparai, D. V. Applegate, M. D. Erickson, L. A. Raphaelian, and I. W. Frank
The Society for Applied Spectroscopy-Chicago Section Poster Session, Northwestern University, Evanston, IL, February 14, 1989

Continuous Emission Monitoring of Hazardous Waste Incinerators Using Fourier Transform Infrared Spectroscopy
J. C. Demirgian
1989 Pittsburgh Conference and Exposition, Atlanta, GA, March 6-10, 1989

Continuous Emission Monitoring of Hazardous Waste Incinerators Using Fourier Transform Infrared Spectroscopy
J. C. Demirgian and M. D. Erickson
1989 Incineration Conference, Knoxville, TN, May 1-5, 1989

CW Verification Concepts
D. W. Green
Verification Technology Conference, Los Alamos, NM, August 29-31, 1989

Determination of Low-Level Radioactivity in Environmental Samples by Gamma Spectroscopy at Argonne National Laboratory
W. E. Streets, R. R. Heinrich, and L. L. Lamoureux
34th Annual Conference on Bioassay, Analytical, and Radiochemistry, Las Vegas, NV, October 17-21, 1988

Determination of Low-Level Radioactivity in Environmental Samples by Gamma Spectroscopy at Argonne National Laboratory
W. E. Streets, R. R. Heinrich, and L. L. Lamoureux
Ninth ACL Technical Meeting, Argonne National Laboratory, November 1, 1988

Determination of Refractory Actinides in Environmental Samples: Is Total Dissolution Necessary?
L. L. Vetter, F. Markun, and T. TenKate
34th Annual Conference on Bioassay, Analytical, and Radiochemistry, Las Vegas, NV, October 17-21, 1988

Determination of Refractory Actinides in Environmental Samples: Is Total Dissolution Necessary?
L. L. Wetter, F. Markun, and T. TenKate
Ninth ACL Technical Meeting, Argonne National Laboratory, November 1, 1988

Development of Computer-Generated Data Packages for EPA Analyses
J. C. Demirgian and M. C. Hansen
1989 Pittsburgh Conference and Exposition, Atlanta, GA, March 6-10, 1989

Development of Computer-Generated Data Packages for EPA Analyses
J. C. Demirgian
Tenth ACL Technical Meeting, Argonne National Laboratory, April 18, 1989

Development of LEU Targets for ^{99}Mo Production and their Chemical Processing Status 1989
G. F. Vandegrift, J. D. Kwok, D. B. Chamberlain, J. C. Hoh, W. E. Streets, S. Vogler, H. R. Thresh, R. F. Domagala, T. C. Wiencek, and J. E. Matos
International Meeting on Reduced Enrichment for Research and Test Reactors, Berlin, Federal Republic of Germany, September 10-13, 1989

Gas Chromatography/Matrix Isolation-Infrared Spectrometry Applications to the Analysis of Fossil Fuel Mixtures
J. F. Schneider and L. A. Raphaelian
The Society for Applied Spectroscopy-Chicago Section Poster Session, Northwestern University, Evanston, IL, February 14, 1989

ICP/AES: A Modern Analytical Tool
C. S. Sabau
14th Annual ARA Congress, Los Angeles, CA, April 20-22, 1989

ICP/AES of the Actinides and Impurities in Plutonium
D. L. Bowers and E. A. Huff
32nd Annual Plutonium Exchange Meeting, Sacramento, CA, October 25-27, 1988

ICP/AES of the Actinide Elements
E. A. Huff
Plasma Spectroscopy Task Group Meeting, American Society for Testing and Materials, Orlando, FL, January 23-29, 1989

Implementing Measures for U.S. EPA Contract Laboratory Program (CLP) Quality Assurance/Quality Control (QA/QC) in the Inorganic Analytical Laboratory
P. C. Lindahl
Society for Applied Spectroscopy Atomic Absorption Workshop, Rosemont, IL, April 5, 1989

Isolation and Purification of Uranium from Environmental Materials for Mass Spectrometric Analysis
A. M. Essling, D. G. Graczyk, and E. P. Horwitz
34th Annual Conference on Bioassay, Analytical, and Radiochemistry, Las Vegas, NV, October 17-21, 1988

Isolation and Purification of Uranium from Environmental Materials for Mass Spectrometric Analysis
A. M. Essling, D. G. Graczyk, and E. P. Horwitz
Ninth ACL Technical Meeting, Argonne National Laboratory, November 1, 1988

Multivariate Statistical Evaluation of Results of Studies of Dioxins from Pulp Bleaching
S. E. Swanson and C. Rappe
Ninth International Symposium on Chlorinated Dioxins and Related Compounds, Toronto, Canada, September 17-22, 1989

The Production of ^{49}V, ^{93}Mo, ^{93m}Nb, and Other Long-Lived Isotopes in Fusion Materials with 14 MeV Neutrons
L. R. Greenwood and D. L. Bowers
Specialists' Meeting on Neutron Activation Cross Sections for Fission and Fusion Energy Applications, Argonne National Laboratory, September 13-15, 1989

Qualitative and Quantitative Analysis of Gas Mixtures Using Fourier Transform Infrared Spectroscopy
J. C. Demirgian and M. D. Erickson
13th Annual Environmental Quality R&D Symposium, Williamsburg, VA, November 15-17, 1988

Quantitation with GC/MI-IR
S. E. Spiro
Tenth ACL Technical Meeting, Argonne National Laboratory, April 18, 1989

Radium in Drinking Water
F. Markun
Tenth ACL Technical Meeting, Argonne National Laboratory, April 18, 1989

Utilization of Gamma Spectrometry and ICP/AES for the Monitoring of Plutonium Recovery from Intractable Residues
R. R. Heinrich, W. E. Streets, D. L. Bowers, and C. S. Sabau
Second Annual Karlsruhe International Conference on Analytical Chemistry in Nuclear Technology, Karlsruhe, Federal Republic of Germany, June 5-9, 1989

C. Awards and Patents

Antoinette G. Engelkemeir
University of Chicago Outstanding Service Award for her exceptional performance in providing high-quality gas mass spectrometry data to researchers and for her contributions to understanding the data she provides.

D. Meetings Attended

Amrit S. Boparai
OSWER Conference, Oakland, CA, October 25-28, 1988

1989 Cryolect Users Group Meeting, Argonne National Laboratory, May 14-17, 1989

Mitchell D. Erickson
1989 Cryolect Users Group Meeting, Argonne National Laboratory, May 14-17, 1989

David W. Green
Ninth Analytical Laboratory Managers Association Meeting, Oak Ridge, TN, October 26-28, 1988

1989 Cryolect Users Group Meeting, Argonne National Laboratory, May 14-17, 1989

Mary H. Picel
OSWER Conference, Oakland, CA, October 25-28, 1988

Stephen E. Swanson
1989 Cryolect Users Group Meeting, Argonne National Laboratory, May 14-17, 1989

E. Professional Organization Leadership

David W. Green
Member of Board of Directors, Analytical Laboratory Managers Association

President, Argonne Chapter of Sigma Xi

John F. Schneider
Chairman, 1989 Cryolect Users Group

Florence P. Smith
Chairman, Organizing Committee for the Annual Conference of the National Organization of Black Chemists and Chemical Engineers (NOBCChE)

F. ACL Seminars

Counting Atoms via Resonance Ionization Mass Spectroscopy: Implications for Surface Analysis
Dr. Dieter M. Gruen, Argonne National Laboratory, Chemistry Division
October 11, 1988

Direct Analysis of Solids by ICP and Glow Discharge Atomic Emission Spectrometry
Dr. I. Brenner, Geological Survey of Israel
October 27, 1988

Optimization of Polyethylene Pipe for Natural Gas Distribution Systems
Dr. John E. Young, Argonne National Laboratory, Chemical Technology Division
November 18, 1988

Atmospheric Studies of Radon and Lead-210 in the Pacific
Prof. Jack Kay, Drexel University
December 15, 1988

Origin and Migration History of Silurian-Devonian Formation Waters in the Illinois Basin
Prof. Alan M. Stueber, Southern Illinois University
March 14, 1989

Is There QA Life After the DOE Survey?
William R. Laing, Oak Ridge National Laboratory
April 6, 1989

Matrix FTIR Spectroscopy of Transient Species
Dr. Lester Andrews, University of Virginia
May 15, 1989

Matrix Isolation and GC: Oh, How Can It Be?
Gerald T. Reedy, Argonne National Laboratory, Analytical Chemistry Laboratory
May 15, 1989

Dioxins in the Bleach Plant
Dr. Stephen E. Swanson, Argonne National Laboratory, Analytical Chemistry Laboratory
May 23, 1989

Particulate/Vapor Phase Interactions of Organic Air Pollutants
Terry F. Bidleman, University of South Carolina
June 15, 1989

Process Optimization Using On-Line Analysis
Robert A. Bredeweg, Dow Chemical Company
June 26, 1989