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ANALYTICAL CHEMISTRY LABORATORY
Progress Report for FY 1988

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MASTER

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

The Instrumental Analysis Group uses nuclear counting techniques in radiochemical analyses on a range of types of samples from low-level environmental samples to highly radioactive ones. Other types of analyses done are 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 spectrometers, a GC/matrix-isolation FTIR 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 1988 are listed in Table 1.

Table 1. Distribution of ACL Work Load for FY 1988 among the Major Divisional Users

ANL Organization/Source	% ACL Effort	
Chemical Technology (CMT)	73.4	
Environmental Survey Program		57.5
Other		15.9
Environment, Safety, and Health (ESH)	3.7	
Energy and Environmental Systems (EES)	3.6	
Chemistry (CHM)	2.8	
Materials and Components Technology (MCT)	2.3	
ACKs (outside procurements)	5.3	

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 previous years, both the funding from CMT and the total funding for the ACL have increased, as shown in Fig. 2. The ACL continued to provide analytical support for programs in ESH, EES, CHM, MCT, Reactor Analysis and Safety (RAS), Environmental Research (ER), and other ANL divisions, as well as nearly $190,000 of technical services for non-ANL clients.

The ACL recovered 97.6% of its total operating costs in FY 1988. Over the last four years, the ACL has recovered 99.2% of its costs. The ACL effort, including divisional administration, includes about one-half recoverable time (sales to users in Fig. 3). The total operating costs for FY 1988, shown in Fig. 4, are largely effort related.

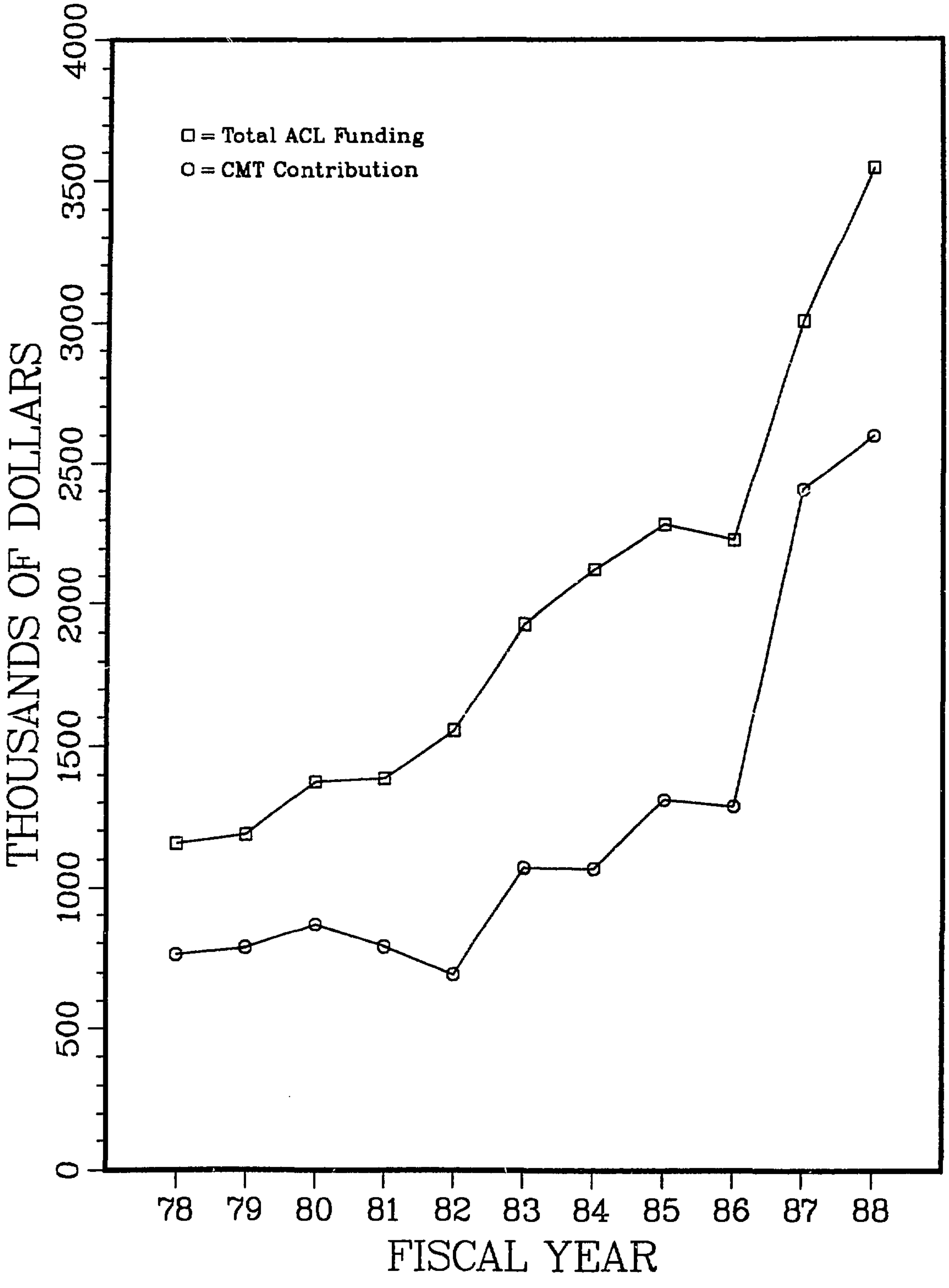

Fig. 2. ACL Funding History, Total and CMT

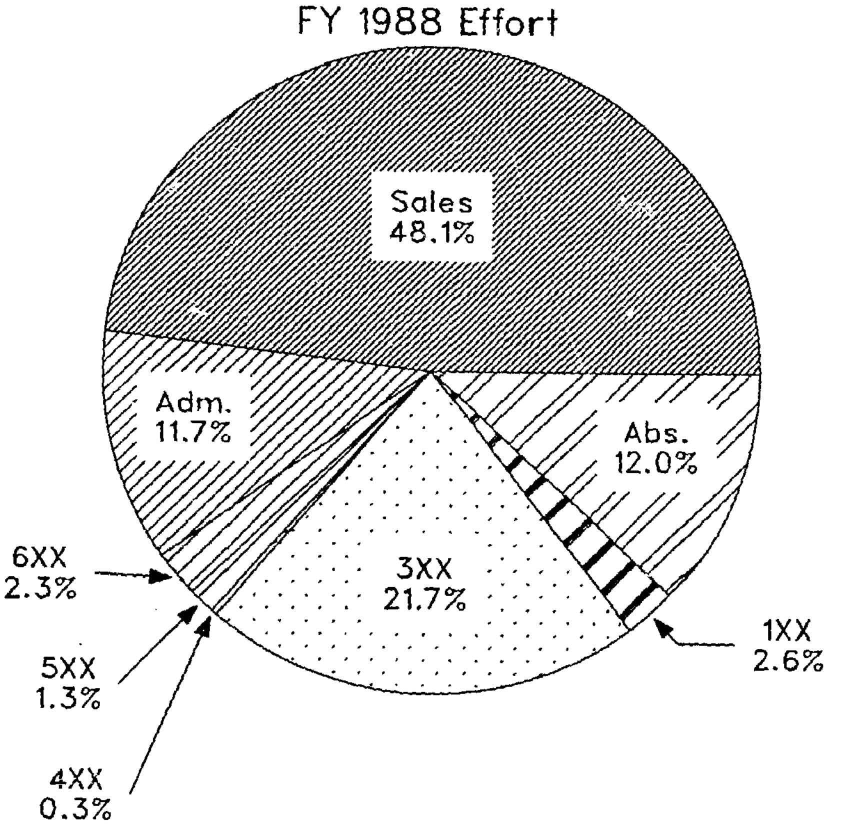

Fig. 3. Analytical Chemistry Laboratory
FY 1988 Effort

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

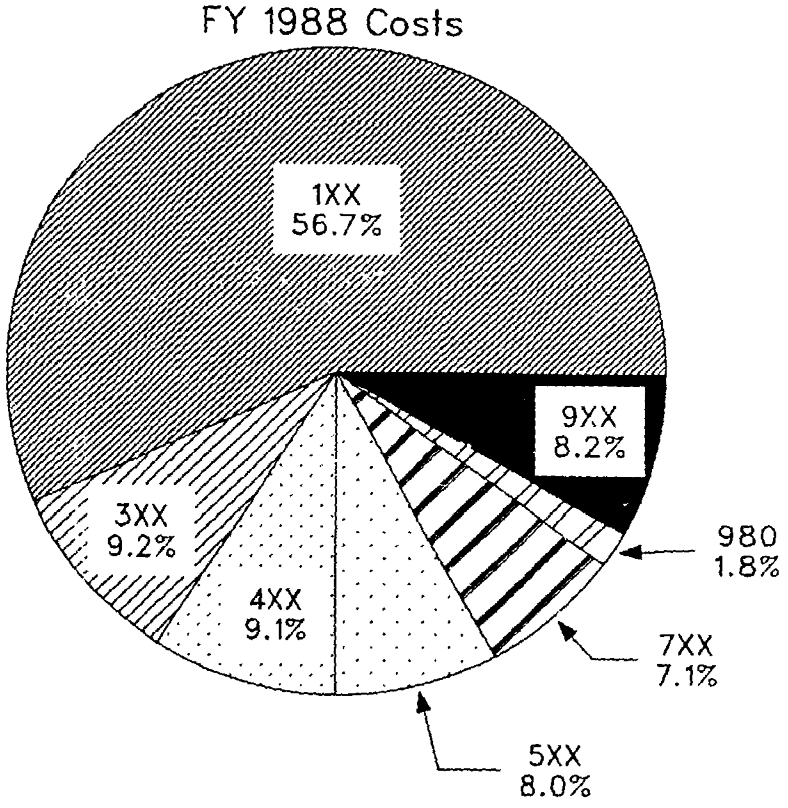

Fig. 4. Analytical Chemistry Laboratory
FY 1988 Total Operating Costs

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 Special Term Appointees (STAs) (see Fig. 1) in FY 1988 to assist with load leveling. About two full-time equivalents were used in FY 1988, and continued help from STAs is anticipated in FY 1989. One intern (IIT's Women in Science - Reentry in Analytical Chemistry Program), three co-op students, one postdoctoral student, two students, and two Faculty Research Leave Appointees supplemented the regular ACL staff.

The ACL continued a small internal funding system to support selected projects leading to publication and development of new capabilities. Projects supported in FY 1988 include the measurement of long-lived radionuclides in fusion materials, mass spectrometry of long-lived isotopes, analysis of PCBs in water-based "oils," quantitative analysis of organic samples by gas chromatography/matrix isolation-infrared spectrometry, and a comparability study of packed and megabore columns for quantitation of PCB/pesticides.

New capabilities of the ACL established in FY 1988 include a rotating anode X-ray generator for the X-ray diffraction apparatus, an infrared microscope, an atomic absorption spectrophotometer for trace element determinations, and a combination gas chromatograph/matrix isolation-infrared mass spectrometer.

III. TECHNICAL HIGHLIGHTS

Engineering Studies of Pyrochemical Processes for Integral Fast Reactor (IFR) Fuels (D. L. Bowers, A. G. Engelkemeir, E. A. Huff, 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.

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. However, where high precision and accuracy are required, the method of choice is still mass spectrometric isotope dilution (MSID). Samples containing no plutonium, which are from processes studying fission product distribution coefficients in the Cd anode and the electrolyte, are also analyzed by ICP/AES using the instrument located in Building 200.

Studies being conducted in CMT to determine the best waste form for these processes have also utilized the ACL. These studies are testing the gamma irradiation behavior of various waste forms and have required mass spectrometric gas analysis 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 fuel reprocessing of the Integral Fast Reactor (IFR) will be contacted with Cd-Li-K alloy 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 that will yield radioactive waste with high compressive strength and low leachability. The leach tests are performed by the standard ANSI6.1 procedure. Leachates are submitted to the ACL. Ion chromatography is used to measure the amount of Cl^- present, which determines the leach rate from the grout.

During FY 1988, approximately 700 samples were analyzed for all the CMT engineering groups associated with this program, with almost 300 samples from electrorefining studies, 200 from fission product distribution studies, and 200 from waste studies. Each of these samples required analysis 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, D. L. Bowers, 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, P. E. Melnicoff, S. H. Newnam, M. H. Picel, E. G. Rauh, L. E. Ross, J. A. Ruskamp, J. F. Schneider, C. Seils, F. P. Smith, C. T. Snyder, W. E. Streets, L. F. Sytsma, T. TenKate, L. L. Wetter, and R. J. Wingender)

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 (EES and ESH staff; S. Ballou, Program Manager) staffed one of the six field sampling teams in the program, and 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) using 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 type of required data is unique. The methodologies for the organic and inorganic analyses [many of which follow the EPA Contract Laboratory Program Statement of Work (CLP SOW)] and 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.

Analytical support for the Survey in FY 1988 included the receipt of approximately 3200 environmental samples for various combinations of inorganic, organic, and radiological analyses. For samples collected by the ANL sampling teams, the ACL has completed inorganic, organic, and radiological analyses of 420 samples from the Portsmouth Uranium Enrichment Complex, Piketon, OH, inorganic and organic analyses of 310 samples from the Kansas City Plant, Kansas City, MO, and inorganic, organic, and radiological analyses of 690 samples from the Y-12 Plant, Oak Ridge, TN. In addition, the ACL has provided technical support to other laboratories, including:

(1) Idaho National Engineering Laboratory (INEL)
   a. 500 samples from the Hanford Nuclear Reservation (Richland, WA) for inorganic and organic analyses.
   b. 220 samples from the Sandia National Laboratories (Albuquerque, NM) for inorganic and organic analyses.

(2) Battelle-Columbus Division/Pacific Northwest Laboratories (BCD/PNL)
   a. 150 samples from the Pinellas Plant (Largo, FL) for inorganic analyses.
   b. 120 samples from the Nevada Test Site (Mercury, NV) for inorganic and radiological analyses.

(3) Oak Ridge National Laboratory (ORNL)
   a. 240 samples from INEL (Idaho Falls, ID) for organic analyses.
   b. 210 samples from the Morgantown Energy Technology Center (Morgantown, WV) for inorganic, organic, and radiological analyses.
   c. 30 samples from the Brookhaven National Laboratory (Upton, NY) for inorganic analyses.

Additional Survey activities have required ACL to participate in several external QC programs (see Section IV of this report): in the inorganic and organic analysis areas, the EPA's Superfund Contract Laboratory Quarterly Performance Evaluation Studies; in the inorganic analysis area, the EPA's semiannual Water Pollution Performance Evaluation Studies; and in the radiological analysis area, the Environmental Measurements Laboratory's Quality Assessment Program and the U.S. EPA EMSL-LV's Intercomparison Study Program.

Detector Upgrade of Thermal Ionization Mass Spectrometer (E. G. Rauh and D. G. Graczyk)

A Daly scintillation detector was added to the VG Isotopes Model 54R thermal ionization mass spectrometer (TIMS) to complement the Faraday cup detection system featured on the original instrument. The Daly detector possesses a sensitivity approaching 10^{-17} A and permits measurement of isotope ratios on samples two to three orders of magnitude smaller (20 to 50 ng) than those required with the Faraday cup.

Applications for the Daly detector at present include isotopic measurements related to environmental or other samples where only tiny quantities of the isotopes of interest are available, and to elements whose ionization characteristics limit the intensity of ion beams achievable with thermal ionization. We have successfully used the new detector for isotopic analysis of uranium in DOE Environmental Survey samples, and in the isotope-dilution determination of U and Th in samples that contained submicrogram amounts of these elements (see below). The precision and accuracy currently available with the Daly detector are limited by our knowledge of mass discrimination effects and by the nonlinearity in the measurement system to a few per cent, relative to each measured isotope ratio. We expect to improve on the reliability of the Daly data as we carry out experiments to characterize the detector's response and account for any nonideal behavior by means of appropriate data-reduction algorithms.

Accelerator Mass Spectrometric Dating with Calcium-41 (A. M. Essling, K. J. Jensen, and E. G. Rauh)

Argonne physicists (W. Kutschera and co-workers, PHY) are using the ATLAS accelerator to develop a new method for determining the age of fossil bones by measuring the amount of radioactive ^{41}Ca that the bones contain. The ^{41}Ca is determined by accelerator mass spectrometry (AMS) relative to ^{40}Ca, the most abundant stable isotope in natural calcium. Ratios of ^{41}Ca/^{40}Ca as low as 10^{-15} are measured with the AMS technique. In support of this work, the ACL prepared a series of isotopic standards for validating the AMS isotope ratio measurements. Preparation of these materials required (1) diluting a ^{41}Ca-enriched (1.23 atom % ^{41}Ca) stock material (in solution) with natural calcium to provide known ^{41}Ca/^{40}Ca atom ratios of approximately 10^{-11}, 10^{-12}, and

10^{-13}; (2) precipitating the calcium isotope mixtures with oxalate; and (3) igniting the product to CaO. A natural $^{41}Ca/^{40}Ca$ blank ($^{41}Ca/^{40}Ca$ ~10^{-15}) was carried through the entire procedure along with the standards. The AMS measurements made with these materials proved quite successful, demonstrating the validity of the AMS determinations on calcium isotopes at naturally occurring levels.

Additional analytical support was provided to the ^{41}Ca dating program through determining the U and Th content of a number of samples, including three limestones, four ancient bones, and three samples of tar sand that surrounded the bones before collection. The actinide elements are of interest in the dating experiments because they may produce neutrons by spontaneous fission or (α,n) reactions. These neutrons may be captured by ^{40}Ca in the sample materials to give ^{41}Ca, which then complicates interpretation of the dating experiments. Two grams or less of each sample was spiked with 2 μg of ^{235}U and 5 μg ^{230}Th and was digested with mixed mineral acids. The U and Th in each digestate were isolated and 10 to 50 ng of each element was loaded on a mass spectrometer filament for isotopic analysis with a Daly scintillation detector. From the measured ratios of natural U or Th to the added spike isotopes, the concentration of each actinide in each sample was calculated. Concentrations ranged from less than 0.1 ppm to 19 ppm in the different samples and were determined to better than 10% in almost all cases. Measurements of such low concentrations in such small samples would not have been possible in previous years when the Daly detector and special separations procedures were unavailable in the ACL.

<u>Gas Analysis for Argonne Premium Coal Sample Program</u> (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 at intervals of the cover gas (N_2) of sealed ampoules of coal to determine their stability. The presence of O_2 is of particular concern, but also of interest are changes in the amounts of CH_4, CO and CO_2, since they may signal chemical or biological degradation of the coal material with time. To date the ACL has analyzed the gas from more than 100 such ampoules.

New Method of Isolating and Purifying Uranium for Isotopic Analysis

(A. M. Essling)

Most existing methods that isolate and purify uranium in one step for mass spectrometric analysis are limited in the number of cations that can be separated quantitatively from uranium. As a result it is necessary to apply several steps in preparing the pure uranium sample required for deposition on the mass spectrometer filament. Isolating and purifying uranium are especially difficult for environmental samples where, often, microgram quantities of U must be separated from large amounts of complicated matrix material. In collaboration with E. P. Horwitz (CHM), we have established a new procedure that utilizes column chromatography on a support material coated with diamyl amylphosphonate. This technique is capable of isolating microgram quantities of U from several grams of matrix material with consistently high recovery (approximately 95%). The uranium product is exceptionally pure and is suitable not only for mass spectrometric analysis but also for determination by other methods such as laser fluorimetry or radioactive counting techniques. We have successfully applied this separation to a variety of dissolved or digested samples (including waters, sludges, sediments, soils, ancient bones, limestones, and vegetation).

Analytical Support for Research on High-Temperature Superconductors

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

Extensive analytical support was provided to superconductivity programs in MCT and CMT. This work principally involved analysis of starting materials, process samples, and products related to fabrication and characterization studies of $YBa_2Cu_3O_{7-x}$ ceramics or other ceramic compositions. This analysis included determining elemental compositions by ICP/AES, anions by ion chromatography, and the oxygen coefficient (i.e., the value of 7-x in the formula $YBa_2Cu_3O_{7-x}$) in selected ceramics by an iodimetry method.

Determination of the oxygen coefficient was established within the ACL as a new capability this year. It was developed from the method of Appelman, et al. of ANL's Chemistry Division [Inorg. Chem. 26, 3237 (1987)], which takes advantage of the relationships between the oxygen coefficient and the valence state of Cu in the sample as measured by iodimetry. We enhanced the published procedure by employing an autoburet for the critical titration of iodine with thiosulfate and by using a specialized colorimeter to detect the tricky starch-blue to copper-blue titration endpoint.

In addition to supporting research in the area of superconducting ceramics, the ACL made substantial contributions to ongoing work in MCT to characterize thin-film superconductors. Elemental composition of thin films, often containing only a few micrograms of material, was characterized by the ICP/AES technique. The results of these efforts were presented at the Spring Meeting of the Materials Research Society. A publication on the same subject was submitted to Physics Letters.

Groundwater Analysis for ANL Geosciences Programs (R. W. Bane, A. M. Essling, I. M. Fox, E. A. Huff, and F. P. Smith)

Groundwater samples have been routinely analyzed by the ACL to determine low-level cations by ICP/AES or atomic absorption and anions (Cl^-, F^-, NO_3^-, $SO_4^=$) by ion chromatography. In response to special needs of ANL's geoscientists, several additions have been made to ACL's capabilities in the area of groundwater analysis. Procedures were established for the ion-chromatographic determination of very low levels of iodide and bromide, which were of interest to geochemists (N. Sturchio, CMT) studying hot springs in

Yellowstone Park. In addition, an automated method for determining the hydroxide, carbonate, and bicarbonate alkalinity of groundwater samples, as defined by ASTM Method 403, was established; this method uses our Metrohm Model 670 titroprocessor to provide fully automated titration of each sample and calculation of the alkalinity results.

The sensitivity of the alkalinity titration proved inadequate for a group of geochemists (T. Beasely, CER) that was studying a deep aquifer by measuring ^{36}Cl and ^{14}C isotopes in the groundwater. Because the acquired isotopic data suggested an intrusion of surface water into the deep aquifer, this group wished to confirm this finding by demonstrating an increase in inorganic carbon content of the groundwater at the point of intrusion. However, the inorganic carbon concentrations before and after the intrusion were lower than the titrimetric limit of detection for carbonate species. To measure these very low concentrations, the ACL set up an analysis train wherein an acidified groundwater sample could be boiled under a purified helium purge, and the CO_2 released by the sample could be collected in a calibrated volume and measured manometrically. This method has the ability to determine inorganic carbon in groundwater at levels well below 1 µg C/mL, at least ten times lower than can be measured by titration. The results obtained from the deep aquifer samples correlated well with the ^{36}Cl and ^{14}C isotopic results and corroborated the hypothesis of surface-water intrusion into the aquifer.

Analytical Support to IPNS Enriched Uranium Booster Target Project (A. M. Essling, E. A. Huff, K. J. Jensen, and E. G. Rauh)

Total ^{10}B content was determined in a series of samples in order to satisfy QA/QC specifications prior to the loading of uranium into a target vessel for a Uranium Booster Target Project experiment. The ^{10}B was determined by an MSID technique after the ^{10}B had been chemically separated from copper, the material used to clad boron powder. The MSID procedure, while very accurate, requires a boron sample essentially free from other elements prior to analysis. After investigating, with little success, a number of ways to make the required boron/copper separation, we finally developed an ion-exchange separation procedure that effectively separates boron from other elements, allowing for precise mass spectrometric boron analysis to provide isotopic-ratio and total-boron-content values. This separation technique is expected to be very useful in future analysis of boron in complex materials.

ACL Support to the Molten Corium Concrete Interaction (MCCI) Program

(A. M. Essling, L. B. Gillis, E. A. Huff, K. J. Jensen, J. A. Ruskamp, and F. P. Smith)

In MCCI experiments, mixtures of uranium dioxide, zirconium, concrete, steel, and fission-product elements are heated to temperatures simulating reactor meltdown conditions. Samples of solidified melt, aerosols collected by impaction or on filters, and gases are examined to study interactions and transport of the simulated reactor materials. The ACL had previously developed specialized procedures for dissolution of the highly refractory materials that are generated in these experiments. The ACL provided detailed analytical data on both major and minor constituents in the samples. 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, and for Si by atomic absorption spectrometry (AAS). Gas samples are analyzed by mass spectrometry. Results for samples obtained during intermediate-scale tests performed during FY 1987 provided some of the most useful data ever produced on aerosol releases, largely as a result of our capability to determine small amounts of such elements as Ba, Sr, and La in small aerosol samples composed mainly of other elements.

This fiscal year, samples from the first of three scheduled large-scale MCCI tests have been analyzed. Determinations included measurement of anions (chloride, nitrate, sulfate) by ion chromatography in some of the aerosol samples. The levels of anions in a few aerosol samples were surprisingly high (several per cent) and contributed substantially to our achieving mass balance from the analytical data.

Analytical Support for ANL High-Sulfur Dry-Scrubber Test (I. M. Fox, S. H. Newnam, E. G. Rauh, L. E. Ross, J. A. Ruskamp, and F. P. Smith)

The ACL has provided analytical support for the ANL High-Sulfur Dry-Scrubber Test conducted for DOE by the EES Division. Feed materials and products were subjected to a variety of analyses, including determination of available lime; total alkalinity; total sulfur and sulfite, nitrite, nitrate, sodium, calcium and silicon; and moisture. Approximately 650 determinations were made during FY 1988 in support of the test.

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

Analytical support for the NAPAP program continued this year. Test briquettes, exposed to a variety of atmospheric conditions at a number of sites throughout the country, are brought to ANL quarterly for sampling and analysis. Hundreds of specimens have been analyzed for anions (fluoride, chloride, nitrate, and sulfate) by ion chromatography and for metals by ICP/AES.

Spent-Fuel Leaching Tests (A. M. Essling, E. A. Huff, E. G. Rauh, and F. P. Smith)

In the Spent Fuel Leaching Tests (E. Veleckis, CMT), leaching behavior of irradiated nuclear fuel by groundwater is being investigated. This year, a preliminary experiment, using nonirradiated UO_2 powder as a stand-in for irradiated fuel, was performed to develop and evaluate procedures for conducting the tests with irradiated material. Samples of the groundwater leaching solution were periodically taken for analysis while the water was in contact with the UO_2 over a period of several months. These groundwater samples were analyzed by ICP/AES for cations and by ion chromatography for anions. Uranium in each sample was determined by either laser fluorimetry or MSID. Uranium isotopic measurements performed as part of the MSID assays showed anomalous behavior in that the isotopic composition (i.e., the ratio of

^{235}U to ^{238}U) of uranium in the leachates differed from that in the original UO_2 powder and changed with time. The explanation for this anomaly was found in the physical composition of the UO_2 powder, which had been prepared by mixing ^{235}U-enriched UO_2 with ^{238}U-enriched UO_2 to produce a mixed powder of a desired $^{235}U/^{238}U$ ratio. Apparently, particles of the different isotopically enriched powders had different morphologies and dissolved at different rates, producing the observed isotopic segregation. In addition, the isotopic segregation persisted when the uranium concentrations in the leachate had reached a steady state, suggesting that the leaching of UO_2 by groundwater may be strongly dependent on the physical as well as chemical properties of the fuel material. Thus, these isotopic data, although not specifically called for in the original test plan, provided unexpected insight into the leaching process and may have important implications for the interpretation of future test results.

Transuranic Extraction (TRUEX) Process Flowsheet Development (E. A. Huff)

The TRUEX program is under the direction of G. Vandegrift (CMT) and consists of the development of a solvent-extraction process for the purpose of separating and concentrating transuranic elements from nuclear wastes. Development of the TRUEX process flowsheet requires determination of the extraction behavior (distribution coefficients, interactions, etc.) of a large number of elements that are anticipated in the wastes to be processed. Enormous quantities of data describing element concentrations in various process stages are being acquired to generate and validate computer models of the TRUEX flowsheet. Hundreds of samples have been analyzed by ICP/AES, with each sample containing ten or more elements for determination. The multielement capability of the ICP/AES system, combined with its wide dynamic range for determination of elemental concentrations, is well suited to this type of measurement and has contributed to the accumulation of an extensive flowsheet data base in a relatively short time.

Industrial Cogeneration: 2-Methylpyridine and Toluene Thermal Degradation Studies (J. C. Demirgian and C. T. Snyder)

Thermal degradation studies were completed for 2-methylpyridine and toluene and the results reported.(1,2) These studies included thermally degrading these fluids in dynamic Organic Rankine cycle loops at several temperatures (300°C to 360°C) followed by complete chemical characterization. Characterization consisted of establishing gas chromatographic separation methods and qualitative identification by GC/MS and GC/IR. Software capable of correlating the GC, GC/MS, and GC/IR data and quantitating the results with external standards has been developed.

Polyurethane Foam Pyrolysis Studies (J. C. Demirgian)

The pyrolysis of selected polyurethane foams was conducted at 210°C and 310°C. Preliminary work consisted of designing the vacuum chambers and conditions for pyrolysis and then chemically characterizing the pyrolysis products. Characterization was performed by GC, GC/MS, and GC/MI/FTIR analysis. The degradation products were compared at two temperatures for foams prepared in air and in vacuum. It was shown that the preparation of the foam was critical in enhancing its thermal stability. In addition, pyrolysis products in vacuum were significantly different than those obtained in air.

FTIR Microscopy (J. C. Demirgian)

The ACL has expanded its capabilities by purchasing an FTIR microscope. This research instrument has a spatial resolution of 10 microns and can be used in reflectance and transmission modes. Users from ANL may operate this instrument themselves after becoming trained on it.

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1. R. L. Cole, J. C. Demirgian, and J. W. Allen, Argonne National Laboratory Report No. ANL/CNSV-TM-202 (1987).

2. R. L. Cole, J. C. Demirgian, and J. W. Allen, Argonne National Laboratory Report No. ANL/CNSV-TM-192 (1987).

Comparison of PCB/Pesticide Quantitation Using Packed and Megabore Columns (M. C. Hansen)

The ACL is measuring polychlorinated biphenyls (PCBs) and pesticides in the DOE Environmental Survey Program, which uses EPA Contract Laboratory Program (CLP) methods. For PCB/pesticides, CLP requires gas chromatograph/electron capture detector (GC/ECD) analysis with packed and megabore chromatographic columns. Currently, samples are quantitated on an EPA-required packed column, while the identification is confirmed by the retention time on a megabore column. The objective of the study was to compare PCB/pesticide quantitation using packed versus megabore columns with regard to precision, accuracy, and resolution capabilities. The results of this study, which included the analysis of over 100 environmental samples by both methods, justified the discontinuance of packed column analysis and the implementation of the megabore quantitation method.

Development of Automated Software for PCB/Pesticide CLP Analysis (J. C. Demirgian and M. C. Hansen)

The analysis of PCB/pesticides by CLP methods requires working with a large data package, which had been hand calculated and hand generated. Software, based upon dBASE III Plus, was developed to automate calculating of results and printing of the forms needed for the data package. This software package is now routinely employed in the ANL PCB/pesticide analysis program. The software was designed to report pesticides and was appended to report PCBs. Software to report PCBs using LOTUS 1-2-3 will be developed to further automate the analysis system.

Advanced Instrumental Methods for Analyzing Organics in Solid Waste (L. A. Raphaelian, A. S. Boparai, and J. F. Schneider)

The objective of this program is to use advanced separation and instrumental techniques to characterize those organics in complex mixtures that cannot be analyzed by GC/MS.

In the companion program "Characterization of Organics in Solid and Sludge Wastes from Emerging Energy Technologies," the organics in the wastes are being identified by GC/MS. Although GC/MS is one of the most powerful organic analytical techniques, it cannot solve analytical problems of certain complex organic mixtures. Two problems that arise in GC/MS analysis are (1) the complex mixtures cannot be separated by high resolution capillary column gas chromatography making mass spectral identification very difficult, and (2) many mass spectra are difficult to interpret unambiguously--e.g., the mass spectra of isomers are often indistinguishable.

To simplify complex mixtures that could not be analyzed directly by GC/MS, supercritical fluid chromatography (SFC) was used. This was done in two ways: (1) the extract was chromatographed to give four fractions and (2) the actual sample, in most cases a feed coal, was extracted in situ with the supercritical fluid (SFE) and chromatographed on-line to give four fractions. The SFC/SFE technique led to better recovery of the organics in the feed coal.

To examine those compounds whose mass spectra do not lead to positive identification, we used the technique of gas chromatography/matrix isolation-infrared spectroscopy (GC/MI-IR). The GC/MI-IR technique was found to have sensitivity of the order of GC/MS and was useful for identifying the isomers of dimethyl naphthalenes in waste mixtures. Each isomer gave a unique infrared spectrum. The sharpness of the peaks in the fingerprint region (as is characteristic of MI-IR spectra) was paramount to obtaining positive identification of the compounds. A study was made on whether overlapping peaks of the dimethyl naphthalenes could be quantitated. It was found that, except for isomers present in small concentrations relative to other isomers, good results could be obtained.

A paper describing parts of this study and entitled, "Gas Chromatography/ Matrix Isolation-Infrared Applications: The Identification of C_2 Naphthalene

Isomers in Complex Fossil Fuel Mixtures," has been submitted for publication in the Journal of Chromatographic Science.

Plastic Pipe Program (L. A. Raphaelian, M. D. Erickson, J. F. Schneider, S. Siegel, and B. S. Tani)

The objective of this Gas Research Institute Program (J. Young, CMT, Project Manager) is to determine and characterize the chemical and molecular properties of polyethylene (PE) resins and to determine the effect of these properties on the mechanical strength of commercial PE pipe presently used for gas distribution. The ultimate purpose of this activity is to characterize the chemical and mechanical properties and correlate these properties with the mechanical strength of pipes, as determined by a standard mechanical test, and then use this information to control, improve, and optimize the mechanical properties and long-term performance of the pipe for use in gas distribution systems.

Carbon-13 nuclear magnetic resonance (^{13}C-NMR) spectroscopy was used to determine the type and degree of branching in the polyethylene. Most commercial pipes had 1 to 4 n-butyl or ethyl branches per 1000 backbone carbon atoms. Generally, pipes with longer branches (n-butyl) and/or more branches performed better in the CTL test than pipes with shorter branches (ethyl) and/or fewer branches. The degree and length of branching varied inversely with the density. It is believed that the branching interferes with crystal formation; single polyethylene molecules crystallize in more than one crystal lattice linking crystals together. Such polyethylene molecules are called "tie" molecules and lead to greater strength in the polyethylene pipe.

A topical report was prepared by ANL and Phillips Petroleum describing methodology for determining molecular weight averages by GPC. This report was in response to the finding that there was poor reproducibility in GPC determination of molecular weight averages by commercial polymer testing laboratories. This work will be continuing with new pipe materials whose chemical properties have been more clearly defined.

Statistical analysis has confirmed a high degree of correlation between the mechanical strength of the pipes with molecular branch type and concentration, density, weight average molecular weight, and number average molecular weight.

Characterization of Organics in Solid and Sludge Wastes from Emerging Energy Technologies (L. A. Raphaelian, I. W. Frank, and J. F. Schneider)

The primary objective of the program is (1) to characterize, by narrow-bore capillary column GC/MS, the organics in solid and sludge wastes from the processes of emerging energy technologies, such as coal gasification and liquefaction and the extraction of oil from shale, and (2) to determine the potential of organics leaching from these wastes after disposal. With a knowledge of the organics in these wastes and their potential for leaching, strategies for treatment of the waste before disposal can be formulated.

Twenty-four waste samples were analyzed. They included feed coals, pulverized feed coal, equalization tank sludge, pitch, tar, and heavy distillates. Two types of analyses were done on each of these samples. The first type was a direct analysis in which the samples were extracted, typically with methylene chloride, and then analyzed by GC/MS. In the second, the samples were leached according to the extraction procedure developed by the EPA, and the leachates were extracted with methylene chloride and analyzed by GC/MS. The leaching procedure would be expected to simulate real situations. The types of compounds found in the wastes included n-alkanes and other alkanes, n-alkenes, cyclohexanes, cyclopentanes, alkyl benzenes, naphthalenes, indans, indenes, tetralins, decalins, biphenyls, acenaphthenes, acenaphthylenes, polynuclear aromatics (PNAs), thiophenes, furans, pyridines, phenols, nitro compounds, and aldehydes.

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

The ACL has participated in this program for approximately one year 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 Department of Energy 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. Then, metal impurities determined by ICP/AES. The impurity measurements of the individual laboratories are statistically evaluated for differences. The ACL has participated in two of these intercomparison studies. Each effort has resulted in metal impurity values that have been consistent with the stated target values being supplied by selected laboratories.

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

The presence of radium in public and private water supplies is currently a very sensitive public issue. Several methods exist for the determination of ^{226}Ra and ^{228}Ra in water, but they are very elaborate and time consuming. Using Argonne Support Development Funds, we have demonstrated the cost effectiveness of a newly developed ion exchange method utilizing a radium-selective Dowex resin. This method is much faster and less involved, but requires a larger sample size.

The water sample, preferably 20 L, is acidified with hydrochloric acid to approximately pH 3. The water is processed through the special Dowex ion exchange resin bed. The resin containing the radium is sealed in a metal can and aged before counting on a NaI detector. The collected counting data are processed by a least-squares gamma spectra analysis program. The sensitivity of this method is 0.5 pCi/L when a 20-L volume of water is used.

This ^{226}Ra and ^{228}Ra determination method was utilized for water samples submitted by N. Sturchio (CMT). The objective was to determine concentrations of ^{224}Ra, ^{226}Ra, and ^{228}Ra in water collected at various strategic points in Yellowstone National Park. In the field, large volumes of water were passed through the special Dowex resin bed, and then these resins were delivered to ANL. At ANL they were sealed in metal cans and counted shortly after collection. An early count was used to first determine ^{224}Ra ($t_{1/2}$ = 3.6 d); subsequently the other two radium isotopes were determined in the conventional manner.

In addition to this application, we have performed numerous radium analyses for municipalities and well drillers in the surrounding area. The reason for this interest is that the EPA's maximum allowable total radium content in drinking water is 5 pCi/L, and all municipalities with water concentrations higher than this level must take steps to comply with EPA guidelines.

Determination of Protactinium in Environmental Samples (L. L. Vetter and F. Markun)

Recently there has been growing concern regarding the increase in the production of protactinium (Pa), which is present as ^{233}Pa ($t_{1/2}$ = 27.4 d, β^-) in all nuclear waste containing its parent ^{237}Np and, more importantly, as ^{231}Pa ($t_{1/2}$ = 3.2 × 10^4 y, α) associated with the uranium-thorium fuel cycle. This interest evolves from the bone-seeking metabolic behavior of Pa, its low maximum body burden (0.1 μCi), and its long biological half life ($t_{1/2}$ biol. = 100 y).

Traditionally, literature methods for the determination of Pa have described the capricious nature of protactinium and have been plagued by low and sporadic yields of utilized tracers. Recently, the ACL has adapted and modified a procedure first suggested by Sill et al.(3,4) for a precise and accurate determination of Pa in environmental samples. This methodology includes the separation of Pa from nonradioactive matrix constituents, as well as from other interfering alpha emitters. Chemical techniques are employed to ensure complete exchange between utilized tracers and indigenous Pa. The protactinium is

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3. C. Sill, K. Puphal, F. Hindman, Anal. Chem. 46, 1725 (1974).

4. C. Sill, Anal. Chem. 38, 1458 (1966).

quantified by isotope dilution and alpha spectrometry. Tracer yields (70 to 80%) have been demonstrated to be highly satisfactory.

Lansdowne, Pennsylvania Project (L. L. Vetter, F. Markun, T. TenKate, W. E. Streets, L. L. Lamoureux, and R. R. Heinrich)

In the early 1900s, a professor from the University of Pennsylvania began to refine uranium ore to extract radium. His work continued for several years and at the peak of production he was a major supplier of radium worldwide for medical applications. Unfortunately, Professor Kabakjian chose to process the ore in the basement of his home. Consequently, his work led to widespread contamination of the household, its contents, and the property itself.

Presently, the Kabakjian property in Lansdowne, PA, is part of a Superfund cleanup project. A group of Argonne employees from Environmental Safety and Health (ESH) is coordinating the destruction and decontamination of the property. In support of ESH, the ACL is analyzing a wide range of sample types for numerous radionuclides. This project is challenging with regard to both the unusual sample types involved (floor carpeting, furniture pieces, bricks, etc.) and the number of analytes required (Th, Ac, Pa, U, and gamma emitters). These multiple-analyses must also be performed rapidly because removal of the contamination is a dynamic, ongoing process requiring coordination between scientific groups and demolition crews.

X-Ray Diffraction of Superconducting Materials (B. S. Tani and S. Siegel)

During the past year, X-ray diffraction (XRD) analysis of superconducting materials has increased (98 jobs out of 379 jobs for the period). XRD analysis is used for phase identification and a sample purity check of powder crystalline material. The minimum detection limit of a minor component is dependent on the matrix and crystal structure of the components and is approximately at the 5% to 10% level.

The $YBa_2Cu_3O_{6.9}$ phase, often referred to as the "123" orthorhombic phase, is superconductive at 90 K. However, the tetragonal form $YBa_2Cu_3O_6$ is not. XRD is used to distinguish between the two forms. In addition to identifying the "123" phase, XRD is used to determine the presence of impurity phases, i.e., CuO, $BaCO_3$, $BaCuO_2$, and "211" phase (Y_2BaCuO_6); their presence is an indicator of incomplete sample preparation.

Currently, scientists are studying the Bi-Sr-Ca-Cu-O (BSCCO) system. The phase diagram for this system is more complex than the Y-Ba-Cu-O phase diagram, which has been fairly well characterized this past year by scientists worldwide. In the BSCCO system, there are three superconducting phases that have been reported with T_c at about 6, 80, and 110 K. The XRD patterns of these phases are sufficiently different, and therefore, XRD is being used to monitor sample preparation procedures. There are also unknown XRD patterns of phases that often appear in the superconducting preparations. Although these phases are not yet characterized, information on just their presence is helpful to the investigators.

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

The principal effort in this category for FY 1988 has been the application of methodologies developed primarily last year for analyzing many important environmental radionuclides. Methodologies were developed for analysis 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-West, ANL-East (ES&H), and the DOE Environmental Survey Program. Approximately 200 samples collected from the two ANL sites were analyzed for most of the radionuclides listed.

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

Several laboratories are working jointly with ANL to devise an economical chemical process capable of recovering plutonium from all types of scrap and residue. The process for the removal of plutonium and other transuranic (TRU) materials from the waste must also be efficient enough that process effluents can be classified and disposed of as non-TRU waste. At ANL, liquid metals and molten salts at elevated temperatures are used as reagents and solvents to effect the necessary separations and chemical conversions. Separations are possible because of the differing stabilities of compounds in the molten salt phase relative to those in the liquid metal phase.

The ACL has significantly contributed to the success of this project by providing high-quality results in a number of areas. It is important to the process development to have reliable analyses of the residues used as feed material. The quantification of the plutonium in this type of sample is done by high resolution gamma spectrometry. Samples generated by the processes have been filtered salts and metals, as well as large metal and salt ingots. Primary elements of interest are Pu, Am, and Si which are determined by complete dissolution of the sample and subsequent analysis by ICP/AES. Small undissolved samples were also analyzed for Pu and Am by gamma spectrometry. Other cationic constituents such as Al, Mg, Cd, Cu, Zn, Ca, K, and Na are also of interest and are determined by ICP/AES. Monitoring of the effluents has indicated that some process systems, such as the copper-magnesium-calcium system, can remove as much as 99.8% of the plutonium from the feed material.

ACL Interaction with Division of Educational Programs (DEP) (W. E. Streets and B. S. Tani)

The ACL participated in the ANL/IAEA (International Atomic Energy Agency) Training Course on X-ray Analysis held at Argonne National Laboratory, January 25-May 13, 1988. One of the participants from Comision National de Energia Atomica in Argentina was then assigned to work in our laboratory until June to gain experience in energy-dispersive X-ray techniques and computer analysis of X-ray fluorescence data.

We have also continued to provide training and equipment in surface area measurements (BET) for graduate geology students from Northwestern University under grants through DEP.

Environmental Protection Agency (EPA) Project (J. F. Schneider and R. J. Wingender)

The EPA (Region V Office) supports a containment room in Building 205 for the handling and analysis of hazardous waste samples. The room is a unique facility that permits toxic sample handling 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) have been installed for the EPA's use. The EPA also continues to support a storage facility on site for legally sensitive samples.

The interagency agreement also gives the ACL the opportunity to participate in various EPA programs if they are within the scope and objectives of the ACL.

Advanced Mass Spectrometric Capability (R. J. Wingender and P. E. Melnicoff)

The ACL's state-of-the-art MS/MS instrument has the capability for GC/MS and tandem GC/MS/MS sample analysis with a mass range up to 2000. In addition, it is equipped with a high-performance liquid chromatography interface (plasma and thermospray) and a programmable solid probe. The instrument has the necessary software to provide forms and data in a format needed to meet the requirements of EPA's Contract Laboratory Program (CLP) for the analysis of priority pollutants in water and soil/sediment samples.

The sensitivity of the instrument is in the low picogram range, which is needed for the quantitation of polychlorodibenzo-p-dioxins and polychlorodibenzofurans (PCDDs and PCDFs). The MS/MS capability has been used to unequivocally determine the presence/absence of PCDD/PCDF in samples with co-extracted interferences of such magnitude that conventional multiple ion detection by GC/MS cannot resolve these compounds at the required detection limits.

The solid probe has been used to determine the mass spectra of impure samples, compounds that are not chromatographable (e.g., porphorines, amino acids), or high molecular weight compounds. The probe is temperature programmed in order to selectively boil off lower volatility components so that the mass spectrum obtained is not influenced by their presence.

The conventional GC/MS capability has been used to analyze samples for the DOE Environmental Survey Program following the CLP guidelines. The instrument was tuned to meet specific CLP criteria so that calibration standards could be reliably run. When all criteria were met, samples were analyzed and the data processed using the resident EPA software.

Analysis of Dioxins from Municipal Waste Incineration (R. J. Wingender and M. H. Picel)

A cooperative effort with the National Bureau of Standards (NBS) on analysis of dioxins from municipal waste incinerators has continued. The NBS effort has been to construct a small-scale incinerator for use in determining combustion conditions for municipal solid waste (MSW) that would minimize the formation of PCDDs and PCDFs. The ACL provided analysis of PCDDs, PCDFs, PCBs, and chlorinated benzenes in various preparations of MSW and in samples generated from the application of various combustion conditions. In addition, standard operating procedures for analysis of these analytes have been written.

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

The ACL, together with the EES Division, began a joint venture that involved the analysis of bottom sediments of the Chicago River and North Branch Canal. 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 the EPA. 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, followed by determination of PCBs using GC with an electron capture detector and using GC/MS analysis for the semivolatiles. Volatiles were determined by a gaseous purge and trap technique, followed by on-line GC/MS analysis. Graphite furnace atomic absorption was used to determine for the presence of As, Se, and Ti; 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.

IV. QUALITY ASSURANCE

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

A large part of the ACL's analytical effort in FY 1988 was sample analysis and other tasks for the U.S. DOE Environmental Survey Program (DES). The ACL's attention to quality assurance and quality control played a major role in the success of the DES in FY 1988.

To help assure high-quality analytical performance, the ACL participated in four EPA-sponsored multilaboratory programs that provided the ACL with quarterly and semiannual blind performance evaluation (PE) samples for analysis. Organic and inorganic PE samples were received quarterly from the EPA Environmental Monitoring Systems Laboratory-Las Vegas (EMSL-LV) and analyzed as part of an EPA multilaboratory qualification program for laboratories active in the Superfund Contract Laboratory Program (CLP). The ACL scores in FY 1988 for the Quarterly Blind inorganic PE samples (QB1 through QB4) were 81.9%, 99.0%, 99.0%, and 95.8%, while the combined scores for the Quarterly Blind organic PE samples (volatiles, semivolatiles, and pesticides) were 88.4%, 72.6%, 93.8%, and 91.7%.

The Chemical Analysis Group also took part in an EMSL Water Pollution Study (WPS) in which 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 reports no scores for these samples but acknowledges that ACL analytical results were in excellent agreement with EPA's known values.

In addition, the EPA Environmental Monitoring Laboratory (EML) and EMSL-LV coordinated multilaboratory Cross-Check PE Programs to provide PE samples for radiological analysis. Water, soil, and air filter samples for radionuclide determinations, e.g., gross alpha-beta, gamma, tritium, radium, uranium, plutonium, neptunium, were received by ACL on a semiannual basis. The ACL continued to do well in these programs, with each of the radionuclides analyzed being correctly reported to within less than one standard deviation of the known value.

An in-house quality control (QC) program continued to provide a means of assuring that analytical systems were performing satisfactorily. With each Quarterly Blind organic PE sample analyzed as part of the ongoing qualification requirement of the DES, commercially obtained QC samples provided the ACL with volatile, semivolatile, pesticide, and PCB (Aroclor) organic standards of known value to be analyzed with the PE samples. Analytical results obtained using in-house standards were compared to the commercial QC known values. Satisfactory performance on this internal self-check is an ACL requirement prior to submitting results to EPA EMSL-LV. Analysis of QC samples has helped provide analysts with insight into problems that might otherwise go undetected. For example, the degradation of an in-house standard used to establish sample peak heights was discovered as a result of comparing analytical data obtained from an in-house standard aliquot to the known value of the commercial QC sample. This problem had the potential for affecting data quality.

Although EPA protocol requires corrective action responses from all laboratories achieving PE scores of less than 90%, the DOE requires corrective action responses from all participating DES laboratories achieving scores of less than 100%. The ACL formalized its corrective action policy as an addition to the ACL QA Plan (Section 12). The policy was defined with a written set of procedural guidelines that provide ACL personnel with steps for addressing situations requiring corrective action.

The ACL QA Plan is subject to an annual review intended to keep it updated and in agreement with current ACL, CMT, and ANL quality assurance policies. Critiques offered by audit teams that reviewed the ACL as late as October 1987, and an internal review of areas thought to be in need of revision, resulted in the plan being revised during FY 1988. The revised ACL QA Plan is scheduled for distribution in early FY 1989.

The ACL participated in the analysis of samples from a number of DES environmental sites in FY 1988, e.g., Fernald, Hanford, Portsmouth, Kansas City, Y-12, and Sandia. In November 1987, with data reporting for the Fernald site completed, the ACL and its Program Office underwent the first site audit of FY 1988. As required by the DOE, a third party team of auditors from Environmental Monitoring and Services, Inc. (EMSI), performed an audit of the Fernald data set and the sample and field documentation. Because the Fernald site sampling and sample analysis were initiated prior to the DES adoption and implementation of full CLP protocol, a judgment on the quality of data and its legal defensibility is pending.

Approximately 275 sample data packages from a number of DES environmental sites were reviewed by ACL 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 sample data packages for three of the environmental sites whose samples were analyzed by the ACL during FY 1988 was submitted to EMSL-LV for evaluation. Information from EMSL-LV indicated that only a few minor areas of data reporting needed attention. Planned corrective actions for those areas were forwarded to EMSL-LV and implemented by the ACL where required.

The total number of ACL standard operating procedures (SOPs) now in place that address analytical and administrative protocol rose to 135 in FY 1988. A bound, two volume SOP manual has been assembled and distributed for use in each of the ACL laboratory areas. A review of those SOPs specifically intended for use in the DES program determined that most of the SOPs might be adapted to serve the ACL for uses other than the DES.

V. PROFESSIONAL ACTIVITIES

A. Publications and Reports

Advanced Instrumental Methods for Analyzing Organics in Solid Waste: The Use of Gas Chromatography/Matrix Isolation Infrared Spectroscopy (GC/MIIR) and Supercritical Fluid Chromatography (SFC) for Waste Characterization
L. A. Raphaelian, A. S. Boparai, and J. F. Schneider
Proceedings of Advanced Research and Technology Development Direct Utilization Contractor Review Meeting, October 14-16, 1987

Analysis of Long-Lived Isotopes by Liquid Scintillation Spectrometry
D. L. Bowers and L. R. Greenwood
Journal of Radioanalytical and Nuclear Chemistry 123, 461-469 (February 1988)

Analytical Chemistry Laboratory Progress Report for FY 1987
D. W. Green, R. R. Heinrich, K. J. Jensen, M. D. Erickson, with contributions from ACL Staff
Argonne National Laboratory Report No. ANL/ACL-87-2, December 1987

A New Automated Continuous Emissions Monitor for Incinerators
M. J. McIntosh, M. D. Erickson, and J. C. Demirgian
Proceedings of the 1988 Pacific Basin Conference on Hazardous Waste, Honolulu, HI, February 1-6, 1988

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)

Organic Rankine Cycle Power Systems Working Fluid Study: Topical Report No. 2 - 2-Methylpyridine/Water
R. L. Cole, J. C. Demirgian, and J. W. Allen
Argonne National Laboratory Report No. ANL/CNSV-TM-202 (1987)

Organic Rankine Cycle Power Systems Working Fluid Study: Topical Report No. 2 - Toluene
R. L. Cole, J. C. Demirgian, and J. W. Allen
Argonne National Laboratory Report No. ANL/CNSV-TM-192 (1987)

Pilot Study of the Relationship of Regional Road Traffic to Surface-Soil Lead Levels in Illinois
S. J. LaBelle, P. C. Lindahl, R. R. Hinchmann, J. Ruskamp, and K. McHugh
Argonne National Laboratory Report No. ANL/ES-154, August 1987

Production of Long-Lived Activities in Fusion Material
L. R. Greenwood and D. L. Bowers
Journal of Nuclear Materials 155-157, 585-588 (1988)

Review of Book: "Mass Spectroscopy," 2nd Ed, by H. E. Duckworth, R. C. Barber, and V. S. Venkatasubramanian, Cambridge University Press, New York, 1986
L. A. Raphaelian
Applied Optics 27(10), 1964 (1988)

Stable Carbon Isotopic Composition of Methane from Some Natural and Anthropogenic Sources
C. M. Stevens and A. G. Engelkemeir
Journal of Geophysical Research 93(D1), 725-733 (January 1988)

A Study of the Permeability of Polyethylene Pipe by Organic Compounds
J. F. Schneider and L. Harty
Argonne National Laboratory Report No. ANL/ACL-87-1, December 1987

Superconductivity in Thin Films of the Bi-CA-Sr-Cu-O Systems
J. H. Kang, R. T. Kampwirth, K. E. Gray, S. Marsh, and E. A. Huff
Physics Letters A 128, 102 (1988)

B. Oral Presentations

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
In Situ Characterization and Monitoring Technologies Worskhop, Idaho Falls, ID, June 7-9, 1988

Are Cleanup Steps Really Needed in Dioxin Analysis?
R. J. Wingender
Eighth ACL Technical Meeting, Argonne National Laboratory, January 8, 1988

A Comparison of PCB/Pesticide Quantitation Using Packed and Megabore Columns
M. C. Hansen, J. F. Schneider, and M. D. Erickson
The Fall ACS Meeting, Los Angeles, CA, September 25-30, 1988

Correlation of Chemical Properties of Polyethylene Pipes with Constant Tensile Load Test Strength
J. E. Young and L. A. Raphaelian
Tenth Plastic Fuel Gas Pipe Symposium, New Orleans, LA, October 27-29, 1987

The Determination of Impurities in Pu Metal by Anion Exchange and ICP/AES
E. A. Huff and D. L. Bowers
12th Actinide Separations Conference, Naperville, IL, May 9-11, 1988

The Determination of Technetium by ICP/AES
E. A. Huff and D. L. Bowers
30th ORNL/DOE Conference on Analytical Chemistry in Energy Technology, Knoxville, TN, October 20-22, 1987

Development of a Methodology for the FTIR Analysis of Stack Gas
J. C. Demirgian
Eighth ACL Technical Meeting, Argonne National Laboratory, January 8, 1988

Evaluation of Multidimensional Gas Chromatographic Enhancement of Quantitative Data
J. C. Demirgian, and C. T. Snyder
39th Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, New Orleans, LA, February 22-26, 1988

GC/MI-IR/MS Applications: Analysis of Coal-Derived Mixtures
J. F. Schneider, L. A. Raphaelian, M. C. Hansen, and M. D. Erickson
9th Rocky Mountain Regional ACS Meeting, Las Vegas, NV, March 27-30, 1988

ICP/AES of Actinide Elements
E. A. Huff and D. L. Bowers
30th Rocky Mountain Conference, Denver, CO, July 31-August 5, 1988

Ion Chromatographic Analysis of Marble and Limestone Surfaces Exposed to the Acidic Outdoor Environment
F. P. Smith
Eighth ACL Technical Meeting, Argonne National Laboratory, January 8, 1988

Measurement of Long-Lived Isotopes
D. L. Bowers
Eighth ACL Technical Meeting, Argonne National Laboratory, January 8, 1988

A New Automated Continuous Emissions Monitor for Incinerators
M. J. McIntosh, M. D. Erickson, and J. C. Demirgian
1988 Pacific Basin Conference on Hazardous Waste, Honolulu, HI, February 1-6, 1988

New Techniques for Enhancing Helium Production in Ferritic Materials
L. R. Greenwood, D. W. Kneff, and D. G. Graczyk
International Conference on Fusion Reactor Materials, Karlsruhe, Federal Republic of Germany, October 4-8, 1987

Non-Productive Activities
D. W. Green
Sixth DOE Analytical Managers Meeting, Brookhaven National Laboratory, September 6-9, 1988

Optimization of the Separation of Polyethylenes and Polystyrenes by Supercritical Fluid Chromatography
A. S. Boparai and D. V. Applegate
30th Rocky Mountain Conference, Denver, CO, July 31-August 5, 1988

PCBs: A Societal, Environmental, and Analytical Problem
M. D. Erickson
Grinnell College, Grinnell, IA, March 1, 1988

PCBs: A Societal, Environmental, and Analytical Problem
M. D. Erickson
Illinois Institute of Technology, Chicago, IL, March 30, 1988

PCBs: A Societal, Environmental, and Analytical Problem
M. D. Erickson
CMT Division Seminar, Argonne, IL, May 18, 1988

PCDFs and Related Compounds in PCB Fires -- A Review
M. D. Erickson
DIOXIN '88, Umeå, Sweden, August 21-26, 1988

Precise Chemical and Radiometric Analysis of Irradiated Fuel Rods from the Light-Water Breeder Reactor
D. G. Graczyk
30th ORNL/DOE Conference on Analytical Chemistry in Energy Technology, Knoxville, TN, October 20-22, 1987

Preparation of Superconducting $YBa_2Cu_3O_{7-x}$ Films by Magnetron Sputtering
J. H. Kang, R. T. Kampwirth, K. E. Gray, A. Wagner, and E. A. Huff
Spring Meeting of the Materials Research Society, Reno, NV, April 5-9, 1988

Production of PCDFs, and Related Products from PCBs - An Overview
M. D. Erickson
EPRI PCB Symposium, Electric Power Research Institute, Kansas City, MO, October 6-9, 1987

Quantitative Analysis by Gas Chromatography/Matrix Isolation Infrared Spectrometry (GC/MIIR)
J. F. Schneider, M. C. Hansen, and M. D. Erickson
The Fall ACS Meeting, Los Angeles, CA, September 25-30, 1988

Sample Analysis for the DOE Environmental Survey
P. C. Lindahl, D. K. Knight, J. R. Barker, J. E. Gebhart, J. S. Jessup, L. W. McMahon, and B. R. Clark
30th ORNL/DOE Conference on Analytical Chemistry in Energy Technology, Knoxville, TN, October 20-22, 1987

Utility Programs for Facilitating Contract Laboratory Program Data Package Preparation
E. Y. Hwang and R. J. Wingender
36th ASMS Conference on Mass Spectrometry and Allied Topics
San Francisco, CA, June 5-10, 1988

The Use of Gas Chromatography/Matrix-Isolation Infrared Spectroscopy (GC/MIIR) and Supercritical Fluid Chromatography (SFC) for Waste Characterization
L. A. Raphaelian, A. S. Boparai, and J. F. Schneider
Advanced Research and Technology Development Utilization Contractor Review Meeting, Morgantown Energy Technology Center, Morgantown, WV, October 14-16, 1987

X-ray Analysis and Fluorescence
W. E. Streets
ANL/IAEA Training Course, Argonne National Laboratory, January 25-May 13, 1988

Awards and Patents

Alice M. Birmingham
Pacesetter Award for "Extraordinary effort in developing procedures for the Analytical Laboratory's sample receiving room," January 1988

Delbert L. Bowers
Pacesetter Award for "Contributions to establishing and demonstrating the laser photoacoustic spectroscopy capability," January 1988

Mark M. Doxtader
Pacesetter Award for "Contributions to establishing and demonstrating the laser photoacoustic spectroscopy capability," January 1988

Peter C. Lindahl
Pacesetter Award for "His support of DOE Headquarters on the DOE Environmental Survey Program, as well as his role coordinating the analytical work for the ACL," March 1988

Fredric J. Martino
Pacesetter Award for "Extraordinary effort in developing procedures for the Analytical Laboratory's sample receiving room," January 1988

Lilia Mojica
Pacesetter Award for "Extraordinary effort in developing procedures for the Analytical Laboratory's sample receiving room," January 1988

D. Meetings Attended

Amrit S. Boparai
1988 Workshop on Supercritical Fluid Chromatography, January 12-14, 1988
Park City, UT

Delbert L. Bowers
31st Annual Plutonium Isotopic Metal Exchange Meeting, Richland, WA, October 27-29, 1987

Susan J. Bussey
EPA Office of Solid Waste and Emergency Response Organics Conference, New Orleans, LA, October 12-16, 1987

Mitchell D. Erickson
In Situ Characterization and Monitoring Technologies, Idaho Falls, ID, June 7-9, 1988

David W. Green
1987 Analytical Lab Managers Meeting and Board of Directors Meeting, Dearborn, MI, October 29-30, 1987

The Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, New Orleans, LA, February 20-24, 1988

In Situ Characterization and Monitoring Technologies, Idaho Falls, ID, June 7-9, 1988

Edmund A. Huff
ASTM C-26 Committee (Nuclear Fuel Cycle) Meeting, Albuquerque, NM, January 26-28, 1988

Peter C. Lindahl
ASTM Committee D-5 on Coal and Coke, St. Louis, MO, October 4-6, 1987

U.S. EPA Office of Solid Waste and Emergency Response Inorganics Conference, Atlanta, GA, October 28-29, 1987

International Organization on Standardization Meeting, Technical Committee 27, Chaired Subcommittee 5 on Chemical Analysis of Solid Mineral Fuels, Argonne, IL, November 16-17, 1987

U.S. EPA Office of Solid Waste and Emergency Response Inorganics Conference, Denver, CO, April 18-19, 1988

Seminar on Atomic Spectroscopy, Denver, CO, April 20-21, 1988

ASTM Committee D-5 on Coal and Coke, Williamsburg, VA, May 22-24, 1988

USATHAMA/EPA Symposium - Quality Assurance in Environmental Measurements, Baltimore, MD, May 25-26, 1988

In Situ Characterization and Monitoring Technologies, Idaho Falls, ID, June 7-9, 1988

Fredric J. Martino

U.S. EPA Symposium - "Waste Testing and Quality Assurance," Washington, DC, July 11-15, 1988

Paul E. Melnicoff

36th ASMS Conference on Mass Spectrometry and Allied Topics San Francisco, CA, June 5-10, 1988

John F. Schneider

EPA Office of Solid Waste and Emergency Response Organics Conference, New Orleans, LA, October 12-16, 1987

The Fall ACS Meeting, Chaired Analytical Chemistry Division Session on Chromatography, Los Angeles, CA, September 25-30, 1988

Florence P. Smith

15th Annual Meeting of the National Organization for the Professional Advancement of Black Chemists and Chemical Engineers, Philadelphia, PA, April 4-8, 1988

E. Professional Organization Leadership

David V. Green

President, Argonne National Laboratory Chapter of Sigma Xi, The Scientific Research Society

Member of Board of Directors, Analytical Laboratory Managers Association

Peter C. Lindahl

Chairman, International Organization on Standardization, Technical Committee 27, Subcommittee 5 on Chemical Analysis of Solid Mineral Fuels

Florence P. Smith

Vice-Chairman, Executive Board, National Organization for the Professional Advancement of Black Chemists and Chemical Engineers

F. ACL Seminars

Developments in Atomic Emission Spectrometry
Dr. Martin Edelson, Ames Laboratory
February 11, 1988

Detection of Ambient Organic Vapors with a Portable Gas Chromatograph
Dr. Richard Berkeley, U.S. Environmental Protection Agency
March 14, 1988

Interpretation of Complex GC/MS Data on the PC
Dr. William Dunn, University of Illinois-Chicago Campus
March 22, 1988

PCBs: A Societal, Environmental, and Analytical Problem
Dr. Mitchell D. Erickson, Argonne National Laboratory, Chemical Technology Division, Analytical Chemistry Laboratory
May 24, 1988

Signal Processing Techniques for Remote Infrared Chemical Sensing
Dr. Robert Kroutil, U.S. Army Chemical Research Development and Research Center
June 9, 1988

Chemical Science at the Advanced Photon Source
Dr. Gopal K. Shenoy, Argonne National Laboratory, Advanced Photon Source
June 23, 1988

Analysis of Nonvolatiles by ^{252}Cf-Plasma Desorption Mass Spectrometry
Dr. Jerry E. Hunt, Argonne National Laboratory, Chemistry Division
June 28, 1988

ICP-MS Applied to the Analysis of Nuclear Materials
Dr. Glenn E. Bentley, Los Alamos National Laboratory
July 29, 1988

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