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ANALYTICAL CHEMISTRY LABORATORY  
Progress Report for FY 1985

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MASTER

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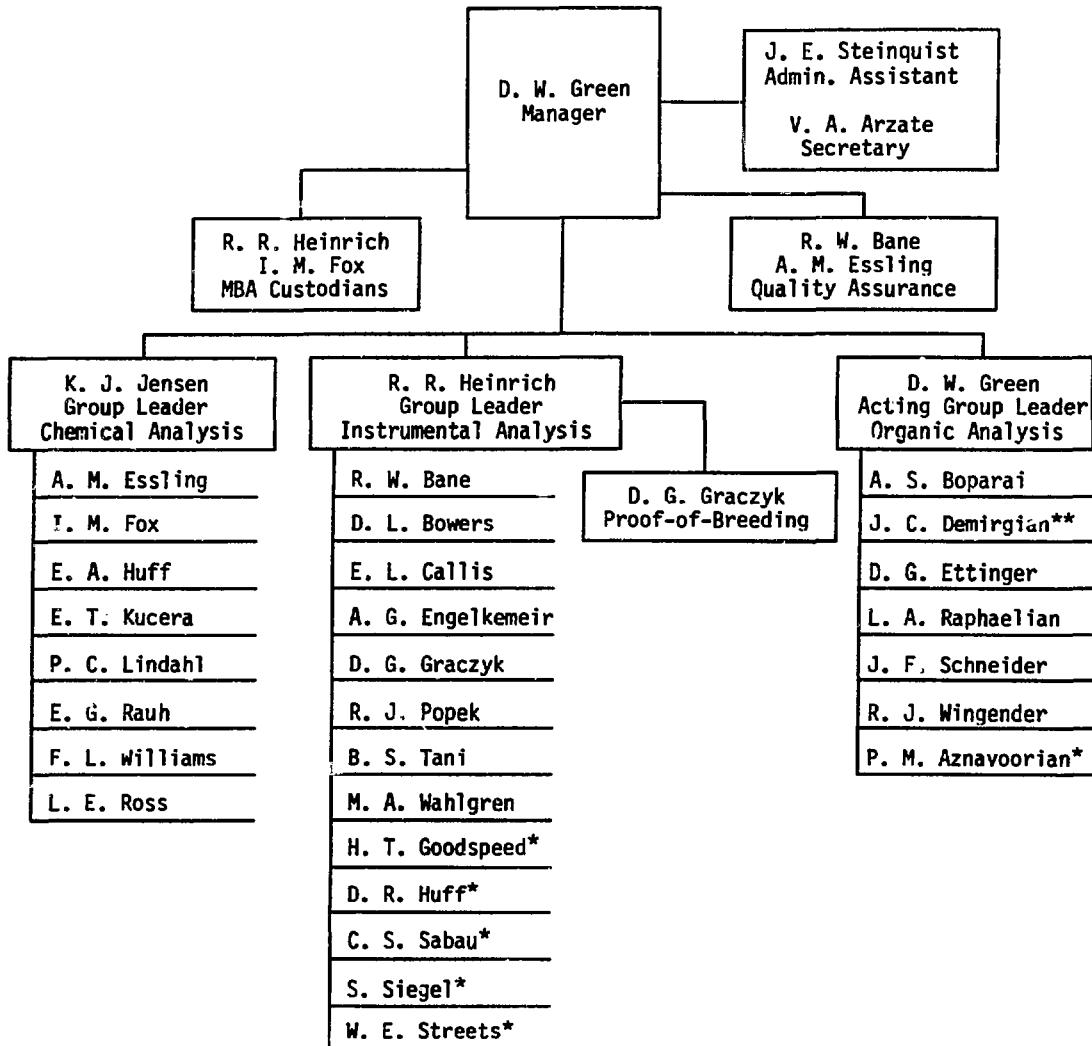
## I. INTRODUCTION

The purpose of this report is to summarize the technical and administrative activities of the Analytical Chemistry Laboratory (ACL) at Argonne National Laboratory (ANL) for Fiscal Year 1985 (October 1984 through September 1985). This is the second annual report for the ACL (ANL/ACL 85-1, March 1985, was the first).

The Analytical Chemistry Laboratory is a full-cost-recovery service center, with the primary mission of providing a broad range of technical support services to the scientific and engineering programs at ANL. In addition, 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 three technical groups -- Chemical Analysis, Instrumental Analysis, and Organic Analysis -- which together include about 30 technical staff members (see Fig. 1). Talents and interests of staff members cross the group lines, as do many of the projects with which the ACL staff deals.

The Chemical Analysis Group performs wet-chemical analyses and instrumental analyses; does spectrochemical analyses and coal analyses; and provides specialized analytical support -- separations, preparations, advice, etc. Major instruments in this group include an ion chromatograph, an inductively coupled plasma/atomic emission spectrometer (ICP/AES), an automated carbon/hydrogen/nitrogen determinator, atomic absorption spectrometers, emission spectrographs, a sulfur determinator, fluorimeters, an X-ray fluorescence spectrometer, and a carbon determinator.



\*Special Term Appointee (STA)

\*\*Term Appointee

7/1/85

Fig. 1. Analytical Chemistry Laboratory Organization Chart

The Instrumental Analysis Group uses nuclear decay counting techniques; performs mass spectrometric and gas chromatographic analyses of gases; analyzes solids with X-ray techniques; and does remote analysis of radioactive samples, dosimetry, neutron activation, inert gas fusion, and isotope analysis. Major instruments in this group include X-ray diffractometers, a variety of multichannel analyzers and associated counting equipment, a scanning electron microscope with X-ray energy dispersive capabilities, mass spectrometers including two thermal-ionization mass spectrometers, gas chromatographs, a surface-area measuring apparatus, and an oxygen determinator.

The Organic Analysis Group uses a number of complementary techniques to separate and analyze complex organic mixtures and compounds, including synthetic fuels, toxic substances, fossil fuel residues and emissions, pollutants, biologically active compounds, pesticides, and drugs. Major instruments in this group include a large variety of gas chromatographs (GC), mass spectrometers (MS), combination GC/MS systems, infrared spectrometers including a Fourier Transform (FTIR) system, a combination GC/matrix-isolation/FTIR (GC/MI/FTIR) system, a combination GC/FTIR/MS system, and liquid chromatographs.

## II. ADMINISTRATIVE HIGHLIGHTS

The major users of ACL services among ANL organizations for FY 1985 are listed in Table 1.

Table 1. Distribution of ACL Work Load for FY 1985 among the Major Divisional Users

ANL Organization	% ACL Work
Chemical Technology (CMT)	57.4
Chemistry (CHM)	12.5
Energy and Environmental Systems (EES)	11.5
Materials Science and Technology (MST)	7.8

The major user of ACL services continues to be CMT (see Fig. 2). The major program within CMT using ACL service is the Proof-of-Breeding Project, which has accounted for about half the CMT total in FY 1983, FY 1984, and FY 1985. The use of ACL services by other ANL divisions, shown in Fig. 3, has changed in recent years. Most decreases can be explained by the termination or substantial cutbacks of particular programs that were previously major users of ACL services [decontamination and decommissioning in the Occupational Health and Safety (OHS) Division, chemical toxicity programs in the Biological and Medical Research (BIM) Division, several Environmental Research (ER) Division sampling programs, etc.]. The increase in ACL use by EES is partially due to administrative consolidation in 1981 and 1982.

The ACL recovered 100.8% of its total operating costs in FY 1985. This year is the first overrecovery for the ACL in the last decade. The ACL effort, including divisional administration, includes about one-half recoverable time (sales to users in Fig. 4). The total operating costs for FY 1985, shown in Fig. 5, are about two-thirds effort related.

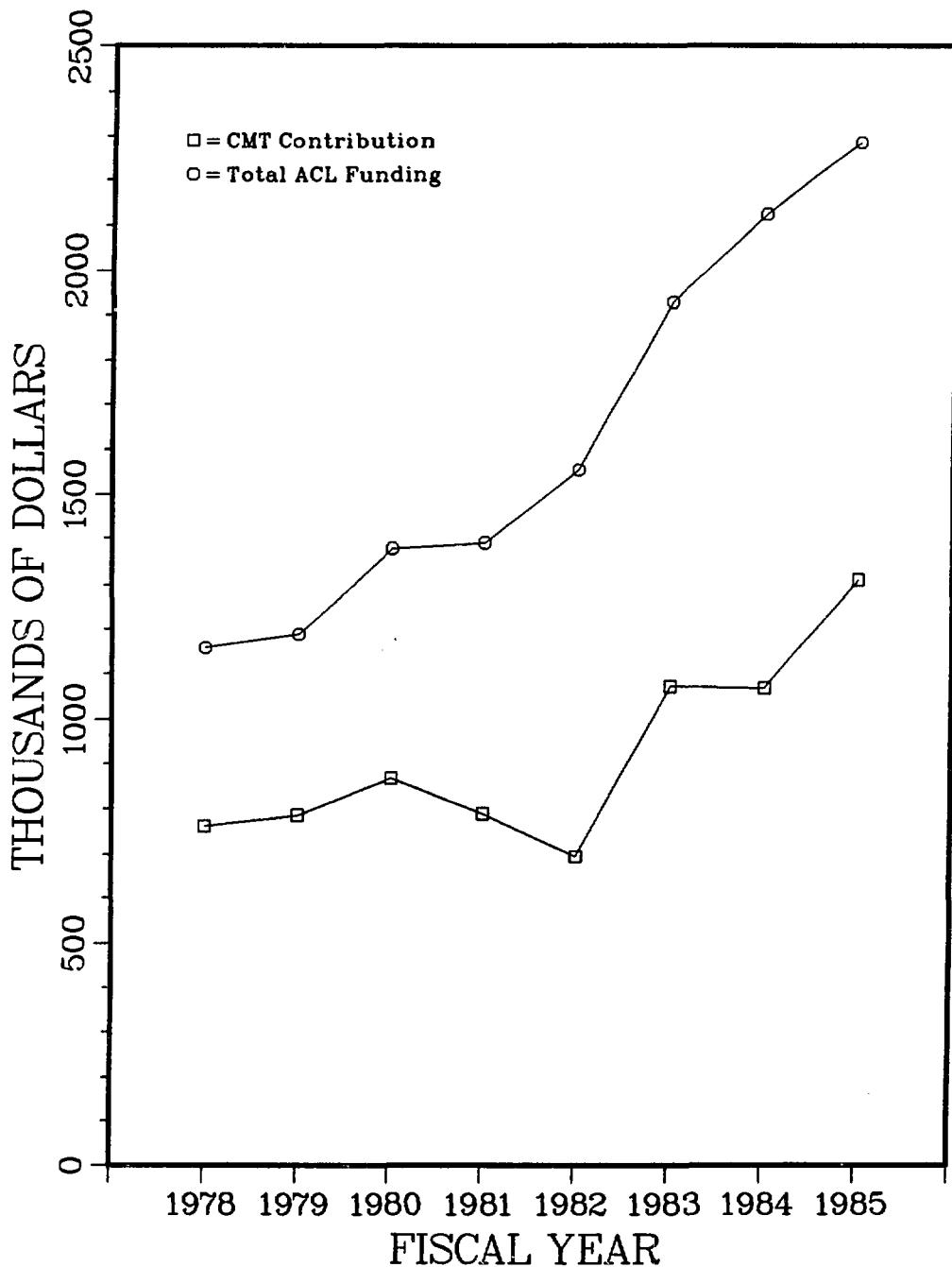


Fig. 2. ACL Funding, Total and CMT

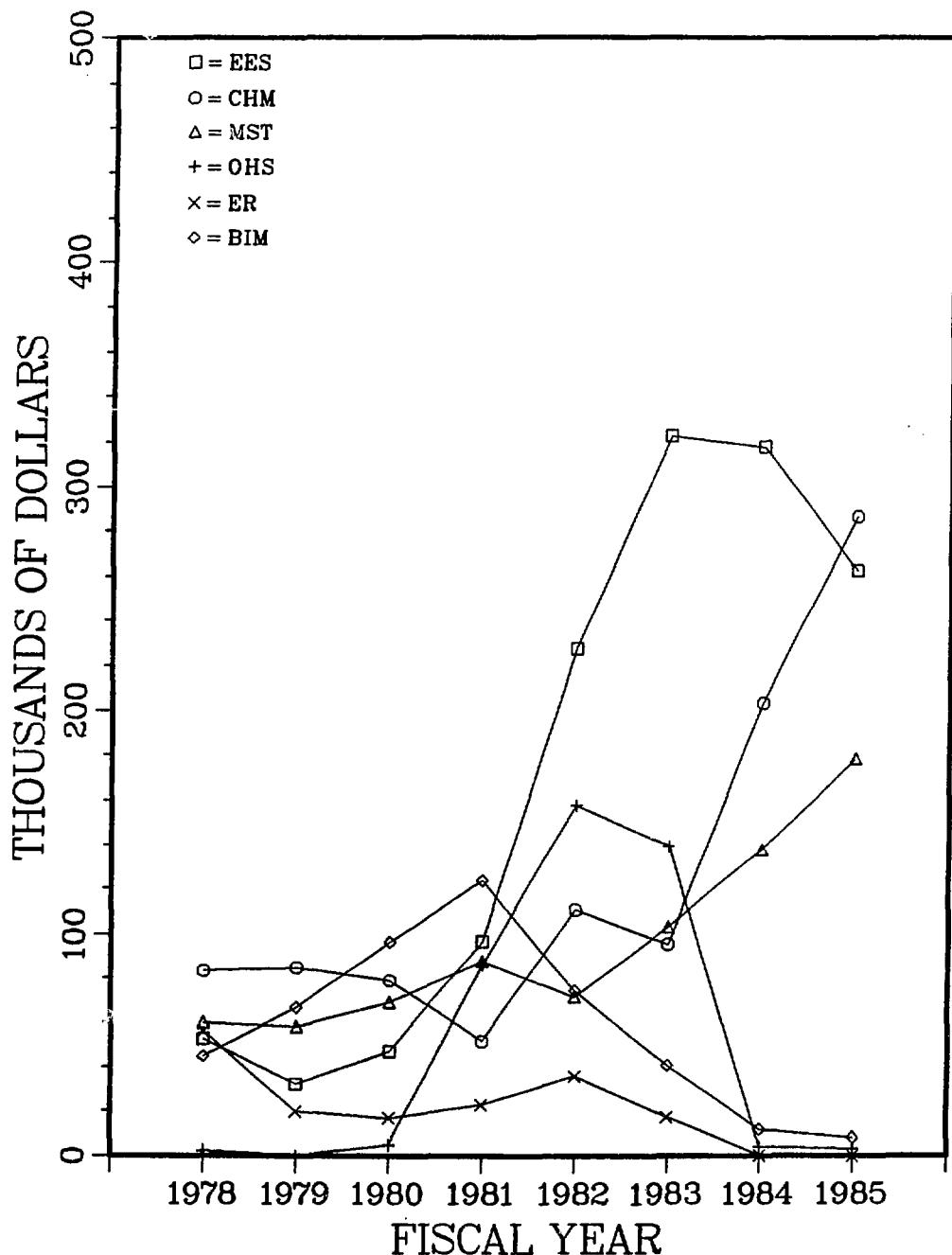


Fig. 3. ACL Funding, Historical Major Users

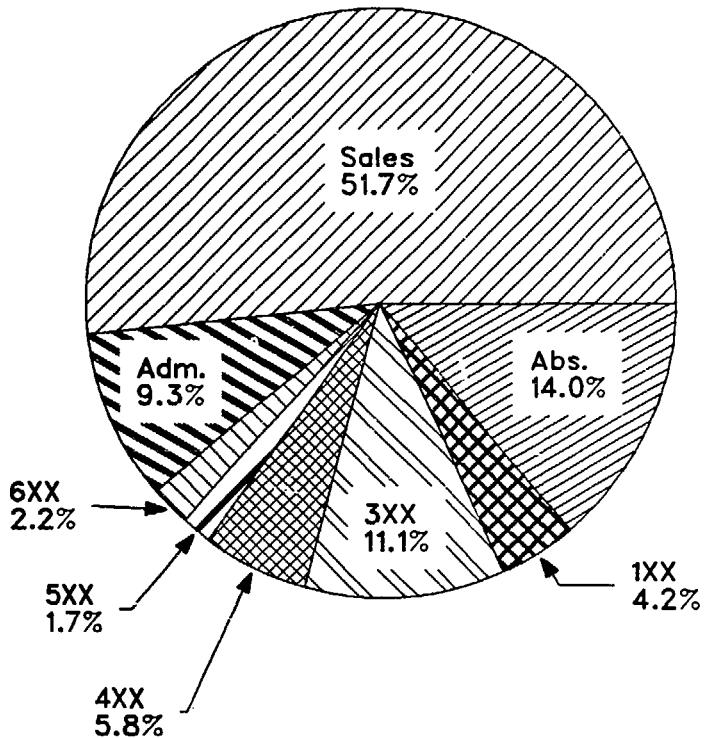


Fig. 4. Analytical Chemistry Laboratory  
FY 1985 Effort

Abs = paid absences

1XX = operations administration

3XX = internal overhead; e.g., instrument maintenance

4XX = general overhead; e.g., safety

5XX = technical and employee development

6XX = experimentation

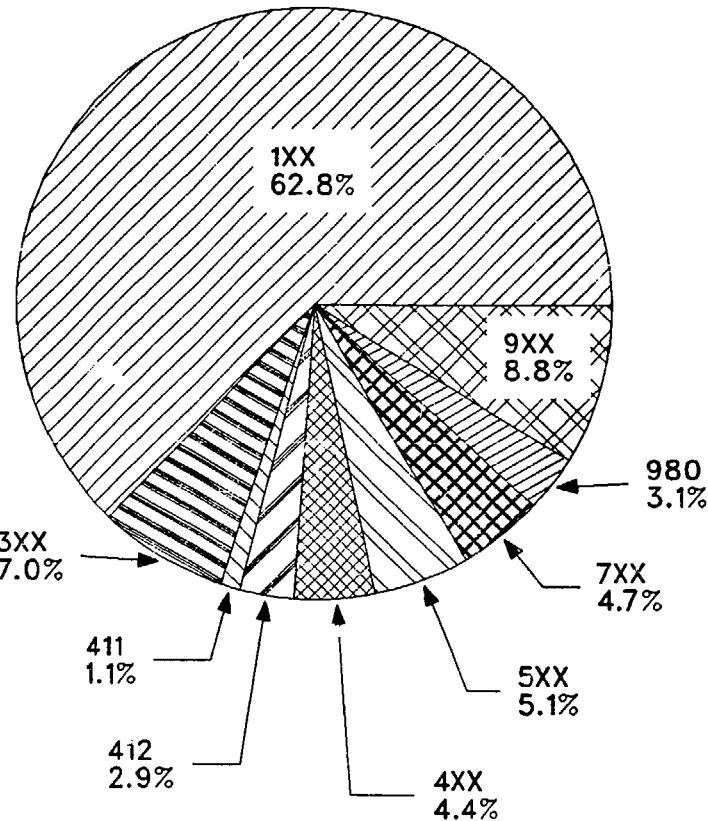


Fig. 5. Analytical Chemistry Laboratory  
FY 1985 Costs

1XX = salaries and fringes

3XX = materials and supplies

4XX = services, subcontracts, etc.

5XX = shopwork and work projects

7XX = redistributions

9XX = indirect costs

The ACL continued to obtain help from Special Term Appointees (see Fig. 1) in FY 1985 to assist with load leveling. About three full-time equivalents were used in FY 1985, and continued use is anticipated in FY 1986. Two visiting faculty members, one intern (IIT's Women in Science - Reentry in Analytical Chemistry Program), and several students supplemented the regular ACL staff.

The ACL continued a program to inform potential ANL users of the analytical services that are available. Thirty single-page summaries of analytical techniques have been issued to ANL staff, eight of them in FY 1985. Several other summaries are in preparation. A description of ACL capabilities for analyses related to hazardous wastes was prepared and distributed. Tours of ACL facilities have been regularly conducted for new CMT employees and other selected ANL users.

The ACL continued a small internal funding system (\$24K in FY 1984 and \$15K in FY 1985) to support selected projects leading to publication and development of new capabilities. Projects supported in FY 1985 include a comparison study of GC/MI/IR with GC/MS, development of methodology for determination of sodium in coal, and a comparative study of the utilities of a light pipe and of matrix isolation as GC/IR interfaces.

The ACL continued to work with the Division of Educational Programs (DEP) in providing access by outsiders to ACL equipment and expertise. Three ACL staff members participated in a DEP-sponsored summer program for gifted students from junior high schools. Several one- or two-day workshops for faculty and students from at least four colleges were conducted by the ACL staff.

A complete safety tour of all ACL laboratories was made during the summer with the CMT Safety Representative. Corrective actions were taken where indicated. An internal audit was made of the ability of ACL staff to retrieve information about samples. An audit was made by the Quality Assurance Division (QAD) of selected portions of the ACL Quality Assurance Plan. Some changes have

been made in the ACL Quality Assurance Plan, and other changes are being considered.

Argonne hosted the sixth meeting of the Analytical Laboratory Managers Association. About 80 attendees were at ANL for the formal program, as well as for tours of selected facilities at ANL.

Technical meetings of the ACL, which were initiated in FY 1984, were continued. Two meetings were held in FY 1985 at which selected ACL staff gave presentations on their work to other ACL staff. Other such meetings will be held in the future.

Improvements in ACL facilities and capabilities were made in FY 1985 (several of these are described in the "Technical Highlights" section). Work has been nearly completed on construction of a containment room to be located in Laboratory X-125 of Building 205. This room will allow the safe handling of potentially toxic samples from field studies. Samples prepared in the containment room can then be safely analyzed by existing GC and GC/MS systems. Funding for construction of the containment room originates from the U.S. Environmental Protection Agency (EPA), which also uses this facility. Work was completed on Room G-117 in Building 205 to prepare for handling and analysis of samples in the Integral Fast Reactor (IFR) Program. Further modifications are expected in G-117 to accommodate the ICP/AES unit dedicated to radioactive samples. Design work has progressed on a clean room for analysis of organic samples. This facility will be located adjacent to the containment room.

A new atomic absorption spectrophotometer (AAS) with state-of-the-art graphite furnace capability has been delivered and is installed in Building 200. This unit will increase the sensitivity with which selected elements can be determined. An ICP/AES unit has been ordered to allow the ACL to do ICP/AES analysis of radioactive samples. This instrument is expected to be used in the IFR Program, the Source Term Experimental Program, and for analysis of core

samples from the Three-Mile Island Unit 2. Other new equipment installed in FY 1985 includes a trace gas analyzer, a multichannel gamma analyzer system, a backscatter detector for the scanning electron microscope, an X-ray generator, and a new data system for a gas chromatograph.

### III. TECHNICAL HIGHLIGHTS

Proof-of-Breeding Program (D. G. Graczyk, R. W. Bane, D. L. Bowers, E. L. Callis, A. G. Engelkemeir, A. M. Essling, R. R. Heinrich, K. J. Jensen, E. T. Kucera, R. J. Popek, E. G. Rauh, and M. A. Wahlgren)

The Light-Water-Breeder Reactor (LWBR) Proof-of-Breeding (POB) Analytical Support Project (N. M. Levitz, CMT) continues to be the largest user of ACL support from both the Instrumental and Chemical Analysis Groups.

During the past year, the final phase of the POB project was nearly completed. The end-of-life (EOL) campaign involves the destructive assay of 17 EOL fuel rods from the LWBR (Shippingport, PA). The ACL role in this campaign focused on providing analyses of dissolver solutions and gas samples obtained during the processing of 152 segments sheared from the individual LWBR fuel rods. More than 1000 aliquots of dissolver solution were prepared and analyzed by thermal ionization mass spectrometry (TIMS) to provide uranium assay and isotopic information; the critical uranium assays were performed using a special internal standard procedure developed within the ACL for the TIMS measurements and showed a standard deviation for duplicate samples over all the segments of less than 0.01%. An additional 550 aliquots of dissolver solution were analyzed by gamma spectrometry to determine the fission products,  $^{137}\text{Cs}$ ,  $^{144}\text{Ce}$ , and  $^{95}\text{Zr}$ , and 200 samples of rod plenum gases or dissolver offgases were analyzed by gas mass spectrometry for the fission products Xe and Kr.

Two separate and independent audits of the LWBR-POB Analytical Support Project, including ACL operations within the project, were conducted during the spring of this year. The first audit was conducted April 9 to 12, 1985, by Bettis Atomic Power Laboratory as part of its investigation of differences between its nondestructively measured fissile loadings for EOL LWBR fuel rods and ANL's destructive assay results. The audit included detailed consideration of ANL's operations and analyses, quality assurance, data handling, and reporting. Special consideration was given ANL's handling of specific off-normal

situations that were encountered during processing of three fuel-rod segments in the Dual Dissolver System. Conclusions reported for this audit indicated (1) validity of the ANL assay results obtained under normal operations and (2) appropriate and reasonable handling by ANL of off-normal situations, leading to satisfactory measurements and analyses within project error requirements, in spite of the operational difficulties that arose.

The second audit was conducted on May 7, 1985, by ANL/QAP as part of a QAD verification of the implementation of the POB Quality Assurance (QA) Plan. The auditors reported no inadequacies in implementation of QA requirements. Final reports describing the results of each audit were published during June 1985.

All measurements proceeded smoothly with rapid turnaround and no significant equipment malfunctions during the 16-month duration of the EOL campaign. Project requirements on the individual analyses were met or exceeded in every case.

Homogeneity Testing for the Argonne Premium Coal Sample Program (R. R. Heinrich and P. C. Lindahl)

The blending, or mixing, process is considered to be the most critical single step in the processing of premium coal samples. The fluidized-bed mixer chosen for this operation was tested under realistic conditions using coal from the ANL Central Boiler Facility and by selecting three indigenous coal constituents as monitors of homogeneity: sodium and potassium, which are components of clay minerals found in coal, and arsenic, which is present in pyrite, are three constituents that are easily detected by neutron activation analysis and can be measured simultaneously, thereby providing a number of monitors of homogeneity.

A suite of 39 vials of coal processed using the fluidized-bed mixer in the Premium Coal Sample Preparation Facility was evaluated for homogeneity using

thermal neutron activation analysis. It was concluded from these measurements that the fluidized-bed mixer is an effective means of homogenizing coal. In April 1985, the first coal to be used as a premium coal was collected and processed in the sample preparation facility. The coal was collected from the Upper Freeport Seam at Homer City, PA. Thirty-four samples of processed coal were taken for homogeneity testing, again using the neutron activation technique. Results on this batch of samples were comparable to the first batch. In addition, total sulfur was determined to assess the sulfur homogeneity in the samples.

Gas Analysis for Exxon Nuclear Corporation (R. R. Heinrich, A. G. Engelkemeir, H. T. Goodspeed, and D. G. Graczyk)

The ACL received three Zircaloy tubes from Exxon Nuclear Corporation for the determination of Kr gas content and  $^{85}\text{Kr}$  enrichment. The puncturing of the tubes was more difficult than first anticipated because of work-hardening of the metal during the fabrication process, but successful penetration was achieved using a special ANL-fabricated puncture valve that contained a hardened puncture needle. Each tube contained about the same volume of gas, but each had a different Kr composition. The isotopic distribution of Kr in each tube was identical, however. From the gas composition and the  $^{85}\text{Kr}$  isotopic distribution, the  $^{85}\text{Kr}$  enrichment was calculated. A comparison of results from gamma spectroscopy and gas analysis yielded excellent agreement between the two independent methods. The successful completion of this work has resulted in an expanded gas handling capability that did not exist previously in the ACL.

Measurement of Oxygen and Nitrogen in U-Pu-Zr Alloy (IFR-029-1) (R. R. Heinrich, H. T. Goodspeed, D. G. Graczyk, D. L. Bowers, and W. E. Streets)

The first measurements to determine oxygen and nitrogen in our newly adapted inert-gas-fusion glove-box facility located in G-102, Building 205, were successfully completed on September 26. The first samples were those of IFR

U-Pu-Zr alloy from Pin #2 (029-1). In this method, the sample is first fused in a platinum bath within a graphite crucible being maintained at a temperature of about 2000°C. A helium carrier gas flows over the crucible and carries reaction gases (CO and N<sub>2</sub>) to a gas analysis train through which the CO is oxidized to CO<sub>2</sub> (using hot CuO). Both the CO<sub>2</sub> and N<sub>2</sub> are collected and measured manometrically. Typically the uncertainties for oxygen and nitrogen measurements using this technique are  $\pm 3\%$  and  $\pm 5\%$ , respectively, at the 95% confidence level.

Isotope Correlation Studies and Measurements (ICSAM) (R. R. Heinrich, R. J. Popek, D. L. Bowers, A. M. Essling, and E. L. Callis)

The change in the isotopic composition of a fuel as a function of burnup is a measure of the irradiation history of that fuel in a power reactor. Experimental data obtained from light water reactor spent fuel indicate that there are relationships between isotopic concentrations that have a predictable, functional behavior over a broad range of reactor operation conditions and burnup. These observed functional relationships, when coupled with reactor analysis and computational methods, may be used to optimize the fuel management of a reactor and fuel reprocessing plant and, perhaps more importantly, can be used as an accountability measure for the safeguards of nuclear materials.

The ACL effort associated with this program (P. Persiani, Applied Physics Division) was to experimentally test computational methods being developed for determining the fuel isotopic dynamics in reactor operations. This was done by the determination of uranium and plutonium compositions on Zion pressurized water reactor fuel that had spanned five power cycles, culminating in a 59,000-to 64,000-MWd per metric ton burnup level. Analyses were performed on ten samples excised from selected sections of the fuel rods. Hot cell operations required the separation of fuel from cladding and the comminution of the fuel. These tasks were successfully accomplished using a SpectroMil, a ball pestle impact grinding and blending instrument manufactured by Chemplex Industries,

Inc., Eastchester, NY. The fuel was dissolved using strong mineral acids and bomb dissolution techniques. Separation of the fuel from fission products was done by solvent (hexone) extraction. Fuel isotopic compositions and assays were determined by the mass spectrometric isotope dilution (MSID) method using NBS standards SRM-993 and SRM-996. Alpha spectrometry was used to determine the  $^{238}\text{Pu}$  composition. Relative correlations of composition with burnup were obtained by gamma-ray spectrometry of the selected fission product,  $^{137}\text{Cs}$ , in the dissolved fuel.

X-Ray Diffraction Analysis (B. S. Tani)

The ACL provides a service for phase identification of crystalline materials by X-ray powder diffraction (XRD) analysis. During the past year, 257 jobs (about 500 samples) were submitted for analysis. The types of samples submitted consisted of the following, with examples of the ACL's role in each.

(1) Quality assurance of materials used in an experiment - An outside lab was contracted to produce a solid solution phase of  $\text{UO}_2$  and a few percent of  $\text{La}_2\text{O}_3$ . The ACL XRD analysis showed two phases present, signifying incomplete reaction. The sample was returned to the lab for another heat treatment, after which subsequent ACL XRD analysis showed a single phase.

(2) Monitoring the result of an experiment - A copper disc was exposed to lithium; XRD analysis showed that a copper lithium compound was formed.

(3) Monitoring doping of materials used in conductivity studies - A change in  $\text{LiFeO}_2$  lattice constant was associated with the extent of Mn doping in the  $\text{LiFeO}_2$ .

(4) Phase diagram studies in coordination with metallographic observations - In the study of Na-S system from batteries, several XRD patterns have been found that were not previously recorded and are probably metastable phases in the system.

(5) Crystalline or amorphous material check - Glass fabrication was monitored by XRD to determine if the product was amorphous, crystalline, or a mixture.

(6) Long-term cell chemistry studies - Electrode products were identified for a series of cells that were charged and discharged to different states to help elucidate the cell reaction, Li-Si/FeS<sub>2</sub>.

Analysis of Environmental Samples from Argonne West (AW) Site (M. A. Wahlgren)

Environmental samples consisting of soil, vegetation, and water collected at the AW site were analyzed for <sup>232</sup>Th, <sup>226</sup>Ra, uranium, and plutonium. Forty-four samples collected over a two-year period resulted in 176 analyses.

Specialized Hair Analyses (I. M. Fox, D. W. Green, E. A. Huff, K. J. Jensen, and P. C. Lindahl)

A hair sample was analyzed by ICP/AES for Cd, Ca, Cu, Fe, Pb, Mg, Mn, Na, and Zn after wet oxidation of small (0.1-g) samples. The analyses were done at the request of W. Walsh (ANL/EES) and the Hair Research Institute (HRI). The sample was hair from the head of James Oliver Huberty -- the man responsible for killing 21 people and wounding 19 others in a southern California McDonald's restaurant in July 1984. Authorities in San Diego requested HRI to provide a specialized chemical analysis of Huberty's hair. Commercial laboratories were not able to provide the required analyses due to the small amount of material available for analysis. The ACL provided HRI with the analyses required (along with control data). The results of the analysis and interpretation of the data were submitted to the Coroner, San Diego County, California, in a report entitled, "Elemental Concentrations in the Hair of James Oliver Huberty," authored by W. J. Walsh, P. C. Lindahl, and E. A. Huff.

A second suite of hair samples was analyzed for arsenic by AAS and other elements by ICP/AES. The samples were head hair from four females, all members

of the same family, who were very ill from some unexplained cause. The ACL data ruled out the possibility of metal poisoning as a cause for the illnesses.

Determination of Arsenic and Selenium in Coal (P. C. Lindahl)

The ACL has participated in an American Society for Testing and Materials (ASTM) interlaboratory testing program on a proposed method for the determination of arsenic and selenium in coal. The testing program, which involved six laboratories, was designed to collect data to evaluate the accuracy and precision of the proposed method. In this study, 12 coal samples were analyzed in quadruplicate using the proposed method, which required combustion of the coal sample with Eschka's mixture and subsequent determination of arsenic and selenium by hydride generation-AAS. In the final report submitted to ASTM, all the ACL data were used in the statistical evaluation of the intra- and interlaboratory precision of the method.

Analysis of Flue Gas Desulfurization System Products (F. L. Williams and P. C. Lindahl)

A second interlaboratory testing program, sponsored by the Electric Power Research Institute and coordinated by the Radian Corporation, was included in our FY 1985 schedule. The program involved the analysis of 21 samples, which included both scrubber liquors and solids. Ion chromatography was used to determine total sulfur and chloride. Sodium, calcium, and magnesium were determined by AAS. To date, the evaluation of the submitted data has not been reported back to the ACL.

ACL Interactions with the Division of Educational Programs (D. G. Ettinger, I. M. Fox, D. W. Green, E. A. Huff, K. J. Jensen, E. T. Kucera, P. C. Lindahl, F. L. Williams, and R. J. Wingender)

A number of workshops were conducted by staff members of the Chemical Analysis Group. Students and faculty members learned to operate and apply state-of-the-art instrumentation to the solution of analytical problems in a

series of one- to two-day workshops, which were sponsored by DEP as part of its Regional Instrumentation Sharing Program. This program was organized and administered by D. G. Ettinger.

Three two-day workshops were conducted by F. L. Williams for students and faculty members from Wright State University (Dayton, OH) and Ball State University (Muncie, IN).

E. T. Kucera conducted a four-hour workshop on energy dispersive X-ray fluorescence (EDXRF). Participating in the workshop were a chemistry professor from Beloit College and two students who were tutored in the principles and operation of EDXRF. The students used the instrument to nondestructively analyze some ancient bronze artifacts.

Lectures on GC/MS were given, and analyses that included both solid probe work and identification of compounds originating from plants and collected on Tenax traps were conducted for several professors as part of the DEP program designed to make high-cost instrumentation available to small colleges.

E. A. Huff conducted a one-day workshop in the application of ICP/AES to the solution of analytical problems. Participants in this workshop included a faculty member and students from Milwaukee Technical Institute (Milwaukee, WI).

Twenty-four gifted junior high school students attended two workshops in analytical chemistry conducted by D. W. Green, I. M. Fox, and K. J. Jensen. The workshops demonstrated an automated carbon/hydrogen/nitrogen (CHN) analyzer and a sulfur determinator to analyze coal and organic compounds.

Ms. Kathryn McHugh, a science teacher at Amundsen High School in Chicago, was granted a Residence in Science and Technology Summer Research Appointment by the U.S. Department of Energy (DOE). She spent six weeks working under the direction of P. C. Lindahl to study the distribution of lead in playground soils.

Dr. John Ruskamp, a science teacher from Hirsch High School in Chicago, also worked on the lead project. His appointment was as a Summer Faculty Research Participant for DEP.

Distribution of Lead-in-Soil Project (P. C. Lindahl)

The ACL has participated in a DOE-sponsored project with S. LaBelle (EES) for testing playground soils for lead content. The intent of this program is to determine the lead content of soils collected in selected sites throughout Illinois and relate these data to traffic patterns to evaluate the potential exposure of lead on children in play areas. Soil samples, about 900 in total, were collected -- 300 in Chicago, 300 from the six counties around Chicago, and another 300 from the rest of the state. The soil analysis was an integrated effort. Samples collected in Chicago were analyzed by the Chicago Department of Health, the samples collected in the counties adjacent to Chicago were analyzed by the ACL at ANL, and those collected in the other areas of the state were analyzed by the Illinois Department of Public Health. The ACL coordinated an interlaboratory quality control program for the analysis part of the project. The three analytical laboratories used AAS to determine the lead. A final report to DOE on this project is pending.

National Acid Deposition Assessment Program (E. A. Huff, F. L. Williams, and K. J. Jensen)

The National Park Service (NPS) is coordinating a study under the National Acid Deposition Program to find ways of maintaining historical monuments and buildings. The ACL is providing analytical service to support a stone exposure project managed by the MST Division for the NPS. Three to four hundred samples of limestone and marble exposed to a variety of atmospheric conditions have been analyzed by ACL to date. Adsorbed anions (especially fluoride, chloride, nitrate, and sulfate) have been determined by ion chromatography. Metals present in the samples have been determined by ICP/AES.

Support for Various ANL Programs (I. M. Fox, A. M. Essling, E. A. Huff, K. J. Jensen, E. T. Kucera, P. C. Lindahl, E. G. Rauh, and F. L. Williams)

A number of different ANL programs have been provided with analytical service. A brief summary is given below.

<u>Name of Program</u>	<u>ANL Division</u>	<u>Analytical Support Services</u>
Materials Development in the Fusion Power Program - Surface Segregating Alloys	MST	Characterization of copper-lithium alloys.
Research in Separations Chemistry	CHM	Numerous samples analyzed for distribution coefficient using ICP/AES.
Corrosion of Refractories in Synthetic Coal Ash Slag with Application to Slagging Coal Gasifiers	MST	Complete chemical analysis of slag and chromite samples.
High-Level Waste Repository Interactions	CMT	Analysis of leached metals by ICP/AES; determination of leached anions by ion chromatography; determination of low levels of uranium by fluorescence analysis; alkali metals determined by flame photometry.
Integral Fast Reactor	CMT	Chemical characterization of salt and product samples.
Diffusion Mechanisms in Alkali Glasses	MST	Chemical characterization of alkali (Na, K, Rb) germanates and alkali aluminum germanates.
Source Term Fission Product Characterization	CMT	Cesium, iodide, and tellurium determination for various phases of the testing program.
Measurement and Control of Alkali Vapors in Pressurized Fluidized-Bed Combustor Flue Gas by a Granular-Bed Sorber Diverter	CMT	Hundreds of samples analyzed for sodium and potassium to evaluate performance of the system.

<u>Name of Program</u>	<u>ANL Division</u>	<u>Analytical Support Services</u>
Reduced Enrichment Research and Test Reactor Program (RERTR)	MST	Accurate chemical assays of test alloys, including $UAl_x$ , $USi_x$ , $UFe_2$ , and $U_3SiCu$ ; wet chemical and MSID techniques have been used for the assays required; inert gas fusion used to determine O, H, and N; ICP/AES for trace impurities.
Premium Coal Sample Program	CHM	Carbon, hydrogen, and nitrogen analyses, sulfur determinations, and chemical characterization of ashes.
Characterization and Reactivity of Coals and Coal Macerals	CHM	Carbon, hydrogen, nitrogen, and sulfur instrumental analyses.
Separations of Coal Macerals	CHM	Carbon, hydrogen, nitrogen, and sulfur instrumental analyses.
Molecular Beam Studies	CHM	Purification of a 3-g sample of $^{56}Fe$ from contaminants that precluded fabrication of the materials into malleable metal.
Battery Program Research	CMT	Deposits of Pb, $PbSO_4$ , and $PbO_2$ mixtures analyzed for Pb and $SO_4^{2-}$ ; lead separated by electrodeposition and weighed as $PbO_2$ ; sulfate determined gravimetrically as $BaSO_4$ after separation of lead; Sb by ICP/AES.
Fusion Power Materials Performance	MST	Characterization of lithium metal-trace metals, nitrogen determination, and lithium assay.
Integrated Two-Step Process for Ultra-Fine Clean Coal	CMT	Carbon, hydrogen, sulfur, copper, iron, and nickel determinations, and energy content.

Miscellaneous Nonroutine Analyses (E. A. Huff, I. M. Fox, E. T. Kucera, K. J. Jensen, P. C. Lindahl, E. G. Rauh, A. M. Essling, and F. L. Williams

A variety of uncommon materials have been characterized using wet chemical and instrumental methods of analysis.

<u>Material</u>	<u>Description of Material and Method of Analysis</u>
Amorphous Boron, Boron Carbide	Materials to be used in fabrication of reactor materials; boron assay and isotopic composition determined by MSID.
Cu <sub>3</sub> Au Alloy	Experimental alloy analyzed for Cu and Au by ICP/AES after dissolution of material.
EuRh <sub>3</sub> B <sub>2</sub> Alloy	Sample dissolved in a sealed tube at high temperature and pressure; assay for major components done by ICP/AES.
U-Ni Alloy	Fuel rod material fabricated by MST. Uranium determined by MSID after separation of Ni; Ni determined gravimetrically.
Single Crystal NiO	Material inert to ordinary acid dissolution; sample dissolved in a sealed tube prior to ICP/AES analysis for trace impurities.
Refractory ThO <sub>2</sub> -ZrO <sub>2</sub> -Y <sub>2</sub> O <sub>3</sub>	Material for materials basic research study; sealed tube dissolution required for dissolution prior to ICP/AES analysis for major components and trace metals.
Corrosion Product Samples from Reactors	Leachable anions determined by ion chromatography; major metal components determined by ICP/AES.

Gas Chromatography/Infrared Spectroscopy Interface Comparison Study (J. F. Schneider)

Test mixtures were run on our GC/MI/IR and GC/light-pipe/IR systems to determine relative instrument sensitivities. These tests should help establish the applicability of the newer GC/MI/IR technique. A paper comparing the two interfaces and suitable applications is being prepared for submission to the Journal of Chromatographic Science. A summer faculty participant, Joseph Stickler, collaborated on this study.

Plastic Pipe Program (J. F. Schneider, L. A. Raphaelian, J. E. Young (CMT), and D. G. Ettinger)

The Gas Research Institute (GRI) sponsors a multidisciplinary program at ANL to characterize, on a chemical and molecular basis, the polyethylene pipe, resins, and other materials used in the manufacture of the pipe and to relate the characterization with the mechanical properties, such as long-term stability, crack resistance, degradation, and strength. Complementary work on mechanical properties is done in MST (D. T. Raske).

Gas chromatography analysis was performed on extracted polyethylene pipe samples, and the results have been reported to GRI. Several short-chain hydrocarbons were found and quantitated. The GC analysis yields useful information about the nature of the polyethylene and is complementary to other available techniques. Studies were undertaken to measure the absorption of short-chain hydrocarbons into polyethylene. The results were incorporated into a report by Lora Harty, a student research participant in the ACL.

Gas Chromatography/Matrix-Isolation/Infrared Spectrometry (J. F. Schneider)

The GC/MI/IR instrument, developed at ANL, is now commercially available. Although no programmatic funding was available in FY 1985, some small projects were undertaken to enhance future funding for additional development work and applications of this technique to analytical problems arising at ANL and elsewhere. Several papers on this new technique were presented at the 1985 Pittsburgh Conference.

Morantown Energy Technology Center Waste Management Program (J. F. Schneider, A. S. Boparai, and L. A. Raphaelian)

In this program, analyses of organics in solid wastes from coal and coal liquids are being done by GC/MS, high performance liquid chromatography, GC/MI/FTIR, and supercritical fluid chromatography. No samples were received in FY 1985. Equipment has been acquired and set up to do this project in FY 1986.

Environmental Protection Agency Projects (R. J. Wingender, J. F. Schneider, and A. S. Boparai)

A small, but constant, weekly effort was required to administer to the upkeep and control of the in-house EPA programs and communicate with Andrea Jirka, the Region V EPA Interagency Agreement Coordinator. Work was continued on the GC/MS and GC/electron capture detection of organic priority pollutant/pesticide/PCB compounds in precipitation samples collected for the Great Lakes Atmospheric Deposition Program. Samples collected from the last quarter of 1983 and the first quarter of 1984 were analyzed, and reports giving the results were submitted. The project was then put on hold and eventually cancelled due to lack of money and disappointment with the sampling technique. A new program will be initiated that will improve the sampling technique and probably involve our analytical expertise. The program will be completed with submission of a summary work report and the standard operating procedure developed specifically for analyzing these samples, after QA approval.

Samples from two Resource Conservation and Recovery Act surveys were received and analyzed for PCBs and pesticides. Results have been reported to the EPA. Still pending are analysis of the samples for GC/MS organics (extractable priority pollutants) and for the purgable priority pollutants. These analyses are under way.

NO<sub>x</sub>/SO<sub>x</sub> Program (R. J. Wingender)

Work was done with Richard Doctor and John Harkness (EES) on developing an efficient technique for the simultaneous removal of NO<sub>x</sub>/SO<sub>x</sub> from stack gases in wet scrubbers used in coal-fired power plants and industrial boilers. The first part of the project required developing a protocol for the preparation of the FeEDTA used in the wet scrubber solution. Next, other active metal chelates were proposed for testing, and the solution chemistry for their role in the system was worked out. Based on the >90% efficiency of the systems tested, a

patent application was prepared and submitted in July. This work was cited for "developing a breakthrough in the capability of simultaneously removing NO<sub>x</sub>/SO<sub>x</sub> from stack gases by wet scrubbing techniques used in coal-fired plants and industrial plants," resulting in the presentation of a Pacesetter Award.

Using a nuclear magnetic resonance technique developed in the ACL, the coordination of some chelates to the ferrous ion were measured. This useful and preliminary information is necessary to elucidate the mechanism occurring in solution to remove the NO<sub>x</sub>. Interest in funding wet-scrubber systems technology has since decreased, but has increased in dry scrubber flue gas cleanup. A report was completed on the feasibility of researching the chemistry of the species adsorbed on the surface of fly ash particulates to help elucidate the chemical processes occurring. In particular, special techniques of FTIR and secondary ion mass spectrometry were found to be useful for this study.

#### Dioxin Work (R. J. Wingender)

Early in the year, several steps were worked out from the EPA Region VII method for 2,3,7,8-tetrachlorodibenzo-p-dioxin in soil/sediment. Later, two samples were received from WGN-TV, Ch. 9, and, in the process of analyzing them, the technique and capability for application of the methodology to real samples was refined. One of the samples contained an amount of materials that overwhelmed the Region VII procedure and was successfully analyzed only after application of the size-exclusion cleanup developed in the ACL. Additional funding is being sought to validate this cleanup method.

#### Energy from Municipal Waste Program (R. J. Wingender)

The Energy from Municipal Waste Program required characterization of the mixture produced from the thermal degradation of cellulose in several forms (filter paper, newspaper, wood chips, etc.). Gas chromatography and GC/MS analyses have been conducted. The latest work involved preparing a GC/MS

library with information obtained from Battelle Northwest and from authentic standards to aid identification of the components in these mixtures. This work has been submitted to James Helt (CMT), the program manager, for inclusion in his FY 1985 annual report to the sponsor and includes the tentative identification of 65 of 86 compounds and their relative concentrations in the sample.

Rush Analysis to Save a Life (J. F. Schneider and A. S. Boparai)

On August 23, 1985, a sample of cardboard that contained an unknown "toxic" material was submitted to the ACL. A dock worker became ill after being exposed to the material on the cardboard, and doctors had no clue as to what was causing the problem. The sample was submitted on a rush "life-or-death-situation" basis to see what information could be obtained immediately, and was analyzed with that in mind. The sample was extracted, then GC/IR and GC/MS were used to try to identify the unknown toxin. Within hours, three major components were identified as benzene, benzoic acid, and trichloroethylene. None of the identified components explained all the symptoms that the dock worker was experiencing, but many possibilities were eliminated. The analysis illustrated the power of GC/IR and GC/MS to perform quick and accurate qualitative organic analysis. The spectral search capabilities of our GC/IR equipment saved hours or days of manually comparing unknown and standard spectra. Each method confirmed the results of the other, and confident identifications were made within minutes of sample injection.

Gas Chromatography/Fourier Transform Infrared Spectroscopy/Mass Spectroscopy  
(J. C. Demirgian)

The capabilities of GC/FTIR/MS were established. The instrumentation separates complex organic mixtures and produces IR and MS data simultaneously in real time. The system has been used to analyze for hydrazine impurities and oxidation products (J. Stetter, EES), toluene degradation products (Sundstrand Corporation, R. Cole, EES), and chewing gum flavors and fragrances (Wrigley Company).

Multidimensional Gas Chromatography (J. C. Demirgian)

Multidimensional GC capabilities have been expanded to include dual detectors -- flame ionization and electron capture -- and the system will be used to analyze Freon degradation products.

Gas Chromatographic Rapid Analysis Protocol (J. C. Demirgian)

A new version of a GC computerized rapid analysis protocol has been written and used for analysis of toluene degradation products. The new version identifies peaks, performs linear regression on retention times, and allows the user to select averaged response factors for concentration calculations. The new revision is completely modular in format and can be easily modified for different materials.

Nuclear Waste Extractants (A. S. Boparai)

A solvent extraction process (the TRUEX process) is being developed in the Chemistry Division to separate and concentrate transuranic (TRU) elements, e.g., plutonium and americium, from acidic nuclear waste streams. A mixture of the bifunctional organophosphorous extractant octyl(phenyl)-N,N-diisobutyl-carbamoylmethylphosphine oxide and tributyl phosphate dissolved in carbon tetrachloride is currently being tested for the separation of TRU elements from an aqueous stream generated during the recovery of plutonium from metallurgical scrap. A method was needed to monitor the concentrations of the extractants to maintain optimum extraction efficiency. A GC method was developed that minimized potential decomposition of the two extractants and resulted in a fast, accurate, and precise means of determining simultaneously the concentrations of the two extractants. The method was written up in sufficient detail that it could be used at the Rockwell Hanford Center analytical chemistry laboratory (the sponsoring agency).

Analyses of Hydrazines (A. S. Boparai and J. F. Schneider)

Hydrazine and substituted hydrazines are important rocket fuels. A U.S. Air Force-sponsored project in the EES Division is to investigate safe ways to neutralize these compounds in the event of a spill. One of the decontamination procedures is to treat the hydrazines with bleach. Samples of hydrazines that had been treated with various hypochlorite solutions were analyzed by GC/MS and GC/IR. Several nitrosoamines and other potentially toxic by-products were identified. Results of this study will allow us to develop safer methods for cleanup of hydrazine.

Work for Others and External Interactions

In addition to the work done for Region V of the EPA, the ACL worked on several smaller projects that were funded by sources outside ANL. Among them are:

- Analysis of Zircaloy tubes from the Exxon Nuclear Corporation for Kr gas.
- Thermal analyses and special gas analyses for Los Alamos National Laboratory.
- Analysis of mildly radioactive corrosion products for Commonwealth Edison Company.
- Analysis for deuterium, helium, hydrogen, and neon of gaseous mixtures from the Fermilab bubble chamber.
- Analysis of coal products for the University of Utah.
- Organic analysis of extracts for the William Wrigley Company.
- Analysis for carbonates and silicates for the Energy Research Corporation.

Support Development Funds

The ACL received funding from Support Development Funds (from Laboratory indirect funding) for four projects for FY 1985. Three related projects were funded to improve the capability of the ACL to analyze coal samples:

1. "Determination of Major and Minor Elements in Coal Ash and Other Siliceous Materials by Energy Dispersive X-ray Fluorescence Spectrometry (EDXRF)," by P. C. Lindahl and E. T. Kucera.
2. "Development of Methodology for the Determination of Total Halogens, Especially Fluorine and Chlorine, Phosphorus, and Sulfur in Coal by Ion Chromatography (IC)," by P. C. Lindahl and F. L. Williams.
3. "Determination of Major and Minor Elements in Coal Ash by Inductively Coupled Plasma/Atomic Emission Spectrometry (ICP/AES)," by P. C. Lindahl, I. M. Fox, and E. A. Huff.

E. A. Huff was funded to work on a project entitled, "The Evaluation of Di-n-hexyl-N,N-diethylcarbamoylmethylenephosphonate (DHDECMP) for Actinide Separations." This project successfully demonstrated a method to extend the applicability of the ICP/AES instrument to samples containing actinides.

IV. PROFESSIONAL ACTIVITIES

A. Publications and Reports

The Applicability of Standard Test Methods to the Analysis of Coal Samples for Coal Research

P. C. Lindahl

American Chemical Society Preprints - Division of Fuel Chemistry 30(4), 184 (1985)

Characterization and Optimization of Polyethylene Resins and Gas Pipe

D. G. Ettlinger, L. A. Raphaelian, and D. T. Raske

Annual Report submitted to Gas Research Institute, June 1985

Computer Assisted Analysis of Energy Related Complex Mixtures by Retention Index Gas Chromatography

J. C. Demirgian and V. C. Stamoudis

Argonne National Laboratory Report No. DOE/MC/49533-1836 (ANL/SER-5), January 1985

Determination of Aluminum in Hydrothermal Reaction Fluids by Graphite Furnace Atomic Absorption Spectrophotometry

P. C. Lindahl, K. C. Voight, A. M. Bishop, G. M. Lafon, and W. L. Huang  
Atomic Spectroscopy 5(4), 137 (1984)

Device for Preparing Reduced-Size Sodium Borohydride Pellets for Hydride Generation

P. C. Lindahl

Atomic Spectroscopy 5(6), 234 (1984)

The Direct Determination of Metals in Organic Matrices by Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES)

E. A. Huff

Argonne National Laboratory Report No. ANL/ACL-85-2, 1985

Electrically Heated Quartz Cell and Holder for an Atomic Absorption Hydride Generation System

P. C. Lindahl

Atomic Spectroscopy 6(4), 123 (1985)

Flue Gas Desulfurization/Denitrification Using Metal-Chelate Additives

J. Harkness, R. Doctor, and R. J. Wingender

Patent Application, August 1985

Fluorescence Quenching and Halide-Ion Nuclear Magnetic Resonance Spectroscopy as Probes for Metal Binding to Proteins

J. J. Pesek, R. J. Dowd, and J. F. Schneider

Analytica Chimica Acta 170, 187 (1985)

GC/Matrix Isolation/FTIR Applications: Analysis of PCBs

J. F. Schneider, G. T. Reedy, and D. G. Ettlinger

Journal of Chromatographic Science 23, 49 (1985)

Half-Life of  $^{60}\text{Fe}$

W. Kutschera, P. J. Billquist, D. Frekers, W. Henning, K. J. Jensen,  
Ma Xiuzeng, R. Pardo, M. Paul, K. E. Rehm, R. K. Smith, and J. L. Yntema  
Nuclear Instruments and Methods in Physics Research B5 233, 430 (1985)

High Resolution Gas Chromatography/Matrix Isolation Infrared Spectrometry  
G. T. Reedy, D. G. Ettinger, J. F. Schneider, and S. Bourne  
Analytical Chemistry 57, 1602 (1985)

Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) in  
Support of Nuclear Waste Management

E. A. Huff and E. P. Horwitz  
Spectrochimica Acta 40B, 279 (1985)

Laboratory Studies of a Breached Nuclear Waste Repository in Basalt

M. G. Seitz, D. L. Bowers, T. J. Gerding, and G. F. Vandegrift  
Argonne National Laboratory Report No. ANL/84-16 and NUREG/CR-3710,  
September 1984

Procedure for Analysis of PCBs, Pesticides, and Priority Pollutants in  
Aqueous Media

R. J. Wingender  
Analytical Chemistry Laboratory Quality Assurance Document No.  
C-0030-0323, August 28, 1985

Procedure for the Determination of Total Organic Carbon in Sediment Samples

I. M. Fox and P. C. Lindahl  
Analytical Chemistry Laboratory Quality Assurance Document No.  
C-0030-0321, November 11, 1985

Products of the Neutralization of Hydrazine Fuels with Hypochlorite

K. Brubaker, J. R. Stetter, J. C. Demirgian, A. S. Boparai, and J. F. Schneider  
in Proceedings of the 1985 JANNAF Safety and Environmental Protection  
Subcommittee Meeting, Naval Post Graduate School, Monterey, CA,  
November 4-6, 1985

B. Oral Presentations

Analyzing the Rock That Burns - Atomic Spectroscopy in Coal Analysis

P. C. Lindahl  
Society for Applied Spectroscopy-Chicago Section, Chicago, IL,  
January 8, 1985

Analyzing the Rock That Burns - Atomic Spectroscopy in Coal Analysis

P. C. Lindahl  
Illinois State Geological Survey, Champaign, IL, June 14, 1985

The Applicability of Standard Test Methods to the Analysis of Coal Samples  
for Coal Research

P. C. Lindahl  
American Chemical Society Annual Meeting, Chicago, IL, September 10,  
1985

Application of GC/MS and GC/MI/FTIR to Fluid Degradation Studies

A. S. Boparai and G. T. Reedy

The 1985 Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, New Orleans, LA, February 25-March 1, 1985, presented by A. S. Boparai

Chemical and Toxicological Characterization of Neutral Components in Coal-Gasification Condensates

V. C. Stamoudis, D. A. Haugen, J. C. Demirgian, P. Heffley, K. Picel, and D. Eikens

First DOE/OHER Contractors Meeting on Research in Complex Mixtures Argonne, IL, June 17-18, 1985, presented by V. Stamoudis

Coal Characterization for the Premium Coal Sample Program

P. C. Lindahl

Department of Educational Programs Seminar, Argonne National Laboratory, March 8, 1985

A Comparison of GC/IR Interfaces: The Light Pipe Versus Matrix Isolation

J. F. Schneider and J. C. Demirgian

The 1985 American Chemical Society Fall Meeting, Chicago, IL, September 1985, presented by J. F. Schneider

Computerized Gas Chromatography Analysis Based on Calibrated Peaks and Relative Retention Indices

V. C. Stamoudis, J. C. Demirgian, D. Eikens, and K. Picel

The 1985 Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, New Orleans, LA, February 25-March 1, 1985, presented by V. C. Stamoudis

Correlations of Chemical, Microstructural, and Mechanical Properties of Polyethylene Piping Materials

L. A. Raphaelian and D. G. Ettinger

Fifth Annual Plastic Piping Materials Workshop, Gas Research Institute, Rosemont, IL, June 19-21, 1985, presented by L. A. Raphaelian

Cryogenic Matrices Applied to Analytic and Spectroscopic Studies

G. T. Reedy, D. G. Ettinger, and J. F. Schneider

The 1985 Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, New Orleans, LA, February 25-March 1, 1985, presented by G. T. Reedy

Data Communication and Control of a Hewlett Packard 5880A Gas Chromatograph by a Perkin Elmer Data Station

J. C. Demirgian

Scientific Computing and Automation Conference and Exposition, Atlantic City, NJ, May 1-3, 1985

Data Processing Technique for Deconvoluting Capillary Column GC/MS Data; Its Use in the Analysis of Priority Pollutants

L. A. Raphaelian

1985 Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, New Orleans, LA, February 25-March 1, 1985

Determination of Metals in Organic Matrices

E. A. Huff

3rd ACL Technical Meeting, Argonne National Laboratory, October 23, 1985

Development and Application of GC/FTIR/MS Capability

J. C. Demirgian

4th ACL Technical Meeting, Argonne National Laboratory, March 14, 1985

Dissolution Technique Using Sealed Glass Tubes at Elevated Temperatures

K. J. Jensen

4th ACL Technical Meeting, Argonne National Laboratory, March 14, 1985

Environmental Analysis by GC/MI/FTIR

J. F. Schneider

4th ACL Technical Meeting, Argonne National Laboratory, March 14, 1985

Environmental Analysis by GC/Matrix Isolation/IR

J. F. Schneider, G. T. Reedy, and D. G. Ettinger

The 1985 Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, New Orleans, LA, February 1985, presented by J. F. Schneider

Experimental Optimization of GC/FTIR/MS Systems

J. C. Demirgian and D. Eikens

The 1985 Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, Paper No. 496, New Orleans, LA, February 25-March 1, 1985, presented by J. C. Demirgian

Gas Chromatography/Mass Spectrometry in the Analysis of Organics in Coal, Coal Liquids, Shale, Tar Sands, and Petroleum Oil

L. A. Raphaelian

Five lectures, University of Wyoming, Laramie, WY, April 1-5, 1985

GC/Matrix Isolation/FTIR Applications: Analysis of PCBs

J. F. Schneider

3rd ACL Technical Meeting, Argonne National Laboratory, October 23, 1985

Luminescence of Adsorbed Molecules

D. G. Ettinger

3rd ACL Technical Meeting, Argonne National Laboratory, October 23, 1985

Migration of Elements in the Sub-Sea Bed Sediments

C. S. Sabau

4th ACL Technical Meeting, Argonne National Laboratory, March 14, 1985

PCB Analysis by Capillary Gas Chromatography Coupled with Matrix Isolation Fourier Transform Infrared Spectroscopy

J. F. Schneider and G. T. Reedy

The 1984 International Chemical Congress of Pacific Basin Societies, Honolulu, HI, December 1984, presented by J. F. Schneider

Precise Determination of Uranium in Spent Reactor Fuel by Mass Spectrometry  
Utilizing Internal Standard Techniques

E. L. Callis and D. G. Graczyk

The 33rd Annual Conference on Mass Spectrometry and Allied Topics,  
San Diego, CA, May 26-31, 1985, presented by E. L. Callis

Preparation and Analysis of Iron Isotopic Standards

E. L. Callis

3rd ACL Technical Meeting, Argonne National Laboratory, October 23, 1985

Uranium and Plutonium Determinations for Evaluation of High Burnup Fuel  
Performance

R. R. Heinrich, R. J. Popek, D. L. Bowers, A. M. Essling, E. L. Callis,  
and P. J. Persiani

Karlsruhe International Conference on Analytical Chemistry in Nuclear  
Technology, at the Kernforschungszentrum in Karlsruhe (KfK), West  
Germany, June 3-6, 1985, presented by R. R. Heinrich

**C. Awards**

David W. Green

Master of Business Administration, Executive Program at the University  
of Chicago Graduate School of Business

Selected for membership in Beta Gamma Sigma

Joseph R. Stetter

Recognized as Senior Member of the Instrument Society of America

Florence L. Williams

Outstanding Service Award for her contribution to the National  
Organization of Black Chemists and Chemical Engineers

Ronald J. Wingender

Pacesetter Award, July 1985, for "developing a breakthrough in the  
capability of simultaneously removing NO<sub>x</sub>/SO<sub>x</sub> from stack gases by wet  
scrubbing techniques"

**D. Meetings Attended**

A. S. Boparai

The 1985 Pittsburgh Conference and Exposition on Analytical Chemistry and  
Applied Spectroscopy, New Orleans, LA, February 28-March 1, 1985

E. Larry Callis

The 33rd Annual Conference on Mass Spectrometry and Allied Topics,  
San Diego, CA, May 26-31, 1985

J. C. Demirgian

Scientific Computing and Automation Conference and Exposition, Atlantic  
City, NJ, May 1-3, 1985

The 1985 Pittsburgh Conference and Exposition on Analytical Chemistry  
and Applied Spectroscopy, New Orleans, LA, February 1985

D. G. Ettinger

Fifth Annual Plastic Piping Materials Workshop, Gas Research Institute, Rosemont, IL, June 19-21, 1985

D. W. Green

Second DOE Analytical Managers' Conference, Sandia National Laboratories, Albuquerque, NM, November 7-8, 1984 (discussion leader)

Information Meeting, Analytical Chemistry Division, Oak Ridge National Laboratory, Oak Ridge, TN, July 23, 1985

Robert R. Heinrich

Karlsruhe International Conference on Analytical Chemistry in Nuclear Technology, at the Kernforschungszentrum in Karlsruhe (KfK), West Germany, June 3-6, 1985

Peter C. Lindahl

American Society for Testing and Materials (ASTM) Committee D5 on Coal and Coke, Norfolk, VA, October 15-17, 1984

American Society for Testing and Materials (ASTM) Committee D5 on Coal and Coke, Denver, CO, May 20-23, 1985

Center for Research on Sulfur in Coal - Characterization and Analysis Task Group, Champaign, IL, June 14, 1985

American Chemical Society Annual Meeting, Chicago, IL, September 10-11, 1985

13th Plenary Meeting of the International Organization for Standardization Technical Committee 27 (ISO/TC 27) on Solid Mineral Fuels, Lexington, KY, September 23-25, 1985

Leo A. Raphaelian

SuperINCOS Electronics, Finnigan MAT Institute, Cincinnati, OH, November 5-9, 1984

The 1985 Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, New Orleans, LA, February 25-March 1, 1985

Fifth Annual Plastic Piping Materials Workshop, Gas Research Institute, Rosemont, IL, June 19-21, 1985

Gerald T. Reedy

The 1985 Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, New Orleans, LA, February 25-March 1, 1985

John F. Schneider

The 1984 International Chemical Congress of Pacific Basin Societies, Honolulu, HI, December 16-21, 1984

The 1985 Pittsburgh Conference and Exposition on Analytical Chemistry and Applied Spectroscopy, New Orleans, LA, February 25-March 1, 1985

FTIR User Training Course, Nicolet Instrument Company, Madison, WI,  
June 17-21, 1985

The 190th American Chemical Society National Meeting, Chicago, IL,  
September 8-13, 1985

University-Industry Poster Session on Research in Chemistry, Chemical  
Engineering and Biotechnology, Amoco Research Center, Naperville, IL,  
October 23, 1985

Florence L. Williams

Twelfth Annual Meeting of the National Organization of Black Chemists  
and Chemical Engineers, Minneapolis, MN, May 21-24, 1985

Ronald J. Wingender

National American Chemical Society Meeting, Third Symposium on  
"Chlorinated Dioxins and Dibenzofurans in the Total Environment,"  
Miami, FL, April 29-May 2, 1985

E. Professional Organization Leadership

Ralph W. Bane

Task Group Leader, Direct Determination of Oxygen, American Society for  
Testing and Materials Committee D.5

Peter C. Lindahl

Chairman, Section 29.01 on Atomic Absorption Methods, American Society  
for Testing and Materials Committee D.5 on Coal and Coke

Convenor (Chairman), Working Group 14 on Trace Elements in Solid Mineral  
Fuels: International Organization for Standardization Technical Committee  
27 (ISO/TC 27) on Solid Mineral Fuels

David W. Green

President-Elect, Analytical Laboratory Managers Association

L. A. Raphaelian

President, Chicago Chemists' Club

Alternate Councilor, Chicago American Chemical Society

Florence L. Williams

Treasurer, the National Organization of Black Chemists and Chemical  
Engineers

Executive Committee, the National Organization of Black Chemists and  
Chemical Engineers