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ORNL-6153

**Chemical Technology Division
Progress Report
for the Period April 1, 1983,
to March 31, 1985**

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Chemical Technology Division Progress Report for the Period April 1, 1983, to March 31, 1985

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Date of Issue—October 1985

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ORNL-5383	Period Ending March 31, 1978
ORNL-5542	Period Ending March 31, 1979
ORNL-5757	Period April 1, 1979, to March 31, 1981
ORNL-5933	Period April 1, 1981, to March 31, 1983

PREFACE

This progress report is a summary of the research and development efforts conducted in the Chemical Technology Division during the period April 1, 1983, to March 31, 1985. More information regarding work in the various program areas can be obtained from the topical reports and journal articles cited as references.

CONTENTS

1. INTRODUCTION AND DIVISIONAL OVERVIEW	1
2. FISSION ENERGY	6
2.1 Preparation of Breeder Reactor Fuel Material	6
2.2 FFTF Fuel Dissolution Studies	7
2.3 SETF Studies	7
2.4 Secondary Cleanup of SRP and ICPP Solvents	8
3. NUCLEAR AND CHEMICAL WASTE MANAGEMENT	9
3.1 Waste Immobilization Technologies	9
3.2 Integrated Data Base Program	12
3.3 Transuranic Waste Management	19
3.4 Repository Waste Characteristics	21
3.5 Sedimentary Rock Program	22
3.6 Assessment of Waste Management Technologies	25
4. ENVIRONMENTAL CONTROL TECHNOLOGY	27
4.1 Support to the Oak Ridge Complex	27
4.2 Fossil Energy Programs	33
4.3 Water Quality Programs	36
5. BASIC SCIENCE AND TECHNOLOGY	41
5.1 Chemical Sciences	41
5.2 Materials Sciences	54
6. BIOTECHNOLOGY PROGRAMS	59
6.1 Biological Research	59
6.2 Bioengineering Research	62
6.3 Bioprocess Development	65
6.4 Health and Environmental Monitoring Technology	73
7. TRANSURANIUM-ELEMENT PROCESSING	76
7.1 TRU Operations	76
7.2 Large Einsteinium Activation Program (LEAP)	77
7.3 Special Projects	78

8. NUCLEAR REGULATORY COMMISSION PROGRAMS	80
8.1 Nuclear Reactor Safety	80
8.2 Repository Licensing Support	90
9. CONSOLIDATED EDISON URANIUM SOLIDIFICATION PROJECT	92
9.1 Introduction	92
9.2 Process Description	92
9.3 Construction Activities	93
9.4 CEUSP Process Flowsheet	96
9.5 Plan of Operation	98
10. RADIOACTIVE MATERIALS PRODUCTION	100
10.1 Uranium-233 Processing	100
10.2 Californium-252 Industrial Sales/Loans	100
11. COMPUTER/ENGINEERING APPLICATIONS	102
11.1 Transportation Program	102
11.2 Process and Systems Analysis	105
12. MISCELLANEOUS PROGRAMS	109
12.1 Fusion Energy Studies	109
12.2 Three Mile Island Support Studies	109
12.3 FUSRAP Stabilization Technology: Development and Applications	110
12.4 Molten Salt Reactor Decommissioning	113
12.5 Recovery of Alumina and Other Useful Minerals from Fly Ash	118
12.6 Studies Related to Postulated Accidents at the Savannah River Production Reactor	118
12.7 Process Monitoring for Coal Conversion	119
12.8 Support to Martin Marietta Energy Systems, Inc., Engineering	121
ADMINISTRATIVE SUMMARY	123
Publications and Oral Presentations	125
Patents	189
Awards, Honors, and Recognitions	191
Special Assignments	197
Consultants	201
Seminars	205
Internal Program Reviews	207
Staffing Level and Financial Summary	208
Chemical Technology Division Organization Chart	209

1. INTRODUCTION AND DIVISIONAL OVERVIEW

The Chemical Technology Division (CTD), which has approximately 250 staff members, is one of the largest research and development (R&D) divisions at Oak Ridge National Laboratory (ORNL). The Division has outstanding facilities and personnel capabilities in chemical engineering and chemistry, as well as significant competence in several other engineering and scientific disciplines. It has a major role in a broad range of programs, with particular emphasis on those for which chemical processing is the principal element. These programs are designed to explore certain aspects of nuclear and nonnuclear energy and to develop advanced technology for selected national priorities. Research and development activities include studies in basic and applied science and engineering, engineering development and assessments, and the operation of various pilot plants and facilities. Most of the research programs are funded by the U.S. Department of Energy (DOE), although some important projects are carried out for other governmental agencies, and limited support is derived from industrial sources.

The nuclear fuel cycle, which traditionally has been one of the Division's most important R&D areas, has continued to decline in funding level during this report period, both because of the national malaise concerning nuclear energy and because of shifts in programmatic emphasis within the Consolidated Fuel Reprocessing Program (CFRP) at ORNL. However, we are continuing to conduct important fuel reprocessing experiments on highly irradiated fuel from the Fast Flux Test Facility (FFTF) at Richland, Washington. Our work in radioactive waste management includes a wide range of projects, such as the development and demonstration of cement grouts for waste fixation, the establishment and maintenance of a national data base on present and future quantities and types of nuclear wastes in the United States, and the performance of evaluations of geologic media as hosts for permanent waste disposal.

Fossil energy programs in the Division have become focused on environmental control technology, especially (though not exclusively) for wastes from coal conversion processes. This is now our largest fossil energy support area, and we do not anticipate changes in funding or emphasis during the near future.

The basic studies in chemistry and engineering not only provide fundamental data that are useful to the scientific community as a whole, but also serve as a source of new ideas and provide impetus and direction for the more applied programs. For example, the results obtained in chemical separations research are often directly applicable to our various technical projects and thus can be used to solve definitive problems or streamline operational details.

The Division's biotechnology programs range from laboratory investigations of enzyme kinetics to wastewater treatment processes that utilize advanced bioreactor systems. We are also continuing a basic program aimed at increasing our understanding of the biophotolysis of water. Bioprocessing R&D is recognized as a "new initiative" area in the current ORNL Institutional Plan, and interest in biotechnology seems to be increasing among federal funding agencies. Division personnel were actively involved in the establishment, in April 1984, of the Oak Ridge Bioprocessing Research Facility at ORNL. This is a user facility/resource with state-of-the-art equipment that is being made available to university, national laboratory, and industrial scientists and engineers. Technology transfer in this area may also be assisted by the discussions now under way between personnel in this Division and at the University of Tennessee to establish a Memorandum of Understanding for collaborative bioprocessing R&D.

We are continuing to operate the facility for transuranium element processing (TRU) and the radiochemical processing plant (RPP) for special nuclear materials. Our transuranium element pro-

cessing activities are being enlarged to include a packaging service for the ^{252}Cf sales program. The RPP is working to solidify nearly 1000 kg of liquid waste (containing mixed uranium isotopes) that has been stored in a tank at ORNL for some time. The solidified material will be stored at the RPP as a highly radioactive waste.

Our programs conducted in support of the U.S. Nuclear Regulatory Commission (NRC) include investigations concerning the transport and deposition of fission products from failed fuels in nuclear reactors and the assessment of DOE proposals for radioactive waste disposal in geologic media. We are also continuing to provide scientific and technical assistance for cleanup activities to the Three Mile Island Unit-2 (TMI-2) Nuclear Power Station.

Some specific highlights of our accomplishments during the period April 1, 1983–March 31, 1985, are listed here:

- Major progress has been made in the Consolidated Edison Uranium Solidification Program (CEUSP). During the past 2 years, construction of the facility was finished, the equipment and process were tested, the Final Safety Analysis Report and Operational Safety Regulations were prepared, and the readiness review by ORNL and ORO safety committees was successfully completed. Despite a major setback in 1984, when 1500 faulty piping components had to be replaced, operation began in April 1985, almost 3 months ahead of the scheduled milestone.
- The Division supported operation of the New Hydrofracture Facility in the disposal of granite tank sludges by establishing operating procedures, setting quality assurance and quality control requirements, and coordinating analytical support services.
- Information on cement-based grouts was provided to support Rockwell-Hanford's decision to begin construction of a Transportable Grout Facility, in FY 1987, for the solidification of low-level waste in shallow trenches.
- Because of the increased safeguards required for fissile materials, the DOE has designated Building 3019 as a national repository for ^{233}U waste from other DOE laboratories. During this report period, safeguards at Building 3019 were upgraded, and essentially all fissile material was moved from the vault to storage wells within the building. Massive cover blocks and locking plates have been installed over the shielded wells, so that theft cannot be accomplished without significant time delays that would allow for security reinforcement.
- Other major improvements to Building 3019 are in progress, such as improved building ventilation, new fans and ducts for cell off-gas, additional containment, reworking of the liquid waste liner, and removal of old equipment.
- Two extensive transuranium-element campaigns were conducted at the TRU Facility in which 26 HFIR targets were processed to produce 72 mg of ^{249}Bk , 809 mg of ^{252}Cf , 3.3 mg of ^{253}Es , and 2 μg of ^{257}Fm , as well as recycle curium and americium.
- An experimental program was carried out in collaboration with the ORNL Chemistry Division to measure ^{253}Es and ^{254}Es cross sections for thermal and epithermal neutrons. Preliminary results indicate that, under properly controlled conditions, $\sim 40 \mu\text{g}$ of ^{254}Es can be produced by a special irradiation of ^{252}Cf shielded with cadmium. This quantity of ^{254}Es (ten times the amount currently available) will afford new opportunities in heavy-element research on chemical properties of transactinide elements and in the study of nuclear properties of the heavier nuclides. It may even be possible to produce elements in the "island of stability" by using ^{254}Es as the accelerator target.
- Solvent extraction tests to study the effects of Purex flowsheet modifications were made with three batches of FFTF fuel; the fuel burn-ups were $\sim 2,200$, $\sim 30,000$, and $\sim 55,000$ MWd/t, with cooling times of 2, 1.3, and 1 year, respectively. A test with FFTF fuel irradiated to 100,000 MWd/t is under way. These were the first efforts toward processing of FFTF fuel and were done in the Solvent Extraction Test Facility (SETF), which is installed in one of the hot cells at the TRU Facility.
- At the request of DOE, we have made plans and started modifications of the Thorium-Uranium Recycle Facility (TURF) to accommodate the DOE ^{252}Cf Industrial Sales/Loan Program. The responsibility for this program is a fitting extension of our present californium production operations.

- Gel-sphere equipment installed in glove boxes at Building 3019 was operated during the period April 1983–September 1984 to produce ~15 kg of mixed-oxide spheres (25% PuO₂) for use in the DIPRES program. The initial quantities were used by the Pacific Northwest Laboratory (PNL) and the Hanford Engineering Development Laboratory (HEDL) to develop pellet-pressing technology; the remaining 11 kg will be used by HEDL to prepare fuel rods for irradiation testing in the FFTF.
- A continuous process sol-gel line was installed, tested, and operated in Building 3053; this facility is capable of producing spherical uranium oxide particles at the rate of 1 kg/h.
- An Environmental Control Technology (ECT) group was established to provide R&D and engineering design support to the Oak Ridge Complex in environmental control work. This group has simulated the Oak Ridge Gaseous Diffusion Plant (ORGDP) Sewage Treatment Plant treating Y-12 site "mop" waters; designed, tested, and evaluated a packed-bed reactor for removal of hydrogen fluoride vapor from vent-gas streams at the Y-12 site; and performed an engineering analysis of the ORNL Process Waste Treatment Plant (PWTP) at ORNL. They are continuing work on the PWTP and are developing information to support designs for the ORNL Nonradioactive Wastewater Treatment Plant.
- A 2-year evaluation of the ANFLOW process for anaerobic biotreatment of municipal wastewater was completed, using a 189,000-L/d (50,000-gal/d) pilot plant in Knoxville. The effluent water met the EPA secondary treatment discharge limits of 30 mg/L for total suspended solids (TSS) and biological oxygen demand (BOD) the vast majority of the time. Sludge production rates for the ANFLOW system were 75 to 80% less than those for systems based on either a primary clarification plus activated sludge process or a primary clarification plus trickling filtration process. A materials balance model prepared by Chem Tech personnel was shown to predict the sludge accumulation, using measurable influent and effluent parameters.
- Volume reduction of simulated cellulosic wastes by anaerobic digestion was demonstrated, using a 75-L digester. More than 80% of the solids were degraded to off-gas and residual, scrubble, organic chemicals. The overall volume reduction ratio is estimated to be ~20/1. A dynamic process model was developed and demonstrated, and preliminary effluent characterization for ultimate disposal was completed. A full-size digester facility for the DOE plants was designed for a General Plant Project (GPP).
- Process condensate wastewaters from two fixed-bed gasifiers have been treated successfully using a laboratory-scale, fixed-film, fluidized-bed bioreactor. Both dilute (1 to 2%) raw wastewater and more concentrated (50%) stripped wastewater were treated continuously for periods of 1 to 3 months. Removal of phenols exceeded 99%, and the decrease in COD was typically 75 to 85% for the 50% stripped wastewater, with a hydraulic residence time in the bed of ~5 to 7 h.
- A new program was initiated in biodenitrification of wastewaters to support National Lead of Ohio's operation of the DOE Feed Materials Production Center at Fernald, Ohio.
- A laboratory system for monitoring the quality of the feed to the Kerr-McGee critical solvent deashing (CSD) unit was developed, fabricated, and installed in the control laboratory of the coal conversion pilot plant located at Wilcoxville, Alabama. Comparisons have shown reasonable agreement between manual measurements of feed solubility in the deashing solvent (requiring ~2 h) and optical measurements done on the ORNL system (requiring ~30 min).
- The Oak Ridge Bioprocessing Research Facility, a user facility/resource, has been established and a descriptive brochure prepared. This is a collaborative undertaking with the Biology Division.
- A 10-kg core-melt apparatus began successful operation under NRC sponsorship. Initial experiments identified an unexpected mode of premature silver control-rod failure and the subsequent formation of an extremely hard phase from the resulting Ag-Zr-Fe alloy. The discovery of this alloy is of importance to the operations for cutting into the damaged reactor vessel at the TMI-2 Nuclear Power Station. Discovery of the control-rod failure mode explained the unexpected presence of silver in the TMI-2 reactor vessel and identified an important design feature in LWRs that needs to be addressed.

- The NRC-sponsored Fission Product Release Program has provided significant new information toward characterization of the chemical forms of fission-product cesium and behavior of these forms following their release from LWR fuel under accident conditions. Cesium was found to be released as CsI and CsOH, with the latter undergoing subsequent reactions with oxidized Zircaloy and silica.
- A special experiment was conducted in the Fission Product Release Program to investigate the mechanism of release of tellurium from overheated LWR fuel rods. The results show this mechanism unambiguously.
- A theoretical model of the post-accident reactor cooling system at TMI-2 was developed that provided quantitative information on the leakage of coolant from the system and the inventory of fission products in the remains of the reactor core.
- A 2-d meeting was arranged and hosted by ORNL for representatives of DOE, the states, Indian nations, and other groups concerned about the transportation of nuclear wastes to prospective repositories. The openness of these discussions was appreciated by all participants.
- The integrity and survival characteristics of the TRUPACT shipping container for transuranic wastes were tested at the ORNL Drop Test Facility. This trailer-truck-sized container was dropped from several orientations for distances up to 30 m onto an unyielding surface.
- Transportation logistics were determined for the U.S. Army's use in assessing demilitarization options for unserviceable chemical munitions.
- Externally applied, pulsed, electric fields have been shown to induce droplet oscillations and generate circulation within and around droplets, resulting in significant increases in mass-transfer rates. This is both effective and much more energy efficient than conventional agitation methods for promoting mass transfer in fluids.
- Colloidal platinum precipitated directly onto chloroplast membranes has been found to be capable of catalyzing the simultaneous photoevolution of hydrogen and oxygen. This demonstrates the direct interaction of a nonbiological catalyst with a biological membrane, and the two-component system is the simplest yet derived for photochemically splitting water molecules.
- The scattering of laser light by extremely fine particles has been shown to reveal complex flow patterns around fluid drops, and the technique is believed to have numerous other scientific applications. The intensity of the laser light allows the utilization of the few minute particles that are present in essentially all fluids. Video recordings, using low shutter speeds, of the light patterns produced by these particles provides for rapid experimental evaluation of both the streamlines and fluid velocities under steady and transient flow conditions.
- A state-of-the-art dynamic laser-light-scattering facility has been developed which features multiple-angle scattering, both autocorrelation analysis and Fabry-Perot interferometry, and computerized control of the experimental procedure, data acquisition, and theoretical analysis. The development of new seed materials has allowed use of the facility to study transport properties of fluids in the critical region and the initiation of a new program to investigate crystallization in the <10-nm range.
- The application of electric fields to stop the motion of suspended particles has provided an opportunity for significant advances in the understanding of hydrodynamics and mass transfer in packed and fluidized beds. The ability to "hold" particles in expanded (fluidized) beds allows studies of fixed-bed hydrodynamics and mass transfer to be conducted for a wide range of packing densities (void fractions). These studies should resolve problems and inconsistencies in theoretical models of both fixed and fluidized beds.
- The design concept for the annular chromatograph was expanded to include a radial electrical field, giving a system in which both chromatography and electrophoresis can be conducted simultaneously and continuously. A simple mathematical model of this separation method was also developed.
- Experimental and theoretical analyses of tritium removal rates by hydride-forming sorbents have promoted understanding of the rate-limiting mechanisms involved in tritium sorption from liquid lithium in fusion reactors. This work also provides a basis for "designing" the structures and compositions of alloy sorbents to provide maximum tritium sorption rates while maintaining acceptable sorption capacity.

- Division personnel have produced a computerized model of an entire nuclear fuel reprocessing plant. The Breeder Reactor Engineering Test (BRET) facility flowsheet was modeled on the Advanced Simulator for Process Engineering (ASPEN) code and was used to establish material and energy balances for the design of the plant.
- A patent application has been prepared for a novel reactor concept having inherently safe features. The Process-Inherent Ultimate Safety (PIUS) type of boiling water reactor uses fluidic valves and novel power-limiting controls to ensure safe operation without the use of moving parts in the safety system.

2. FISSION ENERGY

Fission energy research and development in the Division during this report period has spanned two primary programs. In one of these programs, some unique and important dissolution and solvent extraction work with fast reactor spent fuels was completed. Most of this work was done for the Consolidated Fuel Reprocessing Program (CFRP); a smaller project was completed under contract for Rockwell International Corp., Hanford, Washington. We also conducted specific studies on solvent cleanup under contract for Westinghouse Idaho Nuclear Company, Inc.

Other work done by Chem Tech for CFRP included completing the preparation of 12 kg of uranium-plutonium mixed-oxide microspheres for fuel fabrication and irradiation tests. Detailed results from these studies are not included here, because of regulations governing the distribution of applied technology information. Results of this work are published in CFRP topical quarterly progress reports. The most recent of these publications is the *Consolidated Fuel Reprocessing Program Progress Report for Period January 1 to March 31, 1985*, ORNL/TM-9584, published April 1985.

The second area of Chem Tech's fission energy development has been in support work for the High-Temperature Gas-Cooled Reactor (HTGR) program. Again, the applied technology information is not reported here in compliance with DCE regulations. Most of this work has been reported in internal documents and annual progress reports. During the past 2 years, the annual reports in this area have been the *High-Temperature Gas-Cooled Reactor Technology Development Program Annual Progress Report for Period Ending December 31, 1983*, ORNL-6053, published in June 1984; and the *High-Temperature Gas-Cooled Reactor Technology Development Annual Progress Report for Period Ending December 31, 1984*, ORNL-6192 (in publication).

2.1 PREPARATION OF BREEDER REACTOR FUEL MATERIAL

In 1979 the CFRP at ORNL initiated a program for the development of advanced conversion processes with potential for simplifying and improving the conversion/pellet fabrication flowsheet for recycle plutonium.¹ This activity required major interfaces with breeder fuel fabricators; therefore, a joint conversion/fabrication development program was initiated to be shared between ORNL, the Hanford Engineering Development Laboratory (HEDL), and Pacific Northwest Laboratory (PNL). Under this program, ORNL is responsible for overall program management and conversion process development, PNL for laboratory-scale fabrication development, and HEDL for engineering-scale fabrication development and confirmatory irradiation testing.

An evaluation of advanced conversion processes led to the selection of the DIPRES (Direct PRESS Spheroidized) process as the candidate for development because it has the largest potential for process and product improvements. The DIPRES process utilizes a gel-sphere conversion process to provide a spherical feed material for pellet fabrication. The free-flowing nature of the spherical conversion product allows it to be fed directly to pellet presses (i.e., direct press feed) in place of the conventional, mechanically blended powder feed. This is advantageous for remote fabrication. The DIPRES feed is prepared by an internal gelation process developed at ORNL. Other internal gelation methods have been developed previously at ORNL, in the Netherlands, and in Switzerland.

1. E. D. Collins, M. D. Jackson, C. W. Griffin, D. E. Rasmussen, and R. E. Norman, "Development of the DIPRES Process for the Fast Breeder Reactor Fuel Cycle," *Trans. Am. Nucl. Soc.* 47, 193-94 (1984).

The perceived advantages of DIPRES feed are in flowability and homogeneity, allowing simplification of the fuel fabrication process, improved material handling and transport, decreased dust generation and personnel exposure, and simplified design and maintenance of remotely operated equipment.

During a 2-year critical evaluation of the DIPRES process, its feasibility was established, and pellets were prepared from small (~50-g) batches of mixed-oxide (MOX) feed to meet specifications applicable to fuel for the Fast Flux Test Facility (FFTF). In addition, the perceived DIPRES advantages were confirmed. These results led to a joint recommendation that the DIPRES process should continue to be developed for use in the breeder fuel cycle.

Since the critical evaluation was completed, the DIPRES program has emphasized extending the process to a larger scale. Consequently, the DIPRES MOX batch size was increased from the 40- to 60-g range in FY 1981 to the 200- to 400-g range in FY 1982 and to kilogram size in FY 1983. For the kilogram-scale production, new glove-box-contained equipment was installed and tested in FY 1983 and was used in FY 1984 to produce ~15 kg of microspheres for pellet fabrication studies and irradiation testing. Also during this report period, a continuous, uranium-only DIPRES feed line was developed, installed, and successfully operated at a production rate of 1.5 kg of uranium per hour.

A 5-year program for qualification of the DIPRES process was developed in FY 1982 and was concentrated on making an irradiation test in the FFTF. This project required preparation of the DIPRES feed material at ORNL, fabrication of fuel pellets and FFTF fuel pins at HEDL, irradiation in the FFTF, and examination of the irradiated fuel pins for performance verification at HEDL. The DIPRES feed material was prepared at ORNL during FY 1984, and the kilogram-scale production line has now been placed in standby. Also in FY 1984, scale-up tests on the pellet fabrication process development were made at HEDL, and the DIPRES pellets and fuel pins are being fabricated in FY 1985. During FY 1986 and FY 1987, the fuel pins will be irradiated in the FFTF, and, in FY 1988, a post-irradiation examination of the fuel pins is planned.

2.2 FFTF FUEL DISSOLUTION STUDIES

A study of FFTF fuel characteristics was completed during FY 1984. Details of the results obtained in this study are not included here because regulations stipulate that such information must be distributed only as Applied Technology (category UC-86T). However, the general areas of investigation are summarized below.

Several fuel pins from the FFTF fuel assemblies were sheared in a single-pin shear in preparation for dissolution studies. The fuel represented 36,000- to 55,000-MWd/t levels of peak burnup. Cooling periods for these fuel assemblies were, respectively, 1.5 years and 1 year. The shearing pressure was measured along the fuel pins to provide information for shear design. Fines (particles) were collected and sieved to determine the capabilities that will be required for handling powdered fuel solids. The temperature buildup within a container of fuel was also determined and compared with calculations.

Fuel dissolution was investigated in hot-cell facilities and, on a larger scale, in conjunction with the ORNL Solvent Extraction Test Facility (SETF). Fuel dissolution times and characteristics were evaluated in the hot-cell studies. The stability of dissolved fuel solutions was determined by visual inspection during more than 30 d. During the SETF dissolution tests, the solution temperature, the solution density, and the gas pressure difference between the dissolver and the off-gas header tank were determined. The pressure drop in the off-gas line was measured to provide a qualitative indication of the rate of gas evolution. In addition, samples of the dissolver solution were withdrawn during the dissolution and analyzed for plutonium, uranium, free hydrogen ions, and some of the fission products (those measured in a routine gamma scan). The final sample from the second dissolution was also analyzed for ^{129}I by neutron-activation analysis.

Some glove-box experiments were performed to investigate the effects of high uranium concentrations on plutonium polymerization in solutions with U/Pu ratios up to 500.

2.3 SETF STUDIES

The SETF is located in one of the heavily shielded hot cells of the Transuranium Processing

Plant (TRU), and its operation is supported by CFRP. The SETF, which became operational in 1978, has been used to study Purex flowsheets for the reprocessing of both light water reactor (LWR) and fast breeder reactor (FBR) fuels; the most recent work has been directed at studying flowsheets for reprocessing fuel from the FFTF reactor in the proposed Breeder Reprocessing Engineering Test (BRET) Facility. Results from these tests have provided information on heavy metal recoveries, fission product behavior, comparisons of flowsheet options, evaluation of in-line instrumentation, and general operability of the system.

The processing steps that are done in the SETF include: dissolution of the fuel in nitric acid, clarification and adjustment of the dissolver solutions for solvent extraction, the first cycle of solvent extraction, nitrate anion exchange to purify the plutonium products, and plutonium oxalate precipitation-calcination to make plutonium oxide. The solvent extraction contactors consist of three banks of 16-stage mixer-settlers. Although these contactors are capable of processing several kilograms of heavy metals per day, solvent extraction tests are limited to <1 d in duration because of safeguards restrictions that severely limit the amount of plutonium that can be handled in each test.

Four solvent extraction tests have been completed, using three different batches of FFTF fuel; the fuel burnups included 2,200, ~30,000, and ~55,000 MWd/t with cooling times of 2 years, 1.3 years, and 1 year, respectively. The organic solvent for each flowsheet test was a 30 vol % solution of tri-*n*-butyl phosphate (TBP) diluted with a normal paraffin hydrocarbon. The partitioning of the uranium and plutonium into separate product streams was accomplished by the reduction of plutonium to the poorly extractable Pu(III) valence, using hydroxylamine nitrate. In the most recent run, an in-line photometer sample loop was installed and used to measure the uranium and plutonium concentrations in a stage in the extraction bank. Based on the readings from this instrument, changes were made in the addition rate of the organic extractant to permit the achievement of high metal loadings while maintaining low losses to the aqueous raffinate. The ~0.8 kg of plutonium that was recovered from all four tests was converted to the oxide form and shipped to HEDL for fuel fabrication studies.

2.4 SECONDARY CLEANUP OF SRP AND ICPP SOLVENTS

Normally, the solvent in the Purex process is cleaned to remove radiolytic and chemical degradation products by scrubbing with sodium carbonate solutions. Materials that are not removed by carbonate scrubbing accumulate over time and eventually will degrade the solvent extraction performance. Studies were completed during FY 1984 in which activated alumina was used to treat dry Savannah River Plant (SRP) solvent.² We have now completed a similar series of experiments (under subcontract to Westinghouse Idaho Nuclear Company, Inc.) using Idaho Chemical Processing Plant (ICPP) solvent.

Tests with SRP solvent that had been previously scrubbed with a sodium carbonate solution showed that, when the SRP was dried by sparging with dry air at 60°C, properties such as phase-separation time, interfacial tension, and plutonium retention could be markedly improved by passage of the solvent through activated alumina. The treatment was less effective in removing anionic surfactants and ruthenium.

The degradation of the ICPP solvent in the Purex process was much less severe than that of the SRP solvent, and the ICPP solvent contained only insignificant quantities of retained ruthenium and of plutonium complexers. The quality of the used ICPP solvent was significantly degraded only in interfacial tension (~7.2 dynes/cm compared to ~23 dynes/cm for clean solvent) and in phase-separation time from the 0.2 M Na₂CO₃ scrubbing solution (~30 to 35 s compared to ~15 s for clean solvent).

In our study, small (~0.5-g) beds of activated alumina (Alcoa type F-1, 60-120 mesh) were used to treat ICPP solvent that had been dried by sparging with dry air at 60°C for several hours. Such beds were found to be effective for treating >1 l of solvent before the interfacial tension of the effluent solvent was reduced to 20 dynes/cm. Saturated beds were steam-stripped to remove the bulk of the adsorbed material; this renewed about half of the absorptive capacity of the alumina bed and produced a free-flowing powder that could be easily removed from the column.

2. J. C. Mailen and O. K. Tallent, "Solvent Cleanup and Degradation: A Survey and Recent ORNL Results," pp. 1-431 in *Fuel Reprocessing and Waste Management*, Proceedings of the meeting in Jackson, Wyoming, August 26-29, 1984.

3. NUCLEAR AND CHEMICAL WASTE MANAGEMENT

During this report period, Chem Tech has expanded its role in the management of nuclear and chemical wastes by undertaking work in six major areas: (1) technology of waste immobilization in cementitious materials, (2) Integrated (waste management) Data Base (IDB) Program, (3) transuranic waste packaging and handling, (4) repository waste characterization, (5) evaluation of sedimentary rocks for repository purposes, and (6) assessment of hazardous and low-level waste (LLW) management technologies.

3.1 WASTE IMMOBILIZATION TECHNOLOGIES

The objectives of the Waste Immobilization Technologies (WIT) Group are to provide technical assistance to DOE sites in developing and applying the appropriate, cost-effective immobilization processes to radioactive and hazardous chemical wastes, resulting in reliable, feasible disposal schemes that comply with the evolving regulatory requirements.

During this report period, the major programs continued to be the support of operations of the New Hydrofracture Facility (NHF) and the development of pumpable grouts for the Hanford Transportable Group Facility (TGF). Both of these programs have met major milestones in the resolution of the DOE's waste management challenges.

Other activities of the WIT group are characterized by a rapid turnover of short-range projects that include:

1. formulation of grouts for the in situ solidification of raffinate pits at the Weldon Spring, Missouri, site of the Formerly Utilized Site Remedial Action Program (FUSRAP);
2. preliminary formulation of grout for the Y-12 Plant S3-Pond sludges and the testing of their leachate to ensure that EPA and Tennessee drinking water standards for chemically hazardous elements have been met;
3. development of a hydrofracture-compatible grout that is capable of immobilizing polychlorinated biphenyl (PCB) mixtures, Lindane, polynuclear aromatics (PNAs), and phenols and holds up to 30 vol % of contaminated oil (Fig. 3.1).

Through these activities, the WIT group has established major new initiatives that have begun to transfer technologies in the LLW management of chemically hazardous wastes to satisfy commercial and defense needs.

Technical Support to the New Hydrofracture Facility

The WIT group continued to support the Operations Division by (1) developing formulas and determining operating parameters of the final disposal in the NHF (Fig. 3.2) of the backlogged LLWs from the Laboratory's gunite tanks, (2) performing quality assurance tests on materials used in the various injections of sludges and liquids, and (3) conducting studies on the hydrofracture grout's phase composition, interactions with the Conasauga shale, curing rates, and performance as a barrier to migration.

Work on the location of radionuclides in specific hydrosilicate phases of the hydrofracture grouts was directed toward the fixation efficiency of the various hydrosilicates present in hydrated cement. The separation and concentration of various hydrated phases from a cement slurry were begun. Some success has been achieved in concentrating calcium silicate hydrate and calcium hydroxide. The calcium silicate hydrate is a potential retentive phase for cesium and strontium. The radionuclide retention of this hydrosilicate could be important in the understanding of the overall radionuclide retention of the grout.

10

20

30

OIL, %

4 LBS/GAL GROUT WITH POLY-TERGENT B-300

Fig. 3.1. Fixation of oil emulsions in a pumpable hydrofracture grout.

Fig. 3.2. Cutaway view of the New Hydrofracture Facility at ORNL.

Concrete Grout Bulk Immobilization Study on Hanford Sludges

The use of cement-based grouting techniques is a potential solution for disposing of sludges, which make up a significant portion of the $1.78 \times 10^5 \text{ m}^3$ ($4.7 \times 10^7 \text{ gal}$) of waste at the Hanford site. Since some of these wastes are similar to those handled by ORNL's hydrofracture grout facility, the established technology base for sludge removal, suspension, grout mix development, and transport is adaptable to the Hanford site. Values reported for this study are given in English units only in accordance with an agreement between Chem Tech personnel and Rockwell-Hanford.

Preliminary studies demonstrated the feasibility of grouting the following types of Hanford waste streams: (1) Redox sludge; (2) Purex sludge; (3) $\text{Mg}(\text{OH})_2$ -precipitated Zirflex sludge; and (4) high-nitrate, double-shell slurry (DSS). With ORNL support, Hanford plans to build a TGF for waste disposal in shallow-land trenches or culverts (Fig. 3.3). At present, the waste streams to be processed in the TGF are: Hanford Facility waste (HFW), cladding removal waste (CRW), and DDS. During this report period, the grout formula to immobilize HFW was finalized.

Hanford Facility waste has been defined by Rockwell-Hanford as a 50:50 vol % mixture of sulfate and phosphate wastes. The major components of this waste are shown in Table 3.1.

The phosphate waste, which makes up 50% of HFW, is formed as a result of the decontamination operation performed by United Nuclear Corporation (UNC) at the Hanford site, using a Turco decontamination solution. Turco is a trade name given to a range of products manufactured by the Turco Products Division of the Purex Corporation, Carson, California. The Turco solution used in this decontamination

Table 3.1. Concentrations of major components of Hanford Facility waste

Component	Concentration (M)
Na_3PO_4	0.15–0.5
Na_2HPO_4	0.01–0.02
Na_2SO_4	0.01–0.02
NaOH	0.01–0.02
NaOH_2	0.01–0.02

ORNL-DWG 84-1007

Fig. 3.3. Schematic of Transportable Grout Facility planned at Hanford, Washington.

process (No. 4512-14A) contains the following organic constituents: sodium xylene sulfonate, N,N,N',N'-tetraakis(2-hydroxypropyl) ethylenediamine, ethylene glycol monobutyl ether, 2-mercaptobenzothiazole, nonylphenoxy-polyethoxy ethanol, 2-butyne-1,3-diol, and a complex amine inhibitor (a proprietary product; the supplier will not disclose its chemical composition). For a nuclear reactor decontamination, 20,000 gal of Turco solution was diluted with water to a total volume of 240,000 gal. After decontamination, the stream was again diluted with water to a total volume of 600,000 gal and the pH adjusted to 12 with NaOH. Sodium nitrate (≥ 700 ppm) was added in order to meet a Rockwell tank farm specification. The waste, as received by the tank farm, is defined as phosphate waste.

Sulfate waste, which makes up the other 50% of HFW, results from recharging ion-exchange columns. The sulfuric acid from this operation is neutralized with NaOH to a pH ≥ 12 . This waste, as received by the tank farm, is defined as a sulfate waste.

Grout Performance Criteria. A successful formula is defined as one that, when mixed with HFW, meets all performance criteria as specified by Rockwell-Hanford, ORNL, and Battelle Pacific Northwest Laboratories (PNL). Based on these performance criteria, an acceptable grout must result in the following:

Characteristic	Limiting value
Frictional pressure drop	<11 psi/100 ft
10-min gel strength	<100 lb _f /100 ft ²
28-d drainable water	0 vol %
28-d unconfined compressive strength (ASTM C109-80)	<60 psi
Slump (ASTM C143-74)	Infinite
ANS 16.1 Leachability Index for ⁹⁰ Sr and ¹³⁷ Cs	≥ 6
Solids-to-waste mix ratios	≤ 9 lb/gal
Critical velocity	<70 gal/min
Major dry-solid blend components	Only components used in ORNL process
Dry-solid blend composition	Tolerate $\pm 5\%$ variation

Experimental Results. Experiments were performed to develop a cement-based grout which, when mixed with HFW (Table 3.1), would satisfy all performance criteria. Based on studies with simulated phosphate, sulfate, and HFW, the following formula was recommended for the dry-solids blend: Type I-II-LA Portland cement, 41%; Centralia, Washington, ASTM Class F fly ash,

40%; Attapulgate-150 drilling clay, 11%; and Indian Red pottery clay, 8%. The mix ratio of the grout consists of 7.5 lb of dry-solids blend per gallon of Hanford Facility Waste. However, 0.02 vol % tributyl phosphate (TBP) must be added to HFW as a defoaming agent.

The average properties of grouts prepared with HFW and $\pm 5\%$ variations in the recommended blend are shown in Table 3.2.

Table 3.2. Average properties of grouts encompassing the recommended formula with HFW^a

Parameter	Mix ratio (lb/gal)	
	7	8
10-min gel strength, lb _f /100 ft ²	23	59
2-h phase separation, vol %	2.1	2.1
28-d phase separation, vol %	0	0
Density, lb/gal	11.72	12.06
Apparent viscosity, cP	24	35
28-d compressive strength, psi	395	620
Frictional pressure loss per 100 ft, ^b psi	3.37	4.88
Critical velocity, gal/min	40	50

^aThese grouts are further characterized by the following: ANS 16.1 Leachability Index for ⁹⁰Sr, 9.3; ANS 16.1 Leachability Index for ¹³⁷Cs, 10.6; thermal conductivity, ~ 0.46 Btu/h-ft²-°F; heat capacity, ~ 0.59 Btu/lb-°F; liquid permeability, 10^{-4} to 10^{-7} darcy; and porosity, 58-74%.

^bAt reference conditions.

3.2 INTEGRATED DATA BASE PROGRAM

The IDB Program maintains referenced data on all spent fuel and radioactive waste in the United States. Working with and through the DOE lead sites and major programs, it receives and processes data and provides data output in various formats as required by its numerous users.

The purposes of the program are to create and to maintain a reliable baseline of quality data and information for use in national program planning, decision making, and other management activities. As the official data base for DOE, the IDB is readily available to the DOE community and to DOE-sponsored contractors. Because of the program's wide scope, it is sponsored jointly by three branches of DOE: the Office of Civilian Radioactive Waste Management, the Office of the Assistant Secretary for Defense Programs, and the Office of the Assistant Secretary for Nuclear Energy.

The program provides access to information on spent fuel and radioactive waste inventories and characteristics, including volume and/or mass, age, radioactivity, heat generation, chemical and physical properties, location, packaging, and nuclide composition. Utilizing a modular system of computer models and codes, the IDB Program also provides projections based on anticipated growth rates, schedules for new facilities, waste generation factors, and treatment assumptions. Projections for commercial waste are based on official Energy Information Administration (EIA) electrical growth projections for nuclear safety.

Data are collated and integrated to ensure that all forms are counted only once. Figure 3.4 illustrates the primary sources of data that are included. A major program function is to translate various inputs from the numerous sources into a common and internally consistent data base.

1. to upgrade, improve, and issue the annual report of spent fuel and radioactive waste inventories, projections, and characteristics; and
2. to optimize and document the data-handling methodology used by the program, while providing direct support to other DOE programs and sites in data handling.

Substantial progress was made toward accomplishing these objectives. Plans for future work are also briefly outlined.

Annual Inventory Report

The 1984 Edition. The annual inventory report, *Spent Fuel and Radioactive Waste Inventories, Projections, and Characteristics*, has gained widespread recognition and acceptance. Each issue of the report is in considerable demand, with more than 1000 copies of the 1983 edition (DOE/NE-0017/2) being distributed. Approximately 1100 copies of the 1984 edition were issued in the original distribution, and additional requests are now being filled.

The report includes an overview chapter, followed by chapters devoted to the specific waste forms or sources: spent fuel, HLW, TRU waste, LLW, uranium mill tailings, airborne waste, remedial action programs, and decommissioning. Appendixes provide source terms and flowsheets and projections of spent fuel (or HLW) age at the time of emplacement in a repository. The latter appendix was prepared by the NUS Corporation. Projections, which require assumptions concerning power reactor growth and other waste sources and treatments, are made to the year 2020. The 1984 edition dealt with a base scenario that includes future reprocessing and the EIA's mid-case projection for power reactors. The total accumulation of spent fuel in the absence of reprocessing is projected; the total spent fuel for the reference case and for lower growth projections is also given. Table 3.3, reproduced here from the 1984 annual

Fig. 3.4. The IDB Program encompasses data on all domestic sources of spent fuel and radioactive waste.

Fig. 3.5. Conceptual scheme for the handling of data in the IDB Program.

report,¹ shows the summary of spent fuel and waste inventories through December 31, 1983. Table 3.4, which was derived from data in that report, includes projections to 1990, 2000, 2010, and 2020 for both the reprocessing and the non-reprocessing cases. These tables, as well as all others in the annual report, were generated from data in the core data files. A description of the information contained in the core data files has been published,² and a report of the Statistical Analysis System (SAS) programming used is now in preparation.³

Projections given in the annual inventory report depend on the assumptions, source terms, and models used. The basic assumptions on growth, scheduling factors, and capacities are summarized in the overview chapters (ref. 1, Tables 0.1 and 0.2). Future power reactor capacities and spent fuel discharges were based on the EIA mid-case projections.⁴ The IDB then calculated the resulting requirements for fertile and fissile material and, from that, derived the projected waste streams as a function of time for mining and milling, conversion, enrichment, fabrication, and reactor operation. These calculations were accomplished using the Nuclear Materials Projection Code.⁵ For reprocessing and other waste treatment functions, the Waste Treatment Simulation Code was used.⁵ Waste sources not covered by these codes, such as some projected DOE/defense wastes and projected industrial/institutional (I/I) wastes, were handled

by FORTRAN algorithms written to suit specific cases.

The actual production of the photomasters for the printed report was done on a CPT-8105 word processor that transmitted data and text to an APS $\mu 5$ typesetter. In addition to the text, all of the computer-generated tables were done on the CPT word processor via hardwire hookup to the ORNL PDP-10 for direct access to an IBM-3033 mainframe. Using SAS programming, the desired tables were assembled and formatted, then printed out via the word processor as outlined above. The operator added titles and footnotes to complete the tables.

ORIGEN2 Applications. The ORIGEN2 codes are used by the IDB Program for three purposes: (1) to compute the isotopic content of spent fuel as

1. *Spent Fuel and Radioactive Waste Inventories, Projections, and Characteristics*, DOE/RW-006, Department of Energy, September 1984.

2. C. W. Forsberg, *Data Handling in the IDB Program: I—Data Files for the Annual Inventory Report*, ORNL/NFW-83/30, January 1984.

3. B. L. Stair, *Data Handling in the IDB Program: II—SAS Programs for the Annual Inventory Report*, in preparation.

4. *Projections of U.S. Nuclear Power Plant Capacity, Generation, and Spent Fuel Discharge, 1984–2020*, Service Report SR-NAFD-84-01, Energy Information Administration, Department of Energy, March 1984.

5. C. W. Forsberg, C. W. Alexander, G. W. Morrison, and O. W. Herman, *Integrated Data Base Program: An Overview of Data Handling and Projection Modeling*, ORNL/NFW-83, August 1983.

Table 3.3. Spent fuel and radwaste inventories as of December 31, 1983

Spent fuel	Mass, MTU	Activity, MCI	Thermal power, kW
BWRs (20,702 assemblies)	4,188	4,176	15,000
PWRs (12,520 assemblies)	5,952	8,704	33,000
High-level waste	Volume, m ³	Activity, MCI	Thermal power, kW
Savannah River (DOE)	111,400	776	2,280
Idaho (DOE)	9,700	65	190
Hanford (DOE)	203,000	474	1,380
West Valley (commercial)	2,315	35	104
Transuranic waste ^a	Volume, m ³	Activity, MCI	TRU elements, kg
DOE, buried	171,400	0.296	736
DOE, stored	75,000	1.513	1,238
Low-level waste	Volume, m ³	Activity, MCI	Land used, ha
DOE sites	2,062,000	10.32	128
West Valley (closed 1975)	73,000	0.50	4
Maxey Flats (closed 1977)	135,000	0.99	10
Sheffield (closed 1978)	88,000	0.026	4
Barrow, S.C.	432,000	1.44	22
Beatty, Nev.	95,000	0.24	12
Richland, Wash.	182,000	0.64	21
Active uranium mill tailings	Volume, m ³	Activity, MCI	Thermal power, kW
At mill sites ^b	96,500,000	0.40	9
Remedial Action Programs (DOE)	Volume, m ³	Activity, MCI	No. of sites
UMTRAP (Uranium Mill Tailings)	14,313,700 ^c	0.14	24
FUSRAP (Formerly Utilized Sites)	584,300		21
SFMP (Surplus Facilities)	^d		300
GJRAP (Grand Junction Remedial Action Program)	52,000 ^e		626
Commercial decommissioning	LLW, m ³	Activity, MCI	No. of reactors
Decommissioned reactors	^f		5
Mothballed reactors	^f		10

^aBased on the old (10 mCi/g) and new (100 mCi/g) definitions of TRU wastes. With the new definition, these volumes will decrease as the old wastes are re-assayed.

^bLicensed mills; not all are in operation at this time. Currently, 13 mills are active; 26 mills are licensed.

^cIn addition to mill tailings, up to 8,241,200 m³ of other materials contaminated with mill tailings.

^dSurplus Facilities Management Program; projected to generate 421,210 m³ of LLW and 5,879 m³ of TRU waste in the decommissioning of existing obsolete government facilities.

^eIn form of mill tailings. The GJRAP was folded into the UMTRAP in 1983.

^fVery little of this has been done to date, mostly to small test reactors. The LLW generated is included in LLW.

Source: *Spent Fuel and Radioactive Waste Inventories, Projections, and Characteristics*, DOE/RW-0006, U.S. Department of Energy, September 1984.

a function of the initial enrichment, the burnup, and the decay time since discharge; (2) to calculate the decayed isotopes (also radioactivity and thermal power) of all radioactive wastes, both for the present time and for projections; and (3) to derive isotopic values for unusual situations or purposes.

A regular "maintenance" function is performed to keep the various ORIGEN2 libraries up to date.

This includes, for example, decay parameters (half-life, branching ratios) and neutron functions (cross sections by energy groups). Reactor models must also be upgraded. Because of current interest in higher burnups and longer cycle times, a study of the effects of batch averaging was made, using spent fuel data for a Zion-1 reactor discharge batch of 65 assemblies, where the average assem-

Table 3.4. Current and proposed volume, radioactivity, and thermal power of spent fuel and radioactive waste

Source of material and type	End of CY 1983				End of CY 2000				End of CY 2010				End of CY 2020			
	Volume (10 ³ m ³)	Radioactivity (10 ⁶ Ci)	Thermal power (10 ⁶ W)		Volume (10 ³ m ³)	Radioactivity (10 ⁶ Ci)	Thermal power (10 ⁶ W)		Volume (10 ³ m ³)	Radioactivity (10 ⁶ Ci)	Thermal power (10 ⁶ W)		Volume (10 ³ m ³)	Radioactivity (10 ⁶ Ci)	Thermal power (10 ⁶ W)	
DOE/deliver																
HLW																
Interim storage	324	1,320	3,850		342	1,520	4,620		310	1,150	3,420		286	1,010	3,060	
Glass				0.3		22	60		3.6	223	600		6.8	325	907	
TRU																
Burned	171	0.3	5.7		171	0.3	5.5		171	0.2	5.4		171	0.2	5.2	
Solid	35	1.5	36		112	2.5	63		169	3.9	98		226	6.2	121	
LLW	2,060	10.3	15.6		2,630	12.5	20.4		3,720	15.0	26.0		4,710	16.8	30.4	
RAJ ^a																
Mid tailings	52				21,700				21,700				21,700			
LLW and TRU	50.9				990				990				990			
Commercial																
Spent fuel (10 ³ MT/HM)																
No reprocessing	10.1	12,879	48,000		21.1	23,176	87,000		42.8	35,674	131,000		73.5	60,260	223,000	
With reprocessing	10.1	12,879	48,000		21.1	23,176	87,000		39.8	35,258	130,000		40.8	51,948	196,000	
LLW (no reprocessing)	1,010	3.8	31.3		1,830	6.2	27.2		3,380	9.7	27.2		5,300	12.7	28.3	
D&D ^b	7.5	0.02	0.08		8.3	0.02	0.05		9.1	0.01	0.04		88.3	0.26	1.06	
Mid tailings	96,500	0.40	8.9		130,000	0.61	13.6		188,000	0.99	22.0		280,000	1.58	35.0	
Reprocessing waste																
HLW																
Interim storage	2.3	35	104		0.2	29	87		0.44	347	1,174		3.2	6,513	22,369	
Glass									4.6	0.6	1.2		49.4	11.1	26.6	
TRU									-2.1	-0.003	-0.006		-	-0.046	-0.116	
LLW (reprocessed)																
Authenics																
Isotopes + carbon									0.037	0.4	0.5		0.635	12.4	10.2	
Krypton									0.002	2.7	4.0		0.023	39.8	89	
													1.72	52.1	43.6	
													0.042	35.7	385	

^aBased on data from Spent Fuel and Radioactive Waste Inventories, Projections, and Characterization, DOE/NW-0006, U.S. Department of Energy, September 1994.

^bRevised action program rates and tailings start today, program improves existing disposal rates, thus numbers show existing waste that have been better isolated, existing TRU and LLW waste numbers reported above for 1983; future LLW and TRU waste are from future remedial action activities and are not included in future waste inventories listed above.

^cSpent fuel inventories measured in metric tons usual heavy metal (MT/HM) rather than volume.

^dThermonuclear and decommissioning.

bly burnups varied from 27,900 to 35,850 megawatt-day per metric ton of initial heavy metal (MWd/MTIHM). For each assembly, axial effects were also considered. The results showed a trivial effect on fission products, total radioactivity, and thermal power, but a significant effect on those transuranics resulting from multiple-neutron captures. Some, such as ^{245}Cm and ^{246}Cm , were underestimated by 36 to 49%, respectively, using batch averaging. This work was reported at the Waste Management '84 conference and published in the Proceedings.⁶

Because the ORIGEN2 code is the present model of choice for the generation and decay of isotopic inventories, extensive use will be made of this code in the completion of the extensive spent fuel isotopic characterization for the Repository Waste Characteristics Program. This work will generate the necessary data on spent fuel isotope inventories, as well as radioactivity and thermal loads—all as a function of time up to 1 million years after discharge from reactor for use by repository design and licensing groups.

Quality Assurance Plan. The principal quality assurance (QA) control point is the annual inventory report, since this is the driving force for new data input each year and since the core data files are updated in the process. A preliminary QA plan was prepared and reviewed by the steering committee, with major emphasis on the annual schedule (Table 3.5) and on a mechanism for dealing with the inevitable changes and corrections needed annually. After comments were received, the QA plan was expanded and has been published as a program document.⁷

Future Work. Future plans in matters related to the annual inventory report include the following:

Because of the need for program planning in the area of national spent fuel and HLW repository programs, increasing attention will need to be shown to the question of assuring a complete and accurate inventory of these materials at each site. At present, the national Nuclear Materials Management Safeguards System (NMMSS) is being interrogated to determine the completeness of the information presently included in the IDB annual inventory report. Upon determination of the usefulness of new data and the verification of its accuracy, updates will be included in future additions of the annual report.

A comparison of the data supplied by the various high-level production sites has indicated that

not all sites use the same methodology for calculating isotopic and radioactivity distributions for their HLW. A workshop will be held to determine the existing methodologies used by the various sites and to obtain a consensus on a reporting format to be used for future HLW inventories. This format will become increasingly important as the information begins to be factored into national spent fuel and HLW repository designs.

Summary Data File and User Access

A major objective of the IDB Program is to make the existing data accessible to the DOE family. This objective is met through direct contact with data users and with the distribution of the annual inventory report. In addition, the program has made available the Summary Data File, available in user-friendly format on floppy disks, which does not require programming skills.

Summary Data File. The summary data file was created in the spring of 1983 for use on the IBM-PC and IBM-PC-compatible personal computers. The actual programming was done in dBase II using SAS to create the necessary data files to form the core files. Essentially all of the computerized data in the core files, except detailed compositions, are available through the summary file. By virtue of the menu options offered by the summary file, much data can be extracted that is not directly available in the annual inventory report. These data are all contained on two floppy disks—one for the program and one for the data. These diskettes have been updated on an annual basis after the new annual inventory report was issued.

User Access. In the winter of 1984–1985, a local area network (LAN) was installed that has greatly increased the potential capacity of the IDB to respond to user requests. The LAN links approximately 12 IBM-PCs and a server with 147-megabyte storage into one integrated system that allows sharing of programs, a dedicated printer, and electronic mail. At present, numerous software programs reside on the system in addition to the IDB Summary Data File. In the near future, outside

6. C. W. Alexander and A. G. Croff, "Some Implications of Batch-Average Burnup Calculations on Predicted Spent Fuel Compositions," in *Proceedings of Waste Management '84*, Tucson, Arizona, March 11–15, 1984.

7. K. J. Notz, *Integrated Data Base Program: Quality Assurance Plan*, ORNL/NFW-84/2, January 1984.

Table 3.5. Schedule for IDB Program annual report on inventories, projections, and characteristics

Date	Responsibility	Description
October–November	All	Receive specific comments, questions, or criticisms; response by IDB as appropriate.
December	IDB	Prepare suggestions for next annual report, identifying recommended changes in format or data handling and any problem areas.
January	IDB	Formal call for data to lead sites; response needed by March 30.
January	All	At Steering Committee meeting, review the call for data and the overall format for the next report.
February 15	DOE/HQ	Agreed-to assumptions provided for projection basis.
March 30	Lead sites	Responses received by IDB after initial QA by lead sites; electronic computer output preferred.
April	IDB	IDB review of data received and initial resolution of questions by phone.
April 30	IDB	Letter documentation of any unresolved issues with copy to DOE/HQ; final resolution by lead sites and DOE.
July 1	DOE/EIA	Receipt of spent fuel inventories and projections.
September 15	IDB	Issue and distribute draft report for review; cite any special assumptions and resolutions of comments. Response needed by October 1.
October 31	Lead sites	Final comments provided by lead sites. Detailed review completed at Steering Committee meeting.
November 15	IDB	Incorporation of final comments and preparation of photomasters for reproduction; QA proofing by IDB staff.
December 1	Lead sites and DOE/HQ	Final draft ready for QA proofing by lead sites and DOE. Ten-day turnaround required.
December 15	IDB	Final draft to HQ for printing and distribution.
December	DOE/HQ	Report issued.

call-in capability will be added. This option will allow access to the Summary Data File without having to acquire the floppy disks and, at a later date in the future, will permit the accessing of the detailed ORIGEN2 output for the Waste Repository Characteristics Project and ORIGEN2 decay programs.

Future Work. The major effort in this area, as mentioned above, is to tie call-in capability to the LAN. The vastly increased storage capacity of the system server should allow for improved perform-

ance to the user. The addition of a similar data base management system for the detailed ORIGEN2 output in the Repository Waste Characteristics project and a usable ORIGEN2 decay program should complement the overall objectives of the IDB Program.

Data Processing Methodology and Support to Other Programs

Other major objectives of the program are to develop and apply data processing methodology

that provides both flexibility and accessibility and to share this methodology with other DOE programs. This objective is being met through utilization of widely accepted state-of-the-art technology, with SAS programming, personal computers, and current generation software on floppy disks being the major components. Making IDB capabilities known to others is also important and is achieved through the Steering Committee, site visits, an information booklet and flyer, and participation in national waste management meetings.

Data Processing Methodology. The major hardware components used by the program and the overall concepts and data flow have been described previously.⁸ In general, the core data files are stored on hard disks after generation on IBM-30333 and PDP-10 mainframes. Personal computers are used for the summary data file and numerous other program applications. A CPT-8105 was used to transmit data and text for printing on an APS μ 5 typesetter.

Support to Other Programs. Expertise acquired in the IDB Program has been utilized in support of the Waste Repository Characteristics Project. This task is supplying the critical information support needed by DOE's Office of Civilian Radioactive Waste Management in their National Waste Terminal Storage Program. The work provided by this program is performed in close cooperation with Weston (National Waste Terminal Storage Technical Support Team), Rockville, Maryland. This task provides reference quantities and characteristics for selected wastes and waste forms that are needed to support the various testing, design, and analysis activities of the national repository programs. Major support provided by the IDB Program includes providing fuel assembly inventory and future projection data. In addition, ORIGEN2 runs of both average and high-burnup boiling-water reactors (BWRs) and pressurized-water reactors (PWRs) were provided. This output was provided in both hard copy and menu-driven IBM-PC-compatible floppy disk format.

As the degree of interest in personal computers increases, tremendous capabilities and capacities become available at relatively low cost. The personal computer encourages the direct involvement of technical staff other than computer specialists in actual data processing, which is highly desirable. As the IDB staff has gained experience, we have shared this knowledge with other members of the Steering Committee. Numerous site visits have

been conducted, especially with DOE Headquarters, and we expect that this interaction will continue to be maintained.

As an example of this involvement, the resources of the IDB Program were tapped to provide both upgraded hardware and software capabilities to the Joint Integration Office (JIO), formerly the Transuranic Waste System Office (TWSO). Hardware upgrades centered around LAN similar to that installed at the IDB Program. Software capabilities included the development of the TWSO Waste Management Data Base to provide JIO with a single information resource for long-range waste management planning. Data have been requested from each generating site and have been compiled into an IDB management system by IDB personnel.

Future Work. Future work will continue to concentrate on the increased use of personal computers. The eventual goals will be twofold: (1) to download entire sections of data from floppy disks or electronically from storage sites, and (2) to further implement and expand LAN to allow for the development of simplified models for rapid, easy testing of alternatives and for screening out unrealistic cases.

3.3 TRANSURANIC WASTE MANAGEMENT

The Defense Transuranic (TRU) Waste Program for planning, integration, and technical development for TRU waste management is centered in the DOE's Albuquerque Operations Office, the Defense TRU Waste Lead Organization (TLO), and the JIO. The goal of this program is to achieve permanent disposal and thus eliminate the need for interim storage of these wastes from all defense waste sources. The Waste Isolation Pilot Plant (WIPP) is to be used initially for retrievable emplacement tests and later, if those tests prove successful, for permanent disposal.

The TRU solid wastes are classified by the radiation level at the exterior surface of the waste package. Contact-handled (CH) waste reads <200 mrem/h and may utilize hands-on operations. Remote-handled (RH) waste reads >200 mrem/h and requires special (remote) handling for personnel safety. A third classification, special case (SC), which could be either CH- or (more frequently)

8. K. J. Notz, *Integrated Data Base Program Annual Report for FY-1983*, ORNL/NFW-84/1, March 1984.

RH-TRU waste, is for wastes that are difficult to certify as being acceptable at WIPP [by the WIPP Waste Acceptance Criteria (WAC)].

The CH-TRU waste program is more advanced than the RH-TRU waste program, reflecting the national plan to ship the CH wastes to the WIPP starting in 1989, about 5 years before the RH-TRU waste. The CH program has approved certification documents⁹ and constructed physical facilities, such as the Process Experimental Pilot Plant (PREPP) at the Idaho National Engineering Laboratory (INEL). The RH-TRU waste program is now making the decisions required to complete the needed documents and facilities.

In this report, all our activities are discussed under either the CH- or RH-TRU waste headings, including special-case considerations.

CH-TRU Waste

Within the past year, the program for newly generated CH-TRU waste⁹ has been accepted by the WAC Committee and the state of New Mexico. The last obstacle in preparing certified waste ready for shipment to the WIPP is an audit by the committee that includes one member from the Environmental Evaluation Group (EEG) from the state of New Mexico. This audit is presently scheduled for May 21-23, 1985.

The CH-stored waste is presently being analyzed by nondestructive assay (NDA) and nondestructive examination (NDE) methods and will be examined by the real-time radiography (RTR) unit when it is installed. The RTR unit has been purchased and is presently scheduled for installation in July 1985. Operation of the unit on the ~2000 drums will begin in October 1985.

An ultrasound unit for the detection of drum-wall integrity was tested and found to give reliable results. An eight-detector unit will be ordered for installation at the burial ground and will be used to check all drums for the certification of containers.

Ten boxes (~43 m³) of TRU waste were generated during the decontamination and decommissioning of the Plutonium Laboratory, Building 4508. This waste consists of glove boxes and associated components that were used in the operation of the glove boxes. The boxes containing the waste have been certified as DOT Type A. The contents have been recorded, so certification for shipment to WIPP should pose no problems.

Certification documents for all TRU waste destined for shipment to WIPP are required before the waste can be emplaced in the geologic facility. In order to comply with this requirement, the original document for CH certification is being expanded to cover each of the TRU waste categories (CH and RH, both newly generated and stored) pertinent to ORNL operations.

RH-TRU Waste

TRU waste that is handled remotely is retrievably stored at four DOE sites: (1) INEL, (2) Los Alamos National Laboratory (LANL), (3) ORNL, and (4) Rockwell Hanford Operations (RHO). Data were obtained¹⁰ on the waste quantities and characteristics as well as descriptions of the storage containers, facilities, and methods. Considerable variation exists in storage methods, and an assessment indicated that the retrieval will be extremely difficult and expensive at RHO.

The additional developments/facilities needed to meet the WAC established by WIPP for handling and certifying RH-TRU waste were investigated.¹¹ These developments included:

- a concrete mix to be added to the waste to tie up any free water or fines and to stabilize pyrophoric materials;
2. knowledge of the effects of gamma radiation from RH-TRU waste on the RTR used successfully on CH-TRU wastes;
3. information on the limitations of the nondestructive assay system being developed for CH-TRU waste when used with both ORNL's high-neutron CH-TRU wastes and all RH-TRU waste;
4. the demonstration of an economical, modular interim storage system adaptable to any site (wet or dry);
5. the neutron shielding of casks planned for transporting interstate and for WIPP operations; and
6. a systematic study of the total transportation system to include number of casks required, interfaces at generators and WIPP, and shielding requirements.

9. W. D. Box, *Certification Document for Newly Generated Contact-Handled Transuranic Waste*, ORNL-5985/R2, December 1984.

10. W. L. Carter, unpublished data, May 1984.

11. F. E. Harrington, unpublished data, August 1984.

None of the four sites that store RH-TRU waste has facilities for certifying, loading, welding, and handling canisters. Nonstorage sites (such as Argonne East) will have a similar problem when they change from the current practice of transferring waste to retrievable storage at one of the above sites.

A detailed feasibility study¹² presents a generic facility designed for the minimum processing and repackaging of RH-TRU waste for shipment to WIPP. This facility can process >95% of the RH-TRU defense waste from the four storage sites. It has three shielded process cells in which the wastes can be examined, sorted, stabilized, and repackaged in RH-TRU canisters certifiable for WIPP. Additionally, it cost \$25,000,000 in second-quarter FY 1984 dollars.

A study of neutron shielding possibilities for either a modified high-level defense work cask or the WIPP onsite into-mine transporter showed that shielding is an impractical approach to meeting the very restrictive 75-mrem neutrons at the exterior of the RH-TRU canister now proposed in the WAC by WIPP. The RH-TRU wastes at ORNL will be held in retrievable storage to allow the ²⁵²Cf to decay before storage.

Various options were investigated for changing the ORNL current RH-TRU waste handling and retrievable storage methods. Proposals (Field Task Proposal and Schedule 44 for required capital funding) have been submitted for a processing and repackaging facility to handle nearly all of our RH-TRU waste. A proposal has also been submitted for the generating of an Alternative Disposal Practice Petition (ADPP) for the few packages that are impractical to certify.

Planned future activities include:

1. reworking the cost data in the feasibility study in order to estimate the cost of a facility for repackaging only;
2. documenting the conclusions and recommendations from all parts of the generic process engineering study;
3. initiating a program that will demonstrate the actual shipment to and internment at WIPP of ten TRU waste packages and will include (a) the preparation of one or two canisters of ORNL RH waste and eight or nine canisters of carefully documented, simulated waste to reflect the spectrum of wastes encountered; (b) gas generation studies; (c) canister loading,

welding, and interim storage; and (d) cask loading and transportation to WIPP for final emplacement;

4. generating a certification plan for ORNL's RH-TRU waste form before facilities are available to modify the existing practices of loading and retrievable storage of the RH-TRU form; and
5. determining whether any of ORNL's TRU waste is impractical to certify and, if so, preparing an ADPP for the use of greater confinement disposal (GCD).

3.4 REPOSITORY WASTE CHARACTERISTICS

The Nuclear Waste Policy Act of 1982 authorizes DOE to site, design, and construct mined geologic repositories for the disposal of spent nuclear fuel and high-level radioactive waste and to obtain licenses from the NRC to achieve this mandate.¹³ It also requires the DOE to operate the repositories in such a manner as to adequately protect the public and the environment for an extended period of time (in excess of 10,000 years). In order to achieve these tasks, DOE developed a national repository program that is described in its Mission Plan¹⁴ to fulfill the requirements of the Act.

The Repository Waste Characteristics Program (RWCP) was initiated by Chem Tech to supply the critical support needed by the DOE Office of Civilian Radioactive Waste Management in its National Waste Terminal Storage Program (NWTSP). In close cooperation with Weston, the RWCP is to provide reference quantities and characteristics for selected wastes and waste forms that are needed for various testing, design, and analytical activities in the NWTSP.

The initial thrust of the RWCP has been to augment Appendix B of the report entitled *Generic Requirements for a Mined Geologic System* for

12. Martin Marietta Energy Systems, Inc., Engineering, *Feasibility Study for RH-TRU Waste Handling Systems*, X-OE-254, September 1984.

13. Public Law 97-425, "Nuclear Waste Policy Act of 1982," (NWPA), 42 U.S.C. 10101 (Jan. 7, 1983).

14. *Mission Plan for the Civilian Radioactive Waste Management Program*, DOE/RW-0005 (Draft), vols. I and III, U.S. Department of Energy, July 1984.

commercial LWR spent fuel.¹⁵ A draft report¹⁶ has been prepared which presents pertinent data summarizing (1) mechanical design characteristics and materials of construction for existing fuel assemblies and rods, (2) present and projected generation of spent fuel inventory, (3) burnup distribution of present and projected fuel, and (4) computed values for heat generation rates, radioactivity, and photon and neutron emission rates as a function of time for four model LWRs.

Information used in the report was gathered from a literature survey and from the experts in the field. Requests were made to the major vendors to supply pertinent data on their fuel assemblies/rods. The computerized literature search facilities were used to access the *Energy Research Abstracts*, *Nuclear Science Abstracts*, *Nuclear Safety Information Center Data Base*, and the *Power Reactor Docket Information Data Base*. To augment these sources and to reduce the possible omission of important records, a routine search was made of the more important journals in the field of nuclear energy. A summary that lists just one type of information supplied by the vendors that manufacture PWR fuel is given in Table 3.6.

Calculations were made with the ORIGEN2 computer code for burnups of 27,500 and 40,000 MWd for a typical BWR and 33,000 and 60,000 MWd for a typical PWR. The results are presented in figures depicting the individual contributions per metric ton of initial heavy metal for the activation products, fission products, and actinides and their daughters to the radioactivity and thermal power as a function of time (e.g., Fig. 3.6). Tables are also presented that list the contribution of each major nuclide to the radioactivity, thermal power, and photons and neutrons emitted for disposal periods from 1 to 1×10^5 years (e.g., Table 3.7).

Fig. 3.6. Radioactivity produced by a PWR with a 60,000-MWd/MTIHM burnup.

A computer-searchable data base containing the composition (g), total radioactivity (Ci), and thermal power (W) for the significant nuclides covering 38 decay periods (from 1 to 1×10^6 years) has been developed. Figure 3.7 is a sample output of the NWTSP data base following an interactive session. This system is being expanded to include additional burnups and other nuclear data.

3.5 SEDIMENTARY ROCK PROGRAM

The DOE Office of Civilian Radioactive Waste Management initiated a program at ORNL in

15. *Generic Requirements for a Mined Geologic Disposal System*, DOE/NE/44301-1, U.S. Department of Energy, September 1984.

16. J. W. Roddy, H. C. Claiborne, R. C. Ashline, P. J. Johnson, and B. T. Rhyne, *Repository Waste Characteristics I. Commercial LWR Spent Fuel*, ORNL/TM-9591 (Draft).

Table 3.6. PWR Fuel assemblies shipped by major manufacturers

Vendor	Rod configurations				Total
	14 × 14	15 × 15	16 × 16	17 × 17	
Babcock & Wilcox	^a	3,830	^a	414	4,244
Combustion Engineering	3,100	272	1,076	^a	4,448
Exxon Nuclear	^b	^b	^b	^b	4,000
Westinghouse Electric	4,030	5,600	400	6,800	16,830

^aThis size not manufactured by vendor.

^bData not available per rod configuration.

Table 3.7. Variation in thermal power (W/MTIHM) for significant nuclides as a function of time since discharge from a 60,000-MWd/MTIHM PWR*

(Includes all structural material)

Isotope ^b	Time since discharge (years)					
	1.0E+0	1.0E+1	1.0E+2	1.0E+3	1.0E+4	1.0E+5
Co-60 ^c	1.47E+2	4.50E+1	—	—	—	—
Kr-85	2.00E+1	1.12E+1	—	—	—	—
Sr-89	1.57E+1	—	—	—	—	—
Sr-90	1.32E+2	1.06E+2	—	—	—	—
Y-90	6.29E+2	5.08E+2	5.96E+1	—	—	—
Y-91	4.38E+1	—	—	—	—	—
Zr-95 ^d	1.48E+2	—	—	—	—	—
Nb-95 ^d	3.16E+2	—	—	—	—	—
Ru-106	2.28E+1	—	—	—	—	—
Rh-106	3.68E+3	7.56E+0	—	—	—	—
Ag-110m	6.21E+1	—	—	—	—	—
Sb-125 ^d	5.63E+1	5.34E+0	—	—	—	—
Cs-134	2.66E+3	1.29E+2	—	—	—	—
Cs-137	1.97E+2	1.60E+2	2.00E+1	—	—	—
Ba-137m	6.60E+2	5.36E+2	6.71E+1	—	—	—
Ce-144	2.84E+2	—	—	—	—	—
Pr-144	3.15E+3	—	—	—	—	—
Pm-147	3.37E+1	3.12E+0	—	—	—	—
Eu-145 ^c	2.09E+2	1.01E+2	—	—	—	—
U-233	—	—	—	—	—	2.05E-2
U-234	—	—	—	1.18E-1	1.15E-1	9.10E-2
U-236	—	—	—	—	—	1.55E-1
Np-237	—	—	—	—	—	6.02E-2
Pu-238	2.84E+2	2.68E+2	1.32E+2	—	—	—
Pu-239	1.13E+1	1.13E+1	1.13E+1	1.10E+1	8.84E+0	6.90E-1
Pu-240	2.11E+1	2.15E+1	2.22E+1	2.02E+1	7.78E+0	—
Pu-241	5.84E+0	3.79E+0	—	—	—	—
Pu-242	—	—	—	1.34E-1	1.32E-1	1.12E-1
Am-241	1.92E+1	9.16E+1	1.98E+2	4.74E+1	—	—
Am-243	2.32E+0	2.32E+0	2.30E+0	2.11E+0	9.07E-1	—
Cm-242	1.01E+3	—	—	—	—	—
Cm-243	3.35E+0	2.69E+0	—	—	—	—
Cm-244	5.44E+2	3.85E+2	1.23E+1	—	—	—
Others	7.25E+1	7.00E+0	8.50E+0	5.18E-1	3.42E-1	6.44E-1
Subtotal						
A.P. ^e	1.80E+2	4.61E+1	2.23E-1	2.35E-2	1.69E-2	9.54E-4
F.P. ^f	1.23E+4	1.57E+3	1.59E+2	3.62E-2	3.43E-2	2.10E-2
A.+D. ^g	1.90E+3	7.88E+2	3.80E+2	8.14E+1	1.81E+1	1.61E+0
Total	1.44E+4	2.41E+3	5.39E+2	8.15E+1	1.81E+1	1.63E+0

*MWd/MTIHM = megawatt-day per metric ton of initial heavy metal.

^bNuclides contributing >0.1% of total are listed; — indicates contributions <0.1% of total.

^cOnly activation products contribute to this nuclide.

^dBoth activation and fission products contribute to this nuclide.

^eA.P. = activation products.

^fF.P. = fission products.

^gA.+D. = actinides plus daughters.

Spent Fuel Repository Characteristics Data Base
Developed by: Oak Ridge National Laboratory, Oak Ridge, Tennessee

Type of Reactor: PWR—Normal (33,000 MWd/MTUHM)

Elapsed Decay (years): 1000

All isotopes representing: >0.100% of total

Isotope	Ci	Percentage of total
Ni-59	5.11E+00	0.293
Zr-93	1.93E+00	0.110
Nb-93m	1.83E+00	0.105
Tc-99	1.30E+01	0.747
U-234	2.03D+00	0.116
Np-239	1.56E+01	0.896
Pu-239	3.05E+02	17.528
Pu-240	4.78E+02	27.471
Am-241	8.93E+02	51.321
Am-243	1.56E+01	0.896
Subtotal Ci	1.73E+03	99.488
Total all isotopes	1.74E+03	

Fig. 3.7. Pictorial representation of computer screen after an interactive session with the NWTSP data base.

January 1984 to evaluate five sedimentary rock types other than salt (halite) with respect to their potential for hosting an HLW repository. This effort is the result of an earlier study,¹⁷ reported in the previous Chem Tech progress report,¹⁸ that considered the utilization of the Devonian-Mississippian shales as a repository host rock. The five rock types considered were shale, sandstone, anhydrous carbonate rocks (limestone, dolostone), and chalk. These rock types are not currently being considered in the United States for repository siting purposes. The evaluation was conducted using a non-site-specific approach because its purpose is to evaluate rock types, but the evaluations were based on rock evaluation criteria (RECs) derived from the DOE siting guidelines (10 CFR Part 960).¹⁹ A technical information base consisting of data on the hydrology, geochemistry, rock occurrences, thermal performance, rock mechanics aspects, geology, and natural resources aspects was developed and documented for the five sedimentary rocks. As a part of developing this information base, a report²⁰ constituting a compendium of geological data concerning shale which was prepared under the auspices of another program, was finalized and published.

The result of the information acquisition and evaluation process was a draft report,²¹ currently undergoing review by DOE, establishing the order

of preference of the five subject rock types with respect to their potential as repository host rocks. Shale was determined to be the most preferred of the five rocks, with sandstone a distant second; the carbonate rocks and anhydrous were a more distant third, and chalk was a relatively close fourth. A brief rationale for this ranking is as follows:

1. shale—ranked first on most RECs without having any strong negative aspects;
2. sandstone—intermediate ranking on nearly all RECs, with the only strong negative aspect being its tendency to contain sought-after resources (oil, gas, water);
3. anhydrous—strong rock with good thermal performance, but with negative characteristics of very limited abundance, low ductility, and poor geochemical/dissolution attributes;
4. carbonate rocks—strong rock, but relatively poor hydrology, ductility, and geochemistry and commonly contains natural resources such as oil, gas, and water; and
5. chalk—good ductility, but of limited abundance, poor thermal performance, poor hydrology, and low strength.

Other considerations included cost, previous systems studies, and foreign activities. The costs of repositories in shale and sandstone were the lowest and were estimated to be slightly greater than that for a bedded-salt repository. The cost of a repository in anhydrous was somewhat higher, and costs for those in the carbonate rocks and chalk were still higher. Only two previous systems studies—both generic studies involving shale—were identified. The results of both studies were determined not to be useful for the purpose of deriving conclusions concerning the waste-isolation potential of the five subject rock types. A review of foreign studies concerning the subject rocks found considerable interest in shales and related rocks (clays).

17. T. F. Lomenick, S. Gonzales, K. S. Johnson, and D. Byerly, *Regional Geological Assessment of the Devonian-Mississippian Shale Sequence of the Appalachian, Illinois, and Michigan Basin Relative to Potential Storage/Disposal of Radioactive Wastes*, ORNL-5703, January 1983.

18. Chem. Technol. Div. Prog. Rep. Apr. 1, 1981, to Mar. 31, 1983, ORNL-5933, September 1983.

19. U.S. Code of Federal Regulations, Title 10, Pt. 960; Fed. Regist. 49(236), 47714 (Dec. 6, 1984).

20. S. Gonzales and K. Johnson, *Shales and Other Argillaceous Strata in the United States*, ORNL/Sub/84-64794, April 1985.

21. A. G. Croff, unpublished data, January 1985.

These types of rocks are primary candidates for HLW repositories in Belgium, Italy, and Japan; they are alternatives in France, England, and Canada.

As a result of the evaluation, it was recommended in the draft report that shale should be investigated further as an HLW repository host rock but the study of the other rocks should be deferred at this time. Pending DOE decisions on this matter, work has been initiated to expand the geological data base for shales to accommodate the potential need to screen shale occurrences in the conterminous United States for potentially acceptable repository sites.

3.6 ASSESSMENT OF WASTE MANAGEMENT TECHNOLOGIES

This section provides an overview of the two assessments of waste management technologies performed in Chem Tech: (1) hazardous wastes from defense-related facilities, and (2) LLW from commercial nuclear power stations.

Hazardous Wastes from Defense-Related Facilities

The objective of this task, initiated in March 1984, is to provide a technical review of currently available hazardous waste treatment processes and produce a document suitable as a reference source for DOE. A draft report was completed in August 1984; the final report will be issued in June 1985.

With the assistance of Weston, the technology review document is nearing completion and is divided into six areas in the treatment of hazardous wastes.

Waste Abatement, Reduction, Reuse, and Recycle. The aim of most environmentally conscious proponents of waste abatement, reduction, reuse, and recycle processes is to eliminate the need for much of the expensive treatment and disposal of hazardous wastes that is currently necessary. To employ these desirable options requires intimate knowledge of industrial or DOE processes because they are very much related to the particular operations at a plant within an industry. Thus the subject is discussed generally in the report, and illustrative examples of the major practices are included.

Biological Treatment. Biological treatment encompasses several basic generic processes that

offer potential application to many hazardous wastes generated in DOE facilities (as they have for some time in the industrial sector). The biological processes discussed in this report consist of proven technologies available for application within DOE. They include activated-sludge processes, trickling filters, rotating contactors, fluidized beds, lagoons, composting, landfarming, activated carbon, anaerobic digestion, and enzyme treatments.

Physical and Chemical Treatment. Physical treatment technologies typically include processes that separate components of a waste stream or change the physical form of the waste without altering the chemical structure of the constituent materials. Chemical treatment technologies alter the chemical structure of the constituents to produce a waste residue that is less hazardous. Nineteen physical and chemical processes potentially applicable to DOE operations are discussed in this report.

Thermal Processing. Thermal processing is defined as the treatment of hazardous wastes in a device that uses elevated temperatures as the primary means of changing the chemical, physical, or biological character or composition of the hazardous waste. These processes are useful for reducing volume and detoxifying materials in an efficient and reliable manner. The possibility of energy recovery and recycle or reuse of the ash often exists. All major types of thermal processing devices are discussed in this report.

Solidification/Stabilization and Encapsulation Technology. Stabilization/solidification and encapsulation methods that are currently available or under development have as their goal the preparation of materials for safe ultimate disposal in a permanent manner with a minimum of harm to the environment from such mechanisms as leaching or migration into groundwater. The following major categories of waste-fixation systems are discussed in the report: cement-based processes, pozzolanic (lime-based) processes, thermoplastic techniques (including bitumen, paraffin, and polyethylene), organic polymer techniques (including urea-formaldehyde, unsaturated polyester, and surface encapsulation techniques), and glassification, synthetic minerals, and ceramic processes.

Ultimate Disposal. Ultimate disposal technologies do not treat contaminants but generally ensure that they are contained and controlled for an infinite period of time. Methods discussed in this report are secure landfill, deep well injection, underground storage, and above-ground storage.

Many existing data bases of waste streams and technologies were uncovered during the technology review. The forefront of waste management was found to be the development of models to assist in applying the above technologies to specific waste problems. Additional material developed during the current exercise could form the basis for a follow-on report in technology applications.

LLW from Commercial Nuclear Power Stations

The major purposes of this task, initiated in December 1984, are to provide an assessment of currently available technology suitable for treating LLW from commercial nuclear reactors, to provide a ranking of processes for a given application in a manner suitable for choosing one over the other based on their relative figures of merit, and to identify R&D needed to improve current techniques and decision methodology. A team has been assembled with appropriate work assignments that should permit successful accomplishments of the program objectives.

A working outline has been developed for the proposed report that includes the following major sections: waste forms and sources; regulatory constraints affecting treatment, storage, transportation, and disposal of LLW treatment technologies; packaging, storage, and transport; ultimate disposal of wastes; systems analysis model for selection of technologies; R&D with potential to significantly improve LLW treatment and disposal; and R&D recommended for future support. The treatment

technologies will be organized according to the treatment objective and will include: methods for isolation or concentration of waste constituents from water; removal of water (dewatering); thermal and chemical methods; biological methods; decontamination techniques; mechanical treatment (volume reduction); and immobilization (solidification, stabilization, and encapsulation).

Visits have been made to the EPRI, INEL, Tennessee Valley Authority (Headquarters), Browns Ferry Nuclear Plant, Savannah River Laboratory, and Chem-Nuclear Systems, Inc., including the Barnwell, South Carolina, disposal site. In addition, contacts have been made with U.S. Ecology (operators of the Richland, Washington, and Beatty, Nevada, disposal sites) and Tri-State Motor Transit. Letters have been sent to over 200 suppliers of LLW treatment equipment and methodology requesting information on engineering data, treatment costs, and problem areas. Approximately 20% have responded, and the information is currently being evaluated.

Most of the extant LLW literature has been obtained and is being reviewed. Several data bases and computer models dealing with LLW and similar wastes have been obtained from DOE and EPA. In addition, arrangements have been made to use the volume reduction technology model developed by the EPRI as appropriate.

A request for proposals was issued and a subcontract let to Sargent and Lundy Engineers to prepare a comprehensive analysis of regulatory constraints that affect LLW treatment technologies.

4. ENVIRONMENTAL CONTROL TECHNOLOGY

During this report period, Chem Tech identified environmental control technology (ECT) as an area of emphasis for future planning and resource allocation. The Division plans to continue to perform R&D activities in ECT for external sponsors such as the DOE Office of Fossil Energy (DOE/FE), the Electric Power Research Institute (EPRI), and the Environmental Protection Agency (EPA) while striving for recognition as an R&D center for ECT within the Martin Marietta Energy Systems' Complex. Chem Tech has already played supporting roles in this area for the Y-12 Plant and the Oak Ridge Gaseous Diffusion Plant (ORGDP) and is currently expanding its support to organizations within ORNL responsible for environmental matters. Over the long term, the Division hopes to achieve recognition as a center for R&D in ECT within the wider DOE system. Recent initiatives supporting these plans are discussed below.

4.1 SUPPORT TO THE OAK RIDGE COMPLEX

The ECT group performs R&D and engineering design support tasks within the broad scope of ECT. Historically, the major portion of the activities within the group has been in the area of wastewater treatment, with some involvement in gas-phase treatment and marginal emphasis on solid waste handling. Projects have been conducted for sponsors at the ORGDP, the Y-12 Plant, and ORNL. Currently, five ongoing projects are supportive of the Oak Ridge complex. A synopsis of each of the projects is provided below.

Simulation of the ORGDP Sewage Systems for Handling Y-12 Mop Water

The Y-12 facility discontinued use of the S-3 ponds for waste accumulation and sought other means of handling the liquid wastes from the Y-12 Plant until a central waste treatment facility could

be constructed. One of the Y-12 waste streams is termed "mop" water (wastewater generated by washing down equipment and floors) and was slated to be trucked to the ORGDP for treatment. Following initial pretreatment at the ORGDP, as outlined in Fig. 4.1, the waste was to be routed to the existing sewage treatment plant (STP) at the same facility. Chem Tech was requested to simulate the treatment of the mop water prior to its introduction into the STP in order to both project the performance of the STP and to ensure its continued viability. This was necessary because it could contain heavy metals, which are known to be biotoxic, as well as chlorinated hydrocarbons—many of which are typically not amenable to biodegradation. Figure 4.2 is a schematic of the system constructed at ORNL to simulate the STP at the ORGDP and includes the conditions that were simulated by the system.

The bioreactor was operated continuously for a period of 8 months (January–August 1984). During this time five different batches of mop water were received from the ORGDP and subsequently treated by the system. A significant range in feed heavy-metal concentrations (up to 10X proposed discharge limits) was encountered (Ni, V, Cr) during the operation—none of which appeared to decrease the viability of the biosystem. Varying organic loads were also imposed on the system (acetone, ethylene glycol, and purple and green dyes), and none of these organics appeared to disturb the bioreactor performance.

In summary, the system was used successfully as a real-time predictor of STP performance for 8 months. No indications of harm to the system were seen by any of the mop samples tested.

Removal of Hydrofluoric Acid from Gas Streams by Solid Sorbents

Removal of hydrofluoric acid vapor from vent gas streams by reaction with a solid sorbent such as limestone was investigated as an alternative to

Fig. 4.1. Interim waste treatment system at the ORGDP.

Fig. 4.2. ORNL model of the ORGDP sewage system handling treated mop water.

wet scrubbing. This investigation was performed in a bench-scale system (shown schematically in Fig. 4.3) at ORNL, utilizing a 2.54-cm-diam, 152-cm-high tubular reactor. The reactor configuration studied was a packed bed with gas processed in the upflow mode, which was considered to be the simplest feasible system. Twelve experimental tests were performed with the system. Dolomitic limestone, marble chips, and calcium oxide (CaO or pebble quicklime) were studied as reactive solid sorbents.

Upon reaction with moist hydrofluoric acid vapor, both the dolomite and marble chips formed a large amount of fine particulate ($<5\ \mu\text{m}$), which was entrained out of the reactor. These materials were not investigated further as potential sorbents for the acid vapor.

Most of the testing during this project was focused on evaluating the third sorbent, quicklime. Under certain conditions it was found that quicklime will remove the acid vapor from moist gas streams with very high efficiency. The maximum acid vapor loading measured in this test series was 0.63 g of hydrofluoric acid per g of bed, which translates to a calcium utilization efficiency of 90%. This test was performed with calcium oxide, -6 +9 mesh, that was slaked in situ prior to introduction of the acid vapor. The test results indicate that the initial conversion to the hydroxide form is an important factor in obtaining high conversion of the available calcium to the fluoride form. The test data also show that it may be important to maintain the bed temperature below the temperature at which the hydroxide reverts to the oxide form.

The tests with quicklime indicate that a bed depth greater than 20 to 30 cm is not feasible with a 2.54-cm-diam reactor because of the effects of wall friction that result in compaction and plugging of the bed. A staged reactor with a series of five beds, each with a depth of 15 cm, could be operated satisfactorily with minimal solids binding in the bed. Solids particle size was found to decrease somewhat during reaction. The fines generated did not, however, tend to be transported out of the reactor. Detailed information pertaining to this study is contained in ref. 1.

Evaluation of the Proposed Wastewater Treatment System at the Y-12 Steam Plant

A facility has been conceptually designed to treat the Y-12 Steam Plant and ancillary effluents to meet the "Best Available Technology" (BAT) limitations of the Clean Water Act. A laboratory simulation of the proposed flowsheet was tested in Chem Tech to demonstrate projected performance and to identify any problem areas.

The first step in the study was to obtain and characterize representative samples of the major effluents to be treated in the proposed facility. These samples were mixed, and the various combinations that the wastewater treatment facility could expect to treat were then examined. Upon mixing, the various composite streams had pH values ranging from ~ 2.0 to 2.5; none of the stream mixing studies resulted in solids precipitation. The total organic carbon (TOC) was measured at $<41\ \text{mg/L}$ in some of the samples. The samples were analyzed by gas chromatography (GC) and gas chromatography/mass spectrometry (GC/MS) to identify the organic compounds. None of the following classes of materials were detected by these techniques: volatile organics, methylene chloride extractable organics, and chlorinated hydrocarbons. In addition, the base load feed was analyzed for 5-d biological oxygen demand (BOD_5) and was found to contain $<10\ \text{mg/L}$ of BOD_5 , indicating minimal impact on the oxygen levels of the receiving stream.

Reactor/clarifier jar tests were performed on the various stream mixtures. The concentrations of regulated compounds in the clarified liquid, as well as the settling characteristics of the solids, were monitored. These tests indicate that coagulation with $\text{Ca}(\text{OH})_2$ at a pH of ~ 8.9 results in an effluent that would meet the probable Y-12 Plant discharge limits for all pollutants except chemical oxygen demand (COD) and total suspended solids (TSS). The best results, in terms of sludge settling, were produced by coagulation with $\text{Ca}(\text{OH})_2$, followed by flocculation with PURIFLOC A23. The effect of increasing the reactor/clarifier temperature from 27°C to 71°C (80°F to 160°F) was investigated. In all cases, better performance, in terms of settled sludge volume and TSS of the clarified liquid, was noted at the higher temperature. Also, at temperatures $\geq 38^\circ\text{C}$ ($\geq 100^\circ\text{F}$) the probable discharge limit for TSS would be met at the Y-12 Plant.

1. C. H. Brown, Jr., and V. L. Fowler, *Removal of Hydrofluoric Acid from Gas Streams by Solid Sorbents*, ORNL/TM-9243, January 1985.

Fig. 4.3. Flow diagram of the hydrofluoric acid vapor sorption experiment.

The sludges produced were tested for dewatering characteristics by both centrifugation and vacuum filtration. The results indicate that they are candidates for vacuum filtration but probably not for centrifugation. In terms of percent dry solids, vacuum filtration is better than centrifugation by a factor of >2 . The literature also indicates that the solids content of the sludge could be greatly improved by a "high-density sludge" process. The solids produced were analyzed for extraction procedure (EP) toxicity, and results show that all the regulated elements are present in concentrations well below the level considered EP toxic. Details of this study are reported in ref. 2.

Engineering Design Support Studies for the Nonradiological Waste Treatment Plant

A nonradiological wastewater treatment plant (NRWTP) to treat discharges from the ORNL site is being designed by Martin Marietta Energy Systems, Inc. Engineering. Chem Tech was asked to assess the proposed flowsheet for technical feasibility and to simulate the proposed treatment plant to provide treatability data. These data are needed to support Martin Marietta's negotiations for dis-

charge permits for ORNL. The data are also needed by Martin Marietta Engineering to develop design specifications for the NRWTP.

Three classes of contaminants can be present in these wastewaters: (1) heavy metals, (2) volatile organics, and (3) nonvolatile organics. The flowsheet for the plant has evolved during the project in response to perceived wastewater feed quality and projected discharge criteria. The initial plant flowsheet was rather complex and included the following treatment steps:

1. metals removal by precipitation/clarification,
2. nitrate removal via bionitrification,
3. biological oxidation for gross organics removal,
4. multimedia filtration for suspended solids,
5. granular activated carbon (GAC) to remove trace organics,
6. reverse osmosis (RO) for total dissolved solids (TDS) and mercury abatement,
7. evaporation and drying to treat the RO brine, and
8. ion exchange to polish the RO permeate for removal of residual traces of mercury.

2. J. F. Walker, Jr., C. H. Brown, Jr., and G. B. Dinsmore, *Evaluation of the Proposed Y-12 Steam Plant Wastewater Treatment System*, ORNL/TM-9234, September 1984.

Because of an increased level of understanding of the water character and the probable discharge limits, the initial flowsheet has been reduced to the following: (1) metals removal by precipitation and filtration, (2) volatile organic reduction via air stripping, and (3) removal of nonvolatile organics by GAC.

During the autumn of 1984, a series of tests was performed to simulate the GAC and RO steps in the initial flowsheets. The tests utilized the Building 3544 (process waste treatment plant) effluent and the 190 pond (Building 4500 drainage) influent. A detailed accounting of these tests is given in ref. 3.

These two process streams were doped with mercury and treated by GAC to examine the removal of organics and mercury and also to evaluate GAC as a possible pretreatment for RO. During GAC processing of the 3544 effluent (doped with mercury), feed to the system had a mean mercury concentration of 19.5 ng/mL and a mean TOC concentration of 4.6 mg/L, with mean removals of 98.8% for mercury and 37% for TOC. While operating on the 190 pond influent, the GAC feed contained a mean mercury concentration of 35.7 ng/mL and a mean TOC concentration of 5.3 mg/L, with mean removals of 97.2% for mercury and 43.3% for TOC. The low removal of TOC by GAC indicates the presence of organics that were not adsorbed onto the carbon. For the period of operation (~98,000 L of 3544 effluent processed and ~163,000 L of 190 pond influent processed), column breakthrough was not obtained for either mercury or TOC. Also, GAC had no effect on the nitrates and sulfates present in the wastewaters.

Reverse osmosis was evaluated on the 3544 effluent for the rejection of TDS, nitrate, sulfate, and mercury, using an Osmonics OSMO-192 cellulose acetate membrane and a FilmTec BW30 thin-film composite membrane. While operating with the Osmonics membrane on 3544 effluent, which had been pretreated by GAC and spiked with mercury, the feed recovery [(permeate flow rate ÷ feed flow rate) × 100] was increased from ~65% to ~95%. Within this range of recovery, the nitrate rejection

$$\left[1 - \frac{\text{permeate contaminant concentration}}{\text{feed contaminant concentration}} \times 100 \right]$$

fell from ~66% to ~23%, the TDS rejection fell from ~74% to ~46%, and the sulfate rejection fell from ~92% to ~85%. Mercury was not rejected

at any value of recovery. In comparison, when the recovery across the FilmTec membrane was increased from 60% to 94%, the rejection of TDS decreased from 95% to 83%, the rejection of sulfate held constant at >98.5%, the rejection of nitrate fell from 96% to 68%, and the rejection of mercury fell from 55% to 33%.

The addition of sulfide to the wastewater enhanced the mercury removal across both the Osmonics and the FilmTec membranes. When 0.2 g of sodium sulfide was added to 2650 L of 3544 effluent, the removal of mercury increased from 0 to ~97% with the Osmonics membrane and from ~33% to 54% with the FilmTec membrane. It was postulated that this increase in mercury removal was due to the precipitation of mercury in the sulfide form. This precipitation was confirmed with the use of 5-μm filters upstream of the Osmonics membrane, which removed an average of ~84% of the mercury after sodium sulfide was added and none before it was added.

Results indicate that average effluent mercury concentrations of ~0.1 ng/mL were attained while operating the Osmonics membrane on recycled permeate. Also, RO was not enhanced by pretreatment with GAC over the short term, and no decrease in membrane performance was detected while operating at 95% recovery for the period tested.

Engineering design support studies are under way to evaluate the current plant flowsheet. The work will be conducted in three phases. The first phase will consist of batch simulation of the proposed flowsheet in the laboratory. The Phase 1 study is designed to be a cursory look at the flowsheet and is intended to identify major pitfalls in the concept rather than to provide design data. Jar tests for metals removal will be performed in conjunction with batch air stripping and equilibrium GAC sorption of residual organic compounds. The second phase of the project will consist of more detailed parametric studies of the processes in the flowsheet. As in the first phase, these tests will be conducted primarily in the laboratory. Phase 2 plans will evolve as Phase 1 is in progress and more information becomes available about the system. Phase 3 will consist of flowsheet simulation

3. J. F. Walker and C. H. Brown, Jr., *Scouting Treatability Studies Performed in Support of the Nonradiological Wastewater Treatment Facility Feasibility Study*, ORNL/TM-9667, in preparation.

on a continuous basis, using the existing pilot equipment augmented with equipment as necessary to include processes not currently available. Planning for Phase 3 will be performed during the execution of the first two phases of the study. The results from Phases 1 and 2 will be important in planning the continuous-flow experiments. The Phase 3 results will provide the detailed design data for the NRWTP systems. The first phase of this work is nearing completion.

Engineering Analysis of the Ion-Exchange Portion of the Process Waste Treatment Plant

Process aqueous wastes generated at ORNL are currently collected in an equalization basin and processed through an ion-exchange Process Waste Treatment Plant (PWTP), located in Building 3544, to remove radioactive cations (primarily ^{137}Cs and ^{137}Sr). The Operations Division, which operates the PWTP, requested that Chem Tech perform an evaluation of the PWTP for several reasons. The efficiency of the PWTP was not as high as had been expected, based on laboratory and pilot-scale tests. Notably, resin life and capacity were lower than predicted. In addition, future requirements for the discharge of radioactive contaminants are uncertain; the relationship of the PWTP to the new NRWTP is also in question. Since it was involved in the development of both projects, Chem Tech was asked to ensure that an optimal integration of performance characteristics was achieved.

The primary goal of this project was to perform a detailed functional analysis of the PWTP to facilitate process improvement and to provide a basis for future process design. Of particular interest were (1) an accurate definition of plant feedwater characteristics, (2) an assessment of source and necessity of high plant feed and effluent nitrate concentrations, and (3) a determination of size and type of a new evaporator system.

Results of the individual tests carried out during the evaluation of the performance of the PWTP between mid-August and mid-November 1984 are summarized below:

1. High nitrate present in the plant effluent was the result of plant operating procedures. The plant *could* be readily redesigned to reduce nitrate emissions from 200 parts per million by weight (ppmw) to a value approaching the

influent level of ~ 12 ppmw. However, during the course of the investigation, plant operating procedures were modified to discharge nitrates from column regeneration to the low-level waste (LLW) plant instead of to the equalization pond. This step greatly reduced the level of nitrates going through the plant. Nitrates are accompanied by sodium, which may reduce the plant's capacity for ^{137}Cs removal. Reduction of the sodium level in the plant feed is known to increase column onstream time.

2. Projection of a new evaporator size was found to require complete quantification of the present plant operating parameters, together with a projection of the new NRWTP capacity and emission standards. The required evaporator capacity is sensitive to both column onstream time and the ability to store intermediate products during the sequence of batch treatments comprising the process. Neither the necessary capacity nor the materials of construction for a new evaporator can be predicted at this time.
3. Changing the acid strength used for regenerating the columns has an effect on column life. Evolutionary testing of acid strength in the plant appeared to indicate a significant decrease in column onstream time as the acid strength was decreased from 3.5 N to 2.7 N.
4. Limited testing has shown intense ^{137}Cs releases at the end of a run. These releases may precede the hardness level control point and may be a factor of 5 higher than the influent ^{137}Cs level. Because of delays inherent in direct ^{137}Cs assays, another element must be found which can be correlated with ^{137}Cs levels. Potassium, for which a simple, rapid, and sensitive assay technique is available, appears to be an excellent monitor for column emissions when low ^{137}Cs emissions are necessary.
5. A study of the scheduling problems with two-column operation suggests that operation to ^{137}Cs breakthrough is not the best approach. The variations between columns can cause operational overlaps that force the release of acid to the LLW evaporator system. Adopting a time-scheduled regeneration mode, in which the column with the shortest life is operated close to the breakthrough point, may not change acid consumption significantly.
6. The major unresolved issue is the volumetric capacity of the columns for low-nitrate wastewaters.

Process Waste Treatment Plant Pilot Unit

The Process Waste Treatment Facility is used to treat all of the aqueous waste containing low levels of radioactivity generated at ORNL. In the near future, the facility will be required to meet more stringent effluent limits, whether the discharge is treated further in the NRWTP or the effluent is piped directly to White Oak Creek. Several modifications to the system have been proposed. The efficacy of these changes can be judged only through experimental testing. Over the long term, the best solution to processing LLW may be through an entirely different technology than the current ion-exchange approach, such as RO or electrodialysis.

The immediate objectives of this project are (1) to design, construct, and shake down an engineering-scale facility (ESF) that will perform the same function as the currently used technology (cation exchange); (2) to make improvements on the current technology (anion exchange, rinse systems, and water softening); and (3) to test existing new technologies such as RO and electrodialysis for potential use.

The ESF will be a tool to aid in rapid solution of day-to-day operating problems as well as a vehicle for testing new concepts such as those outlined above. The system will be coupled directly to the diverter box above the equalization basin, thus eliminating the problems inherent to operations with the basin in the circuit. The facility will allow parallel comparison testing of competing technologies (i.e., ion exchange and reverse osmosis). Their ion-exchange portion of the plant has been designed, and procurement shall be initiated in the near term.

4.2 FOSSIL ENERGY PROGRAMS

Fossil Energy Technology Environmental Program

The Fossil Energy Technology Environmental Program (FETEP) is a major initiative undertaken by ORNL which began in May 1983 under the sponsorship of DOE, Fossil Energy-Office of Oil, Gas, Shale, and Coal Liquids (DOE/FE-OGSCL) to develop and maintain the environmental, health, and safety (EH&S) information data base on fossil energy conversion processes. The DOE Oak Ridge

Operations Office (DOE/ORO) is the field project manager for the FETEP project.

Chem Tech was given the responsibility for managing the overall project and executing parts of the program relevant to the Division's areas of expertise.

The initial thrust of the FETEP project in FY 1983 had the following objectives:

1. to develop "lessons learned" and "remaining needs" in the EH&S areas of direct coal liquefaction;
2. to provide reviews on behalf of DOE/FE of environmental monitoring plans submitted to the Synthetic Fuels Corporation (SFC) by sponsors of synthetic fuels (synfuels) projects; and
3. to develop a prototype EH&S Information System for fossil energy conversion processes.

In FY 1985, the FETEP project was expanded to include the identification and resolution of EH&S concerns in fossil energy conversion processes. The specific tasks to be executed under the expanded program include the following:

1. development of data-base benchmarks for physical and chemical characterizations of synthetic fuels;
2. assessment of approaches for prioritizing supplemental (e.g., noncompliance) monitoring activities at synfuels plants;
3. evaluation of the chronic toxicity of raw and upgraded synthetic oils to aquatic ecosystems;
4. development of solid wastes management studies in fossil energy;
5. assessment of the EH&S monitoring needs for synfuels technologies;
6. expansion of the prototype EH&S information system developed earlier to allow for the storage and management of EH&S data derived from and/or relevant to the direct coal liquefaction technologies and major gasification demonstration projects; and
7. extension of information systems support to DOE/FE-OGSCL in the EH&S area, particularly with regard to projects sponsored by SFC.

Activity is continuing in the FETEP area. The FETEP project has already developed a user-friendly EH&S Information System that is regarded by some users as a model data system. In

addition, several reports¹⁵ have been published (or are currently in publication) that present the results of the various tasks conducted as a part of the FETEP project.

KILnGAS® Wastewater Characterization

In the future, coal gasification is expected to provide an increasing fraction of the energy needs in the United States. The different gasification

processes produce wastewaters containing varying amounts of dissolved organics, ammonia, and other contaminants. Research is needed to characterize these wastewaters and to evaluate probable treatment schemes to make the wastewater environmentally safe.

A 544-t/d (600-ton/d) plant based on the KILnGAS process has been built near East Alton, Illinois. The objective of this demonstration facility is to determine and to confirm the ability of the KILnGAS process to convert large quantities of high-sulfur, caking coals to clean fuel gas for electric power generation in a safe, economical, and environmentally acceptable manner.

ORNL's involvement in this project is to assist EPRI in defining the wastewater management problems associated with the operation of the KILnGAS process. This support by ORNL includes:

1. a complete characterization of a grab sample of the wastewater from the process;
2. a 10-d, onsite sampling and analysis campaign; and
3. a comparative assessment of the KILnGAS wastewater with other coal conversion wastewaters.

The results of this study indicate that the KILnGAS wastewater contains a high concentration of organic material, with a large fraction composed of phenolic compounds. The water also has high concentrations of ammonia, cyanide, thiocyanate, sulfate, chloride, TSS, and TDS. Many of these contaminants are generally found in wastewaters from other coal conversion processes.

In comparing the characterization of the KILnGAS wastewater with other coal conversion wastewaters, it was observed that large variations existed in the compositions of the wastewaters from the various processes. In general, it is difficult to make a direct comparison between the wastewater characteristics of the different processes because of variations in the coal types processed and the process operating conditions.

Wet-Air Oxidation

Wastewater treatment has become a significant part of the activities at ORNL. A wet-air oxidation (WAO) system was built to broaden wastewater treatment capabilities at ORNL. This system should be capable of treating coal conversion

4. R. K. Genung, *Environmental, Health, and Safety Assessments for Direct Coal Liquefaction: Volume 1. Executive Summary*, ORNL/FETEP-1, in preparation.

5. N. B. Munro, *Environmental, Health, and Safety Assessments for Direct Coal Liquefaction: Volume 2. Summary of Lessons Learned*, ORNL/FETEP-2, in preparation.

6. M. G. Ryon, *Environmental, Health, and Safety Assessments for Direct Coal Liquefaction: Volume 3. Summary of Remaining Research and Development Needs*, ORNL/FETEP-3, in preparation.

7. A. R. Irvine, S. P. N. Singh, J. F. Villiers-Fisher, and L. M. Roseberry, *Environmental, Health, and Safety Assessments for Direct Coal Liquefaction: Volume 4. Plant Emissions and Control Status*, ORNL/FETEP-4, in preparation.

8. B. R. Clark, L. M. Roseberry, and I. B. Rubin, *Environmental, Health, and Safety Assessments for Direct Coal Liquefaction: Volume 5. Physicochemical Measurements of Process Streams, Products, and Emissions*, ORNL/FETEP-5, February 1985.

9. R. D. Roop, D. B. Hunsaker, Jr., A. W. Reed, F. C. Kornegay, and C. C. Gilmore, *Environmental, Health, and Safety Assessments for Direct Coal Liquefaction: Volume 6. Source and Ambient Monitoring Programs*, ORNL/FETEP-6, October 1984.

10. E. D. Copenhaver, C. S. Dudney, E. E. Calle, G. D. Griffin, T. D. Jones, M. Uziel, P. J. Walsh, and A. P. Watson, *Environmental, Health, and Safety Assessments for Direct Coal Liquefaction: Volume 7a. Potential Health Effects*, ORNL/FETEP-7, February 1985.

11. E. E. Calle, *Environmental, Health, and Safety Assessments for Direct Coal Liquefaction: Volume 7b. Guidance for the Development of an Occupational Health Surveillance System*, ORNL/FETEP-8, February 1985.

12. R. E. Millemann, *Environmental, Health, and Safety Assessments for Direct Coal Liquefaction: Volume 8a. Environmental Transport, Fate, and Effects of Liquefaction Products*, ORNL/FETEP-9, November 1984.

13. L. L. Sigal, *Environmental, Health, and Safety Assessments for Direct Coal Liquefaction: Volume 8b. Environmental Transport, Fate, and Effects of Atmospheric Emissions*, ORNL/FETEP-10, November 1984.

14. C. T. Hunsaker, W. J. Boegly, Jr., and E. D. Smith, *Environmental, Health, and Safety Assessments for Direct Coal Liquefaction: Volume 8c. Characteristics and Environmental Transport, Fate, and Effects of Solid Wastes*, ORNL/FETEP-11, November 1984.

15. R. E. Millemann, G. K. Eddlemon, and R. L. Kroodasma, *Environmental, Health, and Safety Assessments for Direct Coal Liquefaction: Volume 8d. Environmental Transport, Fate, and Effect of Wastewaters*, ORNL/FETEP-12, November 1984.

wastewaters and wastewaters containing bioresistant chlorinated organics. Such a system must be capable of handling waters containing chlorides at temperatures up to 350°C (660°F) and pressures up to 20.8 MPa (3000 psi). The system should also be reasonably resistant to fluorides at tens of ppmw.

A continuous-flow WAO unit has been installed in the southeast cell of Building 3503. Two stirred autoclaves, piped for continuous flow, are on line. One unit is fabricated of 316 stainless steel and the other of Hastelloy C. The working liquid volumes are ~0.8 L. Both are designed to operate at conditions up to 350°C and ~20 MPa. Air or oxygen is injected into the base of the chamber. The gas is dispersed by turbine impellers with power inputs that exceed 9.85 W/L (50 hp/1000 gal) at operating conditions.

Maximum continuous liquid feed capacity is 1.5 L/h. Maximum continuous air or oxygen capacity is 1.5 L/h at 20.8 MPa and 30°C (86°F). The air or oxygen is wet-compressed from pressures of 2.08 to 3.47 MPa (300 to 500 r:i) to achieve the system operating pressure of ~20.8 MPa (3000 psi).

The system includes a tank which contains a 4-d feed supply, a batch stripper, and a batch-stripped liquor feed tank. The feed tank may be heated to about 90°C (190°F) and is equipped with a turbine impeller with a design power input of 3.94 W/L. This power input emulsifies any free oil in the wastewater and ensures constant-composition liquid feed to the feed pump.

All the above units are contained in a high-pressure cell in Building 3503 and are remotely controlled. Four days of continuous operation of the WAO unit is possible.

The system has been operated between 17.3 and 20.8 MPa (2500 and 3000 psi), 300 to 320°C (570 to 610°F), and 0.7- to 1.5-h liquid residence time. The autoclave used in these shakedown tests was the 316 stainless steel unit. The feed consisted of 5000 ppmw ethylene glycol in distilled water. The gas was air flowing at the rate of 2 L/h measured at system operating conditions. Glycol destruction was 87% at 18.0 MPa (2600 psi) and 315°C (600°F). (Based on first-order kinetics, the corresponding conversion in the batch mode is determined to be >98%).

Two serious problems with the WAO unit have been encountered:

1. The lower graphite bearing in the totally enclosed magnetic-drive stirrer failed. On visual

examination, the bearing appearance suggested chemical attack, possibly by oxidation. In future runs, this bearing area may have to be cooled.

2. The performance of the Hastelloy C ball check valves in the high-pressure metering pumps deteriorated rapidly. The cause of failure of the ball check valves has not been determined.

Conductivity probes supplemented the feed tank level measurements in the gas compression system. The Conax fittings used in the system failed at 20.8 MPa (3000 psi). For continuous operation, some changes in the feed water circuit, coupled with level alarms in the feed tanks, are desirable.

The operational leeway at 20.8 MPa (3000 psi) is limited by the use of 22.2-MPa (3200-psi) rupture discs and by the operating band of the back-pressure control valves. The system working pressure is limited to 26 MPa (3750 psi) by the gas-holding cylinders. Use of $25 \pm 3\%$ MPa rupture discs will permit smooth operation at 20.8 MPa (3000 psi).

The WAO unit is presently on standby status. Current plans are to expand the capabilities of the units to permit high-temperature and high-pressure hydrotreatment studies of coal liquids.

Indirect Liquefaction Environmental Control Technology Assessment

The objective of the indirect coal liquefaction environmental control technology assessment (ILECTA) project was to assess the potential impact of designing a coal conversion plant to operate with zero aqueous discharge. The study consisted of evaluating two conceptual treatment schemes for decontaminating the wastewaters likely to be generated in a conceptual dry-ash Lurgi-based indirect coal liquefaction plant. The two treatment schemes are as follows:

1. Case I is the base case for this study. In Case I, the wastewater generated in the plant is treated to the extent that the resulting water can be discharged to a local river with minimal environmental impact.
2. Case II is the zero-wastewater-discharge design. In this scheme, the wastewater is subjected to additional treatment beyond that given in Case I so that the maximum amount of water can be recycled in the coal conversion plant. In Case II, no aqueous effluent is discharged from the plant.

The conceptual indirect coal liquefaction plant examined in the assessment is an integrated (i.e., all utilities generated onsite) facility designed to convert 13,605 t (~15,000 tons) of moisture- and ash-free coal per stream day to motor gasoline, using dry-ash Lurgi coal gasification, Imperial Chemical Industries' methanol synthesis, and Mobil's methanol-to-gasoline processes. The conceptual plant is premised to be located at a generic site in the eastern United States and to process a generic Interior Basin high-sulfur bituminous coal.

The project was conducted for DOE/FE, with the Pittsburgh Energy Technology Center acting as the project manager. The assessment was completed in September 1984, and a report¹⁶ on the study has been prepared.

The following conclusions can be drawn from this assessment:

1. On paper, wastewater treatment facilities can be designed that are projected to treat the indirect coal liquefaction plant wastewaters to a level where the effluent will likely meet the current regulations for aqueous effluents for allied industries such as coke ovens and petroleum refineries. However, no assurances can be given that the treatment schemes will perform as designed or will decontaminate the wastewaters to such an extent that they will have negligible impact on the environment.
2. The estimated capital investments, in 1983 U.S. dollars, for the Case I (surface-discharge) and Case II (zero-aqueous-discharge) schemes are approximately \$440 million and \$550 million, respectively. These costs represent about 15% and 20% of the estimated capital investment for the integrated indirect coal liquefaction plant.
3. Case II (zero-aqueous-discharge) wastewater treatment is likely to result in the accumulation of 900,000 t of toxic solid wastes during the 30 years of plant operation. Major uncertainty prevails in the assessment of the long-term (>500 years) integrity of the EPA-approved hazardous waste landfill design to store the toxic solid wastes.
4. Marine disposal is a potentially viable technique that can be used to dispose of the detoxified soluble salts safely.
5. Because of time constraints, only one design was developed for each case. Other wastewater treatment designs that can reduce the cost of wastewater treatment and/or further mitigate

the hazard potential of the plant effluents may also be possible; however, a lack of operating data on the constituent processes/operations precludes a more exhaustive study of design options at this time.

The following recommendations are made, based on the results of this assessment:

1. Research should be conducted to develop methods to separate the toxic soluble salts from the nonhazardous solid wastes generated in the zero-wastewater-discharge design.
2. Ocean dumping (under regulated conditions) of the detoxified soluble salts generated during the course of treating the coal liquefaction plant wastewaters to meet zero-aqueous-effluent discharge should be seriously evaluated in lieu of dumping the toxic soluble salts in a landfill.
3. Operating data should be obtained from a large-scale integrated indirect coal liquefaction wastewater treatment facility to ensure the design of wastewater treatment circuits that will decontaminate the water to "safe" environmental levels. Such data may be obtained from operating indirect coal liquefaction plants [e.g., the South African Coal, Oil, and Gas Corporation, Ltd. (Sasol) or from planned facilities (e.g., the Great Plains Gasification Project)]. Even the operation of an integrated test facility at a 0.6- to 1.2-L/s (10- to 20-gpm) throughput rate on a long-term continuous basis would greatly enhance our understanding of the wastewater treatment problem.

4.3 WATER QUALITY PROGRAMS

EPA Water Quality Program: Evaluation of Bioassays Used to Estimate Health Hazards of Wastewater Effluents

This 2-year research program has been initiated recently by an interagency agreement with the EPA in collaboration with the ORNL Analytical Chemistry, Environmental Sciences, and Health and Safety Research divisions. The major objective of this interdivisional program is to evaluate bioassay methods for predictive capabilities relative to

16. J. F. Villiers-Fisher and S. P. N. Singh, *An Evaluation of Two Conceptual Wastewater Treatment Schemes for a Lurgi-Based Indirect Coal Liquefaction Plant*, ORNL/TM-8946, September 1984.

human health hazards associated with the discharges of wastewater treatment plants or industrial effluents. This program will require a detailed and thorough examination of selected effluents using a battery of in vitro bioassays in combination with chemical characterization. Initial evaluation will include the Ames mutagenicity test with *Salmonella typhimurium* strains and several Chinese hamster ovary tests. Toxicity tests will be corroborated with some aquatic tests using fish or *Daphnia* species. Sample collection, preservation, storage, and concentration techniques will be examined to determine optimum methods to avoid sample degradation and alteration. Chemical characterization will be coupled with bioassays in order to develop a more complete understanding of the chemical nature of the possible toxicants and the potential use of bioassays for estimating human health risk associated with wastewater discharges. Initial efforts have been the selection of a highly industrialized wastewater treatment plant for sampling; a site visit to plan for the large-scale sampling effort (2600 L); and planning for preservation, storage, and concentration studies.

**Analysis of Goitrogenic Compounds:
Chemical and Biological Characterization of
Goitrogenic Substances in Drinking Water**

This research program is a collaborative effort with the University of Mississippi and DOE. Selected drinking waters are being analyzed in this program as support for a medical study on the incidence of goiter in several Appalachian areas.

Epidemiological studies of this problem indicate the possible correlation of high goiter prevalence with environmental factors, including drinking-water quality. Initial studies have identified resorcinol (a potent goitrogen) and resorcinol-like constituents in drinking waters associated with geo-

graphical and social units having a large percentage of school children with goiter. For example, resorcinol was identified in well water supplying a section of Candelaria, Colombia, in which >25% of the school children were diagnosed as having palpable goiter. Other chemical compounds may be associated with goiter (e.g., sulfides, thiocyanates, etc.).

Several constituents in the drinking-water samples were tentatively identified by GC/MS, and a comparison of the mass spectra was made with published mass spectra. The volatile constituents in fresh water samples are given in Table 4.1. Several water samples were stored for 1 year at -40°C . Volatile constituents identified in the stored samples are given in Table 4.2, and identified constituents separated from the stored samples by macroreticular resin adsorption and ether extraction are given in Table 4.3.

Water Chlorination Conference

The Fifth Conference on Water Chlorination: Environmental Impact and Health Effects was held at the College of William and Mary, Williamsburg, Virginia, June 3-8, 1984. Approximately 120 scientific papers were presented, and 225 scientists participated in the multidisciplinary conference concerning the chemical, biological, and human health effects of water chlorination treatment, including drinking water, wastewater, and cooling water. The conference is cosponsored by the U.S. Department of Energy, U.S. Environmental Protection Agency, Electric Power Research Institute, Tennessee Valley Authority, National Cancer Institute, The College of William and Mary, Virginia Institute of Marine Science, and Oak Ridge National Laboratory. A conference proceedings is being prepared; the scheduled publication date is early autumn 1985.

Table 4.1. Tentative identifications resulting from GC/MS analysis of organics separated from freshwater samples by the Purge and Trap Method^a

Organic compounds	Location ^b								
	1	2	3	4	5	6	7	8	9
Trichloroethylene	✓	✓							
Benzene									✓
Cyclohexane		✓							
Heptane		✓				✓			✓
Chloroform								✓	
Methylpentanol						✓			
Methylbutenoic acid						✓			
Isocyanooctane						✓		✓	
Hydrocarbon		✓				✓		✓	
Toluene	✓	✓	✓	✓		✓		✓	
Octane		✓			✓	✓			✓
Bromochloromethane						✓		✓	
Siloxane (MW207)	✓	✓	✓		✓	✓	✓		✓
Dimethylbenzene	✓	✓				✓			✓
Dimethylbenzene	✓	✓	✓	✓		✓		✓	✓
Bromoform						✓		✓	✓
Dimethylbenzene	✓	✓				✓		✓	
Nonane		✓			✓	✓			✓
Ethynylbenzene									✓
Hydrocarbon					✓				
Trimethylbenzene	✓				✓	✓			
Benzaldehyde									✓
Decane						✓			
Trimethylbenzene	✓	✓							
Trimethylbenzene	✓	✓							
Hydrocarbon					✓	✓		✓	✓
Dichlorobenzene	✓	✓	✓	✓					
Siloxane (281 MW)		✓		✓	✓	✓	✓		
Trimethylbenzene					✓				✓
Hydrocarbon (C ₁₁ H ₂₄)		✓	✓	✓					
Hydrocarbon									
Hydrocarbon									
Siloxane (355 MW)				✓			✓		
Hydrocarbon (C ₁₇ H ₃₆)						✓			
Hydrocarbon				✓					
9-Methoxyanthracene							✓		
Siloxane (MW 341)							✓		
Hydrocarbon									
Hydrocarbon									
Cadinene		✓	✓						
Guaiane		✓	✓						
Di- <i>t</i> -butylcresol ^c					✓		✓		
Phthalates ^d	✓		✓	✓	^d			✓	✓
Ditolylethane			✓	✓					
Methylphenylindane			✓						
Higher-molecular-weight (MW) compounds				✓ ^e			✓		

^aN₂-Purge-Tenax-Trap Method.

^bLocations by number: 1 - SA control, 2 - Darien, 3 - LaCumbre, 4 - Alaska, 5 - KY control, 6 - Center Hill, 7 - Spicer, 8 - Millersburg, and 9 - Indian Spring.

^cThese compounds represent the compound present in higher concentration. They are probably artifacts. This area needs further investigation with respect to whether phthalates may be occurring naturally.

^dGC/MS discontinued after retention time of di-*t*-butylcresol.

^eMS were obtained on several higher-molecular-weight compounds (unidentified).

^fOne compound probably contains sulfur.

Table 4.2. Tentative identification (based on GC/MS) of organics separated from stored-water samples using the Purge and Trap Method^a

Organic compounds	Location ^b											
	1	2	3	4	5	6	7	8	9	10	11	12
Methylenechloride				✓								
Perchloroethylene											✓	
Toluene				✓		✓	✓					
Siloxane (MW207)						✓	✓	✓			✓	
Dimethylbenzene	✓	✓	✓			✓	✓		✓			✓
Dimethylbenzene			✓			✓	✓		✓			✓
Bromoform									✓			
Dimethylbenzene			✓			✓	✓	✓				✓
Styrene	✓								✓			
Trimethylbenzene	✓	✓						✓	✓			✓
Trimethylbenzene	✓	✓										
Nonanone					✓				✓			
Dichlorobenzene						✓	✓		✓			✓
Siloxane (MW281)			✓			✓	✓	✓	✓			✓
Hydrocarbon									✓			✓
Hydrocarbon												✓
Phenol					✓	✓	✓		✓			✓
Methylbenzoate					✓							✓
"Tetramethylbenzenes"	✓	✓				✓						
Dihydroxyacetophenone												✓
Acenaphthalene or biphenyl			✓									
Tetralin						✓			✓			✓
Methylthiophene(?)												
Methylpropylthiophene(?)			✓									✓
Ethylbenzoate					✓	✓			✓			
Tetrahydrosulene					✓							
"Pentamethylbenzene"			✓									✓
Naphthalene				✓								
Propylbenzoate						✓						
Methylnaphthalene			✓	✓	✓	✓						✓
Dimethylnaphthalene			✓									
Dibutylnaphthalene									✓			
Di- <i>r</i> -butylcresol(BHT)	✓	✓	✓				✓	✓	✓	✓	✓	✓
Methylnaphthalene									✓			
Phthalates								✓		✓		

^aN₂-Purge-Tenax-Trap Method.

^bLocations by number: 1 - N₂ control, 2 - Distilled water control, 3 - Cruz, 4 - Matedero, 5 - No. 2 Bennett, 6 - No. 3 Chestnut Gap, 7 - No. 4 Booneville, 8 - No. 6 Jackson, 9 - No. 7 Jackson, 10 - No. 8 Haddix, 11 - No. 9 Spicer, and 12 - No. 10 Neaces.

Table 4.3. Tentative identification (based on GC/MS) of organics separated from stored-water samples by adsorption on XAD and extraction of XAD-adsorbed materials with diethylether^a

Organic compounds	Location ^b							
	1	2	3	4	5	6	7	8
Heptene	✓							✓
Dimethylbenzene	✓						✓	✓
Dimethylbenzene							✓	✓
Dimethylbenzene	✓						✓	✓
Cyclooctatetrine	✓							
Nonane	✓				✓	✓	✓	✓
Trimethylbenzene	✓							
Trimethylbenzene	✓							
Trimethylbenzene	✓							
Decane	✓				✓	✓	✓	✓
Ethylmethylheptane	✓							
Decane	✓							
Phenol		✓	✓	✓	✓	✓	✓	✓
Tetramethylbenzene	✓						✓	✓
Methylbenzoate			✓		✓			
Tetramethylbenzene	✓							
Undecane	✓				✓	✓	✓	✓
Tetralin		✓	✓	✓	✓	✓	✓	✓
Undecane	✓							
Ethylbenzoate		✓	✓	✓	✓	✓	✓	✓
Undecane	✓							
Undecane	✓							
Naphthalene			✓	✓	✓		✓	✓
Tetramethylbenzene	✓							
Ethenyldimethylbenzene	✓							
Hydrocarbon		✓						
Dodecane	✓	✓	✓					✓
Hydrocarbon		✓						
Tridecane	✓							
Butylbenzoate			✓	✓	✓		✓	✓
Dihydroacenaphthalene	✓							
Methylnaphthalene					✓			✓
Di- <i>r</i> -butyl- <i>p</i> -benzoquinone	✓				✓			
BHT	✓	✓	✓	✓	✓	✓	✓	✓
Phthalates			✓		✓	✓	✓	✓
Hydrocarbons		✓						

^aPrincipal artifact was di-*r*-butylcresol, which was a contaminant in the diethylether used for XAD extraction. XAD is a macroreticular resin manufactured by Rohm & Haas Co.

^bLocations by number: 1 - Water control, 2 - No. 2 Bennett, 3 - No. 4 Booneville, 4 - No. 6 Jackson, 5 - No. 7 Jackson, 6 - No. 8 Haddix, 7 - No. 9 Spicer, and 8 - No. 10 Neaces.

5. BASIC SCIENCE AND TECHNOLOGY

Fundamental research in chemistry and chemical engineering within Chemical Technology Division is supported by the DOE Office of Basic Energy Sciences. The work described in this section is supported by the Division of Chemical Sciences and by the Division of Materials Sciences. Work supported by the Division of Nuclear Sciences is described in Sect. 10.2 of this report.

5.1 CHEMICAL SCIENCES

The projects reported here are those investigating the chemistry of actinides and fission products, multiphase studies in external fields, sorption in separations, solvent-solute interactions in separations with supercritical solvents, fundamental processes in sorption pumping, phase equilibria of solvents in the critical region, and continuous chromatography. Other work supported by the Division of Chemical Sciences, on the kinetics of enzyme-catalyzed processes, is described in Sect. 6.

Chemistry of Actinides and Fission Products

This work is directed toward developing knowledge of the fundamental chemistry of the actinide elements and their fission products. Two subtasks are reported here: Solvent Extraction Chemistry and Raman Spectroscopic Studies of Uranyl and Zirconium Species.

Solvent Extraction Chemistry. Experimental work for the program during this report period has emphasized chemistry of the fission products, technetium and rhenium. As reported previously, an exhaustive compilation and critical review of all available data on the solvent extraction of technetium and rhenium by tributyl phosphate (TBP) has been completed.¹ A significant area not addressed in this review was the effects of various reductants [Fe(II), hydrazine, hydroxylamine] on the solvent extraction of Tc/Re in the presence of U, Pu, Zr, and Th. Experimental work to investigate this area has since been completed. Interpretation of the data is in progress, in preparation for publication.

A small effort to gather and interpret thermochemical data on the TBP-diluent-water-nitric acid system continues. Measurements at 25°C have been completed, and those at 40°C are in progress. The existing literature data are inadequate to accurately describe this solvent extraction system, so these data will prove very useful.

Raman Spectroscopic Studies of Uranyl and Zirconium Hydrolytic Species. Previous Raman spectroscopic studies of mixed actinide systems have shown that uranyl ion attaches through hydroxyl linkages to the Th(IV) polymer network, and aging of the polymer converts these hydroxyl links to oxygen bridges.² Such reactions are anomalous in that they occur at considerably lower pH than is usually necessary for the formation of pure uranyl polymer species. Because Zr(IV) hydrolyzes at pH values much lower than other tetravalent cations, its interactions with uranyl ions were examined both to clarify the attachment mechanism and to investigate the utility of Raman spectroscopy as a tool for inorganic polymer studies.

Our investigation of solutions having Zr(IV) concentrations in the range 0.10 to 0.50 M and uranyl concentrations in the range 0.05 to 0.20 M have shown unambiguously that uranyl does attach to Zr(IV) hydrous polymers at pH as low as 1.6. As in the Th(IV) studies, the attachment was detected by observing a shift in the uranyl symmetric stretching frequency. At constant pH and constant uranyl concentration, the intensity of the shifted band increased with increasing Zr(IV) concentration, as illustrated in Fig. 5.1. Aging and/or heating of the solutions resulted in the apparent elimination of uranyl from the polymer network. Solutions that had been allowed to evaporate to

¹D. J. Pruett, *Solvent Extraction of Heptavalent Technetium and Rhenium by Tributyl Phosphate*, ORNL-TM/8668, December, 1984.

²L. M. Toth, H. A. Friedman, G. M. Begun, and S. E. Dorris, "Raman Study of Uranyl Ion Attachment to Th(IV) Hydrous Polymer," *J. Phys. Chem.* **88**, 5574, (1984).

Fig. 5.1. Variation of shifted Raman band at 835 cm^{-1} with increasing Zr(IV) concentration. All solutions contain 0.20 M UO_2^{2+} . (a) 0.10 M Zr(IV) ; $\text{pH} = 2.35$; (b) 0.25 M Zr(IV) ; $\text{pH} = 2.25$; (c) 0.50 M Zr(IV) ; $\text{pH} = 2.25$.

near dryness showed unmistakable phase separation of zirconium from uranium. These results are in marked contrast to those obtained from Th(IV) studies and are attributed to structural differences between the Th(IV) and Zr(IV) polymers. The ease with which the precipitated uranyl and Zr(IV) can be separated has important implications for programs which rely on coprecipitation for extraction of uranium.

The predominant species in strongly acidic Zr(IV) solutions is known from x-ray scattering experiments to be a hydroxyl-bridged tetramer, $\text{Zr}_4(\text{OH})_6(\text{H}_2\text{O})_{16}^{8+}$, with associated anions. Continued hydrolysis results in the replacement of H_2O by OH , aggregation of tetramers and, ultimately, precipitation of hydrous zirconia. We have monitored this process qualitatively through Raman spectroscopy, as shown in Fig. 5.2. In an effort to

Fig. 5.2. Raman spectra of Zr(IV) solutions at various stages of hydrolysis. Spectra of powdered $\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$ and Zr(OH)_4 are included for comparison. (a) $\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$; (b) 0.50 M Zr(IV) in 1.00 M HCl ; (c) 0.50 M Zr(IV) ; $\text{pH} \approx 0.4$; (d) 0.50 M Zr(IV) ; $\text{pH} \approx 0.7$; (e) Zr(OH)_4 ("hydrous zirconia").

quantify correlations between the spectrum of the tetramer and its known structure, we have undertaken a theoretical normal-coordinate treatment of spectra obtained from oriented single crystals of zirconium oxychloride octahydrate, which contains the tetrameric cation. The stretching and bending force constants derived from this analysis will be used to calculate aggregate spectra, which will then be compared with the spectra of hydrolyzed solutions. It is our hope that this work will lead to a better understanding of the structure and behavior of extensively hydrolyzed systems.

Multiphase Studies in External Fields

Several individual projects are included in an overall initiative to seek principles and basic

methods for studying the influence of external electric fields upon mass transport and fluid mechanics. Two-phase, liquid-liquid systems are the current focus of activity. In particular, studies of the effects of electric fields on the droplet formation process and on mass transport in the two-phase liquid systems are progressing. Forcing droplet oscillations is an important facet of this research, and the apparatus designed for this purpose is shared, to some extent, among project groups. Within each of these projects, modeling of the equations for mass, momentum, and electric field is important in deriving a comprehensive theory.

Droplet Formation in Electric Fields. The formation of the dispersed phase in a two-phase fluid system is often the most important single factor in the ultimate mass transfer characteristics of a separation process. We have investigated means of controlling drop size and convection in both phases near the interface to optimize mass transfer and the coalescence with must occur later. The imposition of an electric field on the medium in which

the drops are formed has profound effects on both drop size and convection around the drops. Theoretical and experimental efforts are in progress to provide a better understanding and utilization of these effects.

A system has been built in which the hydrodynamic and mass transfer behavior of two-phase liquid systems can be studied. Droplets are formed at an orifice in a square column with flat viewing windows (shown in Fig. 5.3). The orifice geometry is typical of industrial drop-forming devices. A uniform field is produced by two parallel-plate electrodes, and the water droplets are formed in pure cyclohexane. The effects of flow rate, applied electric field, and nozzle diameter have been studied previously. Theoretically, an applied electric field should produce smaller drops; this is confirmed by our observations. A typical result is shown in Fig. 5.4, where the application of a moderate electric field reduces the drop size by an order of magnitude. Other important system variables such as interfacial properties, electrode geometry, and elec-

Fig. 5.3. Schematic layout for the measurement of interfacial tensions.

Fig. 5.4. Drop volume as a function of applied electric field in the formation of drops from an orifice.

trical conductivity are under experimental investigation. Electric field effects in multidrop systems are also of interest.

Past work in this area has focused primarily on gas-liquid systems. While the theory developed for these problems provides an interesting framework for liquid-liquid systems, the effects of the continuous phase required modifications of the theory. In the presence of a uniform electric field, the electrostatic force on a spherical drop may be theoretically described by the expression:

$$F_e = 4\pi\epsilon r^2 E_0^2 \times \left\{ \sum_{i=0}^{\infty} \frac{b_i}{a_i^2} - \sum_{i=0}^{\infty} \sum_{j=0}^{\infty} \right\} \quad (1)$$

$$6A[(\alpha^2 - 1)A + B]^2 \frac{4(\alpha^2 - 1)A - B}{C^2}$$

where

- ϵ = the dielectric constant of the dispersed phase,
- r = drop radius,
- E_0 = applied voltage,

and where the geometric terms are

$$a_0 = 1, \quad b_0 = \alpha,$$

$$a_{i+1} = \alpha a_i + b_i, \quad b_{i+1} = (\alpha^2 - 1)\alpha_i + ab_i,$$

$$A = a_i a_j, \quad B = b_i b_j, \quad C = a_i b_j + a_j b_i.$$

The term, α , is defined as $(l + r_e)/r_e$, where the nozzle length is l , and its radius is r_e .

The term enclosed within braces in Eq. 1 is a function of the nozzle geometry. From this expression, a balance is taken between the surface forces and the combined gravitational and electric force fields (which are aligned). Since this expression for spherical drops has limited applicability, we are proceeding to develop expressions for ellipsoidal drops, with eccentricity as a factor.

A second initiative in this area seeks improvements in the measurement of interfacial tensions in liquid-liquid systems.³ Potentially, the most accurate means of measurement (particularly in systems with surfactants) is the pendant drop method. The video analysis equipment developed for this project makes it possible to overcome the experimental error inherent in the conventional technique and to automate a tedious and painstaking process.

Droplet Electrohydrodynamics. The goal of the study is to explore the enhancement of mass transfer to and from droplets by oscillation. The mass transfer characteristics and hydrodynamics of single-drop systems are observed using a pulsed electric field that is controlled by resistance in the continuous phase. Finite elemental analyses are being used to develop hydrodynamic models of the drops.

Experimental research is conducted using single water drops fluidized in 2-ethyl hexanol and subjected to a pulsed DC electric field of 3 to 10 kV/cm. Droplet oscillations are observed and recorded using a high-speed video camera and recorder, as depicted in Fig. 5.5. Data acquisition is via microcomputer, and computer software handles the data analysis and storage. Droplet oscillations have been observed to be of two distinct types. At low frequencies (10 to 30 Hz), droplets oscillate between oblate and prolate spheroid forms (shown at the top of Fig. 5.6) while at higher frequencies (100 to 150 Hz), droplets oscillate

³R. A. Malinauskas and C. H. Byers, *Droplet Formation Phenomena in DC Electric Fields*, ORNL/TM-9446 (to be published).

Fig. 5.5. Video camera and recorder used in droplet oscillation studies.

between two prolate spheroid forms (shown at the bottom of Fig. 5.6).

Additionally, liquid-film mass transfer data on the oscillating drops show that the mass transfer coefficients increase as the frequency increases, and this characteristic cannot be attributed solely to the surface area changes. Mass transfer coefficients increase by 35% (shown in Fig. 5.7) when the frequency changes from 0 to 50 Hz, while the surface area increases by <1%. A plot of the surface area ratio of a stretched drop to a spherical drop versus the aspect ratio (ratio of the drop's y axis to its x axis) is given in Fig. 5.8. Our operating range is within aspect ratios of 0.7 to 1.3, where surface area changes are <2%. This finding indicates that previous theoretical results have not adequately considered local convection and drop

hydrodynamics. Current efforts are concerned with reviewing previous models and extending theory to provide a better mathematical description of convection terms.

The theoretical aspects of drop hydrodynamics are being addressed using finite elemental analysis. Initially we will solve steady-state Navier-Stokes problems using the Galerkin weighted-residuals method. Numerical and analytical solutions exist which can be used to verify the algorithm before proceeding to the unsteady-state problem. Time-dependent solutions will be obtained using Newtonian iteration. Eventually we hope to describe the droplet's free surface using a modification of Bernoulli's equation. Operating time has been obtained on the DRAY-XMP supercomputer at Lawrence Livermore National Laboratory

Fig. 5.6. Forms of droplet oscillation at both low and high frequencies in an imposed electric field.

Fig. 5.7. Mass transfer coefficient versus oscillation frequency for 0.3-cm water drop fluidized in 2-ethyl hexanol (natural oscillation frequency = $100 s^{-1}$).

through the DOE's Office of Chemical Sciences, in order to develop the needed computer codes.

Mass Transfer in Oscillating Droplets. In conjunction with the studies of droplet electrohydrodynamics discussed in the previous section a program has been developed to investigate the effect of droplet oscillation upon the rates of mass trans-

Fig. 5.8. Surface area ratio versus aspect ratio for stretched and spherical forms of an oscillating droplet.

fer for dissolved chemical species. The objective of this program is to determine mass transfer coefficients for liquid-liquid systems in which the major resistance to mass transfer lies in the dispersed phase. The initial focus of this study has been in two general areas: theoretical treatment of the mass transport process and development of techniques to acquire experimental data for species dissolved in the water/2-ethyl-1-hexanol system.

The transport of material in the droplet-continuum system has been modeled in terms of a new, hybrid, mass transfer theory which incorporates aspects of both the traditional boundary-layer approach and penetration theory. Solution of the descriptive equations yields concentration profiles for areas near the interface through which a dissolved chemical species is transferring into or out of the droplet. From these concentration profiles, the flux of the dissolved species and the associated mass transfer coefficients for the process may be calculated. The expression for the flux of the dissolved component can be integrated over the entire surface area of the droplet to obtain data for theoretical predictions of the change in concentration of the species in the droplet as a function of elapsed interfacial contact time. Numerical solution of the descriptive equations has been accomplished for short contact times. The resultant concentration profiles are consistent with expected behavior of the system. From these preliminary results, it appears that the model will be useful in predicting mass transfer rates in the droplet-continuum system.

In continuing the work, experimental mass transfer rates will be determined by measuring the change in concentration of a species dissolved in the droplet as a function of interfacial contact time; this will be monitored utilizing spectrofluorescence techniques (see Fig. 5.9). Several industrial dyes have been identified for use as the dissolved chemical species, based on their solubility properties in water and in 2-ethyl-1-hexanol. The components for the experimental apparatus have been specified, and the system is undergoing construction and the initial stages of testing.

Fig. 5.9. Schematic of apparatus for measuring mass transfer in oscillating droplets. The rate of mass transfer for a liquid-liquid system will be determined by monitoring the change in concentration of a fluorescent dye in the drop as a function of interfacial contact time.

Videotapes of images produced by scattering of a helium-neon laser beam from the interface yield detailed pictures of the streamlines for studying the flow of the continuous phase, the flow patterns of interfacial movement, and the circulation patterns inside the droplet. Quantitative measurements of velocity profiles have also been made from examinations of the videotapes. Results from this flow-visualization technique are also being used to assist in the formulation of more realistic models to improve theoretical descriptions and predictions of the mass transfer process.

Sorption in Separations

During this report period, the major emphasis of our sorption research has shifted from a narrow focus on tritium separations science to the broader area of understanding sorption's role in separations. While this work began with, and will continue to be applicable to, the important area of fusion energy research, it will no longer be directed by specific needs of that program. Our purpose is to develop a fundamental understanding of sorption which will be applicable to a broad range of scientific problems. Our emphasis is now on a fundamental investigation of sorption in relation to separations, and our goal is to relate the dynamics of sorption (adsorption rate, desorption rate, surface diffusivity, etc.) and statics of sorption (surface residence times, separation factors, spreading pressures, etc.) to the intermolecular forces that control these effects. This will require a continuation of our theoretical work as well as the development of new experimental techniques to determine the dynamic parameters affecting adsorption.

Our work on tritium separation from lithium has been essentially completed. The final phase involved the development of a new class of sorbers for the removal of tritium from liquid lithium and two studies of sorber regeneration. Hydrogen equilibrium, hydrogen diffusivity, and tritium uptake from liquid lithium were measured for a yttrium-thorium alloy sorbent. These materials form a single-phase solid solution at most compositions. This sorber provides the option of modifying the equilibrium pressure of the getter over a wide range so that the conditions of the regeneration process can be made less severe. It was found that the Sieverts' constant for the mixture can be calculated from those of the pure components through the ideal solution theory.

The recovery of tritium from sorbents into argon was found to be very slow and incomplete even at high temperatures. Two factors contributed to this. First, the very low tritium equilibrium pressure limits the rate at which the sweep gas can remove it from the system. Second, the surface equilibration time is significantly lower than that observed in absorption experiments. Vacuum regeneration gives much faster removal rates, approaching those expected from equilibrium considerations alone. The increased rate is a result of eliminating the gas-phase resistance and significantly reducing the surface effects. The removal rate and temperature

for vacuum regeneration are acceptable for fusion applications.

Solvent-Solute Interactions in Separations with Supercritical Solvents

Late in FY 1983, a fundamental study of the molecular interactions between solvent and solute in supercritical solutions was initiated. Our goal is to develop a predictive capability for use in supercritical separations based on fundamental understanding of molecular interactions as represented by an intermolecular potential function. Both theoretical and experimental approaches are being used.

Theoretical Development. Previous studies (see, for example, the work of Kurnik et al.⁴) have successfully fit supercritical solubility data using an empirical, cubic equation of state, but such an approach fails to provide fundamental understanding or predictive capability. However, with temperature-dependent, empirical, binary parameters, it is possible to interpolate within the region of fit. Johnston and coworkers⁵ have developed a simple, semitheoretical model for solubility in a supercritical fluid based on first- and second-order perturbations from a hard-sphere fluid in a van der Waals one-fluid model. The first-order model fails at densities ~ 1.3 to 1.5 times the critical density, and even the second-order model fails at densities less than the critical density. Finally, because of the empirical simplifications to the model, the physical significance of the regressed binary parameters is questionable.

Jonah et al.⁶ have shown (with perturbation theory calculations) that a Padé approximation of the Pople expansion of the Helmholtz equation of state, with an appropriate potential function for molecular interaction, can represent qualitatively the variation of solubility in a supercritical fluid. While the theoretical basis of this work is sound, the computations are laborious, and the determination of molecular constants from solubility data has not been attempted.

In our work we are exploring a different theoretical approach. Recently it has been shown that the complete virial series for a hard-sphere fluid, corrected in the first few terms with virial coefficients for a realistic potential function having an attractive term, can represent the behavior of gases and liquids in a relatively simple fashion:⁴

$$Z = \frac{Pv}{RT} = Z^{HS} + \sum_{r=2}^m \frac{B_r - B_r^{HS}}{v^{r-1}}, \quad (2)$$

where

- E = pressure,
- v = molar volume,
- R = gas constant,
- T = temperature,
- Z^{HS} = hard-sphere fluid compressibility,
- B_r = r th virial coefficient for a real fluid,
- B_r^{HS} = r th virial coefficient for a hard-sphere fluid.

Although this equation has not been proved rigorously, Gibbs et al.⁷ have provided considerable theoretical substantiation and have shown its excellent agreement with the results of Monte Carlo calculations for a Lennard-Jones fluid.

We have extended the Gibbs equation to model the solubility of supercritical fluid mixtures. Using the Nezbeda⁸ equation for the compressibility of mixed, hard, convex bodies in place of the equation for hard-sphere compressibility allows us to account for the configurational effects at high densities of substantially different solvent and solute molecular size and shape, which are common in supercritical solubility studies. Thus,

$$Z = \frac{Pv}{RT} = Z^{HB} + \sum_{r=2}^m \frac{B_r - B_r^{HB}}{v^{r-1}}, \quad (3)$$

where

- Z^{HB} = mixed, hard-body compressibility,
- B_r^{HB} = r th virial coefficient for hard, convex bodies.

This equation has then been used to derive an expression for the fugacity coefficient of a solute in solution. The resulting equation allows the prediction of solubility from molecular size and shape

⁴R. T. Kurnik, S. J. Halla, and R. C. Reid, "Solubility of Solids in Supercritical Carbon Dioxide and Ethylene," *J. Chem. Eng. Data* **26** (2), 47-51 (1981).

⁵K. P. Johnston, D. H. Ziger, and C. A. Eckert, "Solubilities of Hydrocarbon Solids in Supercritical Fluids. The Augmented van der Waals Treatment," *Ind. and Eng. Chem. Fund.* **21**, 191-97 (1982).

⁶D. A. Jonah, K. S. Shing, V. Venkatasubramanian, and K. E. Gubbins, in *Chemical Engineering at Supercritical Fluid Conditions*, Ann Arbor Science, 1983.

⁷J. H. Gibbs, I. Pavlin, and A. J. Yang, "Vapor Condensation and the Liquid State. An Extension of Mayer's Theory," *J. Chem. Phys.* **83**(3), 1443-49 (1984).

⁸T. Boublik and I. Nezbeda, "Monte Carlo Study of the Equimolar Mixture of Hard Spheres and Spherocylinders," *Czech. J. Phys.* **B30**, 121-27 (1980).

and molecular interaction potential parameters for cases where the first few virial coefficients have been derived. This derivation of the fugacity coefficient for a fluid of mixed, hard, convex bodies from the Nezbeda equation was originally done by Lee and Kanchanakpan;⁹ however, errors in the original derivation have been subsequently corrected.

Although the fugacity coefficient equation is very cumbersome to present in written form, it is readily evaluated by computer. Furthermore, the parameters in the expression have clear physical meanings and can be calculated *a priori* for the molecules of interest. Although only limited testing has been done to date, the equation appears to represent quite well the properties of fluids over a wide range of temperature and density conditions above the triple point including the vapor phase, the two-phase, vapor-liquid region, the liquid phase, and the supercritical fluid phase. It should be noted, however, that this expression is no more successful than any other analytical equation of state for representing properties in the immediate vicinity of the critical point, where scaling laws must be used.

Experimental. We have chosen to focus on the solubility of inorganic solutes in order to broaden the data base for supercritical solubility, which at present consists almost entirely of data for organic solutes. This focus also allows us to build upon Chem Tech's substantial base of experience in solution chemistry and separations of inorganic solutes. Our initial experiments involve simple molecular fluids as solvents, in order to provide a basis for testing the theoretical model discussed earlier. Subsequently, solvents exhibiting stronger and more complex interactions with the solute molecules will be explored, including polar, hydrogen-bonding, and complex-forming compounds.

Scoping experimental studies showed that the first simple solvent of choice, carbon tetrachloride, is far too reactive at temperatures slightly greater than its critical temperature ($\sim 300^\circ\text{C}$) to be used for these studies. Subsequent experiments with sulfur hexafluoride showed that, even at the substantially lower temperatures slightly greater than its critical temperature ($\sim 50^\circ\text{C}$), considerable chemical reactivity would render theoretical interpretation of experimental results very difficult. Consequently, it was decided to use carbon tetrafluoride as the solvent for the first research studies. At

temperatures slightly above its critical temperature ($\sim -40^\circ\text{C}$), it is hoped that the kinetics of the complicating chemical reactions may be so slow as to be insignificant. At such temperatures, several moderately volatile inorganic fluoride salts (e.g., IrF_6 , RhF_6 , UF_6 , MoF_6 , WF_6) are available as solutes for study.

A research apparatus has been designed and is under construction for the planned research (Fig. 5.10). The apparatus may be operated in a flow-through mode for supercritical solubility and separation studies. It may also be operated in a batch mode to permit isochoric pressure-volume-temperature (p-v-t) studies for determining vapor pressure, critical point properties, saturated liquid molar volume, and other thermodynamic properties of pure compounds and binary mixtures of interest. The equipment is designed to operate over a temperature range from -190° to 400°C and over a pressure range from vacuum to 3.45×10^5 Pa (345 bar). The isochoric p-v-t cell (Fig. 5.11) contains $\sim 85\%$ of its volume within a quartz capillary so that a vapor-liquid meniscus may be observed and to facilitate future nuclear magnetic resonance studies. To resist the corrosive effects of fluorides and chlorides, the apparatus is constructed of nickel and quartz, with gold-plated metal seal rings. Should it be necessary, a sapphire capillary can be fabricated by Sapphikon, Inc. to replace the quartz capillary.

Fundamental Processes in Sorption Pumping and Transfer Operations Using Deep Beds of Sorbents

Cryosorption pumping of gases on deep beds of sorbents is becoming an important process in several energy-related areas, including the storage of fuel gases and the development of fusion energy. Both current experiments and future applications relating to fusion energy require clean roughing pumps to evacuate chambers with volumes of up to 10^4 m³. Large cryosorption pumps, in which gases are sorbed on deep beds of molecular sieves at liquid-nitrogen temperatures, are under consideration for this service. However, modeling of the heat- and mass-transport processes that occur as the gas is sorbed requires more detailed understanding than is presently possible.

⁹L. L. Lee, University of Oklahoma, personal communication to H. D. Cochran, Oak Ridge National Laboratory, November 5, 1984.

Fig. 5.10. Schematic diagram of apparatus for supercritical solubility and separation studies.

Our vacuum pumping experiments have utilized cylindrical beds of adsorbents that are immersed in an appropriate coolant (e.g., liquid nitrogen). The apparatus is fitted with valves calibrated to ensure a constant feed rate for several gases. Both the inlet pressure and the differential pressure across the bed are measured as functions of time and feed rate. Data have been obtained for the sorption of nitrogen and carbon dioxide on beds of molecular sieve (4A) pellets.

When gas moves into the bed, hydrodynamic transport takes place as gas flows through the bed interstices, and diffusional transfer occurs as adsorbed molecules move from the surface to the interior of the sorbent particles. The two transport processes can be related by a mass balance around a differential length of bed, relating concentrations,

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Fig. 5.11. Cell for isochoric pressure-volume-temperature studies of supercritical solutions.

c , in the gas phase, and $\bar{q}$, in the solid phase, to time, t , and position, z :

$$\epsilon \frac{\partial c}{\partial t} + \rho_B \frac{\partial \bar{q}}{\partial t} + \epsilon \frac{\partial(vc)}{\partial z} = 0, \quad (4)$$

where ϵ is the void fraction in the bed, ρ_B is the bulk density of the bed, and v is the gas velocity.

Gas drawn into the bed is in molecular flow, so velocity and pressure drop are related somewhat differently than in viscous flow,¹⁰ namely:

$$v = -\frac{\kappa}{p} \frac{\partial p}{\partial z}. \quad (5)$$

The parameter κ is a function of molecular speed and channel geometry, making it difficult to predict for flow through the interstices of a packed bed.

Mass transport of a single-component gas within sorbent particles may be considered a diffusional process using an effective mass diffusivity. Representing particles as spheres of radius, r , the governing equation is

$$\frac{\partial q}{\partial t} + \frac{D}{r^2} \frac{\partial}{\partial r} \left(r^2 \frac{\partial q}{\partial r} \right). \quad (6)$$

A numerical solution of the model equations was obtained,¹¹ and plots of both modeling results and experimental data are shown in Fig. 5.12 (for nitrogen) and Fig. 5.13 (for carbon dioxide).

The model accurately corresponds to the experimental data for nitrogen until the bed approaches saturation. Values of κ that best fit the experimental data are: $8 \times 10^{-3} \text{ m}^2/\text{s}$ for a bed of 0.55-mm pellets, and $1.05 \times 10^{-2} \text{ m}^2/\text{s}$ for a bed of 0.72-mm pellets. Thus the value of κ is proportional to pellet size.¹² The effective diffusivity for nitrogen in the solid phase is $5 \times 10^{-12} \text{ m}^2/\text{s}$, which is very high compared to intracrystalline

¹⁰J. J. Perona, K. S. Crabb, C. H. Byers, and J. S. Watson, "Vacuum Sorption Pumping Studies of Nitrogen on Molecular Sieves," presented at the AIChE Annual Meeting, Washington, D.C., December 1983, Paper No. 52d.

¹¹K. S. Crabb, "Mass Transfer in Vacuum Sorption Pumping of Pure Gases on Molecular Sieves," M.S. Thesis, University of Tennessee, Knoxville, 1984.

¹²K. S. Crabb, J. J. Perona, C. H. Byers, and J. S. Watson, "Vacuum Sorption Pumping Studies with Pure Gases on Molecular Sieves," presented at the AIChE National Meeting, Houston, Texas, March 1985, Paper No. 53c, and accepted for publication in *AIChE J.*

Fig. 5.12. Pressures and pressure drops for N_2 sorption on 0.72-mm 4A sieves at a feed rate of $1.2 \times 10^{-3} \text{ mol/s}$. Lines are calculated using a theoretical model.

Fig. 5.13. Pressures and pressure drops for CO_2 sorption on 0.72-mm 4A sieves at a feed rate of $3.3 \times 10^{-3} \text{ mol/s}$. Lines are calculated using a theoretical model.

values extrapolated to 77 K. Apparently adsorption occurs only on the exterior crystal surfaces, and the effective diffusivity represents macropore diffusion.

For carbon dioxide, the model represents accurately the pressure-time curves above the bed but does not describe with great precision the pressure-drop data across the bed. It is postulated that the controlling diffusion resistance for carbon dioxide is in the micropores of the pellets.

Phase Equilibria of Solvents in the Critical Region

The development of new processes for the synthetic production of natural gas as a part of advanced coal conversion technology has made the study of new concepts in the separation of light gases necessary. The use of liquid carbon dioxide near its critical temperature as an absorption medium has been explored in a preliminary manner by this study, and a simulation of a proposed flowsheet (Fig. 5.14) has indicated feasibility. The process clearly will be more energy efficient than the alternatives that require separation at cryogenic temperatures. Uncertainties in the feasi-

bility studies stem primarily from our inability to predict the behavior of carbon dioxide as a solvent in the near-critical region. Further, other liquid solvents (such as near-critical ammonia) may be superior to the proposed system. The rapidly changing nature of solvents in the critical region requires a strong data base as a foundation for any predictive correlations. A high-pressure, vapor-liquid equilibrium apparatus has been designed and built (Fig. 5.15). Central to the apparatus is a high-pressure cell and a magnetically operated recirculation pump. Operation of the unit is controlled and monitored by a computer, and the data from one run are all stored on one disk file so that theoretical manipulations may proceed without manual data transfer. The construction and debugging of this device and its data acquisition system are virtually complete.

From a theoretical viewpoint, the pure component and mixture data that are being acquired in this program, as well as existing data on these systems, are being compared with the various equation-of-state predictions. Considerable success is claimed for the Peng-Robinson equation in this region, and a newer, more fundamental equation that is the result of work by Whiting and Prausnitz¹³ also shows considerable promise. Systems containing CO_2 and CH_4 , as well as a third component such as H_2S , CO , H_2 , or COS , are under study.

Continuous Chromatography

Development. Multicomponent liquid chromatographic separations have been achieved using a slowly rotating annular bed of sorbent material. Elution chromatography occurs through the continuous introduction of feed material at a stationary point in the top of the resin bed, with eluent distributed evenly around the remainder of the annulus. The rotation of the sorbent bed causes the separated components to appear as helical bands, each with its characteristic stationary exit point at the bottom of the bed, making the separation process truly continuous.

The fundamental work on the theoretical and experimental development of this new separations

Fig. 5.14. Process schematic diagrams for the recovery of methane from a synfuel gasification reactor.

¹³W. B. Whiting and J. M. Prausnitz, "Equations of State for Strongly Nonideal Fluid Mixtures: Application of Local Compositions Toward Density-Dependent Mixing Rules," *Fluid Phase Equilibria*, 9, 119-47, (1982).

Fig. 5.15. Schematic diagram of supercritical fluid VLE apparatus.

concept was completed during this report period, and results (including a dispersion theory for modeling the behavior of the continuous chromatograph) are reported in several publications.^{14,15,16} As shown in Fig. 5.16, increasing the concentration of the feed material to the resin bed causes a deterioration of performance. However, Fig. 5.17 shows that the annulus can be widened to include as much as 95% of the available annular volume without significantly affecting performance, provided a proper feed distribution is maintained over the width of the annulus.

Technology Transfer. The emphasis during this period has been on the transfer of this technology to the public realm. We have developed an ASPEN code that allows the evaluation of continuous chromatography in a processing plant context.

¹⁴J. M. Begovich, C. H. Byers, and W. G. Sisson, "A High-Capacity Pressurized Continuous Chromatograph," *Sep. Sci. Technol.* 18(12&13), 1167-91 (1983).

¹⁵J. M. Begovich and W. G. Sisson, "A Rotating Annular Chromatograph for Continuous Separations," *AIChE J.* 30, 705-10 (1984).

¹⁶R. M. Canon and J. M. Begovich, "Continuous Stationary-Bed Ion Exchange: Incomparable Power for Difficult Separations Involving Higher-Value Products," pp. 113-22 in *Frontier Technology in Mineral Processing*, AIME, 1985.

Fig. 5.16. Resolution achieved by a continuous chromatographic separation of nickel in solution with two cobalt ions (Co-1 and Co-2) as a function of feed concentration.

Fig. 5.17. Resolution of continuous chromatographic copper-nickel separation as a function of feed-to-eluent flow ratio for varying annulus widths (CAC-II, CAC-II-2, and CAC-II-3).

In the code, a FORTRAN routine is used to size the equipment and compute the separation of components for given operating conditions. This can be used for a stand-alone mode or for a plant setting.

The feasibility of continuous electrochromatography has been proven as a part of our technology transfer activities. The combined effects of electrophoresis and chromatography on protein molecules have also been studied, which is a concept with important implications in biological separations.

Contacts with companies who are interested in commercializing continuous chromatography have proceeded at a brisk pace. These contacts include companies seeking to market the technology, as well as firms whose interest is in solving particular separations problems.

Transport Properties of Fluids at Extreme Conditions

The major focus of this program has been on the application of dynamic light scattering in studying high-temperature fluid properties. Light-scattering techniques permit the use of small sample volumes in temperature-controlled enclosures and allow the dynamics of molecular or colloidal species to be characterized and related to the controlling transport properties. The mobility of the light-scattering species is characterized as a diffusion coefficient, while application of the Stokes-Einstein relationship to model colloid systems allows calculation of the suspending fluid's viscosity.¹⁷ Our recent work has concentrated on the measurement of high-

temperature, nonpolar fluid viscosities using model colloids as the suspended scattering species.

Figure 5.18 illustrates the operation of our custom high-temperature spectrometer. An argon-ion laser beam ($\lambda = 488$ nm) is focused through the optical ports of a high-temperature enclosure and onto a sample cuvette. The light scattered at 90° is detected by a photomultiplier. This oscillating signal (oscilloscope) contains the basic mobility information, which is processed to yield an exponential curve. The time constant of this curve is related to the dynamics of colloidal motion and, ultimately, to the fluid viscosity.

Stabilized silica particles ($0.1\text{-}\mu\text{m}$) were used as model colloids for high-temperature viscosity experiments with nonpolar fluids.¹⁸ The viscosities of *n*-pentane, *n*-heptane, cyclohexane, chloroform, and toluene were measured at temperatures ranging from 20°C up to 90% of the critical temperature. An ideal mixture (pentane-heptane) and an interacting mixture (hexane-isopropanol) were also studied at elevated temperatures. Measured viscosities were generally accurate to within 2%.

The results for *n*-heptane are typical (Fig. 5.19). Low-temperature behavior is properly predicted by an exponential (Andrade) relationship. As the temperature rises above the normal boiling point, the fluid behavior is more accurately characterized by corresponding-states model. For both mixtures and pure fluids, we found that only the corresponding-states methods offered any degree of predictive validity. This is particularly true for predictions involving multicomponent systems.

5.2 MATERIALS SCIENCES

The work described in this section is supported by the DOE Office of Basic Energy Sciences through the Division of Materials Sciences. The two materials sciences research programs conducted by Chem Tech are concerned with thermodynamics and sensitivity analyses of energy-related systems and chemical engineering research.

¹⁷D. F. Williams et al., *Determination of High-Temperature Fluid Viscosity Using Dynamic Light Scattering*, ORNL/TM-9648, (in press).

¹⁸A. K. Van Helden et al., "Preparation and Characterization of Spherical Monodisperse Silica Dispersions in Nonaqueous Solvents," *J. Colloid Interface Sci.* 81(2), 354-68 (1981).

Fig. 5.18. High-temperature spectrometer used to study transport properties of fluids at extreme conditions.

Fig. 5.19. Viscosity of *n*-heptane: experimental results, literature values, and predictions.

Thermodynamics of Energy-Related Materials

The objective of this study is the determination and modeling of phase equilibria and other thermochemical data important to energy-producing systems. Current emphasis is on the measurement and application of such data for the actinide oxides used as nuclear fuels, but the methodology is applicable to any oxide solid solution. The present studies involve relevant subsystems of elements U, Pu, O, and specific fission products, particularly lanthanides. Experimental data are obtained under conditions generally not previously investigated. Our own adaptations of chemical and mathematical models from the literature are being used to represent the chemical potential and non-stoichiometry reported in the literature. These

efforts provide a previously unavailable, generalized, chemical thermodynamic description of the actinide-lanthanide dioxide solutions.

The computer program SOLGASMIX-PV, developed in Sweden, is used world-wide for calculating chemical equilibria. Chem Tech personnel have greatly enhanced the usefulness of this program by identifying all variables and publishing a brief description of how they are to be used.^{19,20}

The experimental study and analysis of the $\text{PuC}_{1.5}\text{-PuO}_{1.5}$ system was also completed during this period, and the resulting report²¹ received the 1983 "Best Paper Award" given by the Nuclear Division of the American Ceramic Society. In this report, thermodynamic values for $\langle \text{PuO}_{1.5} \rangle^*$ were assessed using an improved method for estimating $\text{fel}^0\langle \text{PuO}_{1.5} \rangle$ and new data for $S_{298}^0\langle \text{PuO}_{1.5} \rangle$. Based on the assessment, a value of -828 kJ/mol for $\Delta H_{298}^0\langle \text{PuO}_{1.5} \rangle$ is recommended. Measurements of carbon monoxide pressure over the nominal equilibrium, $\langle \text{PuC}_{1.5} \rangle + \langle \text{PuO}_{1.5} \rangle + \langle \text{C} \rangle$, were performed at temperatures ranging from 1348 to 1923 K, yielding pressures from 0.644 to 11,600 Pa. Second- and third-law analyses were used to obtain a value of $-93.3 \pm 3.3 \text{ kJ/mol}$ for $\Delta H_{298}^0\langle \text{PuC}_{1.5} \rangle$.

As another part of this program, the behavior of Cs and Te in breeder reactor fuel was analyzed in a cooperative effort with General Electric Co., Sunnyvale, California.²² A combination of fuel chemistry modeling and equilibrium thermodynamic calculations was used to predict the atom ratios of Cs and Te fission products (Cs:Te) that can be found in the fuel-cladding interface region of irradiated, stainless steel-clad, mixed-oxide, fast-breeder reactor fuel pins. It was concluded

that the ratio of condensed, chemically associated Cs and Te in the interface region, $\langle \text{Cs:Te} \rangle$, which in turn determines the tellurium activity, is controlled by an equilibrium reaction between Cs_2Te and the oxide fuel. The value of $\langle \text{Cs:Te} \rangle$ is, depending on fuel O:M, either equal to or slightly less than 2:1. Since Cs and Te fission products are both implicated as causative agents in FCCI (fission product-assisted inner-surface attack on stainless steel cladding) and in FPLME (fission product-assisted liquid metal embrittlement of AISI-Type 316), the observed out-of-pile Cs:Te thresholds for FCCI ($\sim 4:1$) and FPLME ($\sim 2:1$) have been rationalized in terms of Cs:Te thermochemistry and phase equilibria. The results of this investigation also led to the development of an updated chemical evolution model for the reactive/volatile fission product behavior in irradiated oxide pins.

Our analysis of the chemical thermodynamic behavior of the nonstoichiometry of uranium dioxide, plutonium dioxide, and the uranium-plutonium dioxides was also completed during this report period.^{23,24} All the available data concerning oxygen potential, temperature, and composition for the calcium fluorite-structure, $\langle \text{PuO}_{2-x} \rangle$ phase were retrieved from the literature and utilized in the development of a binary solid-solution representation of the phase. The data and phase relations are found to be best described by a solution of $[\text{Pu}_{4/3}\text{O}_2]$ and $[\text{PuO}_2]$, with a temperature-dependent interaction energy. The fluorite-structure $\langle \text{U}_{1-x}\text{Pu}_x\text{O}_2 \rangle$ is assumed to be represented by a combination of the binaries $\langle \text{PuO}_{2-x} \rangle$ and $\langle \text{UO}_{2+x} \rangle$, and thus is treated as a solution of $[\text{Pu}_{4/3}\text{O}_2]$, $[\text{PuO}_2]$, and either $[\text{U}_2\text{O}_{4.5}]$ or $[\text{U}_3\text{O}_7]$. The resulting equations can reproduce very well the large quantity of oxygen potential-temperature-composition data for the mixed-oxide system that were also retrieved from the literature. Our models are the first that appear able to represent the appropriate oxygen potential-temperature-composition and phase-relation behavior over the entire range of existence for the phases.

¹⁹T. M. Besmann, *SOLGASMIX-PV, A Computer Program to Calculate Equilibrium Relationships in Complex Chemical Systems*, ORNL/TM-5775, April 1977.

²⁰E. C. Beahm, "Description of Variables Used in the SOLGASMIX Program," *CALPHAD* 7, 103-11 (1983).

²¹T. M. Besmann and T. B. Lindemer, "Assessment of the Thermodynamic Values for $\langle \text{PuO}_{1.5} \rangle$ and High-Temperature Determination of the Values for $\langle \text{PuC}_{1.5} \rangle$," *J. Am. Ceram. Soc.* 66, 782-85 (1983).

* $\langle \rangle$ denotes solid phase; $[]$, solid solution component; $\{ \}$, liquid phase.

²²M. G. Adamson, E. A. Aitken, and T. B. Lindemer, "Chemical Thermodynamics of Cs and Te Fission Product Interactions in Irradiated LMFBR Mixed-Oxide Fuel Pins," *J. Nucl. Mater.* 130, 375-92 (1985).

²³T. B. Lindemer and T. M. Besmann, "Chemical Thermodynamic Representation of $\langle \text{UO}_{2+x} \rangle$," *J. Nucl. Mater.* 130, 473-88 (1985).

²⁴T. M. Besmann and T. B. Lindemer, "Chemical Thermodynamic Representation of $\langle \text{PuO}_{2-x} \rangle$ and $\langle \text{U}_{1-x}\text{Pu}_x\text{O}_2 \rangle$," *J. Nucl. Mater.* 130, 489-504 (1985).

The entire $\langle \text{UO}_{2-x} \rangle$ data base for the dependence of the nonstoichiometry, x , on temperature and chemical potential of oxygen (oxygen potential) was retrieved from the literature and represented. This data base was interpreted by least-squares analysis using equations derived from the classic thermodynamic theory for the solid solution of a solute in a solvent. For hyperstoichiometric oxide at oxygen potentials more positive than $-266,700 + 16.5T$ J/mol, the data were best represented by a $[\text{UO}_2]\text{--}[\text{U}_3\text{O}_7]$ solution. For O/U ratios >2 and oxygen potentials below this boundary, a $[\text{UO}_2]\text{--}[\text{U}_2\text{O}_{4.5}]$ solution represented the data. The $\langle \text{UO}_{2-x} \rangle$ data were represented by a $[\text{UO}_2]\text{--}[\text{U}_{1/3}]$ solution. The resulting equations represent the experimental $\ln(p_{\text{O}_2}) - \ln(x)$ behavior and can be used in thermodynamic calculations to predict phase-boundary compositions consistent with the literature (p_{O_2} is defined as the pressure, in MPa, divided by the standard-state pressure, 0.101 MPa). Collectively, this thermodynamic analysis permits, for the first time, a mathematical representation of the total data base.

Kinetics of Nucleation and Growth of Microcrystalline Systems

The study of nucleation and particle growth is an area of increasing importance, particularly in the development of high-temperature ceramics. The solid morphology of materials formed from powders is largely dependent upon control of the powder size. Typical powder synthesis involves complex systems that are not amenable to study or precise control. The goal of this study is to investigate model powder synthesis with the specific objective of identifying the mechanisms by which monodisperse, submicron colloids are formed. Our central effort has been to apply dynamic light scattering to the study of particle growth.

This research was a natural extension of our work on high-temperature fluid transport properties, which explored several methods for colloid synthesis.²⁵ Our initial investigations of particle growth dealt with the synthesis of uniform, submicron metal oxides. Using a method suggested by Matijevic,²⁶ we were able to produce extremely uniform crystalline Fe_2O_3 (hematite) particles (Fig. 5.20). Figure 5.21 shows the growth of this material from a hydrodynamic diameter of 0.05 μm to 0.12 μm . More recent studies have involved

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Fig. 5.20. Uniform microcrystalline Fe_2O_3 particles synthesized in our studies on the development of high-temperature ceramics.

ORNL DWG 85-685

Fig. 5.21. Laser light-scattering data showing particle growth in hematite sol.

²⁵D. F. Williams et al., *Determination of High-Temperature Fluid Viscosity Using Dynamic Light Scattering*, ORNL/TM-9648, (in press).

²⁶E. Matijevic and P. Scheiner, "Ferric Hydrous Oxide Sols," *J. Colloid Interface Sci.* 63 (3), 589-624 (1978).

the synthesis of uniform spheres of SiO_2 from ethyl orthosilicate.^{25,27} Growth from sizes as small as 0.015 μm has been followed using dynamic light scattering.

Further refinement of the experimental procedure has been accomplished by the addition of a custom multiangle spectrometer. This instrument

will permit the investigation of crystal growth to sizes as small as 0.002 μm .

²⁷A. K. Van Helden et al., "Preparation and Characterization of Spherical Monodisperse Silica Dispersions in Nonaqueous Solvents," *J. Colloid Interface Sci.* 81(2), 354-68 (1981).

6. BIOTECHNOLOGY PROGRAMS

Chem Tech has continued R&D in four major areas of biotechnology: (1) biophotolysis and enzyme kinetics; (2) advanced bioreactor systems and microbial immobilization; (3) development of bioprocessing systems for wastewater treatment and anaerobic digestion; and (4) development of bioanalytical concepts for monitoring the physiological effects of environmental pollutants.

6.1 BIOLOGICAL RESEARCH

During this report period, biological research in Chem Tech focused on the fundamental and applied aspects of biological systems for fuels and chemicals production. Specific systems include intact microorganisms, such as bacteria and algae, and extracted organelles and enzymes. An important area of research is understanding the fundamental interactions between biological and nonbiological systems for energy production from renewable inorganic resources.

Platinized Chloroplasts

An important problem that must be solved to gain understanding of the physiochemical mechanisms of biological systems is the electron transfer at interfaces. We have approached this problem in a unique way by precipitating colloidal platinum onto the surface of photosynthetic membranes and entrapping this composition of matter onto fiberglass filter paper. We have demonstrated that this moistened material is capable of the simultaneous photoproduction of molecular hydrogen and oxygen when irradiated with visible light. Experimental data are presented in Fig. 6.1. A diagrammatic illustration of a working hypothesis for the location of the precipitated platinum is shown in Fig. 6.2.

Fig. 6.1. Simultaneous photoevolution of hydrogen and oxygen by entrapped photosynthetic membranes in contact with dispersed metallic platinum. If the platinum is precipitated *before* the chloroplasts are added, no activity is observed.

Fig. 6.2. Colloidal platinum precipitates on the exterior surfaces of the photosynthetic membranes in such a way that photo-generated electrons traverse the membrane interface and drive the hydrogen evolution reaction on the platinum catalyst.

The significance of this work is threefold: (1) this two-component system is the simplest one yet devised for photosynthetically splitting water into molecular hydrogen and oxygen; (2) it eliminates the need for an electron relay (such as ferredoxin or methyl viologen) between the oxygen-evolving chloroplasts and the hydrogen-evolving catalyst; and (3) it demonstrates *direct* interaction of a nonbiological catalyst on a biological membrane, with preservation of functional electron transfer between the biological and nonbiological components.

It is currently believed that the structure of photosynthetic membranes is such that the light reactions generate oxidizing equivalents in the interior (lumen) of the thylakoid and reducing equivalents on the exterior. In all prior research in this area, it had been thought that an electron relay such as ferredoxin or methyl viologen was necessary to transport electrons from the reducing side of Photosystem I to the hydrogen-evolving catalyst, either hydrogenase or a nonbiological catalyst.

In the preparation of metal catalysts on polymeric supports such as polyvinyl alcohol, the metal salt solution is treated with reducing agents in the presence of the polymer. One possible reducing agent is molecular hydrogen. In the research performed during the current report period, we have substituted photosynthetic membranes for the polymer. It is fortuitous that the experimental conditions required for catalyst preparation do not destroy the functional activity of the chloroplasts.

The platinum salt solution and thylakoid membranes are stirred for 90 min in a sealed vessel in which hydrogen flows in the headspace above the liquid. The contents of the liquid are then exposed to the atmosphere and filtered onto fiberglass filter paper. The chloroplasts and catalyst are retained on the filter paper. Visual inspection of the material on the filter paper reveals that the chloroplasts have been blackened by the presence of the metal colloid.

Control experiments indicate that the photosynthetic membranes and catalyst need to be in contact in order for hydrogen and oxygen to be produced. If the catalyst is prepared first and the chloroplasts then added, no activity is observed. Stirring the mixture of platinized chloroplasts (omitting the filtration step) yielded low activity and low stability. These latter results suggest that although the chloroplasts and catalyst are initially in contact, they become separated during the course of prolonged stirring.

These experiments will provide a new opportunity for studying fundamental interfacial electron-transfer reactions as well as a new system for the synthesis of a gaseous fuel from renewable energy resources.

Artificial Photosynthesis and Divalent Cations

The role of manganese in maintaining the stability of the oxygen-evolving complex of Photosystem II in chloroplasts is controversial. We have investigated the effect of exogenously added manganese chloride on the simultaneous photoproduction of hydrogen and oxygen by the chloroplast-ferredoxin-hydrogenase (CFH) system.

Saturating illumination of the CFH system resulted in the simultaneous photoproduction of hydrogen and oxygen whose maximum rates were 16.5 and 3.7 nmol·min⁻¹·mg chl⁻¹, respectively. These rates were stimulated by up to factors of 3 and 5 when manganese was included in the reaction mixture at a final concentration of 2.3 mM (Table 6.1). Preincubation of the chloroplast membranes with manganese in the dark for 1.5 h was necessary to achieve maximum stimulation. The operational stability of the CFH system was increased by a factor of 2 with the presence of manganese. Magnesium and calcium were as effective as manganese in increasing the functional stability of the CFH system. These data are supportive of evidence that suggests divalent cations stimulate the electron donation reactions of Photosystem II of chloroplasts. They are the first of their kind to demonstrate such a stimulatory effect in an *in vitro* reconstituted artificial photosynthetic system. This experiment also marks the first observation of the enhancement of Photosystem I function (hydrogen) by Photosystem II (Oxygen) constituent.

Enzymology Research

Recovery and reuse of cellulase from cellulosic hydrolysates or fermentation broths are necessary if the enzymatic hydrolysis of cellulose is to become commercially viable. Conventional immobilization of cellulase (e.g., covalent attachment to a water-insoluble support) is not suitable as a method for its recovery because adsorption of the enzymes on cellulase will be impaired, and greatly reduced catalytic activity will be apparent.

We have found that if the cellulase enzymes are adsorbed via their carbohydrate moiety onto the

Table 6.1. Maximum rates and half-lives of the simultaneous hydrogen and oxygen evolution by the chloroplast-ferredoxin-hydrogenase system

Addition	Hydrogen		Oxygen	
	Maximum rate ($\text{nmol} \cdot \text{min}^{-1} \cdot \text{mg chl}^{-1}$)	$t_{1/2}$ (min)	Maximum rate ($\text{nmol} \cdot \text{min}^{-1} \cdot \text{mg chl}^{-1}$)	$t_{1/2}$ (min)
None	17.5	82	4.0	75
	15.2	65	2.9	51
	18.1	53	3.7	37
	15.0	48	4.8	60
MnCl_2 (2.3 mM)	60.0	>120	23.7	116
	39.0	>120	12.0	>120
MgCl_2 (2.3 mM)	29.0	123	5.2	123
CaCl_2 (2.3 mM)	31.0	94	4.1	152
NaCl (2.3 mM)	18.2	33	5.1	30

water-insoluble affinity material concanavalin A-Sepharose (CAS) and then contacted with insoluble cellulose, the enzymes become desorbed from the support and adsorbed on cellulose. Scatchard plots of the data obtained from ligand-binding experiments give dissociation constants (K) of 3.1×10^{-3} and 1.2×10^{-2} mg protein·mg adsorbent⁻¹ and E_{sat} values of 1.5×10^{-2} and 1.1×10^{-1} mg·mg adsorbent⁻¹ for the adsorption of cellulase protein to cellulose and CAS, respectively. Thus cellulase has a higher affinity for cellulose than does CAS.

A novel method for cellulase recovery based on these findings has been proposed. In a simultaneous saccharification-fermentation system (e.g., cellulose to ethanol), exposure of an adsorbed enzyme to cellulose will result in desorption of the enzyme from the support and adsorption onto cellulose with subsequent hydrolysis. Readsorption of the enzyme onto the support will occur when all the substrate has been converted to ethanol by the system (Fig. 6.3).

A simple and inexpensive assay for glucose based on the coupling of glucose oxidase (GO) and Fenton's reaction has been developed. Hydrogen peroxide, formed as a result of the action of glucose oxidase on glucose, oxidizes ferrous sulfate (Fenton's reaction), resulting in a peak of maximum absorbance at 340 nm. Using this assay reagent (GO/ FeSO_4), a linear relationship between glucose concentration in the range of 100 to 500

Fig. 6.3. Concept for the recovery of cellulase by its reattachment to an affinity ligand.

mg·dL⁻¹, or 5.55 to 27.78 mmol·L⁻¹, and absorbance at 340 nm was obtained. Furthermore, the assay has been shown to be precise, accurate, and specific for glucose.

Three aspects of this assay are noteworthy: (1) routine assays of β -glucosidase (which catalyze the hydrolysis of cellulose-derived cellobiose to glucose) have been satisfactory, thereby making them

useful for determining glucose produced by other enzymes that hydrolyze energy-rich substrates (e.g., cellulose, starch, glucose); (2) interference with the GO/FeSO_4 reagent by several sugars, metal ions, reduced compounds, and yeast extract that might be encountered in a glucose assay is minimal; and (3) use of the GO/FeSO_4 reagent is less expensive (by a factor of 10) than the commercially available enzymatic reagent that is based on the action of hexokinase and glucose-6-phosphate dehydrogenase on glucose. This cost factor is extremely important for investigators in developing nations that are participating in national programs aimed at developing fuels from rapidly renewable sources of energy such as cellulose, starch, and sucrose.

Microbial Coal Liquefaction

Recent reports^{1,2} of fungi that produce a thick, semiliquid product when cultured on lignite have prompted an assessment of the potential use of this phenomenon in a microbial process to produce useful liquid products from coal. The initial emphasis of this program is to: (1) obtain strains of fungi reportedly capable of growing on and degrading lignite coal, with primary reliance on other researchers supported in this program area by DOE; (2) test lignites from different sources in order to identify those lignites which are subject to microbial action and which are readily available and sufficiently well characterized so as to provide reference source material for current and future investigations; (3) evaluate suspension cultures in parallel with the more conventional surface culture technique originally used to demonstrate this phenomenon; and (4) develop appropriate analytical procedures to characterize and identify the products of microbial action on coal and verify their origin.

Several microbial strains known to interact with lignites have been isolated or obtained from others. Three of these strains, *Trametes versicolor*, *Penicillium waksmanii* ML20, and *Candida* sp. ML13 are being utilized to screen lignite samples. Four lignite samples are currently being studied: a North Dakota lignite and a Vermont lignite (both obtained from the Coal Research Section, The Pennsylvania State University); a Texas lignite used in previous ORNL coal research; and a Mississippi lignite obtained from H. B. Ward, University of Mississippi. Preliminary tests have been

conducted by culturing the fungi on the surface of agars that contain either mineral salts alone or supplemented with a protein hydrolysate and dextrose and by using suspension culture techniques. A summary of recent results is presented in Table 6.2. Some diversity exists in the type of liquid products observed (clear to golden, to brownish-black liquids) and the organism/coal combinations resulting in these products. To date, we have been unable to draw conclusions as to the conditions affecting product formation. Experiments are in progress to resolve these issues.

Although the surface culture technique represents a useful screening procedure, the major goal is to develop a submerged culture procedure that may have subsequent process applications. Preliminary experiments have shown that direct contact between the organisms and coal does occur in submerged culture. It is believed that this direct contact is necessary for the organisms to act effectively on the coal. Future experiments will be designed to enhance this contact by increasing the surface area of the coal available (by size reduction), by chemically treating the coal surface to remove potentially inhibitory compounds, and by adding polyelectrolytes to promote attachment.

Several analytical procedures have been tried to characterize the liquid products obtained. The products are primarily water soluble and only sparingly soluble in organic solvents such as hexane, methylene chloride, and ethanol. Infrared, ultraviolet, and fluorescent spectrophotometry did not reveal significant information about the compounds present. Gas chromatographic analyses of underivatized and silylated products indicate that the components are likely high-molecular-weight, polar compounds. Samples of the liquid product have been submitted for mass spectrometric analysis. Currently, we are working on a liquid chromatographic procedure to separate and to identify the components in the liquid products.

6.2 BIOENGINEERING RESEARCH

Work has continued on the immobilization of microorganisms into biocatalyst beads for use in

1. M. S. Cohen and P. O. Gabrielle, *Appl. Environ. Microbiol.* **44**, 23 (1982).

2. H. B. Ward, "Apparent Bioliquefaction of Lignite by Fungi and Their Growth on Lignite Components," *Bioenergy 84 Proceedings*, vol. 3, *Biomass Conversion*, in press.

Table 6.2. Summary of recent experimental tests

Culturing techniques	Nutrients	Type of lignite	Preliminary results
Coal on agar surface ^a	Sabouraud-dextrose agar	Mississippi lignite (original)	<i>Candida</i> sp. ML13, <i>P. waksmanii</i> ML20, and <i>T. versicolor</i> 12679—all produced black liquid drops on coal surface.
		Texas and North Dakota lignites	<i>Candida</i> sp. ML13, <i>P. waksmanii</i> ML20, and <i>T. versicolor</i> 12679 produced clear to light brown drops on coal surface.
		Vermont and Mississippi (new) lignites	Experiments in progress.
	Mineral salts agar	Mississippi (original), Texas and North Dakota lignites	<i>Candida</i> sp. ML13 produced clear drops on coal surface. Growth minimal on agar and coal. Other organisms failed to grow or showed poor growth on agar and coal.
		Vermont and Mississippi (new) lignites	Experiments in progress—direct inoculation of coal to enhance contact.
Suspension ^b		North Dakota (powdered) lignite	<p><i>Candida</i> sp. ML13—growth and attachment in H₂O/coal suspension.</p> <p><i>P. waksmanii</i> ML20, <i>T. versicolor</i> 12679 and TLig (unidentified isolate)—growth and attachment in nutrient (dextrose ± yeast extract) supplemented flasks after 18 d.</p> <p>Samples of liquid are frozen for later analysis.</p>

^aExperimental conditions: 25–30°C; >90% relative humidity.

^bExperimental conditions: 30°C; flasks shaken at 75 rpm.

columnar bioreactors. Primary emphasis has focused on the use of hydrocolloidal gels, such as alginate and carrageenan, to encapsulate organisms such as *Zymomonas mobilis* into beads small enough for use in fluidized-bed bioreactors.

Most work has been with κ -carrageenan. The biocatalyst beads are formed by forcing a solution of the gel material through a small nozzle at a high velocity. A tuned vibrational force is imposed on the forming nozzle to cause the formation of essentially monodispersed beads. The carrageenan solution is maintained slightly above the gelling temperature (typically, 30 to 35°C) and then forced through the nozzle by a gas overpressure. The resulting beads are chemically fixed by contact with a 0.3 M KCl solution at ambient temperature. Beads in the general size range of 0.5 to 3.0 mm diam can be produced, depending on the nozzle size, flow rate, and physical properties of the gelling solution. A series of tests has been made at varying operating conditions to provide

the basis for the establishment of a correlation of bead size with the various physical properties of the system (see Table 6.3). Some definite effects were noted, including:

1. the physical properties of the gel-forming material are important;
2. nozzle size and stream velocity have a positive link to bead size; and
3. imposed vibration significantly affects the size distribution (standard deviation) of the beads, indicating that an optimum frequency possibly exists for each set of parameters.

Knowledge of the physical properties of the gel-forming fluid (density, viscosity, and surface tension) will also be important variables that will be measured in the future.

It was found that the beads tended to deform on contact with the potassium chloride solution unless the surface tension of the receiving solution was

Table 6.3. Preliminary results from biocatalyst bead formation tests

Material	Nozzle size (mm)	Temperature of gel solution (°C)	Flow rate (mL/s)	Imposed vibration frequency (Hz)	Bead size (mm)	
					Av	Standard deviation
4% κ -carrageenan	0.25	31.8	0.08	0	1.96	0.40
	0.25	31.7	0.10	190	1.96	0.16
	0.25	31.7	0.10	250	1.57	0.18
	0.25	31.7	0.10	275	1.02	0.12
	0.25	31.7	0.10	300	1.18	0.05
	0.25	31.6	0.11	300	1.17	0.04
	0.25	33.7	0.10	280 ^a	0.96	
	0.25	38.2	0.10	325 ^a	1.07	0.08
	0.35	31.6	0.16	300	1.17	0.14
	0.35	31.7	0.17	175	1.55	0.19
	0.35	31.7	0.17	200	1.49	0.06
	0.35	31.7	0.17	250	1.49	0.17
	0.35	31.7	0.17	300	1.33	0.06
	0.35	31.7	0.17	1000	1.91	0.34
	0.35	34.4	0.26	370 ^a	1.57	0.24
	0.35	35.0	0.23	290 ^a	1.57	0.24
	0.35	35.0	0.29	420 ^a	1.59	0.31
	0.35	38.3	0.23	300 ^a	1.41	0.24
	0.35	38.9	0.19	325 ^a	1.30	0.15
	0.78	31.6	0.47	310	2.17	0.12
	0.78	31.6	0.47	350	2.59	0.27
	0.78	31.6	0.47	375	2.76	0.13
	0.78	31.7	0.43	250	2.05	0.43
	0.78	31.7	0.43	300	2.48	0.18
	0.78	31.7	0.50	310	2.21	0.14
	0.78	31.7	0.43	400	2.34	0.40
	0.78	31.7	0.43	800	2.53	0.33
	0.78	34.1	0.37	250 ^a	2.33	0.37
	0.78	34.4	0.43	320 ^a	2.95	0.54
	0.78	35.0	0.53	275 ^a	2.54	0.20
	0.78	38.3	0.23	300 ^a	2.60	0.35
4% κ -carrageenan and 3% Fe ₂ O ₃	0.25	34.0	0.12	260	1.02	0.05
	0.25	34.0	0.12	270	1.00	0.03
	0.25	34.0	0.12	280	1.03	0.12
	0.35	34.1	0.21	310	1.17	0.04
	0.35	34.1	0.21	300	1.22	0.05
	0.35	34.1	0.21	800	1.68	0.19
	0.78	34.1	0.30	260	1.65	0.13
	0.78	34.1	0.30	300	1.51	0.09
4% κ -carrageenan and 10% <i>Z. mobilis</i>	0.25	38.2	0.12	325 ^a	1.16	0.17
	0.25	39.1	0.11	330 ^a	1.08	0.09
	0.35	38.0	0.25	380 ^a	1.48	
	0.35	39.1	0.10	330 ^a	1.74	0.37
4% κ -carrageenan, 3% Fe ₂ O ₃ , and 10% <i>Z. mobilis</i>	0.25	37.0	0.13	360 ^a	1.03	0.15
	0.25	38.2	0.09	350 ^a	0.88	0.11
	0.35	38.2	0.07	340 ^a	0.84	0.14
	0.35	38.2	0.10	350 ^a	1.15	0.16
	0.35	38.2	0.10	360 ^a	1.24	0.16

^aFrequency at apparent maximum stream stability as indicated under stroboscopic light.

reduced by the addition of a surfactant or other chemical. (Apparently the surface tension of the gelling solution is significantly lower than that of the salt solution.) Introduction of 10% ethanol or 200 ppm of an appropriate surfactant to the fixing solution was found to be adequate.

Adjusting Density of the Beads

For some anticipated applications with the biocatalyst, an increase in specific gravity will be needed. For example, in fluidized-bed systems, the shear forces are high and the efficiency of the bioreactor allows very high flow rates of the feed; dilution rates of 3 to 4 h⁻¹ are not uncommon. This condition will tend to wash out suspended organisms and small biocatalyst beads of low density. We are investigating the incorporation of inorganic oxide particulates into the gel structure to increase the bead density. Most tests to date have been performed with Fe₂O₃ powder in the range of 10 to 50 m. Preliminary results indicate that the incorporation of the oxide into the gel is essentially additive. That is, when 3 wt % Fe₂O₃ is added to carrageenan, the resulting gel bead has a density gain of ~0.03 g/cm³.

Stability of Immobilized Microorganisms

Preliminary results indicate that when microorganisms are incorporated into the gel bead, they continue to respire and propagate with ultimate biomass content approaching theoretical packed density. Since it is desirable to retard microbial loss from the biocatalyst bead, the leak rates of various formulations are being determined. Beads, nominally 1 to 3 mm diam, were made with 4 wt % carrageenan and initial *Z. mobilis* concentrations of ~10⁹ cells/mL. In one set of tests, 3 wt % Fe₂O₃ powder was added to the beads for density control, and in other tests the biocatalyst beads were coated with a 0.5- to 1.0-mm film of 5 wt % carrageenan. These materials were then subjected to leak tests in which a measured quantity of the beads was placed in a known amount of nutrient solution. After a prescribed length of time (usually 20 h), the microbial concentration was measured in both the bead material (by dissolution in hot distilled water) and in the bulk solution. The resulting ratio of concentrations per unit of time was considered the leak rate.

As indicated in Table 6.4, the beads with Fe₂O₃ maintained a higher bead microbial concentration

Table 6.4. Preliminary microbial leak rates from carrageenan beads

Material	Microbial concentration in bead (cells/mL)	Leak rate ^a (cells/immobilized cells · h)
4% κ -carrageenan	1.2×10^9	0.031
4% κ -carrageenan	3.7×10^9	0.040
4% κ -carrageenan with 3% Fe ₂ O ₃	2.6×10^{10}	0.002
4% κ -carrageenan with a 5% κ -carrageenan surface film	3.7×10^9	0.010

^aLeak rate is defined as the concentration of cells in solution divided by the concentration of immobilized cells per unit time.

than those with carrageenan alone, and the microbial leak rate was considerably reduced. The dense gel film on the beads also retarded the microbial leak rate, but the film stability has not yet been determined.

6.3 BIOPROCESS DEVELOPMENT

The projects described in this section are the ANFLOW demonstration pilot plant, volume reduction of radioactive cellulosic wastes by anaerobic digestion, biooxidation of coal gasification wastewaters using fixed-film, fluidized-bed bioreactors, and the Bioprocessing Research Facility.

ANFLOW Demonstration Pilot Plant

The development of anaerobic filter technology for the treatment of municipal wastewater has been pursued by ORNL since 1976. The anaerobic, upflow (ANFLOW) process has been operated successfully at the laboratory scale and also at the 19-m³/d and 190-m³/d scales with a pilot plant located at the Loves Creek Wastewater Treatment Plant in Knoxville, Tennessee. Performance has been consistent and predictable; no scale-up problems have been encountered. A commercial ANFLOW unit of the latter size could handle the municipal wastewater produced by 250 to 500 people. Larger systems could be constructed using this basic module.

After ~2 years of operation, the pilot plant was decommissioned and the site was returned to the city of Knoxville, as required by contractual agreements.

During treatment of low-strength municipal wastewater before primary sedimentation [typically, 100 mg/L each of total suspended solids (TSS) and biological oxygen demand (BOD)], the effluent met the EPA secondary treatment discharge limits of 30 mg/L for TSS and BOD the vast majority of the time, with only an occasional increase to the 30- to 40-mg/L range for a brief period of time (perhaps 1 to 2 d). Loading rates were ~ 0.25 kg/m³·d of TSS and BOD each, and the hydraulic retention time was 9 to 10 h.

The bioreactor was operated at ambient temperature from October 1, 1982, to March 16, 1983. At this time the feed preheater was turned on, and the spring and summer warmup period was simulated. A high-strength wastewater study, in which primary clarifier underflow was added to the wastewater feed, was then conducted.

The performance for the different operating conditions throughout this period is summarized in Table 6.5. In general, the good TSS and BOD

removals achieved during the startup period were maintained in ambient cold-weather tests (water temperature, $\sim 12^{\circ}\text{C}$) although the rate of solids accumulation in the bioreactor was higher in cold weather because of decreased biological activity that usually converts solids to off-gas. These solids were partially digested later during the warm-weather operation.

The primary mechanism for removal of TSS and BOD appeared to be biophysical filtration. Approximately 20% of the influent carbon was converted to methane, and 30% remained in the bioreactor as sludge. The remainder of the carbon was converted to CO₂ or was retained in the liquid effluent as TSS and BOD/chemical oxygen demand (COD). Over half of the methane was dissolved in the effluent water; this methane could potentially be recovered along with the methane in the off-gas.

Gas production increased at higher loading rates during the high-strength wastewater study. Solids

Table 6.5. Operation and performance summary for the 190-m³/d ANFLOW pilot plant^a

Operating or performance parameter	Raw wastewater		Raw wastewater plus primary clarifier underflow
	Cold weather	Temperature control to $\geq 20^{\circ}\text{C}$	Temperature control to $\geq 20^{\circ}\text{C}$
Temperature of feed, $^{\circ}\text{C}$	12–18	20–25	22
Period of operation, d	137	213	61
Hydraulic loading rate, m ³ /m ² ·d	10.0	7.3	7.3
TSS loading rate, kg/m ³ ·d	0.15	0.12	0.73
BOD loading rate, kg/m ³ ·d	0.13	0.23 ^b (0.14) ^c	0.40
COD loading rate, kg/m ³ ·d	0.35	0.30	1.2
TSS removal efficiency, %	80	80	78
BOD removal efficiency, %	63	69 ^b (50) ^c	70
COD removal efficiency, %	50	57 ^b (45) ^c	71
Methane yield coefficient, L CH ₄ /kg COD removed	50	160	110
Volumetric CH ₄ production rate, L CH ₄ /m ³ reactor·d	9	24	94
Concentration of CH ₄ in off-gas, %	3–9	63–76	60–80
Sludge production rate, kg dry solids/3800 m ³ of wastewater treated	170	200 ^b (120) ^c	575

^a All average values were derived from monthly averages.

^b Values observed during the startup period.

^c Values observed during the increasing-temperature period.

accumulation also increased, probably due in part to the high solids content of the wastewater formulated by mixing primary clarifier underflow with the raw wastewater. Performance of the ANFLOW process in terms of effluent water quality under these conditions is ambiguous, however, because the bioreactor was heavily loaded with accumulated solids when this test was run.

During the cold-water period, the influent and effluent concentrations of volatile acids were <1 mg/L. These data, along with the fact that very little off-gas was produced during this period, indicated a substantial decrease in anaerobic bioactivity in the ANFLOW column during the cold-weather period. However, the effluent quality remained good because removal of TSS and BOD continued by biophysical filtration.

The amount of sludge in the bioreactor was estimated independently, using a material balance model and the measured change in weight of the ANFLOW column. The total inventory of dry solids in the bioreactor is shown in Fig. 6.4. According to the model, ~ 850 kg of dry solids, or 170 kg of solids per 3800 m^3 of wastewater treated, accumulated in the column during the cold-weather period (days 483 to 588). Figure 6.4 also shows the solids accumulation estimated by the measured change in bioreactor weight. Note the excellent agreement between the model and the

experimental data over the entire operating period of ~ 750 d.

The discharge of solids was carried out several times late in the project. Simple draining of the bioreactor removes some solids, but not as much as would probably be desired. Techniques need to be developed to improve the solids discharge procedures. Solids can be dislodged from the packing very easily by a gentle stream of water from a hose. Approximately 5500 kg of dry solids was removed from the bioreactor when the pilot plant was decommissioned, which is in reasonable agreement with the ~ 6800 kg shown in Fig. 6.4.

Residence-time distribution tests were conducted before inoculation and after ~ 5000 kg of dry solids had accumulated in the bioreactor. An inert fluorescein dye was used. The flow patterns before inoculation were adequately described by three unequal-volume tanks in series, with volume ratios of 1:3:6. The latter flow patterns could be described by two equal-volume tanks in series. However, the mean residence time showed a substantial reduction; approximately one-sixth of the total reactor volume was used, while the other five-sixths was (apparently) occupied by solids and/or stagnant, dead-volume zones.

Type 316 stainless steel test coupons were evenly coated with seven types of paint and placed in the top of the ANFLOW column at the gas-liquid interface for exposure to both the liquid and gas phases. The Amercoat 395/395 sample showed no signs of paint peeling and no indication of reactivity with the corrosive atmosphere after ~ 800 d. The Sikagard 62-gray also demonstrated a high resistance to the corrosive atmosphere. Other paints showed significant deterioration. Overall, the gas phase was more reactive to the paints than was the liquid phase.

ANFLOW is an energy-conserving technology. Earlier studies have indicated that at the $3800\text{-m}^3/\text{d}$ scale, a treatment plant utilizing the ANFLOW process would need only 60% of the energy required for a plant utilizing activated sludge to treat weak wastewaters, and only 30% to treat strong wastewaters. When only the biotreatment step is considered, ANFLOW is projected to need $<10\%$ of the energy required by activated sludge. Methane recovery is not included in these comparisons; however, recovery of methane and conversion to electricity at 40% efficiency are projected to provide more than sufficient energy to operate the ANFLOW-based treatment plant.

Fig. 6.4. Accumulation of solids in the ANFLOW bioreactor.

Anaerobic Digestion of Low-Level Radioactive Cellulosic Wastes

Disposal of solid low-level radioactive waste is an increasing problem for ORNL and the nuclear industry in general. ORNL generates about 2300 m³ of low-level waste (LLW) each year, of which ~350 m³ is cellulosic and readily amenable to biological degradation. This waste is currently placed in trenches in the burial grounds, where it is subject to natural biological decomposition, which leads to instability and subsidence in the burial grounds, readily leachable organics, and an increased need for monitoring and maintenance. An alternative disposal technology is incineration; however, it suffers from substantial off-gas cleanup requirements and the poor economics of small incinerators to handle relatively small volumes of material.

Chem Tech has developed an anaerobic digestion process for volume reduction of low-level cellulosic and organic wastes. Anaerobic digestion reduces the volume of wastes by converting a substantial fraction of the solids to methane and carbon dioxide, producing a biologically stabilized sludge that is better suited for disposal than is the original waste. The anaerobic digestion process is similar to a residential septic tank and the stabilization of sludge as practiced in the municipal wastewater treatment industry. It is a simple, "low-tech" process that operates under mild, near-ambient conditions.

An initial feasibility study explored the rates and extent of microbial digestion of blotter paper, cloth, sanitary napkins, and pine sawdust in shake flasks, batch-stirred reactors, and fed-batch-stirred reactors. Based on these results, a preliminary process design and a cost estimate were developed. The conceptual process flowsheet is shown in Fig. 6.5. An ~95,000-L digester was specified for treatment of ORNL wastes. Further process development work was pursued with a 75-L digester to establish startup and operating conditions, to verify process performance, and to generate effluent for disposal studies.

The process development work with the 75-L digester has led to the following accomplishments, observations, and conclusions:

1. The solids degradation rate and the gas production rate have substantially exceeded the original design values used in the preliminary design of the ORNL facility. A reduction in equipment size or an increase in throughput capacity by a factor of ~2 is indicated. Furthermore, ~80% destruction of solids is projected, with an overall volume reduction of ~20:1 for uncompacted waste. This volume reduction translates to increased capacity and lifetime for current burial grounds.
2. Startup procedures have been developed and tested for the case of sewage sludge inocula. The time required for startup appears to be 1

Fig. 6.5. Process flowsheet for anaerobic digestion of low-level cellulosic waste.

to 2 months, which would be satisfactory from a practical standpoint.

3. The digester was operated for an extended period of time with varied feed composition and feed rate and almost total recycle of the digester effluent supernatant. The results showed that the digester was able to tolerate and recover from process upsets with little loss of efficiency.
4. A simple dynamic process model has been developed that satisfactorily simulates the experimental dynamic behavior under stable operating conditions, as well as some process upset conditions (see Fig. 6.6). The value of this model lies in its utility for process control and operational guidance for a full-scale digester. It is a significant contribution to anaerobic digestion technology.
5. The digester was operated for over 4 months using a recycle condition in which the liquid fraction of the effluent was recycled to slurry the feed, thereby reducing the quantity of liquid effluent for disposal. The solids digestion rates were not noticeably inhibited.
6. The digester was stressed by starvation (no cellulose feed for up to 2 weeks) and fed a high cellulose concentration (a factor of 5 over the normal daily feed amount) with no apparent ill effects. Another stress involved the lack of micronutrients (usually supplied once or twice each week with a feeding) and resulted in a slowing of the rates of gas production and volatile suspended solids (VSS) degradation. The digester recovered rapidly when the nutrients were reintroduced.

A General Plant Project (GPP) proposal was developed and approved for \$667,000 to construct an anaerobic digestion plant at the new volume-reduction facility. Implementation is currently on hold while an incinerator concept is being investigated at Mound Laboratory, Miamisburg, Ohio. Final procedures for effluent disposal have not yet been developed; however, experienced personnel indicate that no problems are anticipated based on preliminary tests that have been completed.

Biooxidation of Coal-Gasification Wastewaters

Technical feasibility has been demonstrated for the biooxidation of coal-gasification wastewaters

Fig. 6.6. Digester gas production rate during starvation and pulse feed test. Legend: + = gas rate data points; — = simulation of the digester operation.

using fluidized-bed bioreactors. Two laboratory-scale bioreactors have been constructed and have been successfully operated for >2 years to treat synthetic and actual wastewaters. Fixed-film, fluidized-bed bioreactors offer several advantages relative to conventional biooxidation technologies. Lower capital costs are projected since the high density of retained cells enables high organic removal rates at low hydraulic retention times and thus permits smaller reactors. The fixed films are expected to provide improved resistance to shock and toxins relative to suspended growth systems, as evidenced by work done at ORNL and elsewhere with similar fixed-film systems. Health and environmental effects associated with open-air systems can be minimized by the use of closed-reactor systems with off-gas control.

Raw condensate wastewaters were obtained from the Holston Army Ammunition Plant (HAAP) in Kingsport, Tennessee, and from the Morgantown Energy Technology Center (METC) in Morgantown, West Virginia, and were stored at 4°C. Prior to biotreatment, the METC wastewater was passed through a cartridge fiber filter to remove a significant quantity of oil (~2%) and was stripped to reduce ammonia and sulfides.

The laboratory-scale bioreactors are shown in Fig. 6.7. Recycle of solids or liquid was possible if desired. Total liquid holdup was ~10 L. An alternate configuration was used with 50%-strength wastewater in which the liquid effluent was taken from the RECYCLE location noted on Fig. 6.7.

Fig. 6.7. Schematic of fixed-film, fluidized-bed bioreactor.

This arrangement lowered the liquid to the overflow level in the angled side arm and reduced the liquid holdup to ~ 6 L.

The solid support particles were 30- to 60-mesh anthracite coal. Considerable earlier work at ORNL with other biological fluidized-bed applications indicated that anthracite coal is especially effective for retaining stable fixed films. Coal is less dense than sand (another common support), which allows fluidization at a lower liquid velocity and hence permits a smaller reactor for a given contact time. Pure oxygen gas was introduced at the bottom of the bioreactor through a metal frit at a flow rate $\sim 50\%$ of the liquid flow rate, which

was sufficient to maintain 1 to 2 mg per L of dissolved oxygen in the liquid phase in the bed.

Phenobac[®] (Polybac Corporation, Berlin, New Jersey) was selected as the inoculum. A mixture of BICHEM[®] 1001 and 1002 (Sybron Biochemical Corporation, Salem, Virginia) was also evaluated. Once fixed films are established on coal particles, samples of the cultures are readily preserved by lyophilization.

To minimize stress on the culture, actual wastewaters were initially diluted to $\sim 1\%$ with process water as they were fed continuously to the bioreactor. The strength of the wastewater was increased gradually over ~ 3 weeks by reducing the process

water diluent, and effluent recycle was initiated with higher-strength wastewaters to maintain 100 to 200 mg per L of phenols in the bioreactor.

Treatment of the HAAP and METC wastewaters was characterized primarily in terms of removal of phenolics. Reduction in COD was also monitored periodically. The reaction rate in the fluidized bed was expressed on a volumetric basis as the amount of phenolics degraded per unit time per volume of settled bed. The equation is:

$$\text{degradation rate} = (C_{in} - C_{out}) \times Q/V,$$

where C_{in} and C_{out} are the concentrations of phenolics entering and leaving the fluidized bed, respectively; Q is the volumetric flow rate of wastewater through the bed; and V is the settled-bed volume in the absence of gas and liquid flow. The expanded-bed volume during operation was typically 50% greater than the settled-bed volume.

The dilute HAAP wastewater was treated continuously for ~6 months, with no operating difficulties or bioreactor instability. No liquid recycle was used. The phenolics degradation rate was typically 5 to 15 mg per L of bed per min, depending on the condition of the culture. Growth of biomass caused bed expansion and necessitated the removal of a portion of the bed approximately twice per week, at which time clean coal was added. Hence the biomass quantity was variable and led to variations in the degradation rate. Thin films were more active on a volumetric basis; the volumetric degradation rate was high shortly after clean coal was added and low just before excess solids were removed.

Dilute METC wastewater (filtered, but unstripped) was treated in a similar manner. Effluent recycle was initiated in preparation for treatment of higher-strength wastewater. Stripped and filtered METC wastewater was successfully treated up to 50% strength for several weeks, with sufficient recycle to maintain the phenols concentration at <100 mg/L in the fluidized bed. On several occasions, 80 to 100% wastewater was fed to the bioreactor for short periods during mechanical malfunctions. However, the hydraulic residence time in the total reactor system was 15 to 20 h, which moderated the effects of the brief periods of higher-strength wastewater. Shedding of films occurred in these instances, but the bioreactor recovered quickly.

Reduction of COD was ~75 to 80%. Effluent COD concentrations were generally in the range of

1150 to 2500 mg/L and appeared to increase with feed strength. The theoretical COD for phenol is ~2 g of O_2 per g of phenol. Thus a comparison of the phenol and COD degradation rates indicates that about two-thirds of the COD reduction is attributable to phenol degradation, while one-third is attributable to biodegradation of other species.

The bioreactor was operated successfully for ~1 week with the phenolics concentration in the 500 to 900-mg/L range and 50%-strength stripped wastewater. The phenolics degradation rates were comparable to those obtained previously at much lower phenolics concentrations.

Batch kinetic experiments were performed to determine maximum degradation rates of monohydric phenol and wastewater phenols under more favorable, controlled conditions. High initial degradation rates of 35 to 40 mg per L of bed per min in all cases indicated that the acclimated culture was equally active on synthetic substrates and actual wastewaters. Preliminary batch treatability tests with METC wastewater and unacclimated cultures showed degradation rates of 20 to 25 mg per L of bed per min. However, the batch reaction rates are substantially greater than the rates in the continuous bioreactor. These results suggest that the mass transfer of phenols and/or oxygen is limiting the reaction rates in the fluidized-bed bioreactor. Increased reaction rates may be possible with improved bioreactor design and/or operating conditions.

An estimate of the sludge production rate can be obtained from the concentration of solids in the effluent and the accumulation of biomass in the bioreactor. Effluent TSS and VSS concentrations were determined over a 25-d period in which 50% METC wastewater was being treated. Overall sludge production during the study was 0.034 g of dry solids per g of phenol degraded, or ~0.34 g of wet sludge per g of phenol degraded. This value is typical of cell yields in suspended growth systems.

An economic comparison was made between a conventional activated-sludge process and a conceptual fluidized-bed process. The wastewater stream to be treated was 2.4×10^6 L/d, containing 1450 mg per L of phenols, which represents a 50% raw wastewater stream. Effluent from the process is to contain <2 mg/L. The conceptual fluidized-bed design was based on a phenols degradation rate of 20 mg per L of settled bed per min. This rate is believed to be a reasonable goal based on batch kinetic studies. The conceptual process flowsheet is shown in Fig. 6.8.

Fig. 6.8. Conceptual flowchart for fluidized-bed bioreactor process.

Results of the comparison indicated that the fluidized-bed process has only ~40% of the capital cost of the activated-sludge process and also ~40% of the operation and maintenance costs. However, both processes were described and costed at the conceptual level, and there are considerable uncertainties in performance and cost. These results do suggest that the fluidized-bed process may have a potential economic advantage if the technical performance of the process can be developed to achieve the (reasonable) design performance used in this analysis.

Oak Ridge Bioprocessing Research Facility

A user facility/resource for bioprocessing research has been established and approved. It is available for use by universities, national laboratories, and industrial scientists and engineers. It includes laboratories for the investigation of advanced bioprocessing concepts using stirred-tank or columnar bioreactors and a fermentation pilot plant for large-scale experiments.

Stirred-tank and advanced columnar bioreactors are available from bench scale to those with capacities greater than 500 L. The columnar bioreactors range from 2.5 to 40.6 cm in diameter and vary in height from ~30 cm to ~5 m. They can be operated as fixed-bed or fluidized-bed bioreactors and are equipped to accommodate a variety of process monitoring and control equipment.

The fermentation pilot plant has two 500-L instrumented stirred-tank fermenters and several 50-L fermenters that can be used for large-scale demonstrations, process scale-up studies, or production of kilogram quantities of microorganisms or biochemicals under controlled conditions. Continuous centrifuges and homogenizers and large chromatography equipment for product separation and recovery are also available.

R&D activities that can be pursued include (but are not limited to):

1. feedstock pretreatment and fractionation,
2. microbial culture selection and improvement,
3. genetic manipulation,
4. microbial and enzyme immobilization,
5. advanced bioreactor concepts,
6. process feasibility and scaleup,
7. advanced analytical concepts,
8. bioprocess monitoring and control, and
9. biochemical separations.

The principal investigator(s) may work independently or may collaborate with ORNL scientists and engineers. Approved users will receive guest appointments at ORNL for the period in which work will be performed onsite. This period may

range from 1 to 2 d to several months. Proprietary work may be carried out after contractual arrangements have been made.

6.4 HEALTH AND ENVIRONMENTAL MONITORING TECHNOLOGY

The purpose of this program is to develop instrumentation and/or measurement concepts and techniques for use as general-purpose monitors for a wide range of environmental applications. This program encompasses measuring toxic compounds in the environment, determining the degree of exposure of personnel to toxic materials, and developing the monitors for measuring specific chemical and biological components in aqueous processes.

A portable fluorometric monitor for detection of surface contamination by polycyclic aromatic hydrocarbons (PAHs) was developed earlier in this program. In addition to its utility in detecting and measuring fluorescent contamination on inanimate objects and surfaces, it is also useful in obtaining some measure of fluorescing compounds on living skin. Studies of its applicability in these areas, coupled with additional field testing of the instrument in coal-conversion pilot plants, completed the development of this "spill spotter." The results of these studies have been published in a comprehensive report.³

While many techniques have been developed to measure various compounds in the environment, few have been developed to measure a physiological response to these compounds. The primary biological receptors for PAHs and other toxic chemicals are the microsomal mixed-function oxidases (MFOs) that initiate biological detoxication mechanisms to rid living organisms of these contaminants. Chemicals are metabolized by the cytochrome P-450 MFO system to yield products that can bind to cellular macromolecules such as deoxyribonucleic acid (DNA), ribonucleic acid (RNA), or protein, resulting in mutagenicity and/or cell transformations and cancer. The carcinogenic potential of these reactive compounds can be either diminished or enhanced by the metabolic activity of the MFO system. Differences in the activities of these enzymes in individuals may be directly related to their exposure or their susceptibility to carcinogenesis by these compounds.

Our studies have been directed toward the development of a microfluorometric technique to monitor for the induction of a subset of the cytochrome P-450 MFO system. This subset is referred to as

cytochrome P-448 MFO. Our technique utilizes 7-ethoxyresorufin as a substrate that is converted by the cytochrome P-448 MFO to resorufin, a highly fluorescent product. This assay has been adapted to the centrifugal analyzer system with its computer system and ancillary automated rotor loading equipment. Liver samples from mice injected with benzo(a)pyrene (BaP) or 3-methylcholanthrene (3-MC) to induce MFO activity were utilized in developing the methodology and instrumentation. The measurements were then expanded to determine MFO activity in other organs, including the lungs, spleen, thymus, skin, and, finally, in mouse blood samples. This approach is designed to lead eventually to the measurement of MFO activity in human blood samples and to correlate this activity with exposure to toxic chemicals.

We previously reported⁴ improvements in the sensitivity of the MFO measurements obtained by incorporation of an argon-ion laser in the instrumentation. This increased sensitivity was utilized to measure the P-448 activity in liver microsomes obtained from mice that were treated topically with different coal liquids. These results have been summarized and published.^{5,6} We have continued to make improvements in instrument capability and extended the MFO measurements to a variety of biological samples, including mouse lung, spleen, thymus, skin, and blood; fish liver; and human blood.

Our data-handling capabilities were improved by the addition of an interactive cathode-ray tube (CRT) monitor. A new 5-W argon-ion laser has been installed to be used as a backup to our present laser, and it is also available for use in developing different optical configurations to improve our measurement capabilities. The new laser has the capability of producing a 200-mW output in the near-ultraviolet wavelength range, which extends our light-source range down to 350 to 360 nm. In addition, the argon-ion laser has been coupled as a

3. C. A. Burtis and J. E. Mrochek, *Development of a Portable Fluorescence Monitor to Detect Surface Contamination by Polycyclic Hydrocarbons*, ORNL/TM-8753, November 1983.

4. Chem. Technol. Div. Prog. Rep. Apr. 1, 1981, to Mar. 31, 1983, ORNL-5933, September 1983.

5. B. Z. Egan, N. E. Lee, C. A. Burtis, J. Y. Kao, and J. M. Holland, "Use of Laser-Excited Fluorescence to Measure Mixed-Function Oxidase Activity," *Clin. Chem.* 29, 1616 (1983).

6. C. A. Burtis, B. Z. Egan, and N. E. Lee, "Development of Laser-Excited Fluorometric Assays for the Monitoring of Toxic Chemical Exposure," *Pure Appl. Chem.* 57, 543 (1985).

pump for a tunable dye laser. This combination allows a large range of wavelengths to be selected in the visible-light range, depending on the specific dye used in the dye laser.

The precision of our MFO measurements was determined from successive measurements over a period of several days. Aliquots of a reference sample of mouse liver microsomes obtained from animals induced with 3-MC were analyzed for both MFO activity and total protein content. Cuvet-to-cuvet (within rotor) variation was estimated to be $\pm 2.3\%$. When normalized for the total protein content in the samples (specific activity basis), the value for the day-to-day variant was $\pm 7.0\%$.

The MFO activity in the lung, liver, spleen, and skin of mice was measured in animals induced by intraperitoneal injection of either BaP or 3-MC dissolved in corn oil. Control animals were injected with corn oil only. Times of induction ranged from 12 to 120 h. In some cases, composite blood samples were obtained by combining blood from several animals (0.5 to 0.8 mL per animal). In one experiment, a composite sample of thymus glands (~ 20 mg each) was also prepared. In a typical sample preparation procedure, the organs were washed, weighed, and homogenized in Tris-ethylenediaminetetraacetic acid (Tris-EDTA) buffer. The homogenates were then centrifuged at $10,000 \times g$, and the supernatant was recovered and referred to as the "S-9" fraction. A portion of the S-9 fraction was centrifuged at $105,000 \times g$ to recover a microsome pellet. The microsome pellets were resuspended in a Tris-EDTA-glycerine buffer and assayed for MFO activity. Several parameters, including sample concentration, substrate concentration, temperature, and pH, were varied in order to determine the best conditions for optimum enzyme activity.

The results of a study comparing 3-MC and BaP as inducers of MFO activity in mice are summarized in Table 6.6. The ratio of MFO activities for the different organs and for the induced:control are summarized in Table 6.7. A total of nine animals was used: three controls were injected with corn oil; three were injected with 4.0 mg of 3-MC in 0.6 mL of corn oil; and three were injected with 4.0 mg of BaP in 0.45 mL of corn oil. After 16 h, the livers, lungs, and spleens were removed, and S-9 and microsome fractions were prepared and assayed for MFO activity. The mean values for the three animals in each set are shown. Data for the

Table 6.6. Comparison of MFO activities induced in mice by 3-methylcholanthrene and by benzo(a)pyrene

Inducer	MFO activity ($\mu\text{mol}/\text{min} \cdot \text{g tissue}$)		
	Liver	Lung	Spleen
Control	1490 $\pm$ 150	18 $\pm$ 9	1.4 $\pm$ 0.1
3-MC	12700 $\pm$ 3400	159 $\pm$ 37	2.1 $\pm$ 0.4
BaP	6800 $\pm$ 2900	113 $\pm$ 29	1.8 $\pm$ 0.7

Table 6.7. Relative MFO activities in organs of induced animals, and relative induction compared to controls

Inducer	Liver	Lung	Induced/Control		
	Lung	Spleen	Liver	Lung	Spleen
3-MC	80	76	8.5	8.9	1.5
BaP	60	63	4.5	6.3	1.3

MFO activity in the livers and lungs were obtained from the microsome fractions. However, the data for the spleens were taken from the S-9 fractions since no MFO activity was measurable in the spleen microsome fractions. A rather large animal-to-animal variation was noted; however, the 3-MC induced higher MFO activity than the BaP in each case. As indicated in Table 6.7, the MFO activity in the liver was 60 to 80 times higher than in the lungs; the activity in the lungs was 60 to 80 times higher than in the spleen. The MFO activity in the liver and lungs of the induced animals was 5 to 10 times higher than in the controls. Only slight MFO induction occurred in the spleen. Similar conclusions were reached from data on animals induced with BaP for 48 h (Table 6.8). The MFO activity in the livers and lungs was higher by a factor of 20 in the induced animals than in the controls; the MFO activity induced in the spleen had

Table 6.8. Summary of MFO activities in mouse liver, lung, and spleen 48 h after induction with benzo(a)pyrene

Organ	Fraction	Activity ($\mu\text{mol}/\text{min} \cdot \text{g tissue}$)	
		Control	Induced
Liver	Microsomes	300 $\pm$ 120	6400 $\pm$ 2100
Lung	Microsomes	2.9 $\pm$ 1.1	55 $\pm$ 7
Spleen	S-9	1.4 $\pm$ 0.4	6.3 $\pm$ 0.8

increased by a factor of 4. The MFO activity was also measured 120 h after induction with BaP. As might be expected, the MFO activity was somewhat less after this length of time after exposure. The MFO activity in the liver microsomes of animals exposed for 120 h ranged from 290 to 990 U ($U = \mu\text{mol}$ of resorufin produced per min per g of tissue); the MFO activity in the lungs of these animals ranged from 17 to 66 U. In all cases, the order of activity was liver > lung > spleen.

Samples of skin and liver microsomes from three different types of mice were obtained from the ORNL Biology Division and analyzed for MFO activity and protein content. The MFO activity in the skin was about 2 to 3% of the MFO activity in the liver of the same animal. These values are comparable to values obtained using a different assay based on measurement of BaP metabolites.

Initial attempts to measure the MFO activity in lymphocytes from mouse blood samples were unsuccessful. Obtaining enough blood from animals exposed to toxic chemicals is a continuing problem. A typical yield is ~ 0.6 mL of whole blood per animal, which results in $\sim 1 \times 10^5$ lymphocyte cells. Consequently, blood from several animals is necessary to obtain enough cells for measurement. In the results described above, the mouse lymphoid tissue could be considered as a surrogate for lymphocytes. Induction of MFO activity did occur to some extent in lymphoid tissues, particularly the spleen, following intraperitoneal injection of either 3-MC or BaP. Although the MFO activity of the lymphoid tissue was very low compared to the lung and liver tissue, these results indicated that systemic administration of these compounds could produce P-448 activity in lymphoid tissues and cells. The difficulty in measuring the MFO activity in mouse lymphocytes may be due to the combination of the lack of cell mass (compared to the relatively large number of lymphoid cells in the spleen) and the low MFO activity.

While the volume of blood is not a problem with human samples, obtaining samples from humans deliberately exposed to toxic chemicals is a problem. In any case, it will be necessary to measure MFO activity in "normal" blood samples as a baseline. We have used human blood samples from three sources: (1) lymphocyte samples from The University of Pennsylvania; (2) commercially purchased whole blood and "buffy coat" preparations;

and (3) fresh, whole blood. Three samples were obtained from The University of Pennsylvania as lymphocyte pellets containing 7.8×10^6 , 11.7×10^6 , and 17.4×10^6 cells, respectively. No enzyme activity was found in samples 1 and 2, but the P-448 activity in sample 3 was determined to be 0.0037 nmol of resorufin produced per minute per 10^6 cells. This sample was identified later as the most likely to have MFO activity, having been obtained from a patient following extended drug treatment. Analyses of blood samples from the other sources are continuing.

Feasibility studies of immunological detection of P-448 MFO have been initiated in collaboration with personnel of the ORNL Health and Safety Research Division. Anti-P-450 antibody with defined specificity has been obtained from the Roche Institute of Molecular Biology through the kind collaboration of Dr. Paul Thomas. Using a standard immunological assay, we have found that this antibody can detect induced mouse microsomal P-448 antibody at titers of at least 1:41,000. Preliminary specificity studies have indicated that a sample of human lymphocytes that showed negative activity by the ethoxyresorufin deethylase fluorescence assay was also negative in the immunologic assay for P-448. In another human lymphocyte sample, the assay was positive at titers above and including 1:20. This result suggests that the immunological procedure may be considerably more sensitive than the 7-ethoxyresorufin deethylase assay. This sensitivity might be further increased by optimizing parameters and coupling the assay to a fluorescence measurement.

In collaboration with personnel of the ORNL Environmental Sciences Division, we have measured the cytochrome P-448 MFO activity in several fish liver samples. The MFO activity in the various samples ranged from 1 to 50 pmol per min per mg of protein, depending on the temperature, environment, and feeding habits of the fish. (For comparison, a normal mouse liver sample had a P-448 activity of about 55 pmol per min per mg of protein.) These results represent basal levels of activity—the fish were not deliberately exposed to a toxic environment. These measurements indicate that it may be possible to measure the MFO activity in fish samples and relate these measurements to the chemical and physical environment of the fish.

7. TRANSURANIUM-ELEMENT PROCESSING

The Transuranium Processing Plant (TRU) is the production, storage, and distribution center for materials used in the heavy-element research program in the United States. The transuranium isotopes produced at TRU continue to be used nationally and internationally in basic studies of the physics and chemistry of the transuranium elements, including experiments designed to produce new, super-heavy elements. Target rods irradiated in the High Flux Isotope Reactor (HFIR) are processed at TRU for the separation, recovery, and purification of the heavy actinide elements, which are distributed to laboratories throughout the country for their research work. Products are usually highly purified prior to shipment and are frequently provided in special chemical forms and/or in special devices required by the experimenter. In addition, TRU is the sole source of the ^{252}Cf that is distributed worldwide by the DOE's californium program at the Savannah River Laboratory (SRL). The californium is used to prepare neutron sources for a variety of applications in both industry and medicine.

7.1. TRU OPERATIONS

The purposes of this section are to report the production and distribution of transuranium materials and to describe recent changes in the processes and equipment being used in TRU.

Production and Shipment

During this report period, 26 HFIR targets were processed to recover the transplutonium elements. Table 7.1 presents data on the amounts of key isotopes obtained during this 2-year period, as well as the total amounts produced at TRU in 19 years of operation.

One batch of high-purity ^{248}Cm , containing a total of 139 mg of ^{248}Cm that had been recovered previously, was further purified and made ready for distribution.

A total of 63 shipments of transuranium elements was made during the report period. The total amounts of each type of product shipped are listed in Table 7.2.

Table 7.1. Amounts of materials recovered in the Transuranium Processing Plant

	April 1983 to March 1985	Total through March 1985
Number of years	2	19
Number of target campaigns	2	46
Number of targets processed	26	540 ^a
Amounts of key isotopes recovered:		
^{242}Pu	0	141 g
^{241}Pu	5.5 g	292 g
^{244}Cm	118 (228) ^b g	2241 g
^{249}Bk	72 mg	732 mg
^{252}Cf	809 mg	6846 mg
^{253}Es	3.3 mg	30 mg
^{257}Fm	2 pg	16 pg ^c

^aThe number of targets includes 343 HFIR targets, 176 SRP slugs and targets, and 21 SRP Pu-Al tubes.

^bThe value in parentheses is the total mass of all curium isotopes.

^cEstimated amount.

Table 7.2. Amounts of transuranium elements shipped during the period April 1, 1983 to March 31, 1985

Major isotope	Total amount	Number of shipments
²⁴⁰ Pu	37 g	5
²⁴¹ Am	1.1 g	2
²⁴³ Cm (57%)	1 μg	1
²⁴³ Cm (8-9%)	9 mg	2
Curium (mixed)	1.5 g	3
²⁴⁸ Cm (97%)	112 mg	6
²⁴⁷ Bk	45 mg	9
²⁴⁹ Cf	2 mg	1
²⁵² Cf	317 mg	23
²⁵³ Cf	3 fg	1
²⁵³ Es (mixed)	1280 μg	3
²⁵³ Es (milked)	208 μg	3
²⁵⁴ Es	6 μg	2
²⁵⁵ Fm	~2 ng	2

Processes and Equipment

Flowsheets used at TRU remain unchanged during this report period. The only significant equipment changes were in replacing the following items: an equipment rack in Cubicle 6, which is used to store miscellaneous process equipment and tools; the calorimeter in Cubicle 2, which is used to assay ²⁴⁴Cm; and the cell off-gas filters.

Target Fabrication

Twenty-three HFIR targets for transuranium-element production were fabricated from a mixture of curium and americium oxides. Feed materials for these targets included recycled americium-curium mixtures recovered from irradiated HFIR targets and ~79 g of curium that had originally been produced in the Savannah River Curium II Program for DOE's Space Nuclear Systems Program.

In addition, nine special targets were made in collaboration with ORNL's Operations Division; these targets included six containing cobalt, two containing ¹¹⁸Sn, and one with ⁶²Ni.

7.2 LARGE EINSTEINIUM ACTIVATION PROGRAM (LEAP)

Four national laboratories active in transplutonium research [Lawrence Berkeley Laboratory (LBL), Los Alamos National Laboratory (LANL), Lawrence Livermore National Laboratory (LLNL), and ORNL] have joined together to pro-

pose a major new thrust, the Large Einsteinium Activation Program (LEAP), which will require the production of about ten times as much ²⁵⁴Es as the 3 to 4 μg that is currently available. A sample of ~40 μg would open new opportunities in heavy-element research on the almost unknown chemical properties of the actinide elements 102 and 103 and transactinide elements 104 and 105 and on the nuclear properties of nuclides having atomic numbers at least as high as 109. It may even be possible to approach the calculated "island of stability" for superheavy elements.

The Large Einsteinium Activation Program is critically dependent on the capability of the HFIR/TRU complex to produce ~40 μg of ²⁵⁴Es. Production of this amount of material in the HFIR by normal irradiation of standard TRU/HFIR targets is impractical because of the very low yields of this highly fissionable nuclide. We have been studying the possibility of accomplishing this production program for several years.^{1,2}

A prerequisite for designing a suitable nuclide production method was new cross-section measurements on ²⁵³Es and ²⁵⁴Es in the thermal and epithermal energy region. The previously existing published values were grossly inconsistent with the observed concentrations of ²⁵⁴Es in HFIR-irradiated targets processed at TRU, so an experimental program to investigate cross-section measurements was developed in collaboration with ORNL's Chemistry Division. There is now cautious optimism that a large sample of ²⁵⁴Es can be produced, based on the new cross-section data resulting from measurements performed by the Chemistry Division using ²⁵³Es and ²⁵⁴Es samples prepared at TRU.³ The new data are listed below, where σ_{th} represents the 2200 m/s cross section, and I is the resonance integral.

Reaction	σ_{th} (barns)	I (barns)
²⁵³ Es (n,γ) ^{254m} Es	178 ± 15	3750 ± 200
²⁵³ Es (n,γ) ^{254g} Es	5.8 ± 0.7	114 ± 7
^{254g} Es (n,γ) ²⁵⁵ Es	28.3 ± 2.5	18.2 ± 1.5
^{254g} Es (n, fission)	2080 ± 250	1200 ± 250

1. Chem. Technol. Div. Annu. Prog. Rep. Apr. 1, 1979, to Mar. 31, 1981, ORNL-5757, November 1981, p. 121.

2. Chem. Technol. Div. Annu. Prog. Rep. Apr. 1, 1981, to Mar. 31, 1983, ORNL-5933, p. 100 (September 1983).

3. Chem. Div. Annu. Prog. Rep. for Period Ending Jan. 31, 1984, ORNL-6037, May 1984, pp. 83-91.

Calculations of ^{254}Es production rates using these cross-section values are in reasonable agreement with observed concentrations in HFIR-irradiated targets.

The cross sections indicate that reactions in the epithermal region are much more favorable to ^{254}Es production than the thermal reactions. The key to enhancing the production of ^{254}Es in the HFIR is the use of cadmium filters around ^{252}Cf targets to drastically diminish the thermal component of the neutron spectrum. However, such a configuration has never been tried in the HFIR.

The presence of cadmium in the HFIR target island introduces several interdependent technical considerations. The high thermal-neutron cross section of ^{113}Cd that makes it a good filter will also cause it to be consumed rapidly in the high thermal flux of the HFIR. The cadmium surrounding the HFIR targets will need to be thick enough to have a useful lifetime, but the total amount of cadmium in the target island will have to be kept small enough to permit the reactor core to achieve and maintain criticality for a useful fraction of the normal fuel lifetime. The cadmium will cause a severe thermal flux depression in the target island, and the core's power distribution will be shifted.

With the assistance of the Nuclear Engineering Applications Department of Martin Marietta's Computing and Telecommunications Division, one- and three-dimensional reactor calculations are being made to examine these effects. In order to make these calculations, it was necessary to update the HFIR cross-section library to include more recent values for many isotopes, add cross sections for the cadmium isotopes, and add cross sections that were not available several years ago for many of the transplutonium isotopes.

There is also the purely physical problem of how to place the cadmium sheaths on HFIR targets in the target island. The space occupied by a single target is not large enough to permit a cadmium sheath to be placed around a full-sized HFIR target. A concept was developed for a trefoil-shaped holder that would fit into the space normally occupied by three adjoining targets. Such a holder would have a minimum dimension large enough to hold an annular cadmium sheath surrounding one central target rod of standard diameter that could be fabricated in existing equipment at TRU. As a part of this project, the ORNL Engineering Technology Division designed and fabricated a proto-

type trefoil assembly and performed heat-transfer calculations to evaluate the configuration.

In a parallel activity, computer calculations have been made to optimize a production scheme, including the irradiation and chemical processing steps. A parametric study has also been made to evaluate the potential impact of uncertainties in the cross-section data.

A short-term, full-scale, irradiation test is planned to validate the calculation results which indicate that an ^{254}Es sample of suitable size can be produced in an irradiation-processing scheme using the cadmium thermal-neutron filters. The irradiation test will (1) confirm the thermal-hydraulic design of the cadmium sheaths and trefoil holders, (2) demonstrate the ability to replace the cadmium sheaths in the holders, (3) provide direct measurements of Δk_{eff} of the cadmium sheaths in HFIR, the perturbed fuel-cycle length, the burnout rate of the cadmium sheaths, and flux perturbations within the cadmium sheaths and at other locations in the target island, and (4) provide confirmation of the validity and accuracy of the overall ^{254}Es production model.

7.3 SPECIAL PROJECTS

The facilities available at TRU are used for a variety of purposes in addition to those associated with the mainline production and distribution of transuranium elements. These customized projects include nonroutine productions, unusual preparations, special irradiations in the HFIR, and fabrication of neutron sources from ^{252}Cf .

Fabrication of Neutron Sources from ^{252}Cf

Some of the californium recovered at TRU is incorporated into neutron sources that are loaned to researchers. Eight sources, containing from 2 μg to 14 mg of ^{252}Cf , were fabricated during this report period. Two of these sources were doubly encapsulated in type 304L stainless steel in a standard TRU configuration (NSD).⁴ One of these sources was also modified and enclosed in an aluminum can furnished by the customer. A single

4. J. E. Van Cleve, Jr., L. C. Williams, J. B. Knauer, and J. E. Bigelow, "Fabrication of ^{252}Cf Neutron Sources at Oak Ridge National Laboratory," p. 25 in *Applications of Californium-252, Proc. of Amer. Nucl. Soc. Topical Meeting*, CONF-720902, 1972.

source was prepared in a special point-source configuration for the National Bureau of Standards.⁵ Five special sources were made for the Naval Reactors Facility in Idaho Falls for a project to assay the fissile content of light-water breeder reactor fuel pins.

Sources Returned

Neutron sources are returned to TRU when the projects for which they were requested are completed or when replacements are ordered to make up for decay of the ^{252}Cf . These sources are available for reassignment until the appropriate time for reprocessing to recover the ^{248}Cm daughter. During the current report period, seven sources were returned and one was reassigned, 84 are now on loan, and 29 (containing from $<1\ \mu\text{g}$ to 6 mg of ^{252}Cf) are available for reassignment.

Electroplated ^{252}Cf Fission Sources

Some varieties of fission sources were prepared by electroplating small amounts of purified californium from dry isopropanol solutions onto platinum foils or disks. The total of eleven electrodeposited sources made during this report period contained from 0.2 to 16 μg of ^{252}Cf .

Preparation of ^{240}Pu

A batch of $\sim 13.5\ \text{g}$ of ^{240}Pu , which was initially recovered in TRU Campaign 63, was prepared for final distribution. This plutonium was first purified by a standard anion-exchange step and then converted to the oxide form by precipitation of plutonium oxalate and calcination of the precipitate. Table 7.3 lists the isotopic composition of this product. During Campaign 65, another batch of ^{240}Pu was recovered that will be prepared for

shipment later. Both batches of plutonium are the decay products of some aged ^{244}Cm that was used to augment the feed for HFIR targets.

Preparation of ^{255}Fm

Approximately 2.5 ng of high-purity ^{255}Fm was recovered from the Campaign 65 einsteinium product and shipped to Oak Ridge Associated Universities (ORAU) for use in medical experiments. The ^{255}Fm (20-h half-life) is the decay daughter of ^{255}Es (40-d half-life), which is present in small quantities in the einsteinium product from irradiated HFIR targets ($\sim 0.03\%$ of the total einsteinium). After the primary fermium product (which contains ^{257}Fm) was separated from the Campaign 65 einsteinium, the einsteinium was stored to permit the ^{255}Fm to grow in by radioactive decay. The ^{255}Fm was then recovered by high-pressure, ion-exchange techniques.

Table 7.3. Isotopic composition of plutonium from Campaign 63

Nuclide	Distribution (at. %)
^{238}Pu	0.008
^{239}Pu	0.005
^{240}Pu	99.463
^{241}Pu	0.003
^{242}Pu	0.497
^{244}Pu	0.024

5. L. C. Williams, J. E. Bigelow, and J. B. Knauer, Jr., "Equipment and Techniques for Remote Fabrication and Calibration of Physically Small, High-Intensity ^{252}Cf Neutron Sources," pp. 165-72 in *Proceedings of the 24th Conference on Remote Systems Technology*, Amer. Nucl. Soc., Inc., 1976.

8. NUCLEAR REGULATORY COMMISSION PROGRAMS

The U.S. Nuclear Regulatory Commission (NRC) programs in Chem Tech focus on two diverse tasks: nuclear reactor safety [primarily light-water reactors (LWRs)] and nuclear waste repository licensing. These two activities are discussed in the following sections.

8.1 NUCLEAR REACTOR SAFETY

Current NRC reactor safety programs focus on the mechanisms and pathways by which fission products are released to the environment from failed fuel elements under hypothetical reactor accident conditions. In the following paragraphs, eight separate projects are discussed, each having as its primary objective the elucidating of the chemistry underlying the important modes of fission product transport.

Fission Product Release from LWR Fuel

Accurate information about the quantities and chemical forms of fission products released from LWRs during overheating incidents is required for the reliable assessment of potential hazards. For several years, the NRC has sponsored experimental studies of these phenomena at ORNL.¹⁻³ In all these tests, small segments of commercial LWR fuel rods were heated under simulated accident conditions, and the released material was collected and analyzed. Complementary tests using fission product tracers and simulated fuel were conducted also. The primary objectives of these studies were: to determine the release rates and the chemical forms of the most significant fission products, to relate these results to changes in the fuel microstructure, and to compare these data with results

from other experimental work. The results will be used by the NRC in computer modeling studies of various accident scenarios to evaluate the resulting consequences.

In the recent Chem Tech HI test series, the fuel specimens, 15- to 20-cm (6- to 8-in.)-long segments of LWR rods of 10 to 40 MWd/kg burnup, were heated in an induction furnace at 1400 to 2000°C for 20 min.⁴ An atmosphere of steam and helium at 0.1 MPa (0 psig) pressure flowed across the heated specimens, oxidizing the Zircaloy cladding and transporting the released material to the collection system. The collection system is comprised of a thermal gradient tube lined with platinum, an aerosol sampler, a series of glass-fiber filters, heated charcoal, and cooled charcoal. Gamma spectrometry, neutron activation analysis, spark-source mass spectrometry, and energy-dispersive x-ray analysis were used to obtain data from the fuel specimen (before and after testing) and from

1. R. A. Lorenz, J. L. Collins, A. P. Malinauskas, O. L. Kirkland, and R. L. Towns, *Fission Product Release from Highly Irradiated LWR Fuel*, NUREG/CR-0722 (ORNL/NUREG/TM-287/R2), February 1980.

2. R. A. Lorenz, J. L. Collins, A. P. Malinauskas, M. F. Osborne, and R. L. Towns, *Fission Product Release from Highly Irradiated LWR Fuel Heated to 1300-1600°C in Steam*, NUREG/CR-1386 (ORNL/NUREG/TM-346), November 1980.

3. R. A. Lorenz, J. L. Collins, M. F. Osborne, R. L. Towns, and A. P. Malinauskas, *Fission Product Release from BWR Fuel Under LOCA Conditions*, NUREG/CR-1773 (ORNL/NUREG/TM-388), July 1981.

4. M. F. Osborne, J. L. Collins, R. A. Lorenz, J. R. Travis, and C. S. Webster, *Design, Construction, and Testing of a 2000°C Furnace and Fission Product Collection System*, NUREG/CR-3715 (ORNL/TM-9135), September 1984.

all apparatus components after the test. The results of six tests have been reported in detail.⁵⁻¹⁰

The fractional releases of Kr, I, and Cs were similar in all tests and varied between ~2% at 1400°C and ~60% at 2000°C,¹¹ as shown in Table 8.1. Measurable amounts of fission products Br, Rb, Mo, Ru, Ag, Cd, Sb, Te, Ce, and Eu were released in some of the tests, but the fractional releases of these elements were generally much lower and more variable. The results correlated reasonably well with physical and chemical changes in the specimens.¹² In addition to the fission products, several structural and impurity elements (Zr, Sn, Mg, Ca, S, and Cl) were released from the furnace and were collected.

The releases of Kr, I, and Cs have been compared, on the basis of a fractional release rate model, with the results of an NRC-sponsored review.¹³ This comparison (Fig. 8.1) assumes an inverse temperature dependence for the fractional release rate, rather than the exponential temperature dependence used in the NUREG study¹³ (and also reported by Albrecht and Wild).¹⁴ Despite considerable scatter, the data appear to fit the Arrhenius form, and in four of six tests, our data fall significantly below the NUREG curve. These data are insufficient, however, to define the effect of other apparently important parameters such as time at temperature, fuel characteristics, and oxidizing conditions during the test.

Deposition and reaction of the various species in the thermal gradient tube and on the filters provided valuable information about the chemical forms. Our results indicate that cesium was present in several forms: as a zirconate on ZrO₂ furnace ceramics and as CsI mixed with other cesium com-

5. M. F. Osborne, R. A. Lorenz, J. R. Travis, and C. S. Webster, *Data Summary Report for Fission Product Release Test HI-1*, NUREG/CR-2928 (ORNL/TM-8500), December 1982.

6. M. F. Osborne, R. A. Lorenz, J. R. Travis, C. S. Webster, and K. S. Norwood, *Data Summary Report for Fission Product Release Test HI-2*, NUREG/CR-3171 (ORNL/TM-8667), February 1984.

7. M. F. Osborne, R. A. Lorenz, K. S. Norwood, J. R. Travis, and C. S. Webster, *Data Summary Report for Fission Product Release Test HI-3*, NUREG/CR-3335 (ORNL/TM-8793), April 1984.

8. M. F. Osborne, J. L. Collins, R. A. Lorenz, K. S. Norwood, J. R. Travis, and C. S. Webster, *Data Summary Report for Fission Product Release Test HI-4*, NUREG/CR-3600 (ORNL/TM-9011), June 1984.

9. M. F. Osborne, J. L. Collins, R. A. Lorenz, K. S. Norwood, J. R. Travis, and C. S. Webster, *Data Summary Report for Fission Product Release Test HI-5*, NUREG/CR-4037 (ORNL/TM-9437), May 1985.

10. M. F. Osborne, J. L. Collins, R. A. Lorenz, K. S. Norwood, J. R. Travis, and C. S. Webster, *Data Summary Report for Fission Product Release Test HI-6*, NUREG/CR-4043 (ORNL/TM-9443), in preparation.

11. M. F. Osborne, J. L. Collins, R. A. Lorenz, K. S. Norwood, and R. V. Strain, "Measurement and Characterization of Fission Products Released from LWR Fuel," *Proceedings of the Fifth International Meeting on Thermal Nuclear Reactor Safety*, Sept. 9-13, 1984, Karlsruhe, Federal Republic of Germany, 1985.

12. R. V. Strain, J. E. Sanecki, and M. F. Osborne, "Fission Product Release from Irradiated LWR Fuel Under Accident Conditions," *Proceedings of the ANS Topical Meeting on Fission Product Behavior and Source Term Research*, July 15-19, 1984, Snowbird, Utah.

13. *Technical Bases for Estimating Fission Product Behavior During LWR Accidents*, NUREG-0772, U.S. Nuclear Regulatory Commission, June 1981.

14. H. Albrecht and H. Wild, "Investigation of Fission Product Release by Annealing and Melting of LWR Fuel Pins in Air and Steam," *Proceedings of the ANS Topical Meeting on Reactor Safety Aspects of Fuel Behavior*, Sun Valley, Idaho, Aug. 2-6, 1981.

Table 8.1. Data from fission product release tests

Test No.	Average test temperature (°C)	Time at test temperature (min)	Specimen burnup (MWd/kg U)	Steam flow rate (g/min)	Inventory ^a released (%)		
					⁸⁵ Kr ^b	¹²⁹ I	¹³⁷ Cs
HI-1	1375	30	28	0.81	3.13	2.04	1.75
HI-2	1700	20	28	0.76	51.8	53.0	50.5
HI-5	1725	20	38	0.30	19.9	22.4	20.3
HI-4	1900	20	10	0.29	31.3	24.7	31.7
HI-6	1950	~1	40	1.70 ^c	31.6	24.7	33.1
HI-3	1975	20	25	0.31	59.3	35.4	58.8

^aBased on operating data, radiochemical analyses, and ORIGEN calculations.

^bIncludes Kr released during irradiation.

^cAverage value; initial rate was 2.4 g/min, but decreased to ~0.24 g/min during test.

Fig. 8.1. Inverse temperature dependence of HI test data at ORNL compared with data reported in *Technical Bases for Estimating Fission Product Behavior During LWR Accidents*, NUREG-0772, U.S. Nuclear Regulatory Commission, June 1981.

pounds on the thermal gradient tube and filters.^{15,16} Iodine was collected primarily as CsI. Antimony and silver appeared to be released as the elements, but behaved in different ways. Antimony combined with the platinum tube, probably forming PtSb₂, and silver seemed to occur as surface deposits on the platinum tube and/or the aerosols.¹⁵ Tellurium was retained by the Zircaloy cladding until the cladding became completely oxidized, then it was released, probably as a telluride.¹⁶

Core-Melt Studies

An experimental investigation of core-melt fission product release and aerosol formation has been conducted for the NRC at ORNL since the Three Mile Island reactor accident in 1979.

The objective of the core-melt release experiments is to address the phenomena associated with LWR Class IX accidents, particularly the postulated large-scale vaporization of fission products, core components, and structural materials. To attain the desired high temperatures and melting rates, radio frequency (rf) induction melting of Zircaloy-clad fuel pins in ceramic crucibles of ThO₂ or ZrO₂ is applied. A uniquely designed furnace permitting rf coupling to the fuel cladding has been chosen as the basis of the experimental system.

Over the period from 1980 to 1983, many core-melt experiments were conducted on a 1-kg (2.2-lb) scale,¹⁷ and the release fractions of almost all fission-product and structural-type material aerosols were measured and reported.¹⁸ Equipment for even larger core-melt demonstrations [10 to 12 kg (22 to 26 lb)] has been completed, offering a unique opportunity to observe physical changes during the course of a meltdown through interrupted steps from initial control rod failure (~1400°C) to complete eutectic oxide melting (2400°C), as shown in Fig. 8.2.

The most significant observations from the ORNL core-melt work thus far are these:

15. K. S. Norwood, J. L. Collins, M. F. Osborne, R. A. Lorenz, and R. P. Wichner, "Characterization and Chemistry of Fission Products Released from LWR Fuel Under Accident Conditions," *Proceedings of the ANS Topical Meeting on Fission Product Behavior and Source Term Research*, Snowbird, Utah, July 15-19, 1984.

16. J. L. Collins, M. F. Osborne, R. A. Lorenz, K. S. Norwood, J. R. Travis, and C. S. Webster, *Observed Behavior of Cesium, Iodine, and Tellurium in the ORNL Fission Product Release Program*, NUREG/CR-3930 (ORNL/TM-9316), February 1985.

17. Chem. Technol. Div. Prog. Rep. Apr. 1, 1981, to Mar. 31, 1983, ORNL-5933, September 1983.

18. G. W. Parker, G. E. Creek, and A. L. Sutton, Jr., "Influence of Variable Physical Process Assumptions on Core-Melt Aerosol Release," pp. 1078-89 in *Proceedings of the International Meeting on Thermal Nuclear Reactor Safety*, Chicago, Aug. 29-Sept. 2, 1982, Vol. 2, NUREG/CP-0027, U.S. Nuclear Regulatory Commission, February 1983.

ORNL PHOTO 4748-86

Fig. 8.2. Photographs showing (a) small bundle core-melt assemblies and components and (b) typical glassy oxide-phase, core-melt residues.

1. Control rod silver-cadmium alloy is observed to rupture the stainless steel cladding at $\sim 1400^\circ\text{C}$, leaving the silver to wet the fuel cladding and form a low-melting zirconium alloy that flows downward. Relatively little silver escapes as a vapor or aerosol.
2. Stainless steel also attaches Zircaloy at about this same temperature and exposes large areas of fuel surface to the steam and hydrogen.
3. The releases of many materials and fission products are lower than those of the long-standing German tests designated SASCHA: [e.g., Ag, Sn (cladding), Fe, U, and Te].
4. The releases of Ba and Sr are higher than in the SASCHA tests; however, this may be an artifact of the low-density fuel simulant used. This point is to be resolved by using high-density fuel simulants in future experiments.
5. The release of europium ($\sim 1\%$) is greater than the release of the other rare earths. Samarium should be similar to europium.

6. The expected form of cesium (Cs_2UO_4) decomposes completely, releasing metallic cesium beginning at 1500°C .
7. The release of boric oxide from B_4C and borosilicate glass is lower than expected ($<1\%$), probably because B_2O_3 reacts readily with both UO_2 and ZrO_2 at high temperatures.
8. The principal aerosol components (by mass) are $\text{Cd} > \text{Mn} > \text{fission products} > \text{Ag} > (\text{In} + \text{Cr} + \text{Ni} + \text{Fe} + \text{Sn})$.
9. Molten stainless steel concentrates the elements Te, Ru, Sn, and probably all of the noble metals, forming a separate phase below the oxide phase.
10. Residual, unoxidized Zircaloy is found uniformly dissolved in the $\text{Zr-ZrO}_2\text{-UO}_2$ oxide phase.
11. A comparison of this work with the release estimates given in the U.S. Reactor Safety Study¹⁹ shows similar values, except for tellurium and the noble metals (Table 8.2).²⁰

Table 8.2. A comparison of fission product release values^a

Fission product	Percent release expected	
	From the Reactor Safety Study ^b	From ORNL work ^c
Xe, Kr	90	>90
I, Br	90	>90
Cs, Rb	80	>90
Sr, Ba	10	$<10^d$
Te	5-25	<1.0
Ru, Rh, Pd, Mo, Tc	3	0.1
Rare earth elements	0.1-1.0	0.1-1.0

^aRelease from fuel before reactor pressure vessel (RPV) failure, nonspecific sequence.

^bData obtained from Appendix VII, Reactor Safety Study, WASH-1400, NUREG-75/014, October 1975.

^cData obtained from G. W. Parker, G. E. Creek, A. L. Sutton, Jr., *Experimental Study of Core-Melt Aerosol Release at the 1-kg Scale*, NUREG/CR-3997, in press.

^dBeing evaluated.

19. Reactor Safety Study, *An Assessment of Accident Risks in U.S. Commercial Power Plants*, WASH-1400, Appendix VII, NUREG-75/014, U.S. Nuclear Regulatory Commission, 1975.

20. G. W. Parker, G. E. Creek, and A. L. Sutton, Jr., *Experimental Study of Core-Melt Aerosol Release at the 1-kg Scale*, NUREG/CR-3997, in press.

TRAP-MELT Validation

Recent calculational results published as part of the Battelle-Columbus Source Term Study²¹ indicate that, for some LWR severe accident sequences, aerosol deposition in the reactor primary coolant system (PCS) can lead to significant reductions in the radionuclide release to the environment. Aerosol transport and deposition in the PCS for the Battelle study was calculated using the TRAP-MELT 2 computer code, which was developed at Battelle-Columbus Laboratories. The objective of the ORNL TRAP-MELT Validation Project, which is sponsored by the Fuel Systems Behavior Branch of the NRC, is to conduct simulated reactor-vessel upper-plenum aerosol deposition and transport tests. The results from these tests will be used in the ongoing effort to validate TRAP-MELT 2. The project includes two experimental subtasks: the Aerosol Transport Tests and the Aerosol Resuspension Tests.

In the Aerosol Transport Tests, aerosol transport in a vertical pipe has been studied; this geometry was chosen to simulate aerosol deposition and transport in the reactor-vessel upper plenum. Four tests have been completed to date in the configuration illustrated in Fig. 8.3. Aerosols were generated by feeding metal powder to a plasma torch aerosol generator; in the tests either iron oxide aerosols were made by oxidizing iron metal powder, or metallic zinc aerosols were made by direct vaporization of zinc metal powder. The generated aerosols can agglomerate, settle on the horizontal floor at the bottom of the pipe, deposit on the pipe sidewalls, or be transported out of the pipe into an aerosol collection bag. Results from these tests were summarized in a series of letter reports to the NRC²²⁻²⁵ and in a recently published paper.²⁶

For the last two tests performed, A103 and A104, the data were compared with TRAP-MELT 2 calculations. Results from the comparisons have not been satisfactory in that the code significantly underpredicted the measured aerosol deposition. The test section illustrated in Fig. 8.3 is now being modified to allow additional measurements to be made; these changes should aid in understanding differences with code results in future tests.

The objective of the Aerosol Resuspension Tests is to provide a data base for developing resuspension-rate models that can be included in TRAP-MELT 2 and other aerosol transport codes;

ORNL DWG 83-884R2

Fig. 8.3. Schematic of test section used for Aerosol Transport Tests A101 through A104.

this is being done because none of the present aerosol transport codes include resuspension models. A series of tests was performed in which various powders and aerosols were deposited by settling on a collection surface mounted in a 0.076- μ m-diam Pyrex pipe. After depositing the materials,

21. J. A. Gieseke et al., *Radionuclide Release under LWR Accident Conditions—Volumes I-VII* (Draft), BMI-2104, Battelle-Columbus Laboratories, 1983-1984.

22. A. L. Wright and W. L. Pattison, *Aerosol Transport Test A101 'Quick-Look' Data Report*, Letter Report to the U.S. Nuclear Regulatory Commission, September 1983.

23. A. L. Wright and W. L. Pattison, *Aerosol Transport Test A102 Preliminary Data Report*, Letter Report to the U.S. Nuclear Regulatory Commission, February 1984.

24. A. L. Wright and W. L. Pattison, *Aerosol Transport Test A103 Preliminary Data Report*, Letter Report to the U.S. Nuclear Regulatory Commission, May 1984.

25. A. L. Wright and W. L. Pattison, *Aerosol Transport Test A104 Preliminary Data Report*, Letter Report to the U.S. Nuclear Regulatory Commission, July 1984.

26. A. L. Wright and W. L. Pattison, "Results from Simulated Upper-Plenum Aerosol Transport and Aerosol Resuspension Experiments," *Proceedings CSNI Specialist Meeting on Nuclear Aerosols in Reactor Safety*, eds. W. O. Schikarski and W. Schock, KfK 3800, CSNI 95, February 1985.

resuspension rates were measured for various flow rates through the pipe. An overall summary of the data from these tests is contained in an NRC letter report;²⁷ a portion of the results is presented in Fig. 8.4. The data shown illustrate a possible influence of deposit-material density on resuspension rate. These results, data from future tests, and information from other investigators will be used to develop appropriate resuspension-rate models for aerosol codes.

Fission Product Interaction with Aerosols

According to current accident evaluations, a principal mode of fission-product movement during a reactor core meltdown occurs by aerosol transport. Calculations made during this project²⁸ indicate that the sorption of fission product vapors

onto aerosols under noncondensing conditions can significantly alter the calculated distribution of the fission products and the calculated quantity released to the environment. The potentially large surface areas of aerosols make them effective competitors for vapors, even in noncondensing conditions. The purpose of this project was to measure the capacity of sorption and the rate of sorption of fission product vapors onto aerosols. In the past year, the semivolatiles, CsOH and CsI, were studied. Chemically pure CsOH or CsI tagged with a small quantity of $^{137}\text{CsCl}$ were studied rather than actual fission products.

A laboratory apparatus was fabricated capable of exposing deposited samples of aerosol or powder to controlled vapor pressures of CsOH or CsI. Initially, all tests were conducted with a carrier gas of He or He-0.5% H_2 . Later, the ability to control the oxygen potential by varying the $\text{H}_2/\text{H}_2\text{O}$ was added.

The potential of different types and levels of interaction based on the chemical composition of the aerosol was recognized from the beginning. Thus testing all potential aerosol materials for interaction is desirable. Although this ideal was beyond the scope of this project, seven aerosol materials most important from the standpoint of quantity generated in a reactor accident, ability to form aerosols at the temperatures (700 to 100°C) proposed, and suitability for our test apparatus were studied. These seven aerosol materials were silver, chromium oxide, manganese oxide, iron oxide, iron, tin oxide, and nickel. A simple set of tests demonstrated that iron and iron oxide reacted in a bulk manner with CsOH vapor and that chromium oxide reacted with CsI vapor. Silver gave ambiguous results, but did seem limited to surface interactions, while the remaining interactions were clearly surface interactions.

Further testing and thermodynamic calculations indicated that although Cs_2CrO_4 can be formed by reaction with either CsI or CsOH, this reaction is quite unlikely at the oxygen potentials expected in a reactor accident. Nevertheless, chromium oxide

Fig. 8.4. Measured resuspension rates, as a function of flow velocity, for tests where powders or aerosols were deposited by settling in aerosol resuspension tests. Tests were done with 0.5- μm -diam tungsten powders, 2.5- μm -diam nickel powders, iron oxide aerosols (mean diam, $<0.2 \mu\text{m}$), and tin oxide aerosols (mean diam, $<0.2 \mu\text{m}$).

27. A. L. Wright and W. L. Pattison, *Series-1 Aerosol Resuspension Test Preliminary Data Report*, Letter Report to the U.S. Nuclear Regulatory Commission, August 1984.

28. R. P. Wichner and R. D. Spence, *Quantity and Nature of LWR Aerosols Produced in the Pressure Vessel During Core Heatup Accidents—A Chemical Equilibrium Estimate*, NUREG/CR-3181 (ORNL/TM-8683), March 1984.

is a significant sorber of CsOH and CsI and can be a significant fraction of refractory aerosols at elevated temperatures. In contrast, both iron and iron oxide proved to reversibly react with CsOH at the conditions expected in a reactor vessel. Thus even a small fraction of iron in the aerosol cloud can be a significant carrier of ^{137}Cs .

The sorption isotherms measured for the surface interactions indicated a complex dependence for the quantity sorbed on the vapor pressure and temperature. First exposure to the vapor led to a large initial sorption, and subsequent increases in vapor concentration led to increased sorption, though not proportional to the initial large sorption. Sorption isotherms were measured for CsOH- Cr_2O_3 at 700 and 900°C in He-0.5% H_2 ; CsOH- Fe_2O_3 at 700 and 900°C in He-0.5% H_2 ; CsI- Cr_2O_3 at 800, 900, and 1000°C for $\text{H}_2/\text{H}_2\text{O} = 0.1$; and CsOH-Ag at 600, 700, and 800°C for $\text{H}_2/\text{H}_2\text{O} = 0.1$.

Results, thus far, indicate that iron in the aerosol cloud can capture a significant fraction of the ^{137}Cs in a reactor core and that ^{137}Cs thus captured can be released if high temperatures are maintained and the CsOH vapor is depleted. Even without such chemical sinks, the large surface area of the aerosol cloud can capture a surprising amount of vapor in noncondensing conditions.

Postaccident Iodine Chemistry

The purpose of this program is to provide experimental information on the chemistry of aqueous iodine species for the development of a reliable fission product release model. Iodine partition coefficients for inorganic iodine have been measured during hydrolysis and radiolysis. The hydrolysis experiments have illustrated the importance of reaction time on iodine volatility.²⁹ However, radiolysis effects can override hydrolysis in determining iodine volatility.^{30,31} In addition, silver metal in radiolysis samples can react to form silver iodide accompanied by a decrease in iodine volatility. We are now in the process of coupling experimental data to an iodine transport and release model that was developed in the Federal Republic of Germany.

The tests were run in U-shaped quartz tubes, with the temperature maintained by a water bath. The iodine volatility was measured by counting tracer ^{131}I in both the gas phase and in the liquid phase. Alternatively, iodine volatility was also measured by placing an iodine-absorbing solution,

such as sodium thiosulfate or sodium hydroxide, adjacent to the sample in the space normally occupied by the gas phase. Additional tests were run on samples containing iodine and metallic silver. These tests were run using silver powder (<100 mesh) that was reduced in an atmosphere of 96% argon-4% hydrogen at 623 K. The iodine partition coefficient (IPC) as used in these studies is defined as:

$$\text{IPC} = \frac{\text{concentration of iodine in aqueous phase}}{\text{concentration of iodine in gas phase}}$$

To enable the use of the experimental results in a description of iodine behavior in containment, the data have been converted into equations that can be incorporated into models. The best approach to the problem of fitting empirical data in a process as complex as iodine hydrolysis lies in the use of regression techniques and multivariate analysis. As a preliminary to multivariate analysis, data from some tests in pure water and tests at pH 7.0 (buffered) have been fit to an equation of the type:

$$\ln \text{IPC} = a + bt \quad (t \text{ in seconds})$$

Table 8.3 provides some experimental test data that have been fitted to this equation.

A series of radiation tests was run on samples that contained $1 \times 10^{-4} \text{ M I}^-$ with $1 \times 10^{-6} \text{ M I}_2$ at pH 6.9 (borate buffered) at 323 K. The conditions were chosen to simulate approximate amounts of I^- and I_2 that may exist in sump water following an LWR accident. Irradiation to 1 Mrad produced a decrease in the iodine partition coefficient from 3700 to 490. When silver powder was added to the sample in a ratio of 40 g-at. of silver per 1 g-at. of iodine, there was no decrease in the iodine partition coefficient with irradiation.

29. L. M. Toth, K. D. Pannell, and O. L. Kirkland, *The Chemical Behavior of Iodine in Aqueous Solutions Up to 150°C. I. An Experimental Study of Nonredox Conditions*, NUREG/CR-3514 (ORNL/TM-8664), April 1984.

30. L. M. Toth and K. E. Dodson, *The Chemical Behavior of Iodine in Aqueous Solutions Up to 150°C. II. Radiation-Redox Conditions*, NUREG/CR-3514, Vol. 2 (ORNL/TM-8664/V2), March 1985.

31. E. C. Beahm and W. E. Shockley, "Iodine Volatility Under LWR Accident Conditions," *Trans. Am. Nucl. Soc.* **45**, 482-83 (1983).

Table 8.3. Partition coefficient parameters determined from test data^a

Test condition	a	b	Coefficient of determination
pH 7 (buffered)			
1×10^{-5} g-at. I_2/L at 298 K	6.46	3.25×10^{-6}	0.978
1.2×10^{-5} g-at. I_2/L at 298 K	5.89	4.35×10^{-6}	0.996
4.7×10^{-5} g-at. I_2/L at 298 K	5.62	1.5×10^{-6}	0.923

^aData fit to an equation of the type $\ln IPC = a + b + bt$ (t in seconds).

Iodine Release from a Steam Generator Tube Rupture

A tube failure within the steam generator of a pressurized-water reactor (PWR) results in the escape of primary coolant into the secondary system. The release of primary coolant from the steam generator tube rupture is a design-basis accident, and for licensing purposes conservative assumptions are made to estimate the release of radioactivity. A better understanding of iodine behavior would help to establish the margin of conservatism. The objective of this task is to conduct laboratory experiments designed to answer basic questions regarding the release of radioactive iodine. The experimental system will determine the vapor-liquid partitioning of iodine within a simulated steam-generator environment as a function of coolant pH, iodine concentration, and chemical form.

Depending on the type of accident being analyzed, radioactive iodine escaping into the secondary system can be released directly through the safety relief valve into the building atmosphere, or be released in greatly reduced amounts through the condenser air ejector. In order to predict mechanisms associated with iodine transport from the steam generator, an experimental system (see Fig. 8.5) was designed to obtain fundamental data using ^{131}I as a radioactive tracer. Basically, the system consists of a 8.9-cm (3.5-in.)-diam, 1.5-m (60-in.)-long pressure vessel heated by a furnace with three temperature control zones to simulate the PWR steam generator and a water-cooled condenser. The connecting flow path contains an orifice and an air-operated valve to permit sampling of the vapor phase. The simulated steam generator will operate at 285 to 300°C [7.0 to 8.4 MPa (1000 to 1200 psia) saturated steam] containing

Fig. 8.5. Experimental system for iodine transport from a simulated steam generator.

dilute aqueous solutions of boric acid, lithium hydroxide (pH control), and iodine. A radioactive tracer is necessary because of the extremely low iodine concentrations (10^{-10} M) existing in PWR primary coolants. Installation of the equipment components has been completed, and the system is presently undergoing preliminary testing.

HTGR Safety Studies

The primary objective of this program is to estimate the time-dependent release of fission products to the environment as a result of postulated severe

ORNL DWG 85-725

accidents for high-temperature gas-cooled reactors (HTGRs). The accomplishment of this objective involves (1) the development of an independent computer code for predicting the consequences of such accidents, and (2) the experimental determination of transport information required by the computer code for the various fission product categories. The emphasis has been on the prismatic fuel element with application to the 240- and the 1170-MW reactors and to Fort St. Vrain. However, a shift to the modular HTGR appears likely.

Basically, the computer code will describe core and PCRV thermal hydraulics, interactions between thermal hydraulics and fission product transport, fuel failure and release from fuel, fission product transport through the graphite core, and physical and chemical interactions of fission products with prestressed concrete reactor vessel (PCRV) components. Major progress has been made in establishing fission product redistribution capability in the ORECA code, which describes thermal hydraulics. Preliminary estimates of initial nuclide inventories were developed using the ORIGEN-S code. The prediction of fission product release from fuel particles according to the Goodin algorithm was incorporated into ORECA. A model of fission product transport in structural graphite was developed. The model accounts for the processes of diffusion, adsorption, and desorption.

The experimental work has concentrated on determining diffusion coefficients in graphite and vapor pressures of fission products data needed by the computer code to describe fission product transport within the core. A graphite resistance

furnace was used for measurements up to 2500°C. The experimental procedure involved loading a mixture of powdered graphite and simulated fission products into a 6.4-mm (0.25-in.)-OD by 3.2-mm (0.126-in.)-ID graphite tube and determining the rate of loss of fission product after exposing the sample to a high temperature over a known time period. A simplified sketch of the experimental setup is shown in Fig. 8.6. By a comparison with the results using a species of known behavior, a vapor pressure and diffusion coefficient can be calculated. Figure 8.7 shows data for cerium plotted according to a Knudsen diffusion model.

In calibration tests with silver, the heat of vaporization of silver based on a gas-phase Knudsen diffusion model was within 3% of literature values. The Knudsen diffusion model was further supported by the absence of effects of pressure and type of carrier gas and by the pore-size distribution of the graphite tube. Experiments were performed with elements from the noble metal group—Pd, Rh, and Ru—and from the rare-earth metal group—Y, La, Pr, Ce, and Sm. With silver as a reference material, gas-phase diffusion coefficients and vapor pressures were calculated.

Differences between experimental and literature vapor pressure values were observed. In the rare-earth metal tests, in which the diffusion samples were prepared from nitrate compounds, thermodynamic calculations of metal oxide vapor pressures were consistent with experimental results. This observation supports previous calculations of the formation of metal oxide gases from CO in an HTGR severe accident. The presence of significant

Fig. 8.6. Sketch of experimental setup for diffusion coefficient and vapor pressure measurement.

Fig. 8.7. Experimental results for cerium plotted according to a Knudsen diffusion model (W in g/s; T in K).

solid-phase diffusion in addition to gas-phase diffusion may explain other differences that were observed.

A final report on the experiments involving fission product diffusion through graphite was written. Plans are to perform an experimental study of fission product interaction with reactor vessel materials on construction.

Severe Accident Sequence Analysis. The Severe Accident Sequence Analysis (SASA) program involves tracing the movement of fission products through an LWR system during a postulated accident sequence. Calculations have been particularized to a specific reactor (the Browns Ferry Unit One, which is a Mark-I type of boiling water reactor) and to specifically defined accident sequences. Fission product transport analyses have been completed for three accident-initiating events: complete loss of station power,³² break in the control-rod drive system outside the containment,³³ and failure of the decay-heat removal system following a reactor scram.³⁴

Given the initiating event, the course of the accident is determined by employing known design parameters and safety systems behavior. The sequence details are worked out by analysts in the Engineering Technology Division. Knowing the details of the accident sequence and the system failure modes, a fission product transport analysis is performed to trace the release of nuclides from

the fuel to the primary systems and then to the containment system, the reactor building, and finally, the environment. Specific models are used to calculate the distribution of species and their subsequent chemical interactions, dissolution in water, and deposition or condensation onto fixed surfaces and aerosols. An efficient algorithm allows computation of the decay for each nuclide in each control volume and currently includes all of the significant isotopes of Te, I, Cs, Kr, and Xe. A model has also been developed to predict the interaction of steam with boron carbide in the neutron control blades. The resulting formation of cesium borates can alter the chemical form of iodine producing more volatile species.

During the postulated accident initiated by loss of decay heat removal, a recent analysis has shown a significant increase in iodine activity release to the atmosphere due to the decay of tellurium precursors.³⁵ Most of the fission product iodine is released from the fuel and retained within the primary containment, whereas a large fraction of the tellurium is released from the dry-well rubble following failure of the reactor vessel bottom head. Tellurium-132 with a 78-h half-life decays to ¹³²I (2.3-h half-life), and consequently, the atmospheric release of ¹³²I is very dependent on the behavior of tellurium in the dry well and reactor building.

Since the Mark-I primary containment is predicted to fail under severe accident conditions, it is most important to assess the capability of the secondary containment to reduce the accompanying release of fission products to the atmosphere. Under accident conditions, the standby gas treatment system (SGTS) at Browns Ferry is

32. R. P. Wichner, C. F. Weber, R. A. Lorenz, W. Davis, Jr., S. A. Hodge, and A. D. Mitchell, *Station Blackout at Browns Ferry Unit One—Iodine and Noble Gas Distribution and Release*, NUREG/CR-2182, Vol. 2 (ORNL/NUREG/TM-455/V2), August 1982.

33. R. P. Wichner, C. F. Weber, A. L. Wright, S. A. Hodge, R. A. Lorenz, and J. W. Nehls, *SBLOCA Outside Containment at Browns Ferry Unit One Volume 2. Iodine, Cesium, and Noble Gas Distribution and Release*, NUREG/CR-2672, Vol. 2 (ORNL/TM-8119/V2), September 1983.

34. R. P. Wichner, C. F. Weber, S. A. Hodge, E. C. Beahm, and A. L. Wright, *Noble Gas, Iodine, and Cesium Transport in a Postulated Loss of Decay Heat Removal Accident at Browns Ferry*, NUREG/CR-3617 (ORNL/TM-9028), August 1984.

35. C. F. Weber, R. P. Wichner, and S. A. Hodge, "Tellurium Precursor Effects on Iodine Transport in a BWR Accident," presented at the Annual Meeting of the American Nuclear Society, Boston, Mass., June 9-14, 1985.

automatically activated and is the final containment barrier. During most of the severe accident sequences, the SGTS maintains a subatmospheric pressure in the reactor building and retains fission products by filtration of particulates [high-efficiency particulate air (HEPA) filter] and adsorption of gas (charcoal bed). In previous studies,^{33,34} the HEPA filters were assumed to fail by tearing at a pressure drop of 2.5 to 3.0 kPa (10 to 12 in.) of water (aerosol loading of 3 kg per filter). Tests were sponsored at New Mexico State University to determine how long the installed HEPA filters would perform their function under severe accident conditions. Experiments were completed with a water spray and dry latex particles (0.3 to 0.4 μm), and no structural damage was observed at filter pressure drops up to 3.7 kPa (15 in.) of water. It seems reasonable to conclude that the type of HEPA filter installed at Browns Ferry would withstand the maximum pressure drop imposed by the SGTS blowers and could become completely plugged during the latter portion of a severe accident sequence. The pressure drop and air flow rate data as a function of mass accumulation (latex particles) on the filter media have been incorporated into the secondary containment model developed in the SASA program at ORNL.

8.2 REPOSITORY LICENSING SUPPORT

Technical support is being provided by staff members from the ORNL Chemical Technology, Chemistry, and Environmental Sciences divisions to the Office of Nuclear Material Safety and Safeguards, NRC, in support of their effort to license geological repositories for high-level waste (HLW) and spent fuel.

Technical Assistance in Geochemistry

During this report period, the technical staff has been assisting the NRC in establishing what geochemical information about a candidate area will be required in order to assess and determine how much credit a particular candidate area can receive for radionuclide retardation in performance assessments conducted in licensing. Technical assistance will also be provided to support the NRC's analysis of the DOE site-characterization activities, the geochemistry portions of the draft Environmental Assessments (EAs), and the Site Characterization Plans (SCPs) on each of the three DOE candidate sites.

Efforts to date have included:

1. collection and review of the DOE geochemical information base for the candidate HLW repository sites;
2. preparation of topical reviews on site-specific geochemical topics (e.g., sorption)³⁶ on the validity of the technical approach as well as the validity of geochemical data, algorithms, and interpretations reported in DOE documents and available through information exchange at DOE/NRC geochemical workshops;
3. participation in DOE/NRC workshops, conferences, and site visits;
4. review and evaluation of the geochemical portions of the EAs provided by the DOE; and
5. review of the technical adequacy of documents developed by the NRC staff.

In order to handle all the information on geochemistry being gathered under the auspices of this project, a computerized, searchable data base was created which is accessible on a timesharing basis, both on-site and off-site.

A workshop on the application of geochemical models in the assessment of HLW repositories was held in Oak Ridge to discuss the current status of geochemical code development, thermodynamic data bases, reaction kinetics, and coupled-process models as applied to site characterization and performance-assessment activities.³⁷ This workshop is the first of a series of workshops designed to bring together experts with diverse backgrounds to discuss various geochemistry topics that may play an important role in analyzing the safety of HLW repositories.

Evaluation of Radionuclide Geochemical Information Developed by DOE High-Level Nuclear Waste Site Projects

The objective of this project is to provide support to the NRC geochemistry staff by making an experimental evaluation of selected radionuclide

36. A. D. Kelmers, *Review and Assessment of Radionuclide Sorption Information for the Basalt Waste Isolation Project Site*, NUREG/CR-3763 (ORNL/TM-9157), September 1984.

37. G. K. Jacobs and S. K. Whatley, *Proceedings of a Conference on the Application of Geochemical Models to High-Level Nuclear Waste Repository Assessment*, NUREG/CP-2062 (ORNL/TM-9585), April 1985.

geochemical information and data acquisition methodology that may be employed by the DOE for HLW repository site projects in performance assessment calculations to show reasonable assurance of expected compliance with regulatory requirements. The project is focused on parameters that are important to the mobility of radionuclides in geologic media (primarily, sorption behavior on host rock and apparent concentration limits in groundwater) under the anticipated geochemical conditions of the repository. Initial emphasis is on information applicable to the Hanford Site candidate repository in the Columbia River basalts of the DOE Hanford Reservation at Richland, Washington, being characterized by the Basalt Waste Isolation Project (BWIP).

Batch contact methodology was employed by the BWIP to develop the published radionuclide sorption information for the Hanford Site. Since the in situ host rock/groundwater system is expected to be strongly reducing, BWIP added hydrazine to the synthetic groundwater to simulate the expected repository redox condition in laboratory experiments exposed to air. We have expressed concern as to the applicability of data developed by this methodology for performance assessment applications and believe that the radionuclide sorption ratios measured under reducing redox conditions may be nonconservative in some cases and inappropriate for performance assessment calculations.³⁸ Much of our work this year has been with neptunium, which is one of the key radionuclides for the Hanford Site (i.e., those expected to be among the major contributors to radioactivity release rates). We now believe that Np(V) (added in tests to the synthetic groundwater as NpO_2^+ , but probably hydrolyzed or complexed in groundwater to anionic species) is removed from solution by chemisorption reactions on the basalt surface which involve reduction to Np(IV) species or compounds. The reduction likely is due to Fe(II) in a basalt phase. In our oxic redox conditions experiments (both batch contact and column chromatographic tests) with Np(V), we measured a lower sorption ratio value (1.7 L/kg) with McCoy Canyon basalt than the value of 10 L/kg (measured with Umtanum basalt) expected under repository conditions by the BWIP. Since the Umtanum

is the only one of four candidate repository horizons, differences in neptunium sorption between different basalt flows could be important to performance assessment modeling. We have shown that the neptunium sorption ratio value obtained is sensitive to many experimental parameters, including: the specific basalt and synthetic groundwater used, radionuclide concentration, temperature, and time. It is difficult to rigorously compare our results with the summary of expected and conservative information published by the BWIP because important test parameters were not described.

Effect of Repository Environment on Performance of Waste Package/Engineered System Components

The primary objective of this project is to provide NRC with input to use in advising DOE about information needs concerning (1) repository environmental parameters affecting waste package performance and (2) the analyses employed in applying these parameters to the determination of waste package reliability. The initial focus of the project was to prepare a report that provided specifications for a model/methodology and approach to be employed in determining postclosure repository environmental parameters relevant to HLW package performance for the BWIP. The specifications provide guidance on (1) the identity and determination of the repository environmental parameters, (2) the various considerations that the models/methodologies must take into account, and (3) the input information needed to determine the output parameters within the specifications for implementing the model/methodology in the context of the overall waste package performance analysis system. In the future this work will be extended to include other waste package designs and repository media.

38. A. D. Kelmers, J. H. Kessler, W. D. Arnold, R. E. Meyer, N. H. Cutshall, G. K. Jacobs, and S. Y. Lee, *Progress in Evaluation of Radionuclide Geochemical Information Developed by DOE High-Level Nuclear Waste Repository Site Projects: Report for October-December 1983*, NUREG/CR-3851, Vol. 1 (ORNL/TM-9191/V1), August 1984.

9. CONSOLIDATED EDISON URANIUM SOLIDIFICATION PROJECT

9.1 INTRODUCTION

A total of 1047 kg of uranium (76.5% ^{235}U , 9.67% ^{233}U , 1.40% ^{234}U , 5.63% ^{236}U , 6.84% ^{238}U , and 140 ppm ^{232}U) is stored at ORNL in the form of ~8000 L of nitrate solution in the Thorium Reactor Uranium Storage Tank (TRUST) Facility, an 18,900-L vessel at Building 3019. This uranium resulted from the reprocessing of a $\text{ThO}_2\text{-UO}_2$ fuel core which had been irradiated in the Consolidated Edison Indian Point Reactor from 1962 to 1965, producing 10.1 PJ of electricity. Reprocessing was performed at the Nuclear Fuel Services Plant in West Valley, New York, and the uranium nitrate hexahydrate (UNH) product solution was transferred to ORNL in 1968-1969 for long-term storage.

The stored UNH solution is maintained at sub-criticality by means of both fixed and soluble neutron poisons. The fixed poison is borosilicate glass raschig rings that are packed inside the storage tank. Two soluble poisons, cadmium and gadolinium, were added to the UNH solution as a safety measure in case the solution should leak from the tank. The concentration of each poison is at a level sufficient to prevent criticality. These concentrations are measured periodically, and the measurements have indicated that some crystallization or deposition has apparently occurred during the 16-year storage period. Therefore several additions of cadmium and gadolinium have been made to maintain the necessary concentration levels.

Since the walls of the storage tank are only 0.53 cm thick, there is concern that localized corrosion could eventually cause leakage. Samples of the solution have been analyzed for stainless steel constituents, and the results have indicated that significant corrosion has not occurred. Nevertheless, both DOE-ORO and ORNL safety committees have warned that tank storage for an indefinite time may be unwise.

The Consolidated Edison Uranium Solidification Project (CEUSP) was initiated several years ago to

convert the uranium to a solid form (U_3O_8) for safer storage. The conversion process includes an evaporation step to concentrate the UNH solution and a thermal denitration step to form the oxide. The denitration will be carried out in situ in each of the storage cans, which will be loaded with ~3 kg of uranium. After solidification is complete, each can will be (1) sealed by welding the lid, (2) enclosed in a secondary containment canister which will be crimp-sealed, and (3) stored in a shielded storage well.

The CEUSP process equipment is located inside Cell 3 at Building 3019. Cell 4 will be utilized to provide holdup space for the decay of ^{220}Rn in the off-gas stream and to house the "earthquake-proof" storage wells for the cans of U_3O_8 .

9.2 PROCESS DESCRIPTION

The CEUSP process is fundamentally simple, but the construction and processing steps are complicated by the following constraints:

1. The ^{232}U has daughters that produce a very energetic gamma radiation of 2.6 MeV and therefore requires massive shielding;
2. Processing of the large amount of fissile material requires equipment designs that are geometrically favorable for subcriticality;
3. Hands-on maintenance requires accessible equipment, which complicates equipment layout;
4. Storage-can transport equipment is intricate; and
5. Available in-cell processing space is limited.

These factors resulted in the need for a complex processing facility.

The CEUSP conversion process involves simply dripping the concentrated uranium-cadmium-gadolinium nitrate solution onto a heated surface in the storage can, thereby evaporating the liquid, decomposing the nitrate, driving off the vapors and gases, and leaving the residual oxide solid. All of

the operations are performed within leak-tight, ventilated, and shielded stainless steel enclosures. The four major steps in the conversion procedure are: (1) periodic transfer of the UNH solution from storage to the processing area in Cell 3, (2) concentration of the UNH solution by evaporation, (3) in-situ denitration and solidification of the UNH concentrate at temperatures up to 800°C, and (4) the sealing and handling steps necessary to complete placement of the sealed cans of solidified oxide in the solids storage facility in Cell 4.

The equipment used for accomplishing these objectives is located in Cell 3, a 6 X 6 X 8 m hot cell at Building 3019. The penthouse area of the building is also used for material transfers of the solidified product from Cell 3 to Cell 4.

The basic layout of the hot cell containing the CEUSP equipment is shown in Fig. 9.1. Enclosures B, C, and H are liquid processing areas and contain vessels, an evaporator, jets, and pumps. Enclosure D and the entrance and exit stations contain one-of-a-kind, dedicated, mechanical mechanisms controlled by handwheels with shafts that extend into the operating area of Cell 3.

Fig. 9.1. Schematic showing orientation of the CEUSP equipment inside the hot cell.

9.3 CONSTRUCTION ACTIVITIES

The design and construction planning constraints described above influenced the type of project management used for CEUSP. The facility and processing equipment design work was performed by the Engineering Organization of Martin Marietta Energy Systems, Inc., with assistance from Chem Tech. The ORNL Central Maintenance Shops fabricated the processing and mechanical handling equipment in the enclosure cubicles. The completed equipment enclosures were then transported to Building 3019 for rigorous functional testing by Chem Tech personnel. The equipment was permanently installed in Cell 3 by Rust Engineering Company. Throughout the design, testing, and construction phases, systems reliability has been an overriding consideration. The CEUSP facility will operate for approximately 15 months, but the components were tested for a 10-year duty life.

Equipment Fabrication

Building the equipment as complete modular enclosures proved beneficial for several reasons:

1. Shop fabrication is more efficient than field fabrication;
2. The enclosures could be sealed in a stainless steel box, thus containing any alpha contamination during the process and facilitating any decontamination efforts that might be needed;
3. The complicated piping and mechanical systems could be constructed in a very compact space; and
4. The systems could be performance tested prior to installation in the hot cell.

Figure 9.2 illustrates the compact piping that is required in the enclosure fabrication. This photograph shows the main processing equipment before enclosure in the stainless steel box. All valves are installed in accessible valve boxes such as the one shown at the left in Fig. 9.2, and all piping terminations are either in valve boxes or external to the enclosure.

Each system component was thoroughly tested while in the shop during fabrication, and a final in-shop functional check of each enclosure was conducted. After transportation to Building 3019, each enclosure was temporarily installed in a mock-up area for an operational checkup. During this phase, the mechanical system was installed

Fig. 9.2. Process piping installed in an equipment module for CEUSP. This was housed in a stainless steel enclosure prior to installation at Building 3019.

and aligned, the processing systems were used, and the sampling station was tested. Several modifications were made as a result of this testing. Notable examples were the replacement of mechanical bearings that corroded and the correction of piping slopes that trapped liquid.

Equipment Installation

The field construction, performed by Rust Engineering Company, included removal of old processing equipment from Cell 3 and installation of a new stainless steel floor and wall liner. Approximately 200 penetrations from Cell 3 to adjacent processing areas were sealed. Other work included preparation of Cell 4 for final storage and installation of the out-of-cell chemical make-up equipment.

The installation of equipment enclosures began in August 1982. Components were installed through a cell roof hatch which measured 3 m by 3 m. The processing enclosures were installed during the spring of 1983. Figure 9.3 shows two of the processing enclosures being rigged for installa-

tion. The enclosure on the left is the same one shown in Fig. 9.2. Note the valve box on the upper part of the enclosure. These 5-m-tall enclosures were lowered into the Cell 3 hatch using the crane visible in the background. A total of 10 enclosures were set up in this manner.

Interconnecting the CEUSP enclosures required extensive pipefitting and electrical work. Piping construction was continuous from spring of 1983 through spring of 1984. Accurate installation of the maze of piping was greatly aided by use of a model built to scale. In Fig. 9.4, the complex piping manifolds built as part of the model can be observed. An engineering draftsman closely monitored the piping installations and modified the model to the as-built condition, making it invaluable as a reference and troubleshooting tool.

Other craftsmen and construction specialists also had large construction jobs in the confined hot-cell space. Approximately 250,000 kg of shielding (half steel and the remainder lead brick) and large, intricate ventilation ducts were installed. Significant additions of structural steel and a mezzanine floor were also required.

Fig. 9.3. Modular CEUSP equipment enclosures rigged for installation. Ten enclosures such as these were installed in a $6 \times 6 \times 8$ m hot cell.

Coordination of the CEUSP construction job required several engineering disciplines. Field engineers checked on progress throughout the job; program engineers wrote at least 50 test procedures and conducted the required testing; the construction engineer provided interfacing between ORNL and Rust Engineering. Project engineers from Martin Marietta Engineering, Rust, and DOE generated schedules and tracked progress and expenditures.

Preoperational Testing

Essentially all construction was completed by April 1984, when preoperational testing commenced. During this testing, a major problem was

discovered in the fittings for interconnecting tubing. The stainless steel surfaces of the fittings had been treated to enhance hardness, and this treatment process lowered their resistance to corrosion. The failure of these fittings required replacement of 1500 piping components, causing a 4-month delay in the project.¹ Preoperational testing resumed in November 1984 and concluded in January 1985. Final construction closure was completed and operational verification conducted in March 1985.

¹ I. R. A. Jacobus, et al., *Report of the CEUSP Tube Compression Fitting Failure that Occurred During June 1984*, ORNL/TM-9495, February 1985.

Fig. 9.4. The CEUSP facility design and construction was greatly aided by the use of this plastic scale model. Here an engineer is verifying pipe layout.

9.4 CEUSP PROCESS FLOWSHEET

A simplified flowsheet for the CEUSP conversion process is given in Fig. 9.5. The first step is to lift the UNH solution from the TRUST Facility to the UNH transfer vessel (V-2) located in Enclosure B of Cell 3. The 0.9-cm ($\frac{3}{8}$ -in) transfer line is enclosed (along with the off-gas and pit-sump sampling lines) within a larger pipe, which provides mechanical support and protection to the smaller transfer line as well as double containment of the UNH solution.

Lifting of the UNH solution to the V-2 transfer vessel is accomplished by means of an air-operated, vacuum-transfer jet. The transfer vessel is then pressurized to move the UNH to the short-term storage vessel (V-1) through one of two parallel sintered metal filters, which remove any glass particles carried over from the raschig ring packing in the TRUST.

The transfer of solution to the 35-L V-2 is done several times to fill the 250-L V-1 storage vessel. Vessel V-1, like all other vessels in Cell 3, is designed to be critically safe under all conditions. From V-1, each 20.8-L batch of material is lifted by vacuum to the evaporator feed vessel. This discrete batch will go through the CEUSP process to be solidified as one container of oxide material (3 kg uranium per can).

After the initial transfer, the next major step is concentration of the UNH solution. The 20.8-L batch of solution, with a uranium concentration of 135 g/L and a free acid concentration of 1.7 M HNO_3 , is pumped from the evaporator feed vessel (V-4) to the evaporator (V-5). Here the heavy metals are concentrated by a factor of ~ 2.5 , and $\sim 98\%$ of the free nitric acid is destroyed by reaction with formaldehyde to yield 8.3 L of product with a uranium concentration of 340 g/L and a free acid concentration of 0.1 M HNO_3 .

Fig. 9.5. Simplified flowchart for the CEUSP conversion process.

The NO_x fumes and water vapor generated in the evaporation step are passed through the condenser, and the noncondensable gases are routed to the CEUSP off-gas waste handling system.

The concentrated UNH solution from the evaporation step is vacuum transferred to one of three furnace feed vessels located in Enclosure H. At this point in the system, three separate solidification process layouts are provided, and these may be operated simultaneously or individually, as desired. Each solidification process layout has a furnace, a UNH concentration vessel, a metering pump, a denitration condenser, and a condensate collection vessel.

To complete the conversion process, each stainless steel storage can (9-cm diam by 61 cm long) is heated in one of the furnaces, and the concentrate is fed at the rate of 9 mL/min into the can, where the heat causes solidification as uranium oxide.

Each furnace has three zones, which are held by a programmable controller at the desired operating temperatures (ranging from 300 to 800°C). Feeding of the concentrate into the container is started when the proper temperature profile in the furnace and the correct air flow pattern have been estab-

lished, if all system indicators are normal. The variable three-zone temperature profile is necessary to deliver maximum heat to the reaction zone without prematurely baking on an insulating oxide crust in the upper region of the container. At slow feed rates, decomposition gases (NO_x , H_2O , CO_2) are produced at rates that minimize the formation of foam. The off-gas vapors are condensed, and noncondensables are removed by an air purge and routed to the off-gas waste handling system.

Following completion of the solidification step, the filled can is removed from the furnace by the main transfer carriage (Fig. 9.6) and transported by another transfer carriage to the entrance station. Within this shielded station, the can is weighed and a lid is welded on. The sealed can is then either lowered into an interim storage well or moved directly to the exit station, as desired. When the sealed can is transported to the shielded glovebox exit station, it is lowered into a thin-walled canister, and a lid is secured to the canister by a double-seaming device.

A shielded transfer cask located above Cell 3 in the penthouse area of Bldg. 3019 is used to lift the filled canister out of the exit station into the pent-

ORNL PHOTO 4629-82

Fig. 9.6. A craftsman is shown adjusting the mechanical transfer carriage used to transport containers of solidified uranium oxide waste from the furnace.

house and to move it over to the Cell 4 storage wells for long-term storage.

In addition to the basic CEUSP processing system located in Cell 3, systems designed to handle the liquid and gaseous wastes are provided to support the solution transfer and concentration steps. Liquid wastes from condensate vessels, sump, and other areas are collected into one vessel (V-7). This 270-L vessel fills approximately once a week and, after sampling, the waste is transferred to the ORNL low-level waste system for disposal. Gaseous wastes from CEUSP are routed through vessel V-41, then filtered and discharged in Cell 4 to allow ^{220}Rn gas (56-s half-life) to decay to solid daughter products before being fed into the Bldg. 3019 cell off-gas (COG) system. In the COG sys-

tem, these gases are filtered to remove the solid daughters of the radon decay and then discharged through the 3020 stack north of Bldg. 3019.

9.5 PLAN OF OPERATION

Both safety and quality assurance documentation reviews were required by several oversight groups within the Laboratory and DOE. The Final Safety Analysis Report (FSAR) was reviewed internally, by the DOE's Oak Ridge and Germantown operations offices, and by a team contracted by DOE from the Los Alamos National Laboratory. These were intensive reviews. When the fitting failure occurred, the program was instructed by the DOE to prepare an Addendum to the FSAR addressing

safety implications of the fitting rework. This document required the same review as the FSAR. An operating contract containing the Operational Safety Requirements was reviewed by ORNL management and by DOE.

Several review bodies assessed the ability of CEUSP to operate. The ORNL Criticality Control Committee and the Radioactive Operations Committee studied the operational preparations thoroughly before recommending to the ORNL Office of Operational Safety that CEUSP be allowed to operate. Similar assessments were conducted by DOE, with representatives from DOE-ORO in the areas of safety, quality assurance, health physics, and criticality, and with personnel from the DOE Washington Program Office participating in the review. Final approval for operation was obtained in March 1985.

Training and preparation of the operating crew was one of the review areas most closely scrutinized. At the beginning of CEUSP, all of the operating technicians were inexperienced in radiochemical processing. An intensive formal training program was developed and implemented using lectures, demonstrations, testing, and on-the-job experience. These training efforts were fully documented and the operating crew certified. The normal operating contingent consists of a supervisor and two technicians, but during startup and initial operation an engineer works with each of the three shifts to lend technical support. Our CEUSP staffing and training program received favorable reviews.

Program engineers and the operating crew jointly developed the operating procedures for

CEUSP, and both equipment checkout and pre-operational testing occurred throughout the project. The results of the testing are documented and have been useful in preparing for plant operation.

Performance Testing

The CEUSP process has been fully performance tested, with 175 batches of synthetic uranium feed denitrated and 60 batches of synthetic feed used for full-scale evaporator studies. No process problems are anticipated, although there is always some potential for failures in the mechanical equipment such as the main transfer carriage and the concentrate feed pump. Spare parts of identified components are on hand, and simulated equipment changeouts have been conducted. Equipment failures could prove to be a nuisance but are unlikely to greatly affect the program.

CEUSP Operation Schedule

Before the project is completed, CEUSP will generate about 375 cans of solidified waste. The facility is designed to produce 9 cans per week by operating the 3 parallel denitration lines concurrently for 3 shifts per day, 5 days per week. The typical processing cycle involves evaporating 3 separate batches of feed to produce concentrate, then denitrating the concentrate in the 3 parallel lines. The evaporation phase requires about 10 h for the 3 batches, and denitration requires an additional 22 h.

10. RADIOACTIVE MATERIALS PRODUCTION

10.1 URANIUM-233 PROCESSING

The Radiochemical Processing Plant (RPP) at ORNL has served as the national repository and distribution center for ^{233}U for >20 years. Several hundred kilograms of uranium, containing approximately 90 to 98% ^{233}U , are stored there in the form of metal, oxides, and nitrate solutions. All of these uranium materials contain small, but significant, concentrations of ^{232}U , ranging from 2 to 225 ppm. Most of the radioactivity associated with the ^{233}U comes from the decay daughters of ^{232}U (74-year half-life). Containment is required for all of the daughters, which are generally alpha emitters. In addition, three of the daughters, ^{228}Th (1.9-year half-life), ^{220}Rn (54.5-s half-life), and ^{208}Tl (3.1-min half-life), require special considerations for handling.

The ^{208}Tl emits a 2.6-MeV gamma ray, which necessitates that the ^{233}U storage and processing facilities must be heavily shielded and remotely operated. Since the ^{220}Rn daughter is a gaseous material, special off-gas treatment facilities are required to enable sufficient retention time of off-gas streams to permit ^{220}Rn decay, followed by high-efficiency filtration to remove the particulate decay daughters of the ^{220}Rn . Since these include the ^{208}Tl , the filters must be either shielded or located remotely.

The ^{228}Th is the decay daughter of ^{232}U . All decay daughters of ^{228}Th (which include ^{220}Rn and ^{208}Tl) are short-lived; thus, by making a chemical separation of thorium from uranium, the gamma-emitting daughters can be temporarily removed from the ^{233}U . Decontamination can be achieved by factors of >100 for several days and >50 for about 1 month before the ^{232}U daughters grow back in.

The RPP contains chemical processing equipment that is used (1) to dissolve multikilogram batches of U_3O_8 , UO_2 , or $\text{ThO}_2\text{-UO}_2$; (2) to process the resulting uranium nitrate solutions by solvent extraction and ion-exchange techniques for separating the ^{228}Th and its daughters from the

^{233}U ; and (3) to convert the purified uranium to the oxide form (U_3O_8 or UO_2). The equipment used to accomplish the first two steps is located in heavily shielded hot cells and is remotely operated (but directly maintained); however, the conversion equipment is located in an unshielded glove-box facility. Only purified ^{233}U containing <10 ppm of ^{232}U can be processed in the conversion equipment without exposing the operating personnel to excessive gamma radiation.

Storage facilities for ^{233}U at the RPP include ten 400-L tanks for liquid storage, shielded storage wells for solids, and a safeguarded vault for small packages and samples. Until early 1985, the vault was used for storage of multikilogram amounts of ^{233}U . However, the safeguards capability was determined to be insufficient for large quantities. Therefore, a significant effort was made to reduce the inventory of special nuclear materials in the vault by transferring most of the material to storage in the shielded wells. Massive cover blocks and locking plates were installed over the shielded wells so that theft cannot be accomplished without a significant time delay. With this delay, guard reinforcements can arrive from the Y-12 site.

During the period between April 1983 and March 1985, several batches of materials containing ^{233}U were received from other DOE sites and were stored. However, only one 10-kg batch was purified and shipped, and only 4 batches, ranging in size from 1 to 3 kg, are scheduled to be purified and shipped during the next 2-year period. Following this, current plans are to solidify all ^{233}U now stored in liquid form and to clean out the processing facilities and place them in a long-term standby condition. This is scheduled to be accomplished by the end of FY 1989.

10.2 CALIFORNIUM-252 INDUSTRIAL SALES/LOANS

The ^{252}Cf Industrial Sales/Loan Program involves loans of ^{252}Cf neutron sources to agencies of the U.S. Government and sales of ^{252}Cf as the bulk

oxide and as palladium-californium alloy pellets and wires. The program has been operated since 1968 in temporary facilities at the Savannah River Laboratory (SRL). The obsolete hot-cell facilities at SRL are now being decommissioned, and the program activities are being transferred to ORNL's Californium Facility in Bldg. 7930, which is managed by the staff of the Transuranium Processing Plant (TRU).

The Californium Facility was placed in operation in 1970 to produce ^{248}Cm (the radioactive-decay daughter of ^{252}Cf) that is highly purified from contamination by other curium isotopes. Since operation began, essentially all of the ^{252}Cf produced in the western world has been processed here, including the ^{252}Cf used in the Industrial Sales/Loan Program. More than a hundred ^{252}Cf neutron sources have been fabricated under the sponsorship of the DOE Office of Basic Energy

Sciences. The Industrial Sales/Loan Program will complement the heavy-element program activities currently being performed by the TRU staff and is a natural extension of the existing program.

Additional facilities and equipment are being installed in the Californium Facility to accommodate the new activities while continuing all of the Basic Energy Sciences program activities already handled by TRU. All operations involving unencapsulated ^{252}Cf will be colocated with the TRU californium purification operations in Cell G, the current Californium Facility. New work stations have been constructed for welding of secondary capsules, final decontamination, ^{252}Cf assay by neutron counting, and loading of shipping casks. These will be located in the adjoining Cell C. A californium storage facility will be installed in the water-filled basin originally intended for handling irradiated fuel elements.

11. COMPUTER/ENGINEERING APPLICATIONS

11.1 TRANSPORTATION PROGRAM

Work for the transportation program in Chem Tech has broadened since the last report period. Activities that support the Transportation Operations and Traffic Management (TOTM) program continue to be funded through DOE-ORO; all R&D is sponsored by the Transportation Technology Center (TTC) at Sandia National Laboratory (SNL). During the past 2 years, emphasis has increased on transportation studies of the movement of chemical weapons for the U.S. Army Toxic and Hazardous Materials Agency (USATHAMA). Much of our work involves the use of several computer programs developed by Chem Tech personnel and refined to their present operational status over the past several years with contributions from the Information Resources Organization and the Computing and Telecommunications and Energy divisions at ORNL.

Federal/State/Indian Nations Transportation Meeting

The Office of Civilian Radioactive Waste Management (OCRWM), DOE, requested that ORNL host a meeting to demonstrate the transportation computer programs utilized by DOE, to establish a dialogue concerning the use of these models, and to define the areas of concern that should be addressed by the OCRWM Office of Storage and Transportation in the near future. The meeting was held November 27-28, 1984, at ORNL, with approximately 100 attendees representing federal, state, and Indian nations entities.

The first day of the meeting was devoted to demonstrations and discussion of the following transportation computer programs: HIGHWAY,¹ INTERLINE,² ORNL Transportation Logistics Program,³ PNL WASTES Logistics Program,⁴ RADTRAN II,⁵ and the associated computer graphics programs.⁶ The participants were divided

into four groups, which rotated among four 1-h technical sessions. At the conclusion of the demonstrations, technical questions regarding the models were discussed. The primary objective of the meeting, a better understanding of the capabilities and utilization of the transportation models by each participant, was achieved through these sessions.

During the second day of the meeting, it was demonstrated how the DOE transportation computer models provide input to support the environmental assessment process. The OCRWM's proposal to meet a state's request for information was also outlined.

The OCRWM agreed to provide to the attendees, by February 1, 1985, a series of maps and tables showing the possible truck and rail routes for the transportation of spent fuel from reactor sites to nine potential repository sites. Maps illustrating these routes superimposed on population density maps were also to be included. This information has been transmitted as promised.

Because of the interest shown by participants, the DOE agreed to provide access to both the highway and rail routing models for state and

1. D. S. Joy and P. E. Johnson, *HIGHWAY, A Transportation Routing Model: Program Description and Revised Users' Manual*, ORNL/TM-8759, October 1983.

2. B. E. Peterson, *INTERLINE, A Railroad Routing Model: Program Description and Users' Manual*, ORNL/TM-8944, November 1983.

3. D. S. Joy and B. D. Holcomb, *Logistics Models for the Transportation of Radioactive Waste and Spent Fuel*, ORNL/TM-6192, March 1978.

4. M. R. Shay, A. L. Thorpe, and G. W. McNair, *WASTE: Waste Systems Transportation and Economic Simulation*, PNL-5413 (SAND84-7173, TTC-0533), March 1985.

5. J. M. Taylor and S. L. Daniel, *RADTRAN-II: Revised Computer Code to Analyze Transportation of Radioactive Materials*, SAND80-1943, June 1983.

6. R. C. Durfee and Geographic Data Systems Section, *Review of Geographic Processing Techniques Applicable to Regional Analysis*, ORNL-CSD/TM-226, in preparation.

other appropriate organizations. The mechanism for such access has since been arranged, pending completion and DOE approval of the applicable agreements.

Alternative Shipping Patterns

During this report period, a study was made of shipping patterns that might develop in the transport of TRU waste shipments by truck and rail to the Waste Isolation Pilot Plant (WIPP) in southeastern New Mexico and of the current and future institutional issues that could impact such shipments.

At least three potential truck shipping routes between each waste source and the WIPP site were developed using the HIGHWAY program. Because of the limited number of viable rail routes, only two source-to-destination routes for that mode were determined for each waste source using the INTERLINE program.

Computer Code Benchmarking

Benchmarking of computer codes is being carried out in several areas dealing with analyses of shipping casks. In the areas of heat transfer and structural integrity, work is just beginning. The TTC has presented ORNL with a thermal benchmark problem that is being analyzed using the newest version of HEATING6. In preparation for supporting an experimental phase of the structural effort, the HONDO II code has also been made operational at ORNL.

Work has been in progress for several years on the application of criticality codes to the design of shipping casks. A recent report on this topic described a criticality analysis that involved five different fissile material forms with several thicknesses of water between the fissile units and a thick water reflector around the outside of the finite arrays.⁷ These were hypothetical transport packages for studying the importance of interspersed moderating materials in the packaging of radioactive wastes.

In an effort to study and validate each parameter separately, the effects of other packaging and structural materials were not considered in these criticality analyses. Experimental systems simulating criticality were chosen for validating the computational procedures to be used for systems with physical and material properties similar to those of Class II packages for fissile materials.

Due to the lack of experiments with large ($\geq 5 \times 5 \times 5$) arrays of fissile materials in which each unit is a substantial fraction of a critical mass, it was not possible to validate the calculations as a function of array size. This left the problem of extrapolating from the available data.

While our work resulted in a recommendation regarding commingling of Fissile Class II packages, it became clear that additional criticality experiments are needed to provide the data needed to ensure the development of a safe and economical transport system for Fissile Class II packages.

Transporting Defense High-Level Waste to a Geologic Repository

The Nuclear Waste Policy Act of 1982 (Public Law 97-425) provides for the development of repositories for the disposal of high-level radioactive waste and spent nuclear fuel and requires the Secretary of Energy to evaluate five potential repository sites. One aspect that is to be examined is the transportation of radioactive materials to the repository. This includes an evaluation of the transportation factors involved in shipments to a defense-only repository vs those for shipments to a site that accepts both defense and commercial wastes.

In response to this requirement, the DOE requested ORNL to undertake an evaluation of costs and risks associated with the potential waste shipments. This evaluation assumed that defense high-level waste (DHLW) is stored in three locations (the Savannah River, Hanford, and Idaho Falls plants) and may be disposed of in (1) a commercial repository or (2) a defense-only repository, either of which could be located at one of the five candidate sites. Two waste-flow scenarios were considered relative to the total quantity of DHLW that will be placed in a repository. The low-flow case assumes that a total of 6,700 canisters will be transported from one site, while the high-flow case assumes that a total of 20,000 canisters will be transported from three sites.

For the scenarios considered, the estimated shipping costs range from \$105 million to \$257 million, depending on the mode of transport and the repository location. The total risks associated with shipping the DHLW to a repository are estimated to

7. CNSI Working Group, *Standard Problem Exercise on Criticality Codes for Large Arrays of Packages of Fissile Materials*, CNSI Report No. 78, August 1984.

be significantly smaller than those predicted for other transportation activities. In addition, it was found that the shipping costs do not depend on whether the site is a defense-only or a commercial repository. Therefore, the transportation considerations are not a basis for the selection of one of the two disposal options. This information was reported in ORNL/TM-9388, *Transporting Defense High-Level Waste to a Geologic Repository*, published in December 1984.

Transportation Information System

The Legislative and Regulatory Information System (LRIS), developed and operated at ORNL, contains information on proposed and approved federal, state, and local legislative actions affecting the transportation of radioactive and hazardous materials.⁸ The information is stored in a computerized data file, which provides great flexibility in retrieving desired information. Since the previous report period, ~800 new bills have been identified, and status changes have been made in ~500 other actions.

Another component of the LRIS that operates as a supporting tool to the overall transportation program is the Operational Restrictions and Emergency Response data base. In addition to highlighting the operational restrictions prohibiting or restricting the use of existing highways and rail lines, this data base identifies the specific state or local contact agency or person responsible for emergencies involving the transportation of radioactive and/or hazardous materials. The data base operates on the INQUIRE data-base management system, and special programs have been written to make it user-friendly.

The data fields of the LRIS pertaining to operational restrictions include information identifying: (1) the agency responsible for regulating transportation; (2) the type of escorts required; (3) areas that require prior notification, permits, or licenses; and (4) areas that ban transportation of all radioactive materials.

TRUPACT Drop Tests

The TRUPACT is a package designed by GA Technologies, Inc., for the TTC that is intended to contain and transfer drums of transuranic waste from generating sources to the WIPP. In order to obtain engineering data on the behavior of the

22,720-kg TRUPACT under accident conditions, the TTC decided to perform full-scale impact tests on a prototype model. These tests were conducted at the ORNL Drop Test Facility. Because of the large package dimensions (2.4 × 2.4 × 7.3 m), the length of the steel impact pad at the Drop Test Facility had to be extended.

Six drop tests were completed within a 2-week period to meet the proposed schedule. In two of these, the package was dropped a distance of 9 m onto the unyielding surface. Four tests were staged by dropping the TRUPACT from a 1-m distance onto a 15-cm-diam piston. The behavior of this package was monitored by measuring deceleration rate and the strain on structural members at a number of points throughout the package. Both normal-speed and high-speed photography recorded the impacts. The package was checked for leaks after each test and returned to the TTC for further evaluation.

PATRAM

The Seventh International Symposium on Packaging and Transportation of Radioactive Materials (PATRAM) was held May 15–20, 1983, in New Orleans, Louisiana — a continuation of the series that began in 1965. At this symposium, an international group (from 20 countries) composed of 538 scientific and technical experts from industry, governmental institutions, and research organizations, gathered to relate their experiences concerning all aspects of packaging, testing, and transporting radioactive materials. PATRAM '83 also considered several important revisions of the International Atomic Energy Agency (IAEA) Transport Regulations. The ORNL responsibilities for the symposium included coordination of all meeting arrangements, publication of a summary volume for distribution at the meeting, and preparation of the proceedings following the meeting.

Shipments of U.S. Army Chemical Weapon Stocks

The U.S. Army is currently involved in a detailed analysis of alternatives for the disposal of

8. C. S. Fore, L. F. Goins, L. F. Patterson, N. P. Knox, and P. T. Owen, *Transportation of Radioactive and Hazardous Materials: A Summary of State and Local Legislative Requirements for the Period Ending September 30, 1983*, ORNL/TM-8860, June 1984.

M55 rockets filled with chemical agents. A portion of the study—that dealing with colocating the weapons at a central site/or sites—is being performed by Chem Tech. The rockets could be moved by truck, rail, or air. Transport by air would require a truck convoy to and from the shipping and receiving airports. Several types of airports are also being considered (e.g., military or civilian).

One factor that has a significant impact on total cost (and possible risk) is the use of an overpack within which the weapons could be placed for shipment.

For this analysis, the costs were categorized according to shipping mode, receiving site, and use of the shipping overpacks. Total transportation costs for shipping the rockets (without overpacks) to a potential disposal site in the western United States are projected to be \$38 million for air shipment from an onsite military airport or \$49 million for transport by truck convoys. If overpacks are used, the projected costs will be \$86 million and \$96 million, respectively, for the modes mentioned above. Costs for the overpacks are not included in these figures.

11.2 PROCESS AND SYSTEMS ANALYSIS

The objective of the Chem Tech Process and Systems Analysis Group is to provide modeling, analysis, and technology assessment assistance to the Division, ORNL, the Oak Ridge Complex, and DOE. Our activities during this report period included (1) support to DOE's Morgantown Energy Technology Center (METC) through the use of the Advanced System for Process Engineering (ASPEN) simulator, (2) support to DOE/ORO for SRC-I modeling and independent verification of the H-Coal Pilot Plant material balance yield calculations, (3) support to ORNL's Department of Environmental Management (DEM), (4) an assessment of direct coal liquefaction technology and an economic evaluation of coal-to-methanol processes for the DOE Office of Fossil Energy (DOE/FE), and (5) ASPEN modeling assistance to the Enriched Uranium Recovery Improvement (EURI) Project at the Y-12 site and to the Breeder Reactor Engineering Test (BRET) Project.

Enhancement of ASPEN's Cost-Estimating Capabilities

The original cost-estimating capabilities of the ASPEN process simulator needed improvement in

at least two areas. First, factors for several types of equipment (e.g., turbines, waste-heat boilers, and slurry pumps) that were needed in ASPEN in order to simulate large-scale synthetic fuels plants were not included. Second, some of the original ASPEN cost relationships appeared to be inaccurate. The METC sponsored our project to enhance ASPEN's capital equipment cost-estimating capability by extending it to include more types of equipment, as well as modifying the cost data for several existing equipment types. This work was carried out on the VAX 11/780 version of ASPEN at ORNL.

The newly developed and revised ASPEN costing models are given in Table 11.1. The cost correlations used in developing these models were derived from published sources, previous studies, and vendor contacts. Each model was tested over a range of sizing parameters, and the results were tabulated and presented in final report.⁹ Additional materials associated with each model (FORTRAN subroutines, ASPEN User and Technical Reference Manual sections, and files for incorporating the model into the ASPEN system) are also included in the report.

ASPEN Analysis of Steam Bottoming Cycles

In order to fulfill and enhance its lead role as the sponsor, manager, and executor of R&D projects related to coal fuel-gas turbine systems and other fossil fuel utilization systems, the METC has

Table 11.1. New and revised ASPEN costing models

Equipment type	Model name
<i>New Models</i>	
Waste heat boiler	COST-WASTEHT
Slurry pump	COST-SLRPUMP
Diaphragm pump	COST-DIAPUMP
Gas turbine with generator	COST-GASTURGN
Gas turbine with driver	COST-GASTDR
Steam turbine with generator	COST-STMTURG
Steam turbine with driver	COST-STMTDR
Conveyors	COST-CONVEYR
Bucket elevators	COST-BKTELEV
<i>Revised Models</i>	
Fabric filter	COST-FABFILT
Electrostatic precipitator	COST-ELPRECR
Steam boiler	COST-FIREDHT

9. P. J. Johnson, M. L. Myers, and J. M. Bogovich, *Enhancement of ASPEN's Capital Equipment Cost Estimation System*, ORNL/TM-9562, in preparation.

identified a need for a general numerical model for Rankine steam bottoming cycles. To support METC and to ensure compatibility with other models being used there, we have developed ASPEN simulation models for representative Rankine steam bottoming cycles of the coal gasification/combined-cycle (GCC) power plants.

A generalized flowsheet for GCC plants was developed from the analysis of reports describing twelve of those plants; then ASPEN was used to obtain a computer program for the simulation of such plants through a generalized model. The ASPEN program, after the modifications necessary to handle the configuration of a thirteenth plant, was successful in its simulation of that plant. However, a careful analysis indicated that each GCC differs enough from the others so that specifying a unique plant configuration while using a generic program is inefficient and prone to error. It was found to be faster and more reliable to build a customized program to simulate the same thirteenth plant using the basic ASPEN unit operation blocks (such as MIXER, PSPLIT, HEATER, FLASH, PUMP, and COMPRESSOR).

The final effort of this project was the preparation of another customized ASPEN program to simulate a very complex and sophisticated steam bottoming plant. This effort confirmed the advantages of using the customized-program approach. As summarized in a report of this work,¹⁰ ASPEN again proved capable of duplicating the vendor's results and useful in examining various flowsheet configurations and process conditions.

Support to DOE Liquefaction Projects

We are providing support to DOE/ORO in reviewing the development by the International Coal Refining Company (ICRC) of both dynamic and steady-state models of the SRC-I process. As part of this effort, the ICRC requested that we develop an ASPEN model of the diethanolamine (DEA) acid-gas removal process that could be integrated with their model for the balance of the plant. To accomplish this, the physical properties for DEA were first added to the ASPEN pure-component data bank. A set of binary interaction parameters for simulation of the physical properties of the quaternary hydrogen sulfide-carbon dioxide-DEA-water system was then developed. A simplified overall model was assembled, based on the separation given by a vendor for the DEA process.^{11,12} Difficulties with the ASPEN absorber

models and the complexity of the quaternary system prevented us from developing a rigorous DEA absorber model; however, a model for a simplified DEA scrubber system was provided that, when used with the binary interaction parameters developed, would allow ICRC to complete the important hydrogen recycle loop.

We are also assisting DOE/ORO in reproducing independently the material-balance yield calculations for the H-Coal Pilot Plant. These calculations were based on information available in the H-Coal data base at the McDonnell Douglas Automation Company (McAuto) and on other information available to DOE, as referenced in the final technical report on the H-Coal Pilot Plant.¹³ During this report period, the data base at McAuto was transferred to ORNL for further use. An extensive analysis of the material-balance calculation methodology and the computer program MATBAL has been made, and a report on this work is currently in preparation.¹⁴ This analysis now allows reproduction of the calculations made by the operators of the Pilot Plant when corrections to the raw data are known. In addition, this analysis has verified the validity of the material balances reported by the Pilot Plant.

Support to the Department of Environmental Management

Demineralizers (ion-exchange columns used to demineralize water) are used to purify primary coolant, pool, and makeup water for the research reactors located at ORNL. Periodic regeneration of the exhausted ion-exchange resin beds with nitric acid generates an effluent stream with a significant nitrate loading. In order to reduce the

10. O. L. Culberson, J. M. Begovich, R. L. Graves, and W. K. Kahl, *ASPEN Modeling of Steam Bottoming Cycles for Gasification Combined Cycle Power Plants*, ORNL/TM-9558, in preparation.

11. J. M. Begovich, O. L. Culberson, P. J. Johnson, and A. A. Khan, "Process Analysis," Sect. 6.3 in *Fossil Energy Program Quarterly Progress Report for the Period Ending June 30, 1983*, ORNL-5990, August 1983.

12. J. M. Begovich, O. L. Culberson, P. J. Johnson, and A. A. Khan, "Process Analysis," Sect. 6.3 in *Fossil Energy Program Quarterly Progress Report for the Period Ending September 30, 1983*, ORNL-6008, November 1983.

13. Ashland Synthetic Fuels, Inc., *H-Coal Pilot Plant. Final Report*, DOE/ET/10143-T37, April 1984.

14. J. M. Begovich, P. J. Johnson, and C. O. Price, *Verification of the H-Coal Pilot Plant Material Balance Yield Calculations*, ORNL/TM-9406, in preparation.

nitrate burden and its attendant cleanup costs, an analysis was made of methods for eliminating nitrates in the waste discharge from these systems.

After a brief study of the demineralizers for the Oak Ridge Research Reactor (ORR) and High Flux Isotope Reactor (HFIR), the following topics were recommended to the DEM for further study: (1) changing the regenerant acid from nitric to either sulfuric or hydrochloric, (2) having a commercial vendor of deionized water service the ORR and HFIR, and (3) directing all acid regenerant rinsewater of the ORR and HFIR pool and primary coolant demineralizers to the LLW evaporator.

The first option listed above would require that new materials of construction be used in the ion-exchange columns and piping. The second option is currently being used by several groups in the Oak Ridge Complex, but an economic analysis comparing costs of the various options needs to be done. The third recommendation appears reasonable if the LLW evaporator bottoms can be sent to hydrofracture.

Another brief study undertaken to assist the DEM concerned the appearance of ^{90}Sr in the ORNL sewage system. A nomograph was prepared so that the amount of ^{90}Sr holdup time in White Oak Lake could be predicted as a function of creek flow rates and current lake elevation.¹⁵ Use of this figure allows a prediction of the time required for White Oak Lake to achieve a given change in water elevation. The nomograph may also be used to determine the flow rate, the final lake elevation, or the initial lake elevation, given the other variables involved. For example, given the initial elevation, the creek flow rate, and the desired number of days for the elevation change, the resulting final elevation may be found.

During the course of this work, it was also determined that the flowmeters for the effluent from the holding ponds were in need of more frequent calibration than is currently done, so that flows will be accurately measured.

Direct Liquefaction Technology Assessment

During this report period, our assessment of the technology for direct liquefaction of coal was completed. The primary objective of the study was to determine the technical readiness of the direct coal liquefaction processes for commercial application. The study consisted of two major tasks. Task 1 was a technological assessment of the principal core

process operations of direct liquefaction. These included coal dissolution, coal-slurry preheating, high-pressure vapor/liquid separations, vacuum distillation of heavy residues, handling and utilization of the residues, and product upgrading. Critical equipment components such as high-pressure pumps, block valves, letdown valves, and instrumentation were also reviewed. Task 2 considered the problems involved in combining these core operations with the hydrogen supply and other ancillary processes and auxiliary systems needed to form a complete, integrated, coal liquefaction facility.

In our final report for Task 1, we concluded that the technology for those process elements which have been studied is sufficiently developed so that commercial-scale equipment can be satisfactorily designed and operated.¹⁶ Yields can be reasonably predicted, and adequate onstream efficiency can be realized. In the areas of coal dissolution and coal-slurry preheating, the potential exists for improvements in technology that would substantially increase yields and/or reduce equipment costs. For example, better coal dissolution techniques could reduce the amount of regressive reaction (polymerization of coal molecule fragments), and development of a means to supply multiple furnace tube-passes from a single pumping source could reduce preheating costs.

The final report of Task 2 discusses the problems involved in making the transition from pilot-plant demonstration of direct liquefaction processes to the design of complete, integrated, commercial-scale facilities.¹⁷ The technical and economic trade-offs in plant integration are illustrated by reference to three major direct liquefaction processes: Exxon Donor Solvent, H-Coal, and SRC-I. The design considerations involved in combining core liquefaction processes with hydrogen supply, ancillary processes, and utilities are discussed in detail. Plant startup problems and the measures needed to ensure adequate plant reliability are also discussed.

15. O. L. Culberson, Oak Ridge National Laboratory, personal communication to J. M. Begovich, 1984.

16. A. R. Irvine, H. D. Cochran, O. L. Culberson, J. F. Fisher, W. R. Gambill, G. E. Oswald, and R. Salmon, *Direct Liquefaction Technology Assessment, Task 1, Technical Readiness of the Developing Plant Functions*, ORNL/TM-9181, in preparation.

17. R. Salmon, *Direct Liquefaction Technology Assessment, Task 2, Plant Integration*, ORNL-6043, July 1984.

Economic Evaluation of Coal-to-Methanol Processes

A report on the economics of methanol production from coal has been submitted to DOE/FE in draft form.¹⁸ Comments will be obtained from other external reviewers prior to publication. The report, presenting a general overview and update of the costs of producing methanol from coal, was prepared by using various published design studies and other sources of information. Costs of conventional methanol production from natural gas are included as a basis for comparison. Sensitivities of costs to economic/financial parameters are examined in detail. The general conclusion reached was that methanol from coal is nearly competitive with methanol from natural gas for use as a motor fuel blending stock, provided that favorable financing arrangements and guarantees can be obtained. However, current weakness in the markets for

methanol and motor fuels makes any near-term, commercial, coal-based projects doubtful. The technology for producing methanol from coal is available and is not in question. The economics will probably become more favorable as world economic growth proceeds and as supplies of petroleum and natural gas become inadequate to meet fuel demands.

Support to the Oak Ridge Complex

Our group has also produced ASPEN simulations of nuclear flowsheets for the EURI and BRET projects. Since this information is considered either classified or applied technology, the details of these simulations are not reported here.

18. R. Salmon, *Economics of Methanol Production from Coal*, ORNL-6091, in preparation.

12. MISCELLANEOUS PROGRAMS

Because of its technical expertise and experience in many areas, Chem Tech has become involved in numerous programs of a varied nature at ORNL, DOE, other governmental agencies, and even with private firms and organizations. Although these efforts may not individually represent a large portion of our activities, many of these programs are extremely important contributions to the agency, to the national program, and to the technical development and growth of the staff members involved. These efforts are viewed as essential parts of our mission to develop and to transfer technology.

12.1 FUSION ENERGY STUDIES

This program involves work with the Fusion Energy and Engineering divisions on the design and fabrication of pellet injectors for the Tokamak Fusion Test Reactor (TFTR). These injectors fabricate up to 4-mm-diam cylindrical pellets of solid hydrogen isotopes and accelerate them to velocities approaching 1.5 km/s with the use of a pneumatic gun. Pellets of this size and velocity are effective for fueling the Tokamak plasma. During this report period, we have designed systems for gas handling, tritium handling, and tritium control. We have also made contributions in the areas of radiation effects, heat transfer, vacuum system design and performance evaluation, and safety analysis. The Repeating Pneumatic Injector has been installed and successfully operated on the TFTR. The Deuterium Pellet Injector, an eight-shot device, is presently being fabricated. The Tritium Pellet Injector design is nearly complete, but this phase is on hold, pending the determination of tritium usage in the TFTR.

12.2 THREE MILE ISLAND SUPPORT STUDIES

The involvement of Chem Tech and other ORNL staff members with recovery operations at the Three Mile Island Unit 2 Nuclear Power Station (TMI-2) has continued since shortly after the

accident in 1979. In addition to the active participation of several of its members, the Division has managed all TMI-2 assistance provided by personnel at the DOE facilities in Oak Ridge, Tennessee.

Early assistance projects were coordinated through the Technology Advisory Group (TAG). The most significant of these projects was the development by Chem Tech and Savannah River Laboratory personnel of the process used for (1) decontaminating the high-activity-level water (HALW) and (2) concentrating the radioactive contaminants into a readily disposable form.¹ The use of the process to decontaminate $\sim 3000 \text{ m}^3$ of HALW that had spilled into the reactor building basement and that remained in the Reactor Coolant System (RCS) was completed in 1982. The process is still being used to clean up miscellaneous batches of water. The use of a similar process is planned during defueling operations.

In December 1981, the Technical Assistance and Advisory Group (TAAG) was established to assist the TMI-2 operating utility, GPU Nuclear (a subsidiary of General Public Utilities Corporation), and its primary contractor for recovery operations, Bechtel National Corporation. The TAAG consists of a variety of consultants from the nuclear power industry, the nuclear naval service industry, the Electric Power Research Institute (EPRI), and the DOE national laboratories. One of the ten members of the TAAG is an ORNL staff member in Chem Tech. While the scope of advice and assistance provided by the TAAG is very broad, primary interests are on reactor disassembly, defueling operations, and facility decontamination. Perhaps the most important contribution has been the suggestion and direction of video examinations of the reactor core, which led to the discovery of

1. E. D. Collins, D. O. Campbell, L. J. King, J. B. Knauer, and R. M. Wallace, "Development of the Flowchart Used for Decontaminating High-Activity-Level Water at TMI-2," *ACS Symp. Ser.*, in preparation.

the large void area and the pile of rubblized fuel and core components. Recently (February 1985), the TAAG sponsored another video examination. This time the subject of interest was the area beneath the reactor core. The video exam showed the presence of a large mass of core materials (estimated to be ~20% of the total core) that appeared to have been melted during the accident. These discoveries have been especially important for determining future defueling methods and equipment that will be required.

Recovery operations have progressed steadily at TMI-2. The GPU Nuclear/Bechtel recovery team completed removal of the reactor vessel head in July 1984. In December 1984, the plenum structure was raised ~19 cm (~7.5 in.) to clear mechanical obstructions in preparation for plenum removal in May 1985. The first defueling operations (vacuuming of the rubble bed) are scheduled to start in October 1985. Decontamination of facilities in the Auxiliary Building and in the upper levels of the Reactor Building have continued, using mechanical surface removal and water flushing methods.

Support projects recently carried out by Chem Tech personnel have included (1) a study to compare the behavior of radiocesium during the TMI-2 accident with behavior during reactor accident experimental work at ORNL,² (2) the analytical characterization of resins removed from the TMI-2 Makeup and Purification System Demineralizers, and (3) the development of a method for elution of the radiocesium from the demineralizer resins.³

Support projects recently carried out in other ORNL divisions included (1) the development of a mathematical model of the degraded TMI-2 core and the use of this model to describe the boron concentration necessary in the RCS to ensure subcriticality during defueling operations (Computer and Telecommunications Division),⁴ and (2) the measurement of the response time and other characteristics of resistance thermometers that were in the TMI-2 RCS during the accident (Instruments and Controls Division).⁵

12.3 FUSRAP STABILIZATION TECHNOLOGY: DEVELOPMENT AND APPLICATIONS

During the period 1957-1966, the Feed Materials Plant at Weldon Spring, Missouri, was used to process uranium and thorium ore concentrates to pure uranium trioxide, uranium tetrafluoride, and

uranium metal for the Atomic Energy Commission (AEC), predecessor organization of the Energy Research and Development Administration, and, currently, the DOE. The raffinate waste streams associated with these operations were discharged to a series of nearby earthen pits, which have proven to be remarkably efficient in containing and controlling the contaminants. Because of these conditions, it is prudent to establish the utility of one or more of these partially filled pits for the storage of other wastes with similar characteristics. This work summarizes the available data on the geologic and hydrologic characteristics of these pits and their surroundings, with emphasis on the suitability of the geohydrological environment to retain additional quantities of radioactive wastes.

Facility Location and Description

The Weldon Spring Plant is situated on a 220-acre tract of land in St. Charles County, Missouri, approximately 40 km (25 miles) west of St. Louis and 21 km (13 miles) southwest of the city of St. Charles.⁶ Locally, the facility lies in the midst of several large tracts of public lands and properties controlled by the U.S. Army. Except for some decontamination activities associated with an aborted effort by the U.S. Army to produce war-related chemicals in the late 1960s, the plant has remained in a caretaker status since AEC operations ceased in 1966.⁷ Responsibility for the Plant was assigned to the U.S. Army in 1967; however, the AEC later reacquired ownership of the 52-acre tract of land comprising the raffinate pits,

2. R. A. Lorenz, J. L. Collins, O. L. Culberson, D. O. Campbell, and R. S. Lowrie, *The Behavior of Fission Product Cesium in the TMI-2 Accident*, Draft Report, in preparation.

3. W. D. Bond, L. J. King, J. B. Knauer, K. J. Hofstetter, and J. D. Thompson, "Cleanup of the TMI-2 Demineralizers," *ACS Symp. Ser.*, in preparation.

4. R. M. Westfall, P. B. Fox, J. R. Knight, D. W. Hermann, and J. C. Turner, *TMI-2 Criticality Studies: Lower Vessel Rubble and Analytical Benchmarking*, ORNL/CSD/TM-222, 1985.

5. H. M. Hashemian, T. W. Kerlin, R. L. Shepard, R. M. Carroll, and K. E. Holbert, *Status of TMI-2 Primary RTDs During and After the Accident*, ORNL-AMS8304R1, September 1983.

6. *Weldon Spring Raffinate Pits and Quarry Task Force Report*, June 1, 1967.

7. *Assessment of Weldon Spring Chemical Plant in St. Charles County, Missouri*, Department of the Army, Office of Department of Army Project Manager for Chemical Demilitarization and Installation Restoration, Aberdeen Proving Grounds, Maryland, Mar. 1, 1976.

as well as a quarry located about 6.5 km (4 miles) south of the pits that was used for the disposal of solid, low-level radioactive wastes.

The predominant operations at the Weldon Spring Plant entailed the conversion of uranium ore concentrates and recycled scrap to pure uranium trioxide, uranium tetrafluorides, and uranium metal. These processes generated several chemical and radioactive waste streams. The most important of the streams were the raffinate streams from the refinery operation and the magnesium fluoride slurry stream from the uranium recovery process. These streams were pumped to the pits, where the solids settled out and the supernatant liquids drained to the plant process sewer (see Fig. 12.1). The solids remaining in the pits consist of silica and other insolubles associated with the feed materials, along with hydroxides and other precipitates formed from the neutralization of the raffinates. Some washed slag residues from the uranium metal production operation were also discharged to the pits. In general, the contents of all

four pits are similar; however, since Pit 4 was used predominantly during the time of thorium processing, it contains a much greater abundance of these residues than the other pits. Soon after the feed materials plant was closed in 1966, samples of sludge were obtained from each pit to ascertain their chemical and radiochemical contents. A homogenized mixture of samples was analyzed for the characterization of the contents (see Table 12.1).

In general, all of the pits were constructed in the same manner, which consisted of excavating into the underlying clay till so that the bottoms, as well as the side walls and/or levees, were composed of "impermeable" clay. In some areas of Pits 3 and 4, the lower extremities of the pits are near to the original land surfaces; thus, levee walls having slopes of 1.5:1 to 2.5:1 were constructed. Because of the desire to maintain a constant elevation for the levees around all pits, some portions of Pits 3 and 4 were constructed with levees that rise up to 10.7 m (35 ft) above the existing grade.

Fig. 12.1. Plot of raffinate pit areas at the Weldon Spring Plant.

Table 12.1. Analyses of composite sample of residues from Pits 1, 2, 3, and 4

General		
pH	9	
Solids (undissolved)	20%	
Solids (dissolved)	5%	
Total acids	5%	
Water content	75%	
Elemental constituents		
<i>Dissolved</i>	<i>Undissolved</i>	
Calcium	Calcium	
Sodium	Magnesium	
Magnesium	Silica	
	Iron	
	Aluminum	
Dissolved chemicals		
Nitrates	3.5%	
Sulfates	0.3%	
Chlorides	0.01%	
Radioactivity		
<i>Source materials</i>		
Uranium	0.15%	
Thorium	0.05%	
Thorium (Pit 4)	1.5%	
Activity	<i>Dissolved</i> ($\mu\text{Ci/mL}$)	<i>Undissolved</i> ($\mu\text{Ci/g}$)
Alpha	1.5×10^{-6}	5.0×10^{-2}
Beta	5.5×10^{-5}	5.0×10^{-2}
Gamma	1.0×10^{-5}	2.0×10^{-3}
Radium	1.0×10^{-6}	1.7×10^{-3}

Geologic and Hydrologic Conditions

Physiographically, the Weldon Spring Plant lies at the southeasternmost end of the Dissected Till Plains Section of the Central Lowland Province. The area is characterized by gently rolling topography and slow, meandering streams. The underlying bedrock consists of rocks from the Paleozoic age which are covered with a moderately thick section of unconsolidated Pleistocene sediments. The generally flat-lying bedrock consists primarily of limestones and dolomites, with some sandstone and shales. These strata dip slightly to the northeast ($<1^\circ$) as a result of doming along the Ozark Uplift lying to the south. Although these rocks show little or no faulting in surface outcrops, joint sets trending northeast and northwest occur in the rock units immediately beneath the residue pits

at Weldon Spring. Typically, a 0.3- to 0.6-m (1- to 2-ft)-thick layer of loess is found at the surface in the area of the pits. This loess layer grades into overburden material of a section of mostly silty clay till that varies from 5.1 to 16.8 m (17 to 56 ft) in thickness.⁸ A thin bed of weathered limestone residuum occurs at the base of the till. This material, which grades into limestone, is predominantly clay but contains an abundance of chert and some limestone fragments. The uppermost bedrock unit at the site is the Burlington-Keokuk formation of Mississippian age, which is between 45 and 60 m (150 and 200 ft) in thickness. This massive-bedded limestone is cherty and, in some areas, dolomitic. It is also characterized by a somewhat irregular-pinnacled surface that contains numerous solution-caverns and clay-filled pockets. Other rocks from the Mississippian age, in descending order, are the Fern Glenn limestone [22.5 m (75 ft) thick], the Chouteau limestone [7.5 m (25 ft) thick], the Hannibal shale [<1.5 m (5 ft) thick], and the Sulphur Springs sandstone [2.4 to 9 m (8 to 30 ft) thick]. A major unconformity marks the contact between the Sulphur Springs sandstone and Kimmswick limestone of Ordovician age. The 15-m (50-ft)-thick Kimmswick formation is a coarsely crystalline limestone that is white to light gray in color and is medium to massively bedded. Corals, brachiopods, and cephalopods are numerous throughout the unit, as are chert modules. Immediately underlying the Kimmswick is a gray-to-dark-gray, fine-to-medium-grained limestone known as the Plattin Formation. It is ~ 30 m (100 ft) thick in the Weldon Spring area. The Joachim dolomite occurs next in descending order and is between 21 and 27 m (70 and 90 ft) thick. Some thin shales are interbedded in this unit, along with some massive dolomite and a rather thick sandstone bed near its base with the St. Peter sandstone. The St. Peter sandstone is widespread in the Mississippi Valley and often serves as an important source of groundwater. The formation, which is ~ 39 m (130 ft) thick at the pits, is predominantly a fine-to-medium-grained quartz sandstone.

Groundwater in the vicinity of the pits is generally confined to the Burlington-Keokuk limestone

8. R. M. Richardson, *U.S. Geol. Surv. Rep.*, June 1960.

and deeper rocks.⁹ Indeed, the watertable lies some 18 m (60 ft) below the pits, with the glacial till overburden lying in the unsaturated zone. Although the Burlington-Keokuk is the nearest water-bearing rock underlying the pits, the water is generally hard and in limited supply. The Sulphur Springs formation, lying some 90 m (300 ft) below the bottom of the pits, is a major source of potable groundwater in the area as is the St. Peter sandstone located at a depth of ~210 m (700 ft).⁹ Surface drainage from the pits is north toward the Mississippi River. Thus, the groundwater and surface water from the area would flow into Dardenne Creek and its tributaries. Because of the impermeability of the glacial till overburden, little infiltration of rainfall occurs in the pit area.

Summary of Results

Since the glacial till is instrumental in the containment and control of the raffinate residues and would also be critical to the retention of additional wastes that might be emplaced in the pits, detailed data on the characteristics of this material have been obtained through exploratory drilling programs. Indeed, sample borings of the till show it to consist primarily of silty clay, with some sandy clay occurring in the lower half of the section. Tests on "undisturbed" samples of the till show permeabilities of 6.4×10^{-9} cm/s at 2.7 m (9 ft) below the bottom of Pit 4, 1.7×10^{-8} cm/s at 3 m (10 ft), and 8.3×10^{-9} cm/s at 3.2 m (10.5 ft). The moisture content of these deposits was found to be ~20%, which indicates that rather small quantities of rainfall infiltrate into the subsurface at the pit site. These data indicate that the pits are likely to be rather impermeable to the downward or lateral migration of raffinate residues. Furthermore, since only a minimal amount of rainfall infiltrates into the clay till, little leaching of the wastes occurs. The quantity of rainfall does not exceed the quantity of evaporation in the Plant area; thus, water will not tend to accumulate from rainfall in the catchment area of the pits.

12.4 MOLTEN SALT REACTOR DECOMMISSIONING

The Molten Salt Reactor Experiment (MSRE) was a homogeneous-fueled reactor built to investigate the practicality of the molten salt reactor concept for central power station application. It was operated from June 1965 to December 1969. The

circulating fuel solution was a eutectic mixture of lithium and beryllium fluorides, containing uranium fluoride as the fuel and zirconium fluoride as a chemical stabilizer. The initial fuel charge was highly enriched ^{235}U , which was later replaced with a charge of ^{233}U . Following reactor shutdown, the fuel salt was drained into two critically safe storage tanks and isolated in a sealed hot cell, along with a third tank containing the flush salt. Table 12.2 summarizes the properties of these salts and the stored secondary coolant salt, which is not radioactive.

This facility, along with the stored salts, is supervised by the Surplus Facilities Management

Table 12.2. Stored MSRE salts

	Fuel salt	Flush salt	Coolant salt
Total mass, kg	4650	4290	2610
Volume, ^a ft ³	65.4	69.9	42.5
Composition, mol %			
LiF	64.5	66	66
BeF	30.3	34	66
ZrF ₄	5.0		
UF ₄	0.13		
Uranium content, kg			
^{233}U	<i>b</i>	<i>b</i>	
^{235}U	30.82	0.19	
^{238}U	2.74	0.02	
^{235}U	0.85	0.09	
^{238}U	0.04	0.00	
^{239}U	2.01	0.19	
Total	36.46	0.49	
Plutonium content, g			
^{239}Pu	657	13	
^{240}Pu	69	2	
Other Pu	2	0	
Total	728	15	
Lithium composition, %			
^6Li	0.001	0.009	
^7Li	99.991	99.991	

^aAt room temperature.

^bPresent at 220 ppm, U-basis.

9. Preliminary Investigation of Groundwater Occurrences in the Weldon Spring Low-Level Radioactive Waste Disposal Site, Weldon Spring, Missouri, Lawrence Berkeley Laboratory, LBL-11481, September 1980.

Program and is scheduled for eventual decommissioning. However, the presence of the highly radioactive fluoride fuel salt (and, to a lesser degree, the flush salt) has created some unique problems. Plans for site decommissioning will first have to address the issue of disposition of the fuel. Early studies generally assumed that the stored fuel would be removed from the MSRE cells [with or without reprocessing (fluorination) to strip out the uranium and some of the fission products] and then taken to a final repository or to retrievable storage. However, these operations are complex and potentially hazardous and, therefore, expensive. Thus far, no need has been demonstrated for the recoverable ^{233}U or for the cell space. Therefore, there has been no incentive from that direction to proceed with decommissioning. Further, there is no known site that will accept the fluoride fuel salt for disposal in its present condition, either now or in the near future. Thus, regardless of future possibilities, needs, or decisions, it appears that the solidified salts must continue to be stored in their present mode and location. Fortunately, the salts, their containment, and the facility are in excellent condition to do so. It is essential, however, to address two issues not directly considered in prior studies:

1. Is it rational to continue storing these salts in their present situation and, if so, what should be done to enhance this storage in terms of safety and stability for an extended period of time?
2. What other options are there for the future, and what steps should be taken in the near term to facilitate future decision making?

A recently completed feasibility study gives an affirmative answer to the first question and identifies specific areas where improvements should be made.¹⁰ It also identifies needs that should be addressed in the near term to provide necessary information for future selection among available options. The following sections outline three of the major areas of the feasibility study: a review of available options, projections of radioactivity out to 1 million years, and the summary and conclusions.

Prior studies of MSRE decommissioning have focused on the final disposition of both the salts and the facility. Selection among options involved many possible decision points, including the following:

1. Should the salt be chemically processed to separate the uranium from the bulk of the fluoride salts?
2. Should the fluoride salts be chemically converted to another form?
3. Should the salts be packaged and sent to an off-site repository, or sent to an on-site storage/disposal area, or disposed of in the MSRE facility itself?
4. Should the MSRE facility be totally or partially dismantled and portions entombed in concrete?

At present, no sound basis exists for making selections among these options. Options 1 or 2, if exercised, require sophisticated chemical processing of highly radioactive materials; new flowsheets and processing equipment would be required (and this would ultimately also have to be disposed of). The cost of processing has been estimated at roughly \$20 million. No choice can be exercised on Option 3 at this time because no off-site or on-site location is available to accept this material, and we have no basis upon which to recommend permanent disposal in the MSRE facility. Option 4 could cost as much as \$10 million (for partial dismantlement, plus entombment) and requires a prior decision on Options 1, 2, and 3. In short, at this date, we need to "buy time." Continued storage of the fuel and flush salts is forced upon us by external conditions. The issues of concern, then, revolve around three aspects:

1. Is it reasonable and safe to plan continued storage-in-place for up to 20 or so years?
2. Before deliberately embarking on such extended storage, what (if anything) should be done to enhance the storage environment?
3. During this storage period, or prior to it, what should be done to facilitate a future decision of a final nature?

In order to gain a sense of perspective, it is instructive to define a range of options and to systematically evaluate these options against various criteria. The six options that have been treated this way as a part of this evaluation are listed and defined in Table 12.3. Options 1, 2, and 3 focus on

10. K. J. Notz, *Feasibility Evaluation of Extended Storage-In-Place of MSRE Fuel Salt and Flush Salt*, ORNL/TM-9756, September 1985.

Table 12.3. Options and evaluations for decontamination and decommissioning

Option	Description	Score ^e		
		A ^b	B ^c	C ^d
0	Continue as-is, with no decision. Requires continued surveillance and maintenance, introduces maximum uncertainty, and eventually still needs a decision between the "real" options outlined below.			
1	Removal of fuel and flush salts; complete dismantlement of hot cells.	15	185	5
2	Removal of fuel and flush salts; entombment of drain tank cell.	11	147	4
3	Entombment of drain tank cell, with fuel and flush salts in-place.	3	62	2
4	Enhanced near-term storage of solidified salts (which still leaves all other options available).	3	69	0
5	Enhanced storage in-place, including remelt and addition of getters (which still leaves Options 1, 2, 3, and 6 available).	4	78	0
6	Enhanced storage in-place, including remelt, addition of getters, and repackaging (which still leaves Options 1, 2, and 3 available).	6	98	0

^aIn all cases, a low score is superior.

^bNumber of process steps involved.

^cSemiquantitative rating of process steps.

^dNumber of limiting factors.

the final decision and bracket a wide range of possibilities; this viewpoint provides a broad, long-term perspective. Options 4, 5, and 6 focus on three variations of enhanced storage-in-place in order to bring near-term needs into view. Evaluation was done on the basis of three categories:

- A. a qualitative comparison of the process steps involved;
- B. a semiquantitative evaluation relative to process readiness, economics, near-term hazard, long-term hazard, and conservation (i.e., beneficial utilization of the buildings and contained materials); and
- C. an identification of the limiting factor or factors.

The first evaluation simply lists the major process steps that would be required for the various options. The steps were defined in such a way that each step represents a more-or-less equivalent effort or cost. Evaluation then simply requires counting the number of steps for each option (Column A in Table 12.3). From this point of

view, Options 1 and 2 are prohibitively complex (and costly) and would not be selected unless an overriding reason existed for doing so.

The second evaluation involves "scoring" each option for each applicable process step. Scoring was done on five different bases, as listed above under evaluation (B). These results are summarized in Column B of Table 12.3. On this basis, Options 3, 4, and 5 are distinctly superior to the other three, with Option 6 a possibility.

The third evaluation identifies limiting factors which, in their own right, would disqualify an option from further consideration at this time. These are factors which, if not within an acceptable range, would completely scuttle that option no matter how favorable other aspects of that option might be (Column C of Table 12.3). On this basis, Options 1, 2, and 3 should not be considered further. In summary, in terms of these three evaluations, and at this time:

- Options 1 and 2 are rejected on the basis of all three evaluations;

- Option 3 is rejected on the basis of its limiting factors; and
- Options 4 and 5 would be available for near-term implementation (and possibly Option 6).

The original decay calculations, done with ORIGEN and using MSRE-specific input data, were truncated at 5 years. However, any decisions regarding permanent emplacement require decay data out to 1 million years, as well as a detailed knowledge of radiological properties in the 10- to 10,000-year time span. Ideally, the entire calculation (generation and depletion) should be repeated using ORIGEN2—the updated and improved version of ORIGEN. However, ORIGEN2 does not have a molten salt reactor model, and it would be prohibitively expensive to construct such a model. So far, what has been done is to take the old ORIGEN output data at discharge and then do the decay calculations with ORIGEN2. In cross-checking these two sets of data at 5 years after discharge, only inconsequential or readily explainable differences were noted. This knowledge gives us confidence in using the ORIGEN results as input to ORIGEN2 decay, which were generated for these time spans (years): 1, 10, 10^2 , 10^3 , 10^4 , 10^5 , and 10^6 . Figure 12.2 shows the total fission product (FP) activity and the major contributors out to 1 million years. Figure 12.3 shows the total actinide activity and the major contributors, also to 1 million years. Both drawings are to the same scale. The former is essentially all beta-gamma, while the latter is mostly alpha. The actinide decay chain does include ^{208}Tl (from the ^{232}U chain), which is noteworthy because of a very energetic gamma (2.6 MeV) accompanying the beta decay to ^{208}Pb . In both cases, relatively few isotopes contribute most of the activity over rather distinctive time spans. Two observations are particularly noteworthy:

1. FP activity dominates at present (by a factor of 15) but will decline by a factor of 10 in the next 100 years and be at a minimal level after 1000 years.
2. Actinide activity, presently controlled by Pu and the ^{232}U chain, is decaying slowly, while the ^{233}U chain is very slowly growing in. The result is a minimum in alpha activity at 1000 years, along with the depletion of the ^{208}Tl gamma, followed by a maximum in alpha activity at about 40,000 years, after which permanent decay occurs.

Fig. 12.2. Fission product radioactivity of MSRE salts.

Fig. 12.3. Actinide radioactivity of MSRE salts.

The total activity has decayed dramatically since discharge, from 1.8 million Ci to 34,000 Ci. Most of this decline is from the depletion of short-lived FPs which accounted for 200,000 Ci 1 year after discharge, and now (after 15 years) are at 32,000 Ci. The actinides have decayed slowly—from 2,550 Ci at discharge, to 2,520 Ci after 1 year, to 2,000 Ci after 15 years.

Neutron activity of the salts derives almost equally from (α, n) reactions and spontaneous fission (S.F.). At present, there are about 1.6×10^6 neutrons/s from each of these two sources. This level fluctuates a bit with time and eventually declines after 100,000 years (as the alpha activity itself declines).

The transuranic alpha activity of the salts is relatively high—about 70,000 nCi/g in the fuel salt and about 1,000 nCi/g in the flush salt. Removal of the uranium (and neptunium) by fluorination should lower the activity of the flush salt below the threshold level for TRU waste, but the fuel salt would still be a TRU waste.

The following conclusions were drawn:

1. Extended storage is not only feasible, but preferable at this time because:
 - a. the fuel salt is located in a safe place;
 - b. no repository or disposal site is available to receive the waste if it were removed, either with or without reprocessing;
 - c. the full range of future disposal options or locations is not yet known;
 - d. any enhancement actions taken now would probably benefit whichever option is finally exercised; and
 - e. extended storage would provide time to collect additional data that would be of direct help in selecting a final option at a future date.
2. Entombment should be deferred at this time because:
 - a. it is not necessary;
 - b. it is essentially nonreversible; and
 - c. additional technical data are needed before such a decision could be supported.
3. Some enhancements in direct support of extended storage should be evaluated in the near future:
 - a. cell penetrations should be reviewed by Martin Marietta Energy Systems, Inc., Engineering, inspected by Operations Division, and, where deemed necessary, sealed permanently;
 - b. cell internals should be reviewed to identify interfering structures, if any, and plans made for their removal;
 - c. remelting of the fuel salt, with possible addition of a fluorine getter (and possibly a neutron poison), should be evaluated for implementation, since this procedure would eliminate both the need for annual reheating and long-term concerns about radiolysis-produced fluorine;
 - d. repackaging (if it is to be done at all) should be done in conjunction with remelting; this step should also be evaluated;
 - e. enhancements seem prudent since the alpha activity (and the resulting neutrons from α, n reactions) will decline very little in future years, even though the gamma activity will drop significantly;
 - f. these enhancements would not interfere with the exercise of options at a later date, and would, in the interim, improve on the already sound and secure condition of the facility;
 - g. these enhancements, while not necessary for continued short-term storage, should be done prior to initiating extended storage while the assured capability to do so still exists, in terms of both people with first-hand knowledge and equipment that is still functional.
4. Pertinent data or studies should be obtained over the next few years in these areas:
 - a. observations of groundwater levels around the site;
 - b. possible improvements in the area of civil engineering to lower the groundwater level;
 - c. Hastelloy N corrosion behavior in the presence of moisture plus alkali and/or fluoride;
 - d. suitability of candidate materials for use as a fluorine getter, in-cell desiccant, tailored grout, and neutron poison.
5. Beneficial utilization of the facility should be encouraged and aided because:
 - a. it will help satisfy our own facility requirements;
 - b. the office space at the site is fully utilized today, and this need is expected to grow;
 - c. the high bay area is being used to store bulky, mildly radioactive samples, and this need is expected to continue;

- d. the three more active hot cells (reactor, drain tank, and reprocessing) could be used for storage of other radioactive waste;
- e. the remaining cells could be used for hot operations or radioactive waste storage; and
- f. the ^7Li content of the cooling salt is non-radioactive and may have enough value to justify recovery.

Based on the above considerations, a work plan for FY 1986 and beyond is being planned.

12.5 RECOVERY OF ALUMINA AND OTHER USEFUL MINERALS FROM FLY ASH

Results from the initial ORNL study of alumina recovery from fly ash reported in the last Chem Tech progress report indicated that, although simple, direct acid leaching of ash would not dissolve large portions of the aluminum in many ashes. The cost, however, for recovering the 30 to 50% of the aluminum by this route was lower than that required for higher recovery by alternative, more aggressive approaches. Lower recovery does not harm the economics greatly because of the low value of the fly-ash feed material. After several firms reviewed the preliminary economics presented in the initial ORNL report, EPRI subsequently initiated two additional studies.

The first study included further laboratory tests by ORNL to evaluate some of the more critical process steps and tests by commercial vendors to determine if unforeseen technical problems could be found. A more-detailed economic analysis was prepared by Raymond Kaiser Engineers, Oakland, California—a commercial engineering firm experienced in alumina technology. The efforts focused upon a single representative fly ash, that from the Kingston Steam Plant of the Tennessee Valley Authority (TVA). Although the Kingston plant does not produce as rapidly as needed for the reference design, sufficient additional ash is "stored" in ash ponds to make up the required feed materials for several decades. ORNL provided laboratory support for the study and prepared samples of materials for vendor testing. The results of that work¹¹ essentially confirmed the general conclusions of the earlier ORNL study, but the economic analysis demonstrated a clear need to find uses for

the large quantities of solid wastes produced by the process. Although the wastes are believed to be nonhazardous (most potential problem materials have been removed) and present no disposal problems, the large quantity of materials do have potential value, and the economics need some input from the sale of these materials. Furthermore, removal of all or most of the waste products may be important in attracting interest from many utilities. Kaiser Engineers has found several potential uses for the solid residue from the processing of fly ash, which include uses in cement and as industrial filler materials. The value of some potential uses is high enough that some additional processing of the waste materials may be justified. Such processing is likely to involve little more than physical separations.

The other and most recent EPRI study is still in progress and focuses its attention on two main areas of development: (1) the evaluation of potential uses of the solid residue(s) produced by direct acid leach of fly ash, and (2) a national survey of the ash available and suitable for processing. ORNL is providing samples of residue (waste) materials and evaluating ashes from several utilities throughout the country. Exploratory efforts are also being made to determine if relatively simple physical separations can isolate more valuable materials from leached ash residue, principally by removing iron and other "colored" materials from the residue. The reference ash for these studies is supplied by the Southern Company (a utility holding company), Birmingham, Alabama, and is generated by a plant in the Georgia Power system. More than twenty other utility companies have contributed smaller samples that are being tested to determine their composition and aluminum leachability. The results of these tests will provide a basis for estimating the availability of ash suitable for mineral recovery.

12.6 STUDIES RELATED TO POSTULATED ACCIDENTS AT THE SAVANNAH RIVER PRODUCTION REACTOR

The purpose of this analysis is to provide an estimate of the rate of organic iodide generation in the

11. R. F. Wilder, P. J. Barrett, L. W. Henslee, Jr., and D. Apri, *Recovery of Metal Oxides from Fly Ash. Volume 1: Executive Summary—Financial Evaluation*, EPRI-CS-3544, vol. 1, June 1984.

reactor room of the Savannah River Production Reactor (SRPR) under two postulated reactor accident conditions: (1) a relatively mild event involving localized damage to ~1% of the core and (2) a more severe event involving complete core degradation. The results will be utilized in an evaluation of the existing organic iodide filtration system for the SRPR.

A mathematical model has been developed to predict the net rate of organic iodide production under the postulated accident conditions. The model is based upon a description of the reactor room conditions during the accident, as provided by Savannah River Laboratory (SRL), and upon an ORNL review of iodine reactions to organic iodides.

In the scenario addressed by the model, iodine is suddenly released in the reactor room (volume ~9000 m³), where organic iodides may be formed by the reaction of iodine with organic species in the atmosphere and on the room surfaces. An outside air supply, which replenishes the room atmosphere approximately every 8.5 min, carries the iodine species to a bank of filters and adsorbers. The iodine may also adsorb onto the various surfaces (metal, concrete, and paint) in the room, as well as be adsorbed into a pool of water on the room floor. This pool of water, formed by a water spray activated under the more severe accident conditions, drains to a storage tank.

Predictions of organic iodide production will be made as a function of the model parameters. These parameters include the reaction rate constant and the equilibrium constant for organic iodide formation, mass transfer rate, air supply flow rate, etc.

12.7 PROCESS MONITORING FOR COAL CONVERSION

Fundamental work related to process development in coal conversion has diminished rather sharply from that reported in the previous Chem Tech progress report.¹² This decrease has been partially due to a rather widespread diminution of research efforts on fossil energy at ORNL and elsewhere because of a reduced emphasis in the area on the part of the DOE. An apparent "glut" of oil on the market, coupled with the cancellation of coal conversion demonstration plants, has contributed to a lessening of interest in near-term fossil energy R&D. Current ORNL research related to coal conversion involves a continuing effort to

develop a rapid-monitoring capability with the potential to optimize deashing performance for the Kerr-McGee critical solvent deashing unit in operation at the Wilsonville, Alabama, pilot plant.

The previous divisional progress report described the development of a rapid two-solvent separation of feed to the Kerr-McGee critical solvent deasher (CSD). The feed sample, dissolved in pyridine, is dispersed onto a sand column employing chloroform as the initial eluent, followed by pyridine. The two fractions are frontally eluted through an optical detector (broad-beam source at 360 nm with a photoconductive detector). The chloroform solubles approximate an oil + asphaltene fraction, while the pyridine solubles represent a preasphaltene fraction of the CSD feed material. An automated (except for sample preparation and loading) analytical system was fabricated and installed in the control laboratory at the Wilsonville pilot plant. The system (see Fig. 12.4) consists of two high-performance liquid chromatographic pumps, an integrator-plotter, an ORNL-developed optical detector, a sample injection valve, a sand column (50 × 1.2 cm), and a programmable timer. The system, shown schematically in Fig. 12.5, is under complete operational control of the programmable timer. The operator is only required to rotate the injection valve twice—first, to start the system, and then to inject the sample. Its entire analytical cycle (sample elution with two solvents and return to initial conditions) is completed in 13 min, and the system automatically powers down awaiting the next sample injection. Operation by laboratory personnel on the pilot plant staff has shown the system to be easy to operate, rugged, and reliable over its 2-year operational existence.

Operating Results

Initial operating experience with the feed quality monitor (FQM) at Wilsonville, Alabama, demonstrated a good correlation between chloroform solubles and the Soxhlet-measured ratio [(oil + asphaltenes)/preasphaltenes]. However, Wilsonville operations changed from a nonintegrated, once-through solvent refined coal (SRC) process to an integrated, two-stage liquefaction (ITSL), or a doubly integrated two-stage liquefaction (DITSL)

12. Chem. Technol. Div. Prog. Rep. Apr. 1, 1981, to Mar. 31, 1983, September 1983.

Fig. 124. Photograph of CSD feed quality monitoring system.

process—either of which resulted in uniformly high SRC recoveries ($>90\%$). Such uniformly high SRC recoveries negated the need to monitor incoming feed to the CSD unit for purposes of process control. However, control and optimization of deashing in the first stage of the CSD unit to minimize energy rejection (loss of soluble carbon values with the ash underflow) and maintain smooth deashing operation have become more important to overall pilot plant operation.

The exact mechanism by which antisolvent deashing processes (of which the Kerr-McGee CSD process is just one example) work is not completely understood. However, Winschel and

Burke¹³ report that it is generally believed that part of the solubilized coal products precipitate upon addition of antisolvent (a paraffinic or highly alkylated aromatic oil). The precipitate coats the solids, increasing their diameter and causing them to settle faster. Wilsonville controls the deashing solvent (DAS) formulation by means of two solubility measurements performed in the laboratory. These tests, requiring ~ 2 h, are solubility measurements in the current DAS and in pyridine. The

13. R. A. Winschel and F. P. Burke, "Recycle Slurry Oil Characterization," *Second Annual Report for Period October 1, 1981-September 30, 1982*, DOE/PC/30027-39, 1983.

Fig. 12.5. Schematic diagram of CSD feed quality monitoring system.

ratio of these two solubilities, termed solvency index (SI), is employed as a control measure for the CSD unit. At least one sample per shift is analyzed, and the results are used to determine the need to alter the DAS.

The ORNL FQM, capable of analyzing a sample in <30 min, has the potential to be a significant labor-saving device if optically monitored results correlate with the standard beaker tests. The initial eluent on the ORNL FQM was changed from chloroform to a particular DAS without difficulty. Comparison of the manually measured SI with that determined on the FQM has shown reasonable correlation, especially when the CSD unit was undergoing gross changes. Current testing of this configuration at the Wilsonville pilot plant is concerned with a direct head-to-head comparison, using the same samples for both measurements on a once-per-shift basis over a total time period of ~ 2 weeks. These results will be employed to determine the feasibility of substituting the instrumental for the manual measurement in the pilot plant environment. This FQM system is amenable to full automation and on-line automation, which could result in further time savings and more expeditious availability of the data.

12.8 SUPPORT TO MARTIN MARIETTA ENERGY SYSTEMS, INC., ENGINEERING

During this report period, Chem Tech continued to work closely with Energy Systems Engineering on a number of projects. This initiative provides a service to both organizations, allowing a work-broadening experience for Chem Tech personnel and experts on loan for Engineering projects. Two of the Division's newly developed engineers spent ~ 1 year under Engineering tutelage as part of a transition from nonexempt to exempt status. Seven Chem Tech staff members are currently on loan to Energy Systems Engineering, working on projects at the Y-12 Plant and the ORGDP.

Technical Support to the GCEP

Work on the Gas Centrifuge Plant (GCEP) at Portsmouth, Ohio, is being stretched out as competing enrichment technologies maneuver for dominance. The enrichment program is currently in the process of evaluating and comparing advanced gas centrifuge technology with the atomic vapor laser isotope separation (AVLIS) technology as future enrichment options. Our personnel have participated in each phase of the process. The support

effort is expected to decline as the work elements are completed.

**Technical Support to the
Y-12 Safety Analysis Program**

Chem Tech is providing technical support to Energy Systems Engineering in assisting the Y-12 Plant Safety Analysis Department in the preparation of Final Safety Analysis Reports (FSARs) for some areas of the Y-12 production facilities. The primary Chem Tech responsibilities are the preparation of facility and process descriptions and the development and analysis of accident scenarios for a chapter in the FSAR.

**Technical Support to the Y-12 Enriched
Uranium and Plant Improvements Programs**

Chem Tech is providing technical support to Energy Systems Engineering in coordinating the renovation and upgrading of the existing chemical recovery equipment to meet the production needs and environmental protection requirements. This task entails working with the architect-engineer as well as doing original design work. In addition, state-of-the-art emission control systems are being designed and installed to control air emissions with DOE's ALARA criteria.

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AWARDS, HONORS, AND RECOGNITIONS

<p>E. C. Beahm
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 1985 Publications Award, Martin Marietta Energy Systems, Inc., Awards Night</p> <p>J. T. Bell
 1984-1985 <i>Who's Who in Frontier Science and Technology</i>, Marquis Who's Who
 1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.</p> <p>T. M. Besmann
 1983 Materials Science Research Competition Award, Significant Implication for Energy Technology Category, "Thermodynamic Investigations and Modeling of Energy-Related Chemical Systems," U.S. Department of Energy
 1983 Best Paper Award, Nuclear Division, American Ceramic Society
 1983 Appointed Membership Chairman, Nuclear Division, American Ceramic Society
 1983 Appointed to the National Public Information Committee, American Nuclear Society
 1984 Appointed to Program Committee, Nuclear Division, American Ceramic Society
 1984 Appointed Membership Chairman, Knoxville-Oak Ridge Section, American Nuclear Society</p> | <p>J. E. Bigelow
 1983 Union Carbide Corporation Community Service Award</p> <p>R. E. Blanco
 1981 Fellow, American Nuclear Society</p> <p>W. D. Box
 1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.</p> <p>C. A. Burtis
 1984-1986 Board of Directors, American Association of Clinical Chemists
 1985 Technical Communication Award of Distinction in Scholarly and Professional Articles Category, East Tennessee Chapter of the Society for Technical Communication
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 1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.</p> <p>D. O. Campbell
 1981 Advancements in Nuclear Technology Special Award, American Nuclear Society
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- S. D. Clinton**
1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.
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- L. K. Felker**
1983 Technical Communication Award of Achievement in Journal Articles and Conference Papers Category, East Tennessee Chapter of the Society for Technical Communication
- E. J. Federick**
1983 Certificate of Appreciation from the U.S. Department of Energy for organizing the Alpha-Contaminated Waste Management Workshop, August 1982
1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.
- H. A. Friedman**
1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.
- W. R. Gambill**
1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.
- H. W. Godbee**
1983 Certificate of Appreciation from the U.S. Department of Energy for organizing the Alpha-Contaminated Waste Management Workshop, August 1982
1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.
- E. Greenbaum**
1984 Fellow, American Physical Society
1985 Technical Communication Award of Merit in Scholarly and Professional Articles Category, East Tennessee Chapter of the Society for Technical Communication
1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.
- P. A. Haas**
1984 Fellow, American Institute of Chemical Engineers
1985 Golden Acorn Award for patent development, Martin Marietta Energy Systems, Inc.
- F. E. Harrington**
1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.

J. R. Hightower

- 1982 Engineer of the Year Award, American Institute of Chemical Engineers, Knoxville-Oak Ridge Section

D. W. Holladay

- 1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.

A. R. Irvine

- 1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.

R. L. Jolley

- 1981 Vice-Chairman and Secretary, Division of Environmental Chemistry, American Chemical Society
- 1982 Chairman, Division of Environmental Chemistry, American Chemical Society
- 1983 Union Carbide Corporation Community Service Award
- 1984 Program Chairman-Elect, Division of Environmental Chemistry, American Chemical Society
- 1984 Chairman, Fifth Water Chlorination Conference, June 3-8, 1984
- 1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.

D. S. Joy

- 1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.

F. A. Kappelmann

- 1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.

A. D. Kelmers

- 1983 Technical Communication Award of Achievement in Journal Articles and Conference Papers Category, East Tennessee Chapter of the Society for Technical Communication
- 1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.

N. E. Lee

- 1985 Technical Communication Award of Distinction in Scholarly and Professional Articles Category, East Tennessee Chapter of the Society for Technical Communication
- 1985 International Technical Communication Award of Distinction in Scholarly and Professional Articles Category, Society for Technical Communication

R. E. Lenze

- 1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.

T. B. Lindemer

- 1981 Vice-Chairman, Nuclear Division, American Ceramic Society
- 1982 Chairman, Nuclear Division, American Ceramic Society
- 1983 Materials Science Research Competition Award, Significant Implication for Energy Technology Category, "Thermodynamic Investigations and Modeling of Energy-Related Chemical Systems," U.S. Department of Energy
- 1983 Best Paper Award, Nuclear Division, American Ceramic Society
- 1985 Publications Award, Martin Marietta Energy Systems, Inc., Award Night
- 1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.
- 1985-1988 Trustee, Nuclear Division, American Ceramic Society

M. H. Lloyd

- 1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.

R. S. Lowrie

- 1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.

J. C. Mallen

- 1985 Golden Acorn Award for patent development, Martin Marietta Energy Systems, Inc.

- A. P. Malinowski**
1981 Special Award, Advancements in Nuclear Technology, American Nuclear Society
- E. W. McDaniel**
1983 First Place in Ceramographic Competition, Unique Techniques Category: "Autoradiography of Cored Hydrofracture Grout Shows No Significant Migration of Radionuclides"
1985 Technical Communication Award of Excellence in Scholarly and Professional Articles Category, East Tennessee Chapter of the Society for Technical Communication
1985 Session Chairman of the Conference on Hydrofracture and Grout Techniques, jointly sponsored by the U.S. Department of Energy and the University of Arizona
1985 International Technical Communication Award of Achievement in Scholarly and Professional Articles Category, Society for Technical Communication
- W. J. McDowell**
1981 I-R 100 Award for PERALS (Photon-Electron-Rejecting Alpha Liquid Scintillation) Spectrometer, *Industrial Research*
- C. P. McGinnis**
1982 Certified Professional Engineer
1984 Newsletter Editor, Division of Nuclear Engineering, American Institute of Chemical Engineers
1985 Chairman, Knoxville-Oak Ridge Section, American Institute of Chemical Engineers
- D. R. McTaggart**
1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.
- J. R. Mrochek**
1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.
- K. J. Netz, Jr.**
1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.
- W. L. Pattison**
1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.
- R. L. Pearson**
1985 Publications Award, Martin Marietta Energy Systems, Inc., Awards Night
- J. J. Perona**
1982 Chairman of the Energy Transport Research Committee, American Institute of Chemical Engineers
1983 Fellow, American Institute of Chemical Engineers
1983 Chairman of the Energy Transport Research Committee, American Institute of Chemical Engineers
1984 Chairman of the Energy Transport Research Committee, American Institute of Chemical Engineers
- W. W. Pitt, Jr.**
1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.
- D. J. Pruett**
1982-1984 Appointed to Membership Committee, Division of Nuclear Chemistry and Technology, American Chemical Society
1983-1985 Appointed to Training of Nuclear and Radiochemists Committee, Division of Nuclear Chemistry and Technology, American Chemical Society
1984 Appointed Chairman of Membership Committee, Division of Nuclear Chemistry and Technology, American Chemical Society
1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.
1984-1985 Appointed to Subcommittee on Undergraduate Awards, Division of Nuclear Chemistry and Technology, American Chemical Society

J. R. Reddy
1982 Technical Communication Award of Excellence in Topical Reports Category, East Tennessee Chapter of the Society for Technical Communication

B. R. Rodgers
1981-1983 Director of the Fuels and Petrochemicals Division, American Institute of Chemical Engineers
1984 Appointed Program Coordinator, Fuels and Petrochemicals Division at American Institute of Chemical Engineers' Spring National Meeting, Anaheim, California
1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.
1985-1987 Director of the Fuels and Petrochemicals Division, American Institute of Chemical Engineers

R. G. Ross
1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.

R. Salmon
1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.

F. M. Scheitlin
1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.

C. D. Scott
1983 Research Fellow, Union Carbide Corporation
1983 Appointed to Committee on Separation Science and Technology, National Research Council
1985 Golden Acorn Award for patent development, Martin Marietta Energy Systems, Inc.

F. G. Seeky
1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.

S. E. Shell
1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.

W. E. Shockley
1984 Technician of the Year Award, American Society of Certified Engineering Technicians
1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.

W. E. Shockley
1984 Technician of the Year Award, American Society of Certified Engineering Technicians
1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.

S. P. N. Singh
1984 Appointed National Laboratory Consortium Representative to the Control Technologies Task Group (Task Group H), National Acid Precipitation Assessment Program (NAPAP)
1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.

R. D. Spence
1983 Technical Communication Award of Achievement in Journal Articles and Conference Papers Category, East Tennessee Chapter of the Society for Technical Communication

M. G. Stewart
1983 Technical Communication Award of Achievement in Journal Articles and Conference Papers Category, East Tennessee Chapter of the Society for Technical Communication
1983 Technical Communication Award of Merit in Periodic Progress Reports Category, East Tennessee Chapter of the Society for Technical Communication
1984 Technical Communication Award of Achievement in Periodic Activity Reports Category, East Tennessee Chapter of the Society for Technical Communication

- 1985 Technical Communication Award of Distinction in Scholarly and Professional Articles Category, East Tennessee Chapter of the Society for Technical Communication

O. K. Tallent, Jr.

- 1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.

L. M. Toth

- 1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.

J. F. Villiers-Fisher

- 1985 Golden Acorn Award for patent development, Martin Marietta Energy Systems, Inc.

J. S. Watson

- 1984 Appointed National Research Committee, American Institute of Chemical Engineers
- 1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.

H. O. Weeren

- 1983 First Place in Ceramographic Competition, Unique Techniques Category: "Autoradiography of Cored Hydrofracture Grout Shows No Significant Migration of Radionuclides"
- 1983 ORNL Environmental Protection Award for work in the development and implementation of hydrofracture disposal techniques, Oak Ridge National Laboratory

R. M. Wham

- 1984 Certified Professional Engineer
- 1985 Secretary, Oak Ridge-Knoxville Section, American Institute of Chemical Engineers

S. K. Whatley

- 1982-1983 Second Vice-President, Society of Women Engineers
- 1983 Distinguished New Engineer Award, Society of Women Engineers
- 1983 Women of Achievement Award, The University of Tennessee
- 1983-1984 National First Vice-President, Society of Women Engineers
- 1984 Appointed to Management Committee, Engineering Affairs Council, American Association of Engineering Societies
- 1985 Nominated for President, Society of Women Engineers

J. Woodward

- 1984 Librarian, Division of Microbial and Biochemical Technology, American Chemical Society
- 1985 Technical Communication Award of Merit in Scholarly and Professional Articles Category, East Tennessee Chapter of the Society for Technical Communication

R. G. Wymer

- 1985 Silver Acorn Award for patent development, Martin Marietta Energy Systems, Inc.

SPECIAL ASSIGNMENTS

CHEMICAL DEVELOPMENT SECTION

D. O. Campbell

Assigned to: AERE Harwell, U. K.
Period: February 1984 to February 1985
Assignment: Participation in research and development work on nuclear fuel dissolution and on improved extraction flowsheets

R. L. Fellows

Assigned to: Process Support Division, ORGDP
Period: January 1985 to September 1985
Assignment: Development of instrumentation for analyzing volatile components in centrifuge materials

P. A. Haas

Assigned to: Fuel Recycle Division
Period: January 1985 to September 1985
Assignment: Participation in integrated pilot plant operations for fuel reprocessing

C. W. Hancher

Assigned to: Development Division, Y-12 Site
Period: January 1983 to September 1983
Assignment: Assistance in waste treatment process development

D. J. Pruett

Assigned to: A. Zucker, ORNL Associate Director and Finance Manager for the Physical Sciences
Period: January 1985 to September 1985
Assignment: Preparation of a position paper on separations science and technology

R. D. Spence

Assigned to: Fuel Recycle Division
Period: January 1985 to August 1985
Assignment: Participation in the Integrated Engineering Test (IET) of fuel reprocessing operations in the Fuel Recycle Division

ENGINEERING COORDINATION AND ANALYSIS SECTION

W. J. Armento

Assigned to: Martin Marietta Energy Systems, Inc., Process Engineering Division
Period: (K-25) August 1983 to January 1984;
(Y-12) January 1984 to date
Assignment: At K-25, technical assistance in program analysis and documentation for the Gas Centrifuge Enrichment Plant Program (GCEP)
At Y-12, technical support to the Enriched Uranium Recovery Improvements (EURI) project

C. D. Bopp

Assigned to: Martin Marietta Energy Systems, Inc., Engineering Division at K-25
Period: February 1984 to February 1985
Assignment: Technical assistance in program analysis and documentation for the GCEP

J. M. Chandler*

Assigned to: Martin Marietta Energy Systems, Inc., Engineering Division at K-25
Period: May 1982 to date
Assignment: Technical assistance in determination of staffing and maintenance requirements for the GCEP and process evaluation activities for the Atomic Vapor Laser Isotope Separation (AVLIS) project.

W. Davis, Jr.

Assigned to: Martin Marietta Energy Systems, Inc., Engineering Division at K-25
Period: May 1982 to December 31, 1984
Assignment: Technical assistance in program analysis and documentation for the GCEP, including safeguards and UF₆ purity determinations.

L. S. Dickerson

Assigned to: Martin Marietta Energy Systems, Inc., Process Engineering at ORNL
 Period: May 1984 to January 1985
 Assignment: Technical support on special projects.

E. J. Frederick

Assigned to: Martin Marietta Energy Systems, Inc., Process Engineering at Y-12
 Period: April 1982 to date
 Assignment: Assistance to the Y-12 Plant Safety Analysis Department in the preparation of Final Safety Analysis Reports (FSARs) for existing Y-12 site production facilities

W. R. Gambill

Assigned to: The Center for Neutron Research
 Period: March 1984 to date
 Assignment: Project leadership for the overall core design of a very high-flux research reactor

S. M. Gibson

Assigned to: Martin Marietta Energy Systems, Inc., Process Engineering at ORNL
 Period: May 1984 to April 1985
 Assignment: Technical support on special projects

J. H. Goode[†]

Assigned to: Martin Marietta Energy Systems, Inc., Process Engineering in the Y-12 Safety Analysis Program
 Period: February 1982 to December 31, 1984
 Assignment: Assistance to the Y-12 Plant Safety Analysis Department in the preparation of FSARs for existing Y-12 production facilities

D. W. Holladay

Assigned to: Martin Marietta Energy Systems, Inc., Engineering Division at K-25
 Period: February 1983 to May 1984
 Assignment: Technical assistance in program analysis and documentation for the GCEP

A. R. Irvine

Assigned to: Martin Marietta Energy Systems, Inc., Process Engineering at Y-12
 Period: May 1984 to October 1984
 Assignment: Technical assistance on design and construction of wastewater treatment facilities

K. H. Lin

Assigned to: Martin Marietta Energy Systems, Inc., Process Engineering at K-25
 Period: May 1982 to May 1984
 (transferred to K-25)
 Assignment: Technical assistance in program analysis and documentation for the GCEP

J. W. Nehls, Jr.

Assigned to: Martin Marietta Energy Systems, Inc., Process Engineering at Y-12
 Period: February 1984 to date
 Assignment: Technical support to the EURI project

R. L. Pearson

Assigned to: Martin Marietta Energy Systems, Inc., Engineering Division at K-25
 Period: May 1982 to January 1985
 Assignment: Technical assistance in program analysis and documentation for the GCEP

H. C. Savage

Assigned to: Martin Marietta Energy Systems, Inc., Process Engineering at Y-12
 Period: May 1982 to February 1984
 Assignment: Assistance to the Y-12 Safety Analysis Department in the preparation of FSARs for existing Y-12 site production facilities

R. R. Shown

Assigned to: Martin Marietta Energy Systems, Inc., Engineering Division at K-25 and Process Engineering at Y-12
 Period: May 1982 to September 1984 (K-25); October 1984 to date (Y-12)
 Assignment: At K-25, technical assistance in program analysis and documentation for the GCEP;
 At Y-12, assistance to the Y-12 Plant Safety Analysis Department in the preparation of FSARs for existing Y-12 production facilities

J. W. Snider

Assigned to: Martin Marietta Energy Systems, Inc., Process Engineering
 Period: October 1981 to date
 Assignment: Technical assessment of environmental problems and production processes

W. C. Ulrich

Assigned to: Martin Marietta Energy Systems, Inc., Engineering Division at K-25

Period: April 1982 to date

Assignment: Technical support for the Centrifuge Program in long-range logistics and planning activities related to the GCEP

H. O. Weeren**

Assigned to: ORNL Operation Division

Period: 1978 to date

Assignment: Technical assistance on the use of hydrofracture for the disposal of nuclear wastes

E. L. Youngblood

Assigned to: Martin Marietta Energy Systems, Inc., process Engineering at Y-12

Period: February 1982 to date

Assignment: Assistance to the Y-12 Plant Safety Analysis Department in the preparation of FSARs for existing Y-12 production facilities

ENGINEERING DEVELOPMENT SECTION**M. T. Harris**

Assigned to: Martin Marietta Energy Systems, Inc., Process Engineering

Period: October 1983 to May 1985

Assignment: Technical support in the development of environmental control technology

W. W. Pitt, Jr.

Assigned to: Mixed-Waste Study conducted by the Environment, Safety, and Health Program of Martin Marietta Energy Systems, Inc.

Period: July 1984 to March 1985

Assignment: Evaluation of alternatives for treating and/or disposing of mixed wastes at DOE/ORO facilities

A. L. Rivera

Assigned to: F. J. Homan, ORNL Nuclear and Chemical Waste Management Programs

Period: October 1983 to September 1985

Assignment: Technical support for liquid radioactive waste systems

S. M. Robinson

Assigned to: R. M. Counce, Consolidated Fuel Reprocessing Program

Period: February 1984 to December 1984

Assignment: Development of a fluidic pump

B. R. Rodgers

Assigned to: Hazardous Chemical Defense Waste Management Program

Period: November 1983 to June 1985

Assignment: Review of currently available technology relating to hazardous chemical waste management

PILOT PLANT SECTION**J. M. Chilton**

Assigned to: ORNL Operations Division

Period: 1983 to date

Assignment: Technical consultation and operations analyses for ion exchange methods used in process waste treatment

*Retired May 31, 1985.

†Retired December 31, 1984.

‡Retired December 31, 1984.

**Retired May 31, 1985.

CONSULTANTS

CHEMICAL DEVELOPMENT SECTION

S. K. Beal

Affiliation: Stanford Cohen & Assoc.
Expertise: Aerosol treatment and
particulate fouling in
heat exchangers
Time Employed: 6 months

K. E. Spear

Affiliation: Pennsylvania State University
Expertise: Actinide chemistry
Time Employed: 3 d

J. C. Giddings

Affiliation: University of Utah
Expertise: Separation science and
technology
Time Employed: 3d

ENERGY RESEARCH SECTION

E. Bon

Affiliation: Federal University of
Rio de Janiero
Expertise: Biochemistry
Time Employed: 1 d

J. A. Quinn

Affiliation: University of Pennsylvania
Expertise: Chemical engineering
Time Employed: 3 d

A. E. Humphrey

Affiliation: Lehigh University
Expertise: Trends in biotechnology
Time Employed: 1 d

H. B. Ward

Affiliation: University of Mississippi
Expertise: Coal Liquefaction
Time Employed: 1 d

C. J. King

Affiliation: University of California
at Berkeley
Expertise: Separations science
Time Employed: 3 d

D. W. Weeter

Affiliation: University of Tennessee
Expertise: Design and operation of waste-
water treatment facilities
Time Employed: 4 d

ENGINEERING DEVELOPMENT SECTION

R. L. Berger

Affiliation: University of Illinois
Expertise: Cementitious materials
Time Employed: 24 months

ENGINEERING COORDINATION AND ANALYSIS SECTION

J. T. Bartis

Affiliation: EOS Technologies, Inc.
Expertise: Fossil energy technology
Time Employed: 5 d

D. W. Byerly

Affiliation: University of Tennessee
Expertise: Geologic disposal
Time Employed: 105 d

J. R. Couper

Affiliation: University of Arkansas
Expertise: Computer modeling for
equipment capital cost
analysis
Time Employed: 2 d

S. Gonzales

Affiliation: Earth Resource Assoc.
Expertise: Geology
Time Employed: 105 d

K. S. Johnson

Affiliation: The University of Oklahoma
Expertise: Geology
Time Employed: 75 d

P. E. Muhlmeister

Affiliation: Applied Management Sciences
Expertise: Fossil energy technology
Time Employed: 10 d

PILOT PLANT SECTION

A. L. Boch

Affiliation: Independent
Expertise: Management
Time Employed: 4 d

SEDIMENTARY ROCK PROGRAM

D. Brookins

Affiliation: University of New Mexico
Expertise: Geochemistry
Time Employed: 5 d

G. D. Brunton

Affiliation: University of Mississippi
Expertise: Geology of shales
Time Employed: 3 d

L. B. Cobb

Affiliation: L. B. Cobb & Assoc.
Expertise: Field geology
(core drilling)
Time Employed: 59 d

S. Gonzales

Affiliation: Earth Resource Assoc.
Expertise: Geology of shales
Time Employed: 26 d

D. E. Hattin D. Rye

Affiliation: Indiana University
Expertise: Geology of chalk
Time Employed: 29 d

P. A. Domenico

Affiliation: Texas A & M University
Expertise: Geohydrology
Time Employed: 33 d

R. H. Dott, Jr.

Affiliation: University of Wisconsin
Expertise: Geology of sandstone
Time Employed: 4 d

S. G. Driese

Affiliation: University of Tennessee
Expertise: Geology of sandstone
Time Employed: 40 d

J. E. Russell

Affiliation: Texas A & M University
Expertise: Rock mechanics
Time Employed: 21 d

Affiliation: Yale University
Expertise: Geochemistry
Time Employed: 2 d

K. S. Johnson

Affiliation: The University of
Oklahoma

Expertise: Geology

Time Employed: 26 d

H. H. Mills

Affiliation: Tennessee Technological
University

Expertise: Geomorphology

Time Employed: 24 d

M. Monaghan

Affiliation: Yale University

Expertise: Geochemistry

Time Employed: 2 d

P. E. Potter

Affiliation: University of Cincinnati

Expertise: Geology of shales

Time Employed: 16 d

K. Turekian

Affiliation: Yale University

Expertise: Geochemistry

Time Employed: 2 d

W. S. Twenhofel

Affiliation: Independent

Expertise: Geology of shales

Time Employed: 3 d

K. R. Walker

Affiliation: University of Tennessee

Expertise: Geology of carbonate rocks

Time Employed: 30 d

SEMINARS

May 9, 1983	"Interactions of Humic Acids with Actinides in the Marine Environment,"
R. A. Roberts, Florida State University, Tallahassee, Florida
June 6, 1983	"Electrohydrodynamics of Drops and Bubbles,"
O. A. Basaran, University of Minnesota, Minneapolis, Minnesota
October 13, 1983	"Kinetic Studies of Carbon Gasification Reactions and Their Application to the Design of Coal Gasifiers,"
S. Katta, Westinghouse Research and Development Center, Pittsburgh, Pennsylvania
October 13, 1983	"Comparison of Platinum and Platinum-Iridium Catalyst for Heptane Reforming,"
R. W. Rice, Clemson University, Clemson, South Carolina
October 21, 1983	"Barnwell Nuclear Fuel Plant—Criticality Safety Analysis Highlights,"
R. C. Ashline, Allied-General Nuclear Services, Barnwell, South Carolina
October 24, 1983	"Experience with the Advanced Systems for Process Engineering (ASPEN),"
F. R. Muncaster, McDonnell Douglas Automation Co., Chesterfield, Missouri
November 8, 1983	"Molecular Beam Electric Resonance Spectroscopy and Its Applications,"
S. A. Sherrow, University of California, Berkeley, California
December 12, 1983	"Fischer-Tropsch and Slurry Reactors,"
G. A. Huff, Jr., Practice School of Engineering, Massachusetts Institute of Technology, Allentown, Pennsylvania
December 15, 1983	"Fixed-Bed Sorption with Recycle,"
J. T. Casey, University of Missouri, Rolla, Missouri
January 16, 1984	"Ammonium Nitrate in Atmospheric Aerosols,"
A. W. Stelson, Exxon Research and Engineering Co., Annandale, New Jersey
March 1, 1984	"Measurement of Tritium Bred in Lithium-End Lithium Oxide,"
P. C. Bertone, Princeton University, Princeton, New Jersey
March 15, 1984	"Sedimentation in Quiescent and Sheared Suspension,"
E. D. Lynch, California Institute of Technology, Pasadena, California
March 19, 1984	"Characterization and Modeling of Time-Resolved Coal Pyrolysis and Hydro-pyrolysis Product Distribution,"
J. R. Bautista, Princeton University, Princeton, New Jersey

- March 20, 1984 "Hydrolysis of Lactose in Cottage Cheese Whey Ultrafiltrate by the Immobilized Enzyme β -Galactosidase,"
T. C. Scott, University of Wisconsin, Madison, Wisconsin
- April 2, 1984 "Effects of High Pressure on Dye Sensitization of Wide-Bandgap Semiconductors,"
E. R. Roberts, California Institute of Technology, Pasadena, California
- May 3, 1984 "Determination of Surfactant Micell Formation by Ultrafiltration,"
I. W. Osborne-Lee, University of Texas, Austin, Texas
- August 3, 1984 "Potential Health Risks Associated with the Chlorination of Drinking Water,"
R. J. Bull, U.S. Environmental Protection Agency, Cincinnati, Ohio
- August 17, 1984 "Formation, Structure, and Characteristics of Ceramic Alumina Membranes,"
A. F. M. Leenears, Twente University of Technology, Enschede, the Netherlands
- August 27, 1984 "The Effect of pH Oscillations on the Experimental Stability of a Competing Mixed Culture,"
B. H. Davison, California Institute of Technology, Pasadena, California
- November 2, 1984 "Water Quality-Based Approach for Control of Toxic Substances Discharged to the Environment,"
L. Condit, U.S. Environmental Protection Agency, Cincinnati, Ohio
- February 20, 1985 "Fuel Cycle Research and Development in the Chemical Technology Division,"
J. T. Bell, Chemical Technology Division

INTERNAL PROGRAM REVIEWS

DATE	PROGRAM	COORDINATOR
June 14, 21, 1984	Biotechnology	C. D. Scott
July 17, 1984	Environmental Technology (FETEP, ECT in EC&A)	R. K. Genung
September 25, 1984	NRC—Waste Programs	S. K. Whatley
October 9, 1984	Waste Programs (Cement, Assessments, Technical Assistance)	W. W. Pitt
November 13, 1984	Reactor Programs (TMI, HTGR, LMFBR)	J. R. Hightower
December 4, 1984	Nuclear Fuel Cycle (Reprocessing, Refabrication)	J. T. Bell
January 15, 1985	Waste Programs (IDB, Transportation)	J. A. Klein
February 11, 1985	Engineering Support (GPP, CEUSP, K-25, Y-12, ORNL, Waste Assistance)	V. C. A. Vaughen
March 12, 1985	NRC—Safety	R. P. Wichner
April 9, 1985	Chemical and Materials Sciences	J. S. Watson

**CHEMICAL TECHNOLOGY DIVISION STAFFING LEVEL AND FINANCIAL SUMMARY
FY 1984**

	Funding (in thousands)	Chem Tech Scientific/Technical^a (person-years)	Total Scientific/Technical^b (person-years)
DOE PROGRAMS			
Coal and Petroleum	\$800	5.7	5.9
Breeder Reactor Systems	25		
Converter	674	3.2	4.6
Remedial Action	247	1.6	1.6
Commercial Waste Management	50		
Defense Waste Management	2,222	12.9	14.3
Nuclear Fuel Cycle	3,040	19.3	22.7
Nuclear Waste Fund	1,242	4.9	5.3
Solar Energy	123	0.8	0.8
ANFLOW	16	0.1	0.1
Transportation	115	1.1	1.1
Multi-Sector	5		
CEUSP and ²³³ U Dispensing Facility	5,809	18.2	20.4
Environmental R&D	321	2.2	2.3
Basic Energy Sciences	7,589	47.3	52.4
TOTAL DOE PROGRAMS	\$22,278	117.3	131.5
WORK FOR OTHERS PROGRAMS			
Nuclear Regulatory Commission	\$ 3,603	18.4	21.6
SRC and H-Coal	99	0.5	0.5
Rockwell	917	6.1	6.2
Gas Research Institute	111	1.1	1.1
Department of the Army	113	0.5	0.5
Y-12 and K-25	1,262	14.7	15.0
Du Pont	614	3.0	3.0
Miscellaneous	126	0.6	0.6
TOTAL WORK FOR OTHERS	\$6,845	44.9	48.5
TOTAL	\$29,123	162.2	180.0

^aChem Tech research staff supported by the Division in FY 1984.

^bTotal research staff supported by Chem Tech funding in FY 1984, including personnel on loan to Chem Tech from other divisions.

CHEMICAL TECHNOLOGY DIVISION

JULY 1985

INTERNAL DISTRIBUTION

1. C. W. Alexander
2. W. J. Armento
3. T. D. Anderson
4. R. C. Ashline
5. S. I. Auerbach
6. J. M. Begovich
7. L. H. Bell
8. J. T. Bell
9. D. E. Benker
10. T. M. Besmann
11. J. E. Bigelow
12. W. D. Bond
13. C. D. Bopp
14. C. H. Brown
15. W. D. Burch
16. C. A. Burtis
17. J. M. Butler
18. C. H. Byers
19. E. B. Cagle
20. D. O. Campbell
21. W. L. Carter
22. S. D. Clinton
23. H. D. Cochran
24. E. D. Collins
25. J. L. Collins
26. A. G. Croff
27. B. H. Davison
28. L. S. Dickerson
29. S. R. Dinsmore
30. L. R. Dole
31. T. L. Donaldson
32. B. G. Eads
33. B. Z. Egan
34. K. A. Erb
35. L. D. Eyman
36. D. E. Ferguson
37. J. F. Fisher
38. P. W. Fisher
39. C. W. Forsberg
40. E. J. Frederick
41. H. A. Friedman
42. A. S. Garrett, Jr.
43. R. K. Genung
44. S. M. Gibson
45. T. M. Gilliam
46. R. W. Glass
47. E. Greenbaum
48. R. A. Griesemer
49. W. S. Groenier
50. P. A. Haas
51. R. Hall
52. B. A. Hannaford
53. J. R. Hightower
54. D. W. Holladay
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