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Chemical Technology Division Progress Report for the Period January 1, 1987, to June 30, 1988

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Contents

Abstract

This progress report summarizes the research and development efforts conducted in the Chemical Technology Division (Chem Tech) during the period January 1, 19[illegible], to June 30, 1988. The following major areas are covered: waste management and environmental programs, radiochemical and reactor engineering programs, basic science and technology, Nuclear Regulatory Commission programs, and administrative resources and facilities.

The Administrative Summary, an appendix, presents a comprehensive listing of publications, oral presentations, awards and recognitions, and patents of Chem Tech staff members during this period. A staffing level and financial summary and lists of seminars and Chem Tech consultants for the period are also included.

1. Overview

The Chemical Technology Division (Chem Tech), which has ~350 staff members, is the largest research and development (R&D) division 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 major roles in a broad range of ORNL programs for which chemical processing is a critical element. These programs are designed to explore selected aspects of nuclear and nonnuclear energy technologies and to develop advanced processes and systems to meet national priorities. Research and development activities include basic and applied research and engineering, engineering development and assessments, and the operation of various pilot plants and specialized 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 major objectives of Chem Tech are:

- to produce, separate, package, and distribute transuranium elements and other radioactive and stable isotopes for use in research and in applications for medicine and industry throughout the free world;
- to perform R&D related to advanced isotope separation processes and isotope usage;
- to perform chemical processing R&D related to nuclear fuel cycle processes;
- to perform R&D related to advanced waste management and environmental control technologies;
- to perform basic R&D related to chemical separations and biotechnology;
- to provide process chemistry and chemical engineering support to other divisions and programs within ORNL, other parts of Martin Marietta Energy Systems, Inc. (Energy Systems), and other DOE facilities; and
- to enhance the transfer of new technology developed within Chem Tech and elsewhere to U.S. industries, thus improving the technical competence and the industrial competitiveness of the nation.

During this reporting period, the division has been involved in numerous specific activities related to the pursuit of the preceding objectives.

Chem Tech is deeply involved in nearly all aspects of ORNL's current waste management activities as well as in planning for future waste operations that will be acceptable to state and federal regulatory bodies. The Waste Management Technology Center (WMTC), which is managed by Chem Tech personnel, has a major responsibility for participating with industry in the demonstration of innovative and potentially relevant waste management processes on Energy Systems' sites and on vendor sites where wastes from Energy Systems' sites are used. In this regard, the WMTC serves as the technical arm of Energy Systems' Central Waste Management Office (CWMO). Working with groups at various other DOE and Department of Defense facilities, we are also developing and demonstrating in the field a variety of advanced waste management and environmental control technologies. Chem Tech is involved in DOE's work on permanent federal waste repositories for commercial radioactive wastes, providing support in systems integration, project management, waste packaging, and transportation. We continue to maintain DOE's national data base on present and projected radioactive waste in the United States and have been given recent responsibility by the CWMO for the development of data bases encompassing wastes generated on all five Energy Systems sites.

Chem Tech's basic research programs in photobiochemistry, enzyme kinetics, and fixation of enzymes on substrates continue to be internationally respected. Our activities in the development of bioprocessing applications for fossil energy have been favorably reviewed by DOE and will be expanding in FY 1989. Collaborative programs with the ORNL Environmental Sciences Division and with the University of Tennessee on in situ biodegradation of organic pollutants in the earth are also producing promising results. Several of our basic programs in biotechnology and chemical separations attracted industrial attention during the reporting period.

Radiochemical processing programs within Chem Tech emphasize operation of the Radiochemical Engineering Development Center

(REDC) for DOE's Office of Basic Energy Sciences (BES) for the isolation, purification, and packaging of Bk, Cf, Es, and Fm for research uses. The REDC was created during FY 1988 and utilizes facilities that historically have been referred to as the Transuranium Processing Plant and the Thorium-Uranium Recycle Facility. In addition to these BES activities, we expect that the REDC will begin a long-term program for the recovery of isotopes of americium for other DOE sponsors during FY 1989. We continue to conduct hot cell programs for the Nuclear Regulatory Commission on the transport and deposition of fission products under hypothetical nuclear reactor accident conditions. Chem Tech is also engaged in supporting activities for ORNL's High Temperature Gas-Cooled Reactor and Consolidated Fuel Reprocessing programs.

During June 1988, ORNL consolidated isotope activities, which were at that time divided between the Operations and the Chemical Technology divisions, into the Chemical Technology Division, thus returning many stable and radioactive isotope activities of the Operations Division to their earlier home. Within Chem Tech, isotope activities have been combined with related activities and organized into functional groups that allow considerable synergism for research, development, and production activities. The current organization includes a strategic planning group, an R&D group, two isotope preparation groups (one for transuranic and stable isotopes and one for nontransuranic radioactive isotopes), and the Isotopes Distribution Office.

The organizational integration of these elements will allow several issues surrounding the isotopes program to be more efficiently addressed than has been previously possible, thus helping to ensure the continued health of the program. The strategic issues that we will address on a continuing basis include (1) reviewing the business policies that govern interactions among DOE, other federal agencies, and the private sector; (2) maintaining the international competitiveness of production and separation technologies upon which the isotopes programs are based; and (3) reensuring that needed facilities and operations are in full compliance with current regulations, orders, and policies. Activities that are associated with these new organizational elements will be covered in detail in Chem Tech's next technical progress report.

2. Waste Management and Environmental Programs

During this report period, Chem Tech's involvement in waste management and environmental control activities continued to expand along the lines of previously initiated work. The activities can be aggregated into two major areas: support for the Department of Energy's Office of Civilian Radioactive Waste Management (DOE/OCRWM) and waste treatment and environmental control technology (WT&ECT).

Work areas related to the DOE/OCRWM objectives included:

1. transportation of highly radioactive spent nuclear fuel, where we performed work related to all three of the lead offices (i.e., hardware, environmental/economic, and operations);
2. repository program activities, where we performed studies concerning the potential use of shales as repository host rocks, as well as provided support to the main thrust of the program at the Nevada Test Site;
3. system integration studies and analyses to ensure that the various multicomponent DOE/OCRWM systems will fit together and work in a near-optimal way; and
4. the Integrated Data Base (IDB) project, which constitutes the DOE's reference source of data on radioactive wastes.

Chem Tech's WT&ECT activities encompass a broad spectrum of sponsors and applications. The first type of activities involves the DOE-sponsored work related to the management of DOE site wastes. The focal point is wastes generated by DOE/Oak Ridge Operations (DOE/ORO) sites, but this also extends to wastes at other DOE sites, which often have similar characteristics. Work is being performed to develop bioprocesses to treat existing waste management problems, to support the Martin Marietta Energy Systems, Inc., (Energy Systems) Central Waste Management Office (CWMO) in developing and demonstrating technologies to manage DOE/ORO site wastes [i.e., the Waste Management Technology Center (WMTC)], to provide support for the design and safety assessment of processes and capital projects to manage site wastes, to develop technology for the decontaminating and decommissioning (D&D) of existing sites and facilities, to characterize the leach rates of site wastes so that appropriate management strategies can be developed, and to develop processes and waste forms to immobilize wastes.

The second type of WT&ECT activity concerns DOE-sponsored work that is not directly related to site wastes. This work includes providing continuing support for the cleanup of the Three Mile Island Unit 2 reactor, investigating neptunium biosorption, and developing membranes for off-gas cleanup.

The third type of WT&ECT activity involves work sponsored by organizations other than DOE [e.g., Department of Defense (DOD)], although much of the technology is directly applicable to DOE sites. As with the first type of activities mentioned previously, this work is related to waste management activities on specific sites. The wastes included are virtually all hazardous chemical wastes because the organizations are not significantly involved in the handling and processing of radioactive materials. Specific technologies being investigated include:

1. remediation of soil and water containing volatile organic chemicals through the use of soil venting and air stripping, respectively;
2. characterization of an entombed test reactor and developing plans for its D&D;
3. minimization of hazardous wastes produced by ordnance plants;
4. development of hyperfiltration processes;
5. environmental evaluation of fire training pits;
6. development of methods for solidifying oil-contaminated soil and sludges; and

7. preparing numerous transportation routing options and safety analysis reports for the demilitarization of chemical warfare agents for the U.S. Army Toxic and Hazardous Materials Agency.

Analysis of the pervasiveness of various waste management problems and the extent to which they are currently being addressed indicates relative stability for DOE/OCRWM areas and DOE non-site-related work. Growth is expected in Chem Tech's site-related work for both DOE and non-DOE sponsors, predominantly in areas involving wastes containing significant concentrations of hazardous chemicals.

2.1 DOE/OCRWM Programs

DOE/OCRWM was established by the Nuclear Waste Policy Act of 1982. In this act, DOE is directed to take title to the spent fuel and other high-level wastes (HLWs) from civilian and defense programs and to manage them safely. This broad mandate incorporates all aspects between picking up the spent fuel and HLWs at the reactors (or other sites) and final closure of the geologic repositories where the wastes will be emplaced for ultimate disposal. In other words, DOE is charged with putting into place the system to fulfill its mandate and then acting to discharge its legal obligations.

Chem Tech is assisting DOE in this program with a series of related tasks that include collecting the basic data necessary for the design and licensing of the various facilities, studying the options for packaging and transporting the wastes and spent fuels, determining the development needs of the program, and ensuring that all of the facilities and their system interfaces match properly. Much of this work is carried out by subcontractors who are specialists in their fields.

In the following subsection, summaries of our work in the areas of transportation operations, routing, and shipping cask testing are presented. The subsection on systems integration includes specifying, in detail, the characteristics of all the wastes and spent fuel that DOE will need to handle, the logistics required to accomplish this task, a detailed assessment of the capabilities of the existing reactors and facilities to ensure that no interfaces will be mismatched, and an assessment and qualification of computer codes used in the design and operation of the system to provide assurance that the licensing can proceed with confidence in the validity of the code results.

An additional program partially funded by the OCRWM is the IDB, the official source of information about the characteristics of all past, present, and future nuclear wastes generated and stored within the United States. As a special case describing one use of information contained within the IDB, the plan is presented for working off the accumulated inventory of transuranium element (TRU) wastes at ORNL. While Yucca Mountain, Nevada, has been chosen as the preferred repository site for nuclear wastes, DOE has maintained a small secondary effort on alternative host rocks for future repositories. The work that Chem Tech does for the sedimentary rock program lies within this category. And DOE's work would not be complete without keeping abreast of the actions and trends in the regulatory arena. Chem Tech is assisting the ORNL Environmental Sciences Division (ESD) in helping DOE to respond to and to maintain compliance with the U.S. Environmental Protection Agency (EPA) proposed rulings.

Transportation Program

The Transportation Program within Chem Tech provided extensive technical and management support to DOE/ORO as well as a number of other sponsors. The Transportation Program is broadening the scope of its activities in keeping with its multidisciplinary approach to solving transportation problems. It is expected that the program will continue to expand into new areas as opportunities are developed.

The DOE/OCRWM originally assigned ORO the lead responsibility for the development of an operational transportation system to support its spent fuel transportation needs. ORO relied on Chem Tech to take the lead in the development of this project and to serve as the program integrator for the myriad of activities that are required. [In June 1988, DOE Headquarters (HQ) expressed its intention to consolidate the primary responsibility for transportation operations with its other civilian transportation program elements at the Chicago Operations Office (CH) beginning in FY 1989. Chem Tech's role is expected to remain similar in scope and magnitude to that being performed for DOE/ORO.]

The analysis of transportation logistics and routing by Chem Tech personnel continued to support the technical analysis of options related to the movement of chemical weapons by the military, hazardous materials by private industry, and spent fuel and HLW by DOE. Analyses and drop tests were conducted on designs and prototypes of radioactive materials packagings. These performance evaluations were directed at determining the ability of specific packaging designs to comply with the regulatory requirements and to investigate the application of computer codes for analyzing performance. Plans were prepared for the improvement of these capabilities through the development of an integrated test facility at the Tower Shielding Facility, where all types of radioactive materials packaging tests (drop, low-temperature, high-temperature, and immersion tests) can be performed.

Transportation Operations Support

OCRWM assigned ORO the responsibility for the development, implementation, and management of an operational transportation system. This system must be capable of transporting the wastes for which OCRWM has responsibility; these wastes include civilian spent fuel and HLW as well as defense high-level waste (DHLW). All aspects of transportation must be developed (except for package designs), and operational capabilities for highway, rail, and waterborne transport need to be available. While the primary responsibility for transportation operations will be transferred to DOE/CH, it is expected that Chem Tech's role in this area will continue.

Chem Tech has provided broad-based support and assistance in the development of the OCRWM transportation operations system and has also provided ORO with direct technical and management support, including the integration of other contractor efforts into a cohesive overall effort.

Management Support

Management support was provided to ORO in planning for the development and management of the transportation system, including the development of activity logic networks and schedules. A set of planning assumptions and bases was developed to ensure that all levels of management agreed with the basic premises underlying the activity plans. On the basis of these planning assumptions, logic networks with detailed activities were prepared and loaded onto personal computer-based project management software. Building on the logic networks, schedules were developed to assist in the planning and budgeting for future activities. In many cases, subcontractors provided their specialized expertise in these areas.

In support of ORO, a Transportation Project Office Project Management Plan (PMP) was completely redrafted and delivered to ORO for further DOE consideration. A proposed charter for the Transportation Operations Projects Office was included as an appendix to the PMP; the charter had been forwarded in draft form to DOE/HQ. With the planned change of DOE responsibility from ORO to CH, modifications in the PMP will probably be required. In addition to the PMP, a draft of the Transportation Operations Project-Quality Assurance Plan was drafted and placed in review.

Technical Support

In the fulfillment of its overall responsibility to support ORO in the OCRWM transportation activities, Chem Tech has used a system engineering approach to the development of the transportation system. The approach that has been adopted is depicted in Fig. 2.1. The process begins with identifying functional requirements, then involves allocation of these requirements, and continues through other steps to the operation of the system.

The first step in this procedure, identifying functional requirements, was partially completed with the development of a comprehensive listing and description of the functions that must be performed. This will assist in ensuring that the transportation system eventually developed will actually perform as required. These functions were organized in accordance with the structure of higher-level OCRWM documents and provided both detailed descriptions of the functions and flow diagrams that show the interrelationships between the functions. Functional Flow Block Diagrams (FFBDs) were used in this process to graphically depict functional relationships. The top-level FFBD for the OCRWM transportation operations system shown in Fig. 2.2 indicates that there are four primary functions in the Transportation Operations System. The FFBDs were carried to as low a level as necessary for each of the four primary functions. For example, Fig. 2.3 depicts the functions

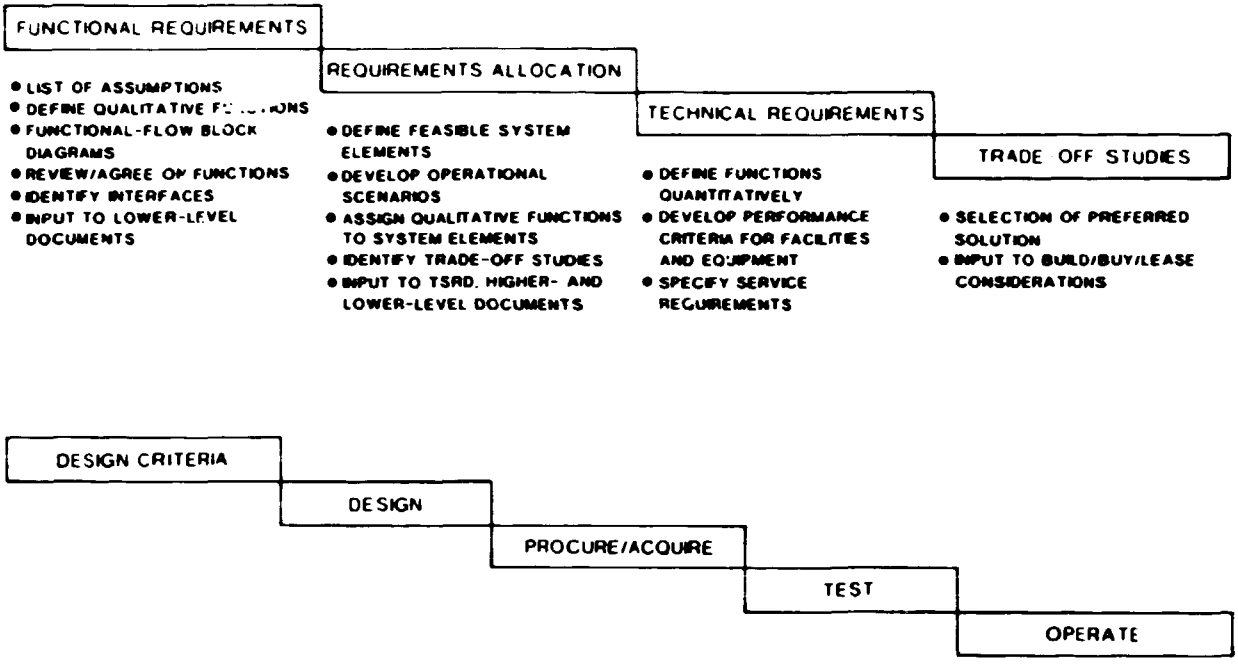
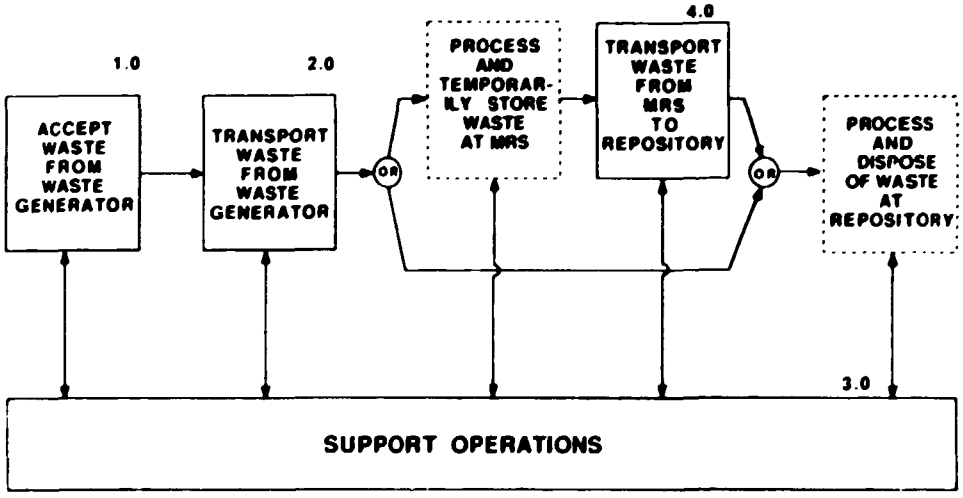


Fig. 2.1. Planning, using a system approach, to accomplish the transportation operations project.



LEVEL 1
FUNCTIONAL FLOW BLOCK DIAGRAM
TRANSPORTATION OPERATIONS SYSTEM

Fig. 2.2. Functions of the Transportation Operations System.

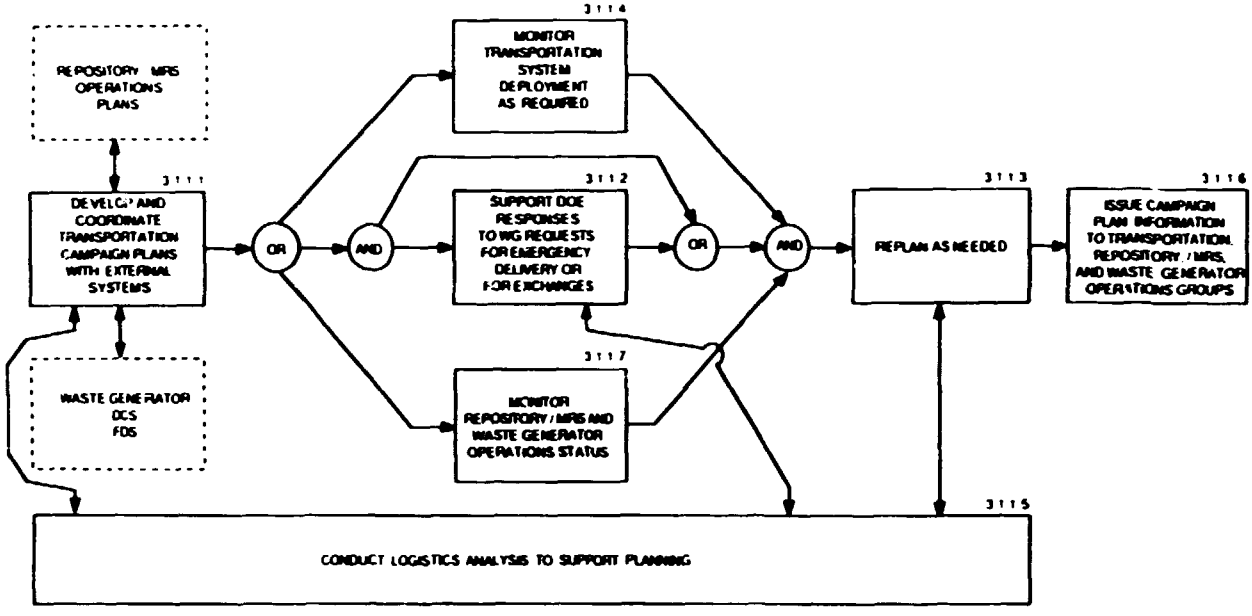


Fig. 2.3. Functional flow block diagram for integrated campaign planning and analysis.

associated with integrated campaign planning and analysis (i.e., function 3.1.1).

A report summarizing the functional identification activity was issued in June 1988.[1] This functional requirements report will serve as a basis for allocating responsibilities to various parts of the OCRWM program and for guiding the quantification of requirements for the transportation program.

To initiate activity for function 3.1.1.5 (Conduct Logistics Analysis to Support Planning, see Fig. 2.3), a preliminary estimate was made of the cask fleet that will eventually be required to support the civilian radioactive waste management activities. Eight different scenarios for transportation requirements through the year 2028 were addressed. The scenarios led to significant differences in the number and types of casks that will be required (see Table 2.1). The most significant factors impacting the fleet size and content are cask loading and unloading times, the number of casks allowed per dedicated train, whether rail shipments are made by dedicated train or as general freight, and whether shipments are made to a Monitored Retrievable Storage (MRS) facility before being forwarded to the repository or are made directly to the repository. The maximum number of casks was found to be between 48 and 110 for these scenarios.

Two of the country's leading spent fuel cask owners and operators provided, through contracts, reports that summarize their available cask fleets as well as reviews and assessments of their experience in shipping spent fuel. The Nuclear Assurance Corporation (NAC) reported that it has 13 casks available.[2] Transnuclear, Inc., reported that it has three casks available and operates one cask owned by a utility.[3] In addition to a description of their current cask fleet, the companies reported on operating practices, loading and unloading procedures, and problem areas. The second phase of the contracts will focus on developing solutions to the reported problem areas.

The OCRWM transportation system must provide not only the equipment and carrier services necessary to move the nation's spent fuel but also the support facilities and services required to keep the system operating. One of the primary considerations in this area is the maintenance of the transport equipment, particularly the spent fuel casks. In this regard, the design and costs of a Cask Maintenance Facility were investigated. Chem Tech, in conjunction with Central Engineering, initiated a Cask Maintenance Facility Feasibility Study (preconceptual design). This study will be used to investigate the major issues related to a cask maintenance facility, to establish the functions and requirements of such a facility, and to develop

Table 2.1. Summary of cask fleet projections for different assumed scenarios

Scenario	Maximum number of casks required		
	Truck	Rail	Total
1. Basic TSLCC* assumptions includes an MRS	14	58	72
2. Basic TSLCC except five casks per unit train	14	38	52
3. Basic TSLCC except rail casks transported as general freight	14	34	48
4. Basic TSLCC assumptions but does not include an MRS	24	31	55
5. Basic TSLCC except five casks per unit train and no MRS	24	28	52
6. Basic TSLCC except rail casks transported as general freight and no MRS	24	27	51
7. All shipments in truck casks; no MRS	61		61
8. All shipments in truck casks; no MRS, extended cask loading/unloading times	110		110

*TSLCC (total system life cycle cost) assumptions, including: For truck—legal-weight truck shipments; casks available 345 d/year; dispatched as general freight. For rail—100-ton for from-reactor shipments and 150-ton for from-MRS (monitored retrievable storage) shipments; casks available 330 d/year; dispatched in single dedicated train from each reactor once per year.

initial designs and cost estimates for the buildings and site.

The evaluation of management configuration options for the transportation operations system was performed in more detail than in earlier studies. A proposed study list of possible management configurations was developed along with a list of issues that related to the desirability of the individual configurations. Ten separate management configurations were identified, and each is being assessed against 24 separate issues. A coarse evaluation of each configuration was completed. This preliminary determination of strengths and weaknesses for the various management configurations will serve as a basis for guiding future efforts to arrive at the preferred management structure for the OCRWM transportation operations.

Transportation Routing Analysis

The Transportation Routing Analysis project has primary responsibility for the support of DOE/OCRWM transportation projects managed by the Chicago Transportation Program Office. There are four areas of support: highway routing analysis, rail routing analysis, population density statistics, and mapping. Activities in each of these areas will be described briefly.

Highway Routing Analysis

The HIGHWAY routing model was modified to incorporate the capability of automatically predicting alternative routes. Previously, in order to estimate different routes between the same origin and destination, it was necessary to manually adjust the

data base. This was a slow, labor-intensive operation and was not suitable for estimating a large number of alternative routes for risk analysis projects. The automatic calculation technique provides a convenient and efficient way to estimate alternative routes. Figure 2.4 contains an example of four alternative routes between the Robinson reactor in South Carolina and the Yucca Mountain site in Nevada. The route labeled with a "1" is the primary interstate highway route, which minimizes the routing objective function. The other three routes are alternatives and are labeled in the order calculated. The maximum variation in distance along the routes shown in Fig. 2.4 is 235 miles, or 9.1%. The driving time along the alternative routes is expected to vary by about 6.5% (3.5 h).

The HIGHWAY data base contains a description of over 240,000 miles of roads in the United States. There are approximately 25,000 highway segments connecting 13,500 intersections or nodes. Each segment is described by the highway name, road classification, distance between endpoints, estimated driving speed, and geographical coordinates of the end points. The HIGHWAY data base is updated approximately every 2 or 3 years so that recent changes in the U.S. highway system can be included. Most of the changes are caused by new construction, rerouting of existing highways, renaming of highways, and so forth. The changes incorporated during the 1987 validation check primarily involved secondary rural highways.

Rail Routing Analysis

Activities in the rail routing analysis involved a validation check of INTERLINE, the rail routing data base, which contains a description of the U.S. railroad system. The data base is updated periodically to reflect recent rail abandonments and rail line mergers. The data base now represents the status of the U.S. railroad system as of May 1988. Mainline classifications, which are based on traffic density along a particular section of track, are used in the prediction of likely rail routes. Rail lines

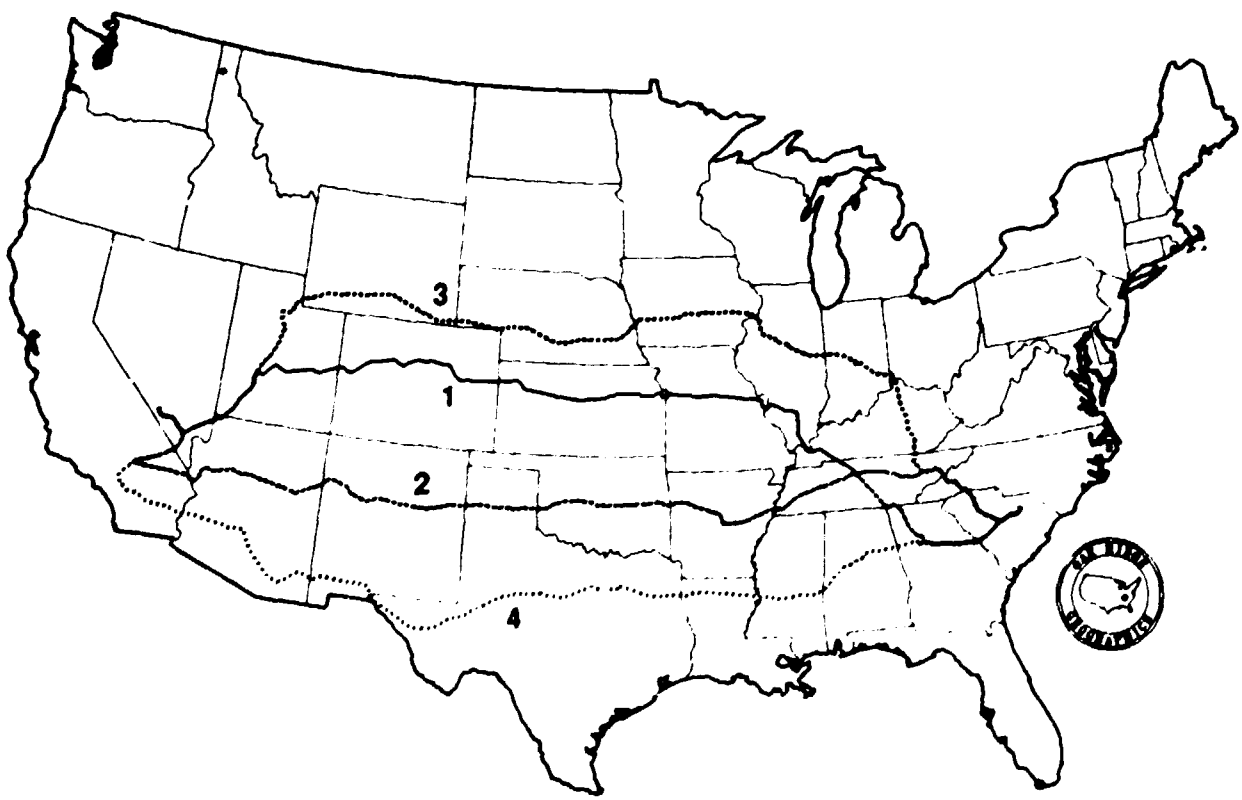


Fig. 2.4. Example of alternative routes, Robinson Reactor to Yucca Mountain.

that carry a higher traffic density are called mainlines; the lesser-used lines are called branchlines. The routing algorithm incorporated in the INTERLINE rail routing model is designed to use mainline tracks whenever possible. Hence, the model tends to direct shipments over the more heavily used rail lines. The 1984 freight traffic density information, the most current data available, was used to update the mainline classifications in the data base.

Population Statistics

The ORNL data base for the distribution of population in the United States was modified to include an additional set of population density contours. The incorporation of the new contours was requested by DOE/CH to support the Transportation Risk and Cost Analysis Model (TRICAM). The contour data were intersected with both the HIGHWAY and INTERLINE routing networks, and a population density distribution was calculated for each link in the routing data bases. Including the population density data with the routing data bases simplifies the calculation of population data for routes of interest to DOE.

Mapping

A number of map sets were prepared for DOE, and each map set included a detailed route description. A new piece of information requested by DOE/CH was the number of the urbanized areas traversed by the route. This information is used to estimate escort costs for highway shipments of spent fuel. A special set of maps, prepared for DOE, included both the outline of federal Indian reservations and the estimated highway or rail routes to the Yucca Mountain site.

Cask Testing

The Drop Test Facility (DTF) is unique in that it has the capability to lift and drop large weights from great heights. It is the site at which packages and casks are tested to meet certain federal requirements. Over the past 13 years, more than 50 drops have been made on packages ranging in weight from 100 to 25,000 kg. The drop heights have ranged from 9 to 27 m. Recently, questions have been raised as to whether the DTF would be able to drop test a 100-ton cask. Since this is expected to be important to future DOE programs, it is being investigated.

During this report period, the testing of full-scale and model shipping containers at ORNL has become more formalized. There has been an increased emphasis on obtaining very specific information from tests of the sponsor's package, and procedures are being standardized to attain this goal. In addition, because the tests are carried out at the DTF, which shares the physical equipment with the Tower Shielding Facility (TSF), the safety of the testing complex is also being examined.

The Quality Assurance (QA) Program has been strengthened for the testing of packages and casks. The Drop Testing QA Plan was written to establish the QA program requirements for performing drop tests in accordance with ANSI/ASME NQA-1, "Quality Assurance Program Requirements for Nuclear Facilities." The QA requirements imposed on each activity are intended to provide adequate confidence that tests will be performed safely and that reliable results will be obtained. The plan will be applied to all drop tests in a graded manner, imposing more stringent requirements over the tests as the need for safety, reliability, and reproducibility becomes more stringent.

Testing Capabilities Study Task

The DTF has been used since 1975 to drop casks, packages, and overpacks from 9 m onto an unyielding surface. The largest cask tested weighed ~23 tons, and the largest overpack tested weighed about 25 tons. Two 100-m-tall towers are used to elevate the package. These towers were originally designed for another purpose, which included the lifting of large weights to heights greater than 60 m. While studies have led ORNL engineers to conclude that a 100 ton cask could indeed be dropped from 9 m, an actual lift of this magnitude has not yet occurred. A related question deals with the effect of the release and impact of heavy weights on equipment that is in close proximity to the impact pad. It is necessary to confirm that no damage to the towers or to neighboring machines and equipment will result from the release or impact of 100-ton weights.

A program plan and a QA plan have been set up to confirm that the testing capacity of the DTF does meet the 100-ton and 9-m expected capacity. An engineering review has been carried out; it examined the entire lift system, including the towers, cabling, and winches. The preliminary

results indicated that the system has the necessary capacity. Further analyses are planned to monitor the behavior of the neighboring equipment during actual drop tests. These results, which are expected to be available within the next few months, should provide assurance that the DTF has the capacity and capability to safely test 100-ton casks to meet the regulatory limits.

The ORNL Uranium-Shielded Shipping Cask

In order to experimentally verify the structural adequacy of the ORNL Uranium-Shielded Shipping Cask [which has the identification number USA/5595B(U)], one of the models (L) was dropped from 9 m onto the unyielding surface at the ORNL DTF. The cask, a steel-jacketed, uranium-shielded package, has a 28- by 45-cm-deep cavity, weighs about 6.8 t, including its skid, and is used primarily to ship ^{137}Cs and ^{60}Co. The specific test was carried out to investigate the movement of the cask lid relative to the body, which, if significant, might provide the potential to release a portion of the cask contents under extremely severe accident conditions. The package was dropped at an oblique angle (30°) onto its top to impact the edge of the lid, which would load the lid bolts in shear. No impact limiter was used.

The cask lid remained in place but was slightly damaged as a result of the impact. The lid bolts did not shear or exceed their elastic limit.

The NAC Quarter-Scale Model Cask

NAC has designed a single-element, lead-shielded, steel-jacketed cask. In order to support the structural analysis in its licensing application to the NRC, a one-quarter-scale model of the package was put through a series of drop and punch tests at the DTF at ORNL. The model, weighing 390 kg, was equipped with aluminum honeycomb energy absorbers on both ends (see Fig. 2.5), contained a dummy payload, and was subjected to the following tests:

1. a vertical end drop from 9 m onto the closure end,
2. an oblique drop from 9 m onto the closure end,
3. a side drop from 9 m,
4. an oblique drop from 9 m onto the bottom end with the closure end free to slap down following the initial impact, and

Fig. 2.5. Quarter-scale NAC shipping cask being readied for drop testing.

5. a 1-m side drop onto a 3.8-cm-diam punch.

The cask was pressurized to about 30 psig and was equipped with both piezoelectric accelerometers and strain gages. The cask dimensions were recorded before and after the test series.

Accelerometer and strain gage data are still being analyzed, but preliminary results indicate that the cask met the expected goals. Following the drops, measured changes to the internal dimensions were negligible.

The Army RDX Drum Drop Tests

The Picatinny Arsenal has responsibilities for transporting RDX and HMX explosives in bulk quantities. This material is presently shipped in Department of Transportation (DOT) specification 21C fiberboard drums. Twenty-three kilograms of bulk explosive is normally placed in cloth sacks; four of these sacks are placed inside two plastic bags used to line the drum. The explosives are dampened with a mixture of water and alcohol. The tests were used to study the containment capabilities of the drums when cooled to approximately −34°C (−30°F) and dropped from 1.2 m (4 ft) onto a solid surface. In addition, the effect of

replacing cloth bags with plastic bags was examined.

A sand-sawdust mixture formulated to have a density similar to that of the explosive was used in place of the RDX/HMX. Three different combinations of water and isopropyl alcohol were placed in sacks; the water-to-alcohol ratios used were 100:0 (100% water, no alcohol), 60:40, and 30:70. The sand-sawdust mixture dampened with 100% water was placed in plastic bags, whereas the mixture dampened with the two water and alcohol mixtures was placed in the traditional cloth bags. Three sets of four drums were filled, each set containing one of the three dampening agents. Each set of four drums was tested by dropping individual drums from a height of 1.2 m (4 ft) in the following orientation:

1. Top chime, diagonally onto a solid surface.
2. Bottom chime, diagonally onto a solid surface.
3. Sidewall over a 2-in. by 6-in. timber resting on a solid surface with the 6-in. leg vertical. The drop was made with the drum in a horizontal position at right angles to the timber.
4. Horizontal, onto a solid surface.

The preliminary conclusion is that the drums containing some alcohol were damaged less severely than those containing only water. The bags dampened with water only were frozen solid at −34°C (−30°F), whereas bags containing some alcohol were softer and, on impact, tended to mold themselves into the shape of their surrounding support structure. Not only the drums, but also the sacks dampened with some alcohol sustained less damage.

Y-12 Drum Tests

The Y-12 Plant requested a drop test of their newly designed shipping package, the outside of which is a standard 55-gal drum. The package was to be tested to meet the regulatory drop test standards. The container was dropped from a height of 9 m onto a solid, unyielding surface and then dropped a second time from a height of 1 m onto a 15-cm-diam punch. The package sustained little damage and was returned to Y-12 engineers for further evaluation and testing.

Proposed DOE Cask-Testing Complex

On September 17, 1987, a proposal to establish a cask-testing complex at ORNL was presented to DOE. The presentation evaluated the needs of DOE to test packages, particularly full-scale casks weighing up to 100 tons, and the capabilities of ORNL to meet those needs. The plan was based, in part, on information from the OCRWM Business Plan and the From-Reactor Cask Program.[4]

The testing complex would build on the existing capabilities at ORNL to carry out two of the four accident test requirements specified in both the DOE and Nuclear Regulatory Commission (NRC) regulations.[5,6] These present capabilities include the ability to test full-scale packages by dropping them from 9 m onto a solid, unyielding surface or by dropping them 1 m onto a steel punch.

The presentation identified the OCRWM's testing requirements, the regulations, and the major cask-testing capabilities in the United States; it also proposed an approach to support DOE's needs, which included a number of possible ORNL activities. The discussion described the development of a thermal facility at ORNL and a water immersion test facility. (ORNL has subjected full-scale radioactive material shipping packages to the prescribed DOE accidental fire test requirement at a distant facility.)

DOE is in the process of determining the merits of establishing a cask-testing complex at ORNL. One of the components involved in this decision (the capability to drop 100-ton casks) is described under the Testing Capabilities Study Task.

Systems Integration Programs

A significant activity in the development of the DOE Federal Waste Management System (FWMS) is integrating its components, which include handling, packaging, transportation, storage, and final disposal of spent fuel and HLW. The objective of the DOE Waste Systems Integration program is to ensure that the components of the FWMS will constitute an efficient and safe system that operates as a unified entity. A major

factor in accomplishing this objective involves collection of data and development of information and capabilities used by all program elements.

In support of this objective, Chem Tech manages the ORNL Waste Systems Data and Development (WSDD) program, providing DOE/OCRWM information and technology with which to integrate the FWMS. Typically, the WSDD activities have been focused on systems studies, waste characterization, classification and treatment, technical computer code assessment, verification and validation, and system modeling.

Characteristics Data Base

The OCRWM is responsible for the spent fuel and HLW that will eventually be disposed of in a geologic repository. The two major sources of these materials are commercial light-water-reactor (LWR) spent fuel and DOE nuclear materials production plants. Other wastes that may require long-term isolation include non-LWR spent fuels and miscellaneous sources such as activated metals. Detailed characterizations, which include physical, chemical, and radiological properties, are required for all of these materials. Radiological properties must take into account decay as a function of time. In addition, the present inventories and projected quantities of the various wastes are needed. This information has been assembled in a Characteristics Data Base, which provides data in four formats: hard-copy standard reports, menu-driven personal computer (PC) data bases, program-level PC data bases, and mainframe computer files.

This data base is an integral part of the systems integration approach being used by OCRWM. Specifically, the data base provides a standard set of self-consistent data to the various areas of responsibility within OCRWM, including systems integration and waste stream analysis, storage, transportation, and geologic disposal. The data will be used for design studies, trade-off studies, and system optimization by OCRWM and their supporting contractors.

A brief summary of this work was given in the previous progress report, which cited the first draft report.[7] Since then, two additional draft versions have been issued, and the final journal report was issued in eight volumes and two printings.[8] A summary of this work is given in the following subsections.

Overview

The Characteristics Data Base is organized by four waste stream categories: LWR spent fuel, immobilized HLW, non-LWR spent fuels, and miscellaneous wastes. These four categories, plus a summary, comprise the five major sections of the first characteristics report, which was recently issued as an eight-volume set.[8] Six of these volumes consist of 16 appendices that provide supporting information to the other 2 volumes.

Five of the appendices are Users' Guides to the menu-driven PC data bases described in Table 2.2. No programming skills are required to use these data bases. Two more such data bases are under development, one with data on LWR fuel pin characteristics and one for LWR assembly serial numbers. These data bases are designed for use on IBM-PCs or PC-compatibles and are available through ORNL (ordering information is given on p. v of ref. 8).

The previously mentioned data bases, as well as many of the internal working files, are written in dBASE III, which permits tabulation of special

Table 2.2. Menu-driven personal computer data bases available from the Characteristics Data Base

Light-Water Reactor (LWR) Radiological Data Base—Contains radionuclide compositions, heat generation rates, curies, photon spectra, and other information as a function of spent fuel type, burnup, and decay time
LWR Assemblies Data Base—Contains detailed physical descriptions of fuel assemblies and radiological properties of spent fuel disassembly (SFD) hardware
High-Level Waste (HLW) Data Base—Contains physical, chemical, and radiological descriptions of HLW, both as the interim form and the immobilized form in canisters
LWR Nonfuel Assembly (NFA) Hardware Data Base—Contains physical and radiological descriptions of NFA haruware (i.e., nonfuel-bearing hardware other than SFD hardware)
LWR Quantities Data Base—Contains data on discharged fuel, as historical inventories and as projected quantities, based on Energy Information Administration data supplied to them by the utilities

reports and interactive output. Mainframe files are used to generate some of the files and some of the hard-copy reports. Their use involves Statistical Analysis System (SAS) and FORTRAN programming.

The radiological properties are calculated using ORIGEN2[9] and include:

1. quantities of each nuclide (grams or gram-atoms);
2. radioactivity, total and by nuclide (curies);
3. thermal power, total and by nuclide (watts);
4. photon energy spectra, by energy group (18 groups);
5. neutrons from spontaneous fission (per second);
6. neutrons from (α,n) reactions (per second); and
7. quantity of each element (grams or gram-atoms).

The above properties are decayed, over time, for 24 (or more) periods from 1 to 1 million years. For LWR spent fuel, the integral heats are also available between any 2 times out of a set of 38 times, also spanning periods of 1 to 1 million years.

.WR Spent Fuel

Data on LWR spent fuel constitute the largest portion of the overall data base, because of the large quantities of material involved and because of the complexity of the data. The data base presently incorporates 78 different assembly types or models over a wide range of fuel burnups, both inventories and projections for future discharge, and provides the radiological properties over time for the fuel itself and for the associated hardware. In addition, although most of this fuel is presently in the form of uncanned intact assemblies, some (i.e., defective fuel) is canned. More canned fuel can be expected. Furthermore, future practices may result in extensive disassembly and consolidation (either at a Monitored Retrievable Storage Facility or at reactor sites), which will also give rise to activated metals from the hardware components.

Although there are 78 different assembly types listed in the data base, many of these are virtually identical, differing only in minor details or manufacturer, and their basic design or configuration being controlled by reactor core design. There are 21 such "assembly classes" (Table 2.3), plus a

Table 2.3. Major classes of light-water reactor assembly models

Pressurized-water reactors (PWRs):	Babcock & Wilcox, 15 × 15
	Babcock & Wilcox, 17 × 17
	Combustion Engineering, 14 × 14
	Combustion Engineering, 16 × 16
	Westinghouse, 14 × 14
	Westinghouse, 15 × 15
	Westinghouse, 17 × 17
	South Texas, 17 × 17 XLR
	Palisades, 15 × 15
	Saint Lucie II, 16 × 16
	San Onofre I, 14 × 14
	Yankee Rowe, 15 × 16
	Indian Point I, 13 × 14
	Fort Calhoun, 14 × 14
	Haddam Neck, 15 × 15
Boiling-water reactors (BWRs):	General Electric BWR/2, 3 7 × 7, 8 × 8, 9 × 9
	General Electric BWR/4, 5, 6 7 × 7, 8 × 8, 9 × 9
	Lacrosse, 10 × 10
	Big Rock Point, 9 × 9, 11 × 11
	Dresden I, 6 × 6
	Humboldt Bay, 6 × 6, 7 × 7

number of variants that represent a limited number of discharged assemblies.

Figure 2.6 shows schematically the flow of input and output data for LWR spent fuel. Data on discharged fuel are provided by the utilities to the Energy Information Administration (EIA) via the RW-859 annual data submittal; quantities of spent fuel are given in metric tons of initial heavy metal (MTIHM). More detailed information on this part of the data base is provided in a paper by Moore et al.[10]

Defective LWR spent fuel is of interest because it may require special handling procedures that have not yet been defined. This aspect of the data base is described in a paper by Lawson et al.[11]

High-Level Waste

Immobilized HLW will derive from one commercial operation (West Valley) and three defense sites: Savannah River, Hanford, and Idaho. The first three will produce borosilicate glass by a vitrification process, while Idaho, which is storing its HLW in the form of a calcine (rather than liquid, sludge, or salt cake), plans to produce a high-density ceramic waste form. All four sites plan to use canisters 10 ft long by 24 in. in diameter. For completeness, and to utilize current (rather than projected) data, the data base also includes the characteristics of the interim waste forms at the four sites.

The HLW data base includes detailed information, year by year, on the number of canisters and their radiological and thermal properties. A condensed summary of this type of data is given in Table 2.4.

Non-LWR Spent Fuel

This category includes high-temperature gas-cooled reactors (HTGRs), research and test reactors, and miscellaneous fuels from various reactors now stored at nine locations. Table 2.5 gives a summary of these fuels. The HTGR fuel is stored

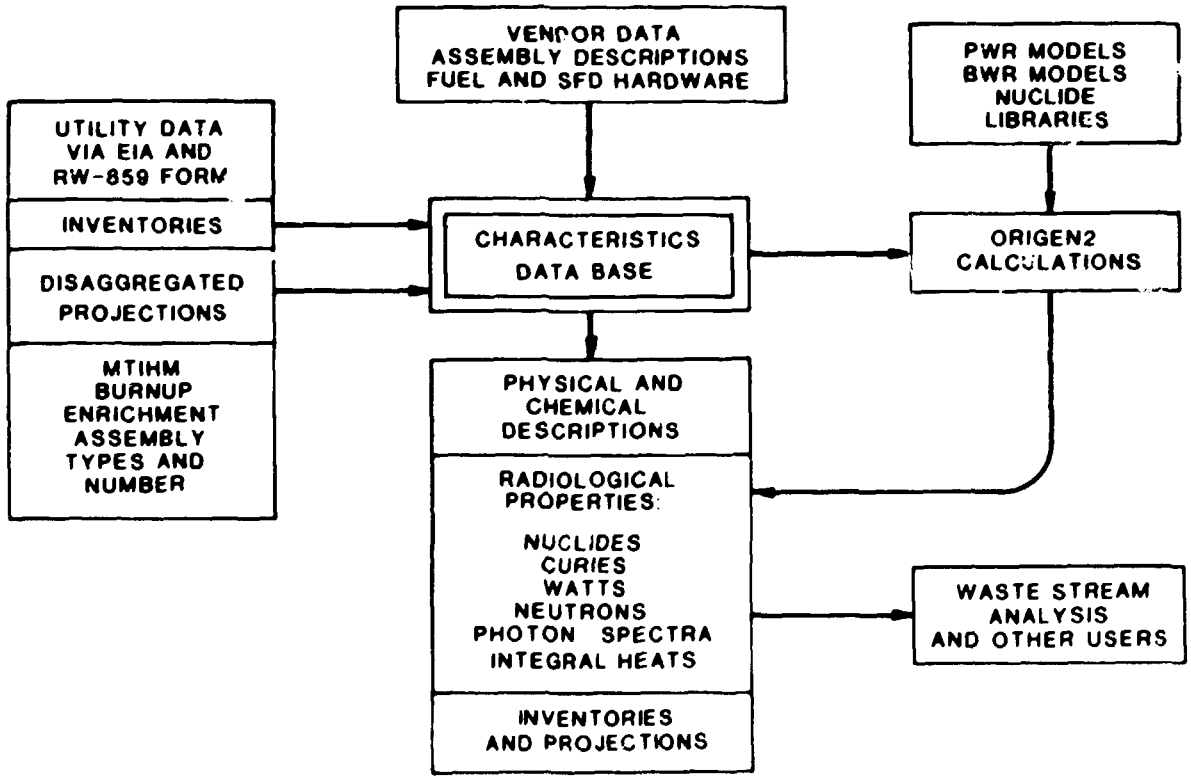


Fig. 2.6. Characteristics data for light-water reactor spent fuel.

Table 2.4. Characteristics of immobilized high-level waste

	West Valley	Savannah River	Hanford	Idaho
Weight of canister, kg	252	500	500	500
Weight of contents, kg	1895	1682	1650	1825
Total weight, kg	2147	2182	2150	2325
Kilocuries per canister[a]	125	234	416	143
Watts per canister[a]	360	700	1150	340
Projected startup (year)	1990[b]	1990	1996[b]	2011
Projected no. of canisters (cumulative) in:				
1992	275	922	0	0
1995	275	2152	0	0
2000	275	4202	653	0
2005	275	5302	1305	0
2010	275	5890	1860	0
2015	275	6350	1860	3800
2020	275	6810	1860	8800

[a]These are maximum values at time of loading. For West Valley, with fixed input, the average is 10 to 20% lower. For the defense sites, with future input undefined, the averages may be even lower.

[b]Unofficial updates have adjusted these dates to 1992 and 1999.

Table 2.5. Summary of non-light-water reactor spent fuels

Reactor/site	Quantities (1987 data)
HTGRs (number of fuel elements)	
Peach Bottom I	
Core I	804
Core II	804
Fort St. Vrain	725
Research/Test Reactors (number of assemblies)	
MTR Plate Fuel	
TRIGA	
UO_2-in-polyethylene	
PULSTAR	
FFTF	170
Miscellaneous Fuels (metric tons of heavy metal)	
Argonne	0.31
Babcock & Wilcox	0.05
Battelle-Columbus	1.50
Battelle-PNL	2.25
Hanford	0.07
Idaho	38.06
Los Alamos	0.13
Oak Ridge	1.28
Savannah River	19.02

at the Idaho facility. The Idaho total does not include the Shippingport breeder core, which contains 770 kg of uranium, mostly ^{233}U, and 47.21 t of thorium. In addition, the HTGR fuel is also at the Idaho facility. The thermal outputs of these spent fuels are generally quite low. However, the diversity of chemical compositions and physical configurations, coupled with the high enrichments of many of the spent fuels, may impose some challenging disposal considerations.

Miscellaneous Wastes

This category includes wastes that can be defined neither as spent fuel nor as HLW, and, in addition, may not qualify for commercial low-level disposal or for handling as defense TRU waste. Many of these wastes are in the category of Greater-Than-Class-C Low-Level Waste and are not eligible for shallow-land burial without an exemption from the NRC.

In the absence of an intermediate-depth or greater-confinement disposal facility, these materials may be future candidates for consideration for repository disposal. They are included in the

Characteristics Data Base in order to provide technical data on these materials.

Other wastes presently included in this section are OCRWM-generated wastes, commercial TRU waste, reactor decommissioning waste, radioisotope capsules, and certain LWR operational wastes.

Future Work

After comments from users have been received, a major revision and update of the entire report is planned for 1989. This revision is currently planned to include two additional PC data bases, one on LWR fuel pin characteristics and one on LWR assembly serial numbers. Descriptive data on recently introduced LWR assembly models will be added. All inventories and projections will be updated, and improved descriptions of HLW will be incorporated. Radiological calculations for LWR fuel and hardware will be improved, using data and techniques being developed at this time.

Code Verification and Validation

The OCRWM, through its many contractors, uses computer models and software programs to perform facility designs, environmental assessments, and licensing activities. Because of the disparate nature of the OCRWM programs they support and the dispersed locations of the participants in the overall system, the OCRWM has realized that a program is needed to (1) identify these generic software programs and methodologies, (2) assess their current status with respect to OCRWM and NRC licensing requirements, (3) determine necessary development and QA needs, and (4) perform the required computer code development. Chem Tech has called upon several divisions at ORNL (Analytical Chemistry, Computing and Telecommunications, Environmental Compliance and Health Protection, and Health and Safety Research) to assist in this activity.

From a survey of the project offices and contractors for specific information concerning their use of computer codes in support of OCRWM programs, Chem Tech personnel produced a draft report that contains descriptions of 318 computer codes divided into 22 separate categories.[12] Seven of these categories were selected, and assessments of computer codes in four of the more mature areas—source term, criticality, shielding, and thermal analysis—have been completed.[13-16] Codes that model atmospheric dispersion, food chain transport, and dose effects are now being appraised.

One of the important needs of OCRWM is to be able to predict the composition, decay heat, and radioactivity of spent fuel and HLW over extended periods of time. A number of computer codes, called source term or radionuclide generation/depletion codes, are available for simulating nuclear fuel cycles and estimating these characteristics. A program has been initiated to provide OCRWM with a single source term code that can withstand licensing scrutiny. To do this, the code must have undergone all of the necessary QA steps and be verified and validated with experimental tests. Such a code can be used to produce the required data with a high degree of confidence.

During this period, a specific QA plan was written and approved by DOE/HQ.[17] A graded QA approach was used for each of the activities of assessment, verification, validation, and enhancement. Three source term codes, CINDER-2, ORIGEN-S, and ORIGEN2, were chosen for a benchmark test to compare their predictions for composition, decay heat, radioactivity, and radiation spectra when modeling four generic LWRs.[18-20]

Chem Tech has initiated a joint effort with the Materials Characterization Center at Pacific Northwest Laboratories (PNL) to provide analytical data that will be used at ORNL to validate one of the source term codes. Phase I activities of the experimental program were started and include identification of important variables, a survey of available spent fuel analytical data,[21,22] and selection and optimization of analytical techniques. The benchmark test will generalize the results to the other codes.

Logistics Modeling

ORNL is currently engaged in supporting the DOE/OCRWM Systems Integration Team by developing two logistics models: (1) the Waste Stream Analysis Model (WSA) and (2) the Systems Integration Operations/Logistics Model (SOLMOD). These models form an integral part of an overall modeling capability being put together by DOE. Other activities included in this effort are a Scenario Description Data Base, the Spent Fuel Characteristics Data Base (an ORNL product described previously), and an economics model (COST) being developed at PNL.

Waste Stream Analysis Model

The WSA model is being developed to specify and characterize radioactive waste packages at any location at any point in time within the DOE Waste Management System. Characterization includes identifying the contents and reporting physical, thermal, radiological, and isotopic distributions for individual packages or groups of packages. The primary effort during this fiscal year has been to describe and track the spent fuel flows. Basic data for this effort are primarily obtained from the EIA Assembly Tracking System and the ORNL Spent Fuel Characteristics Data Base.

A number of selection and allocation rules have been included in the WSA program for estimating the quantity and identity of the spent fuel assemblies being accepted annually by the Waste Management System. These rules allow the WSA to simulate and characterize the movement of spent fuel for a wide range of scenarios of interest to DOE. The programming logic in the WSA model is able to simulate the management of spent fuel storage at the reactors, transportation from the reactor to the MRS or a repository, the consolidation of fuel at the MRS, subsequent transportation of material from the MRS to the repository, and the preparation of emplacement packages at the repository.

The various capabilities of the WSA model were demonstrated by analyzing a series of sample problems. The first scenario considered was the classical oldest-fuel-first scenario. While DOE can regulate the amount of fuel taken from each reactor storage pool, there is no control over which assemblies will be provided by the utility. Any assemblies transferred to DOE must meet the basic waste acceptance criteria. In general, a utility would prefer to transfer younger fuel to the DOE rather than the oldest fuel because removing the younger fuel from the storage pool would reduce the radiation exposure at the utility site. At a number of utilities, the oldest fuel will probably be stored outside the storage pool in an approved storage cask. Transferring the oldest assemblies to DOE would involve a number of handling steps. The heat content of the repository packages would be higher if younger fuel is accepted, which may be an advantage at the tuff repository. A warmer package will increase the temperatures in the rock formation and tend to drive water away from the waste packages. This is a somewhat different approach than was used in the salt repository designs. For salt repositories, cooler packages were desired to prevent water from migrating toward the waste packages–hence, the oldest-fuel-first concept. In order to simulate the acceptance of younger fuel, the second scenario investigated was based on the assumption that the youngest fuel in the storage pool would be accepted by the Waste Management System, provided that fuel was at least 5 years old.

A number of techniques, both tabular and graphical, have been developed to display the desired characteristics. Figure 2.7 shows a comparison of the average fuel age at the time the spent fuel assemblies are picked up at the reactors for the two scenarios previously outlined. In the oldest-fuel-first scenario, the first fuel shipped to the MRS is expected to have an average age of ~25 years, and the average age will slowly decrease as a function of time. The minimum age, ~10 years, occurred at the end of the scenario. In the second scenario, the reactors will be shipping short-cooled fuel (at least 5 years old). At the start of the study period, the average fuel age will be ~9 years, and as shown in the figure, the average age increases rapidly toward the end of the scenario, reaching a value of 30 years in the year 2022.

A number of other reports can be produced from a typical WSA analysis. Physical property distributions can be generated for fuel burnup, assembly length, heat content, shielded gamma radiation, neutron radiation, and isotopic content. Figure 2.8 shows the ^{14}C content (which is an important aspect for repository management) of the fuel being emplaced in the repository. The WSA also produces a number of data files that are used in SOLMOD.

Systems Integration Operations/Logistics Model

SOLMOD is being developed to simulate, in detail, the movement of radioactive waste packages through the various components of the DOE Waste Management System. The model will be capable of tracking waste packages and their contents from pickup at the reactors to emplacement at the repository. Transportation and waste processing at the MRS and or the repository are included in SOLMOD. SOLMOD has five basic capabilities: assembly tracking, package tracking, estimation of system throughput, evaluation of system status at

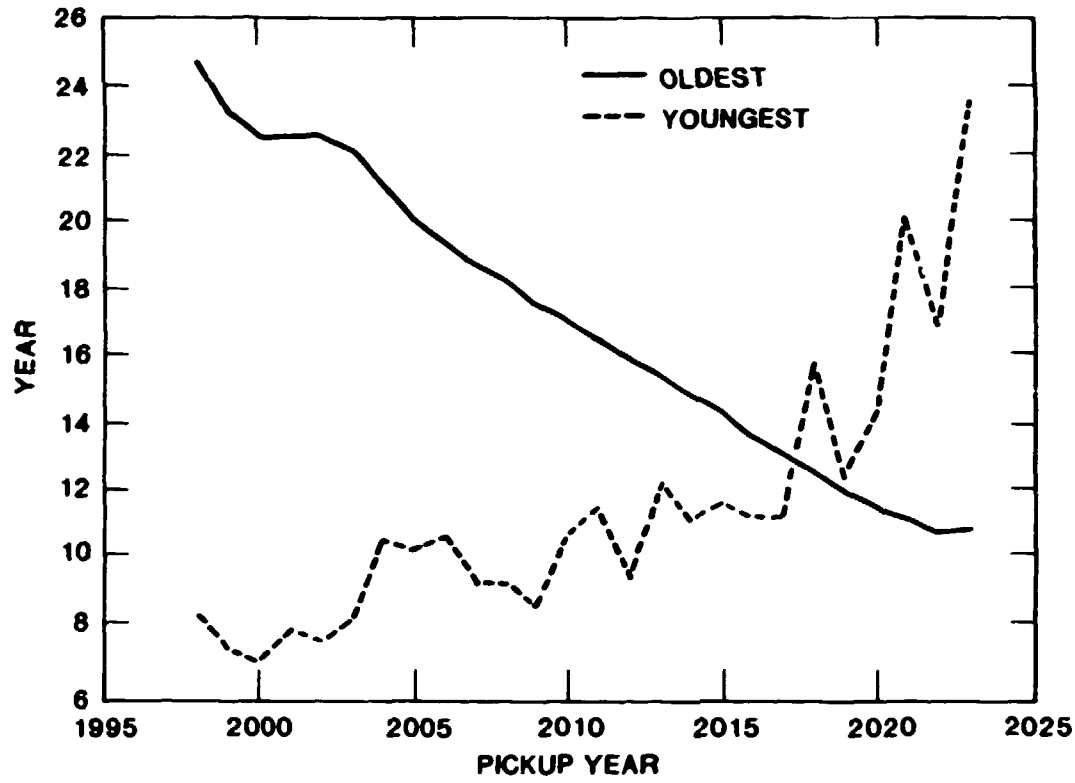


Fig. 2.7. Comparison of average fuel age at time of pickup. Oldest first vs youngest fuel >5 years. Type: PWR.

or during any time period, and reporting resource utilization. Development of SOLMOD was guided by a number of principles, including simplicity, modularity, and flexibility. The application of these principles resulted in the development of a programming language in which processing flowsheets are described as data bases rather than being directly incorporated as part of the computer code. This characteristic provides the ability to change any or all of the flowsheets without having to do any reprogramming.

The structure of SOLMOD is shown in Fig. 2.9. An applications generator provides a user-friendly means for inputting data and detecting syntax errors before execution. The transportation module organizes the dispatching of transportation casks and supplies the simulation module with the shipment arrival time and amount of material being transported. The simulation module, which uses a discrete event simulation technique, is the heart of SOLMOD. The movement between processes is controlled by a series of user input rules. Each process is simulated by a queue. A number of different types of queues can be used to simulate the various waste processing schemes, including first in–first out, first in–last out, random, and overflow queues. In addition, a sink is used to simulate placing radioactive materials in a repository. User-defined rules direct the flow of waste from process to process. These rules can be based on the capacity of other processes in the system or on the amount of material processed.

SOLMOD will provide DOE with the capability of analyzing the entire waste management system to determine how the various components will operate as an integrated unit. The use of stochastic calculations will help identify the way in which the waste management system would operate in a real-world environment and show which critical processing steps may be limiting system throughput. The ability to change the system flowsheets through data input provides a flexible tool for easily evaluating alternative design concepts. For example, should a repository have five or six cask unloading bays?

The requirements for and the utilization of resources can also be explored with SOLMOD. DOE will be able to determine the number of

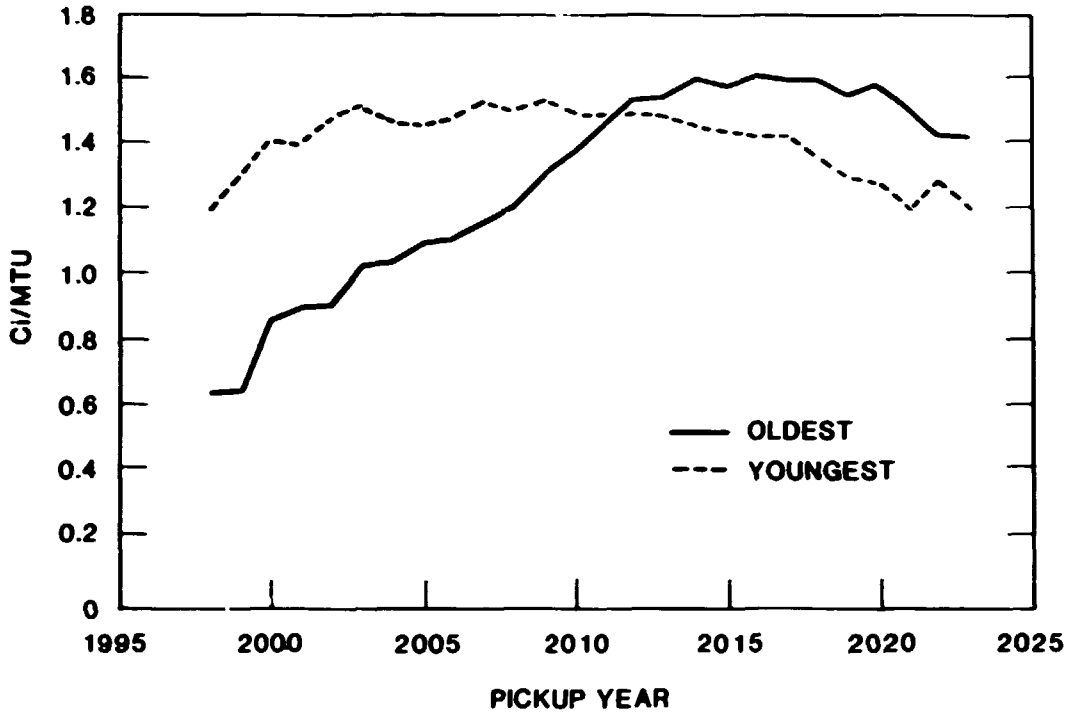


Fig. 2.8. Comparison of average ^{14}C at time of pickup. Oldest first vs youngest first >5 years. Type: PWR.

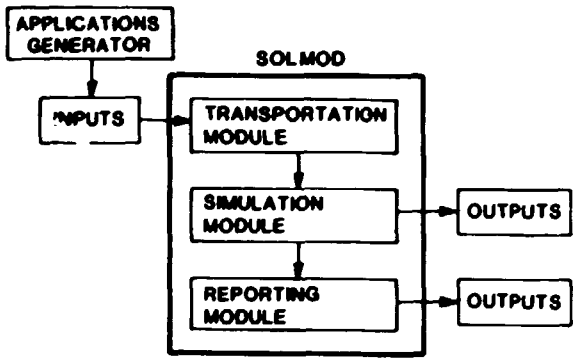


Fig. 2.9. Structure of systems integration operations/logistics model (SOLMOD).

resources needed to perform a specified action (e.g., the number of transportation casks needed to move the fuel between facilities). In addition, it will be possible to evaluate how the system would operate during periods when sufficient quantities of particular resources are not available. Alternatively, one can evaluate equipment reliability factors needed to maintain the desired production level.

Development of SOLMOD was initiated early in fiscal year (FY) 1988. By summer, SOLMOD had progressed to the point where a sample problem could be processed. The purpose of the sample problem was to demonstrate the ability to automatically pass data between the WSA and SOLMOD. Subsequent tests have shown the feasibility of passing SOLMOD output data to the PNL COST model.

Facility Interface Capability Assessment

Interfaces between the nuclear reactor waste generators, the waste transportation system, and the waste repository can have a significant impact on the FWMS performance since a common item, the shipping casks, must be handled by all of them. In an effort to understand and characterize these interfaces, a subcontract was initiated in 1986 with NAC to perform a two-phase Facility Interface Capability Assessment (FICA) study. The study involves evaluating a utility's capability to handle, store, and ship spent fuel and determining where facility upgrades could benefit the FWMS. Phase I

of the study involved generating a master list of data parameters to determine the type of reactor information desired, searching the open literature and licensing documents to obtain the information desired, and developing an information data base. Phase I was completed in FY 1987. Phase II activities involve visitations to each reactor site to verify information previously obtained in Phase I and to obtain any missing information. A utility working group was established by the Utility Nuclear Transportation Group to assist the subcontractor in establishing lines of communication and to facilitate the utility visits. Phase II was begun in June 1987 with test visits to ten selected reactors. These reactors had extensive experience in spent fuel technology and enabled the subcontractor to verify the Site Visit Plan developed for interaction with the utility before, during, and following a site visit. Visits to 26 of the remaining reactors were completed during the reporting period. The FICA study is scheduled to be completed in FY 1990.

Repository Programs

Chem Tech participates in several programs supporting the development of a geologic repository. The contributions to these programs from Chem Tech during the reporting period were in four principal areas: (1) the DOE siting record, (2) model pillar tests, (3) international cooperative work, and (4) mechanical properties of clay-rich rocks.

The DOE Siting Record

A siting record document has been prepared for DOE to provide a historical account of the siting process of a geologic repository for HLW from the inception of this concept in the mid-1950s through December 1987. The many site investigations are recounted that have led to the current site in Nevada, which has been recommended and approved by the President for characterization. The record also relates to the important issues and events that were instrumental in shaping the course of siting over the last four decades. More than 60 geographical areas have been studied for this document.

With the passage of the Atomic Energy Act of 1954, a need for the siting of radioactive waste repositories was established, as the legislation permitted the construction and operation of nuclear reactors by private industry. Almost immediately, the Atomic Energy Commission (AEC), in collaboration with the National Academy of Science (NAS), initiated studies to determine and define the most promising means for the permanent disposition of these wastes.

In general, the siting of a geologic repository for HLWs can be divided into three rather distinct phases. The first phase, which covered the period from 1954 through 1972, was centered on the selection of a site in rock salt deposits in Kansas. A unique feature of this early siting process was the requirement of an existing excavation (disused mine) to handle alpha-contaminated wastes. This period concluded with the cessation of activities at Lyons, Kansas. The second phase of siting, which covered the period from 1973 through 1982, was a time during which many rock types and geographic areas of the country were investigated for multiple repository sites. A number of siting criteria documents were developed during this time, along with elaborate plans for site searches and plans for participation by state and other organizations in the siting process. This phase of siting ended with the signing into law of the 1982 Nuclear Waste Policy Act. The third phase covers the siting work laid out by procedures in the act and its 1987 amendment.

Although many issues and events have had some bearing on the search for repository sites throughout the country, it is likely that no single factor has been instrumental in bringing the work to the present point. Rather, the siting problem has evolved through several stages of development that would be expected in light of the growth and importance of the nation's nuclear reactor industry and the generally increasing environmental concerns over the disposition of nuclear and other types of hazardous wastes. This document is expected to be published as a DOE report in 1989.

Model Pillar Tests

Several model pillars of clay were fabricated from samples of three formations and then tested at various temperatures and pressures to determine their creep and deformational properties. These preliminary, or scoping, tests showed that the clays deform initially at a high rate of closure, then enter into a transient creep phase where the deformation rates decrease with time. The soft and pliable clays deform at ambient temperature in a

manner similar to other plastic, but harder, rocks like salt and shale. However, unlike rock salt and shale, which become more plastic at elevated temperatures, the clays lose moisture when heated and exhibit greater strength. The dominant element in the mechanical behavior of clays is clearly their water content. Future studies will focus on moisture-related creep phenomena and on porewater pressure buildup.

International Cooperative Work

As part of DOE's work on the development of technical data for geologic repositories, these documents are being prepared: (1) *Clay Deposits in the United States and Europe as Potential Hosts for High-Level Radioactive Waste Repositories;* (2) *A Program Plan for International Work on Sedimentary Rocks;* and (3) *In Situ Test Plan for International Cooperative Work in Clay-Rich Rocks.* The first report details the characteristics of clay-rich rocks in the United States and Europe and also documents current underground research activities on these rocks in Belgium and Italy. The program plan outlines the justification and objectives of cooperative work related to in situ testing of clay-rich rocks and also points out the universal need for obtaining fundamental data on the behavior of clays at nonambient repository conditions, which can only be found in underground excavations such as those that exist at Mol, Belgium. The third document, the in situ test plan, describes investigations that were made to measure the coupled thermal/mechanical/hydrological response of clay-rich rocks when subjected to thermal loads by buried heaters.

Clay-rich rocks are currently considered to be primary candidates for hosting geologic repositories in several European countries; and while they are not as prominently regarded for such use in this country, it is clear that the same behavioral data on clays are needed in DOE's Waste Technology Program, as clay-rich materials occur immediately below the projected disposal horizon at the Yucca Mountain site in Nevada. Also, clays are to be used as an encapsulating packing agent for waste canisters, regardless of the repository host rock type. Finally, the use of clays for the sealing of shafts, haulageways, drift openings, and boreholes in repositories would require the gathering of such basic information.

Mechanical Properties of Clay-Rich Rocks

As a first step toward development of a thermomechanical model for clay-rich rocks, standard baseline material properties were determined for the Yazoo and Porters Creek Clays. These clays are typical Cenozoic clays and were easily accessible from near-surface excavations. Mineralogically, the Yazoo Clay was found to contain about 73% clay minerals, while the Porters Creek Clay contained about 42% clay minerals. Each of the clay percentages is approximately 90% smectite. The Yazoo Clay is indurated and stiff and contains 26 wt % water; however, the Porters Creek Clay is soft and moldable and contains 45.5 wt % water. Neither the Yazoo Clay nor the Porters Creek Clay is naturally saturated in the undisturbed state, as both clays were observed to absorb additional water upon saturation. Their strengths were also found to be reduced upon saturation.

Baseline mechanical properties, including deformability and strength, are vastly different between the Yazoo and Porters Creek Clays. The Yazoo Clay has a nearly linear stress-strain response and displays distinct, catastrophic failure. On the other hand, the Porters Creek Clay exhibits a stress-strain response typical of wet clay—large strain accumulation at low stress differences and continuous strain hardening. As a result, the Porters Creek Clay is easily remolded and has little loss of strength upon remolding (insensitive clay). The Yazoo Clay, by contrast, disaggregated completely upon failure and could be remolded only with significant compactive effort. A failure envelope can be constructed for the Yazoo Clay but not for the Porters Creek Clay, which deformed to axial strains as high as 25% without loss of load-bearing capacity.

Integrated Data Base Program

The IDB is the official DOE data base for spent fuel and radioactive waste inventories and projections through the year 2020. The IDB Program is jointly sponsored by DOE's Office of the Assistant Secretary for Nuclear Energy, Office of the Assistant Secretary for Defense Programs, and the OCRWM. The purposes of the IDB Program are

to create and to maintain a reliable baseline of quality data and information to be used for national program planning, decision making, and other mangement activities. As the official data base for DOE, the IDB is readily available to the DOE community and to DOE-sponsored contractors. Work on maintaining and upgrading the IDB continued during this report period.

The IDB Program provides access to information on spent fuel and radioactive waste inventories and characteristics, including volume and mass, age, radioactivity, heat generation, chemical and physical properties, location, packaging, and nuclide composition. The IDB, which uses a modular system of computer codes, also provides projections based on expected growth rates, schedules for new facilities, waste generation factors, and treatment assumptions. Projections for commercial waste are based on official DOE/EIA electrical growth projections.

IDB Annual Report

The annual report of the IDB Program presents summary-level tables and figures that compare volumes and radioactivity levels of all forms of spent fuel and radioactive waste [HLW, TRU waste, and low-level waste (LLW)].[23] These materials are generated by commercial fuel cycles, including D&D activities, defense programs, remedial action programs (RAPs), and institutional and industrial operations.

A formal system is in operation to ensure the necessary quality control for both summary and detailed levels of data. These procedures were developed in cooperation with the DOE sponsors and the IDB Steering Committee. The quality control procedures are based on the requirements of the DOE data users. A commitment has been made to document all actions. This documentation produces a record of events and ensures that library files, published data, and computerized files are internally consistent and accurate.

Recent efforts have been extended toward acquiring quality data in the area of miscellaneous, highly radioactive materials that may require geologic disposal and in mixed waste. The first area contains a variety of materials, including intact spent fuel elements or solids remaining after experimental testing and for which no reprocessing is planned. These materials constitute less than 1% of the total waste but, because of their complex and varied nature, may require elaborate and costly pretreatment and packaging. Efforts in mixed-waste data acquisition have been initiated with the downloading of summary mixed-waste data from the Hazardous Waste Disposal, Development and Demonstration (HAZWDDD) Program for the five Energy Systems sites. This material has been presented to the national Hazardous Waste Remedial Action Program (HAZWRAP) as a prototype for their upcoming call for 1988 data. It is anticipated that the IDB Program will be highly involved in the QA aspects of acquiring and manipulating these mixed-waste data.

Summary Data Tables and Figures

The usefulness of the IDB is shown in its summary tables and figures, in which data for a variety of waste forms can be listed and displayed in a self-consistent manner. As an example, Table 2.6 shows a summation of spent fuel and waste inventories through December 31, 1987, and Table 2.7 includes projections to years 2000, 2010, and 2020. These tables were generated from the core data files through the use of SAS programming.

Figure 2.10 presents the volume percentages of commercial and DOE/defense wastes and spent fuel accumulated through 1987. It provides a clear pictorial overview of the relative amounts of this country's radioactive wastes. In Fig. 2.11, the radioactivities of the same wastes are presented in a similar fashion. On this basis, LLW comprises over 85% of the volume and spent fuel over 90% of the radioactivity for disposal. It is obvious from these two figures why spent fuel and LLW are the major areas for concern in recent discussions on the storage and disposal of radioactive waste.

In summary, the IDB provides, in a wide variety of formats, technical information on spent fuel and radioactive waste in terms of inventories, projections, and characteristics. This information is useful in various program-planning exercises and in response to data requests by DOE, state and local governments, and interested third parties. Ongoing efforts are concentrated on (1) continuing the presentation of consistently high-quality data; (2) streamlining the methodology for collecting data for the various waste types; (3) using desktop publishing methods for simplifying the production of the annual inventory report; and (4) acquiring and presenting new areas of radioactive waste data, including miscellaneous material and mixed wastes.

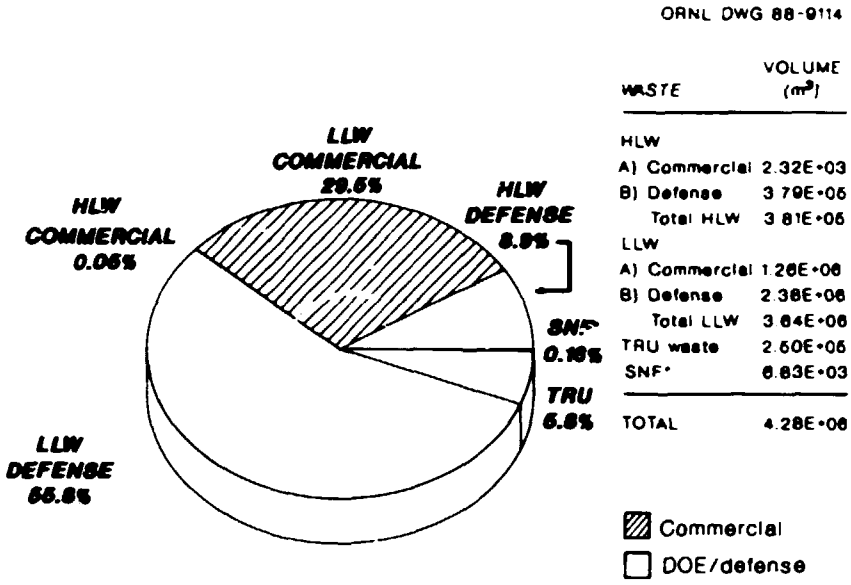
Table 2.6. Spent fuel and radioactive waste inventories as of December 31, 1987

Spent fuel	Mass (MTU)	Activity (MCi)	Thermal power (kW)
BWRs (34,007 assemblies)	6,178	5,064	18,500
PWRs (22,877 assemblies)	9,725	12,583	48,300
HLW	**Volume (m^3)**	**Activity (MCi)**	**Thermal power (kW)**
Savannah River (DOE)	127,600	731	2,099
Idaho (DOE)	11,900	63	183
Hanford (DOE)	239,700	481	1,414
West Valley (commercial)	2,320	30	89
TRU waste (DOE)	**Volume (m^3)**	**Activity (MCi)**	**TRU elements (kg)**
Buried TRU waste	190,837	0.25	771
Stored TRU waste	58,749	3.87	2,064
Stored as LLW waste	35,824		13
LLW	**Volume (m^3)**	**Activity (MCi)**	**Land used (ha)**
DOE sites	2,381,000	13.99	224
Commercial sites	1,257,900	4.83	72
Uranium mill tailings (commercial)	**Volume (m^3)**		
Licensed mill sites	116,000,000		
Remedial action programs (DOE)	**Volume (m^3)**		**No. of sites**
UMTRAP (Uranium Mill Tailings)	4,408,550		25
FUSRAP (Formerly Utilized Sites)	150,631		29
Defense D&D Program	*a*	220	
SFMP (Surplus Facilities Management Program)	*a*		100
GJRAP (Grand Junction)	52,070		593

*Information not available.

Table 2.7. Current and projected volumes of spent fuel and radioactive waste

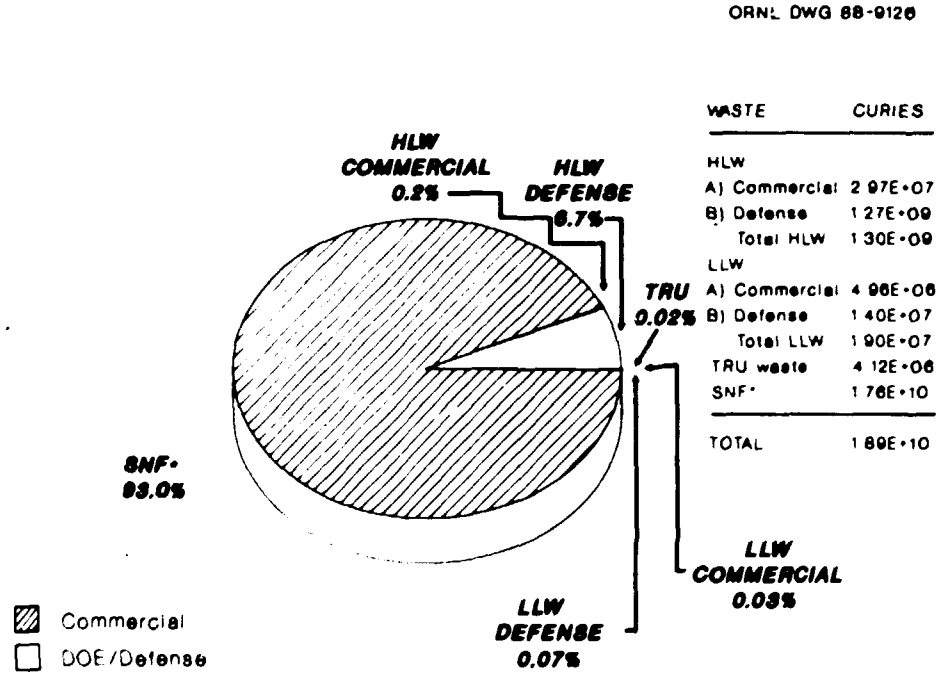
Source of material and type	End of CY 1987		End of CY 2000		End of CY 2010		End of CY 2020	
	Volume (10^3 m^3)	Radioactivity (10^6 Ci)	Volume (10^3 m^3)	Radioactivity (10^6 Ci)	Volume (10^3 m^3)	Radioactivity (10^6 Ci)	Volume (10^3 m^3)	Radioactivity (10^6 Ci)
			DOE/Defense					
HLW								
Interim storage	379	1,274	328	1,281	329	1,025	335	1,039
Glass	0	0	2.2	159	3.4	306	3.5	347
TRU waste								
Buried	191	0.2	191	0.2	191	0.2	191	[illegible]
Stored	59	3.9	99	17.1	130	27.3	N/A	N/A
LLW	2,381	14.0	4,344	19.8	5,611	22.9	6,783	24.8
UMTRAP and GJRAP								
Mill tailings and other wastes	4,461		29,482		29,482		29,482	
FUSRAP	151		1,087		1,101		1,101	
Defense D&D Progam			149		150		150	
SFMP			2,340		N/A		N/A	
			Commercial					
Spent fuel (10^3 MTIHM)	15 9	17,646	40 4	31,700	60 1	38,900	77 4	34,600
Commercial HLW glass (WVDP)	0	0	0 21	22 0	0 21	17 6	0 21	13 9
LLW	1,258	5 0	1,978	5 5	2,556	5 8	2,991	5 0
D&D (LLW)			0		66 9		746 1	
Mill tailings	116,200	0 9	124,000	1 3				



WASTE	VOLUME (m^3)
HLW	
A) Commercial	2.32E+03
B) Defense	3.79E+05
Total HLW	3.81E+05
LLW	
A) Commercial	1.26E+06
B) Defense	2.38E+06
Total LLW	3.64E+06
TRU waste	2.60E+05
SNF*	6.83E+03
TOTAL	4.28E+06

*Includes spacing between fuel assembly rods.
Does not include DOE/defense spent fuel to be reprocessed.

Fig. 2.10. Volumes of commercial and DOE/defense wastes and spent fuel accumulated through 1987. SNF = spent fuel.



WASTE	CURIES
HLW	
A) Commercial	2.97E+07
B) Defense	1.27E+09
Total HLW	1.30E+09
LLW	
A) Commercial	4.96E+06
B) Defense	1.40E+07
Total LLW	1.90E+07
TRU waste	4.12E+06
SNF*	1.76E+10
TOTAL	1.89E+10

*Does not include DOE/defense spent fuel to be reprocessed

Fig. 2.11. Radioactivities of commercial and DOE/defense wastes and spent fuel accumulated through 1987. SNF = spent fuel.

Special Studies

The TRU Waste and Integration Program for planning, integration, and technical development for TRU waste management is centered in DOE's Albuquerque Operations Office. The program's goal is to implement permanent disposal of TRU wastes from all defense waste locations, thus eliminating the need for interim storage. The Waste Isolation Pilot Plant (WIPP), located in southeastern New Mexico, is being designed to serve as this central, permanent depository. Retrievable emplacement tests are now being conducted at the site to demonstrate that the site could be approved as a permanent disposal facility for TRU waste. In support of this activity, the Operations Division at ORNL has the primary responsibility of preparing certification documentation, design and construction of an ORNL TRU Waste-Handling and Packaging Plant (WHPP), and the later shipment of the packaged wastes to the WIPP. Chem Tech has assisted with several of these preparatory tasks.

During this reporting period, Chem Tech assisted with the development of an Activity Description Memorandum (AcDM) for the retrieval and investigation of a remote-handled (RH) waste cask, aided in the preparation of several waste characterization documents (such as the *Generator Assistance in Preparation of WIPP RCRA Permit Application* and *TRU "Mixed" Waste Characterization Data*), and continued to provide the Joint Integration Office (JIO) with updates of ORNL's TRU waste volumes. It is in the role of waste characterization that Chem Tech contributed most during this period. As the TRU waste certification effort advances, Chem Tech is expected to continue to be active in all phases of the TRU waste-handling effort.

TRU Waste Inventory Work-Off Plan

In response to the JIO Program Office's directive that all major defense facilities being managed by DOE and possessing large inventories of TRU waste must provide a plan for disposal of this waste, Chem Tech prepared the ORNL inventory work-off plan. This work-off plan covers all stored and newly generated contact-handled (CH), RH, and special case (SC) TRU wastes. TRU waste included in this work-off plan is defined under DOE Order 5820 as containing >100 nCi/g of alpha-emitting radionuclides with atomic numbers greater than 92 and half-lives >20 years.

The *Final Inventory Work-Off Plan (IWOP) for ORNL Transuranic Wastes* addresses ORNL's strategy for retrieval, certification, and shipment of its stored and newly generated CH and RH TRU wastes to WIPP.[24] This Chem Tech document considers the WIPP/Waste Acceptance Criteria (WAC) certification compliance and is consistent with the Long-Range Master Plan for Defense Transuranic Waste Management. Because many changes and developments have occurred since the original strategies and plans presented in the IWOP were considered, the document is subtitled "1986 Version."

This task characterizes ORNL's TRU waste by type and estimates the number of shipments required to work it off; presents the methods, facilities, and systems required for certifying and shipping the waste; presents work-off strategies and schedules for retrieval, certification, and transportation of the waste; presents the resource needs and additions that will be required for the effort and projects costs for the long-term TRU waste management program; and lists the public documentation required to support certification facilities and strategies.

The strategies for certifying and shipping TRU waste are being redefined and reevaluated—even the definition of TRU waste is undergoing modification. To address these changes and provide a yearly update of the TRU waste inventories listed in the IWOP, Chem Tech continues in its support work. The "1988 Transportation and System Integration Request for Data" is such a task. This update of ORNL's IWOP is further used to update the IDB.

The March 1988 update report gives the inventory of RH TRU as of December 31, 1987, as 1290 m^3 (of which ~950 m^3 are sludges) and that of CH TRU as 600 m^3. An estimated 2520 cumulative newly generated (NG) and stored RH TRU waste canisters are to be worked off by the year 2013, and 4950 cumulative stored and NG CH TRU waste drums are to be worked off, not including the 85 standard boxes of CH TRU waste that are expected to be generated. Because the design and payload of shipping containers are currently being debated, it is not practical to project the number of shipments per year.

Other Chem Tech support work includes continued characterization of TRU waste, especially RH

TRU wastes. A data base is currently being formed for RH TRU wastes that will lend support to the WIPP certification effort and the structuring and layout of the WHPP by providing more detail on the contents of the individual casks in storage at ORNL.

Regulatory Analysis

Legislation that affects DOE is continuously being generated. Also, regulatory agencies, such as EPA and NRC, are promulgating new regulations related to past legislation at an ever-increasing rate. Often, the regulations have significant effects on the way DOE carries out its business; thus, an evaluation of these impacts should be made as early as possible in the process to allow DOE time to respond. This effort monitors the *Federal Register* (FR) for regulations that affect DOE and prepares comments for submission to the appropriate DOE headquarters organization; at present, only DOE's Office of Environmental Guidance and Compliance (OEG) and OCRWM have availed themselves of this service.

Effect of RCRA on DOE/OCRWM Programs

Using published information, it was determined that nonradioactive materials that could potentially come under the Resource Conservation and Recovery Act (RCRA), by applying DOE's recent by-product rule interpretation, are contained in spent fuel and HLW. Further, it was determined that none of these materials were RCRA "listed wastes" and that the wastes would be considered hazardous only if they failed the EPA Extraction Procedure Toxicity Characteristic Leaching Procedure (TCLP) test. Based on the most conservative assumption of 100% leachable material, it could be predicted that only one RCRA substance in spent fuel, cadmium, could fail the TCLP concentration test. However, it is almost certain that an actual test at these low concentrations would not leach cadmium out of spent fuel in large enough quantities to fail the TCLP test. Hence, spent fuel would not come under the RCRA.

Conversely, most of the RCRA materials found in some types of HLW are present in large enough quantities to cause the proposed TCLP test to classify the waste as hazardous under RCRA, based on concentration alone. Thus, a TCLP leach test would be necessary on each waste to resolve the issue. However, the EPA TCLP test is designed to test wastes from a municipal landfill and is shown not to be applicable to the conditions of the repository. Finally, based on comments by DOE and others to the EPA regarding the inequities of the TCLP test, they are proposing to remove the grinding requirement. HLW in glass logs or cementitious waste forms would surely pass the amended test. The conclusion is that there is a high probability that neither spent fuel nor HLW is subject to the RCRA.

Guidance on the Effect of EPA Legislation on DOE Operations

The DOE/OEG is responsible for evaluating regulations that have a potential effect on DOE operations. The EPA actively promulgates rules in response to legislation, such as the RCRA through the FR process. OEG has asked ESD to continually monitor the FR for proposed EPA rules that could affect DOE and to thoroughly evaluate the potential impact in time to draft DOE comments to the rules before the promulgation process is complete. These rules are often very specific to chemicals and chemical processes, requiring comments by chemists and chemical engineers.

During this report period, the following proposed EPA rules have been evaluated:

1. proposed restrictions on underground injections into deep wells,
2. methods of detecting and controlling groundwater contamination by uranium mill tailings,
3. determination of waste treatment standards for hazardous wastes,
4. incineration of contaminated soils,
5. dilution and attenuation factors related to transport of wastes after land disposal,
6. TCLP effluent reference levels for organic constituents, and
7. TCLP particle-size reduction.

2.2 Waste Treatment and Environmental Control Technology

During this report period, Chem Tech continued to expand and broaden its research, development, and demonstration (RD&D) efforts in waste treatment and environmental control, both for DOE-related wastes and other federal agencies' wastes.

These efforts ranged from applied research and development of physical, chemical, and biological treatment methods for specific and generic waste problems through design and engineering support to technical support for the development of technical information needed for remedial actions and compliance strategies, including test and evaluation demonstrations of new and innovative waste treatment technologies. In all of these activities, Chem Tech emphasized multidisciplinary and multidivisional approaches, drawing on or providing the appropriate skills as needed.

DOE-Funded Waste Management RD&D

Major portions of the Chem Tech Division personnel and facilities have been dedicated to RD&D of technologies for application in waste management needs within the DOE, both on the Oak Ridge Reservation (ORR) and other sites. The Division staffed and managed the WMTC and its support group. The WMTC plays a technical leading role in the development and implementation of the DOE Model for Waste Management. Division staff have conducted research in the application of various bioprocesses to waste management problems and assisted in the test and evaluation of a biodenitrification plant for the Feed Materials Production Center (FMPC) in Fernald, Ohio. Chem Tech personnel have provided design support for ORNL waste treatment facilities by managing environmental capital projects and performing waste stream characterization and systems analysis. Chem Tech staff have been instrumental in improving the treatment of ORNL liquid and gaseous effluents. The division is a major contributor in developing and applying technology to the decontamination and decommissioning of ORNL facilities. Chem Tech has also continued to provide technical assistance to DOE sites in developing and applying appropriate, cost-effective processes to immobilize radioactive and hazardous chemical wastes.

Biotechnology Applications to Waste Treatment

Microorganisms are capable of detoxifying and destroying a wide variety of chemical wastes. Two projects are currently under way to use these capabilities for new bioprocess technologies for remediation of sites contaminated with polychlorinated biphenyls (PCBs) and chlorinated alkenes such as trichloroethylene (TCE). In addition, the ORNL biodenitrification process for treatment of nitrate wastewater has been successfully implemented at the DOE Feed Materials Production Center in Fernald, Ohio. The recent technical accomplishments in these projects are summarized in the following subsections.

Bioremediation of PCB-contaminated Soils

A new project has been initiated to develop a bioremediation technology for soils contaminated with PCBs. This work is being carried out by an interdisciplinary team of professional and technical staff members in Chem Tech and ESD at ORNL, the University of Tennessee, and the Oak Ridge Research Institute.

Both field and laboratory studies are under way. Six 100-L field lysimeters were placed at a PCB-contaminated site on the Bear Creek floodplain in June 1987 (Fig. 2.12). This site contains about 20 mg/L of PCBs in the soil. Different conditions of nutrients, carbon sources, and microbial inocula were established in each lysimeter. The lysimeters were watered and aerated weekly during the summer and fall of 1987, and then they were allowed to lie undisturbed over the winter. Weekly maintenance was resumed in June 1988. Soil samples are taken periodically for PCB and microbial activity assays.

Concurrently, laboratory studies were done to assess the potential natural PCB-degrading bioactivity in these soils and to evaluate microbial cultures believed to be capable of PCB degradation. Two 100-L slurry bioreactors were also operated in the laboratory using contaminated soil from the Bear Creek site. One bioreactor was inoculated with a strain of *Alcaligenes*, believed to be capable of PCB degradation, while the other bioreactor served as a control.

Fig. 2.12. Field lysimeters at PCB-contaminated site on the Bear Creek flood plain.

Laboratory results have shown that soils at the Bear Creek floodplain site do contain indigenous microorganisms capable of biodegradation of chlorinated biphenyls. Tests with radiolabeled 4-chlorobiphenyl (4CB) have shown production of substantial radiolabeled CO_2. Gene probe assays have shown that indigenous microorganisms have the genetic potential for degradation of 4CB. In addition, tentative evidence has been obtained for anaerobic dechlorination of PCBs under laboratory conditions using microbial cultures isolated from other contaminated sites on the ORR.

No unequivocal evidence has yet been obtained for degradation of the PCB contamination at the Bear Creek site either in the slurry bioreactors or in the lysimeters. However, aeration and watering of the lysimeters appear to increase the population of microorganisms that exhibit potential PCB-degrading characteristics under laboratory conditions. The various positive laboratory results are quite encouraging and suggest that it may be possible to establish conditions for biodegradation of PCBs in soil. The emphasis in current field and laboratory work is on anaerobic dechlorination of PCBs in order to improve the subsequent aerobic biodegradability.

Biological Degradation of Trichloroethylene in Groundwater

The purpose of this project is to demonstrate the technical feasibility of a methanotrophic bioreactor for the remediation of TCE and 1,2-*trans*-dichloroethylene (DCE) contamination in groundwater. The work is being conducted by personnel from Chem Tech and ESD.

Methanotrophic microorganisms use methane as a sole carbon source. Methane is initially oxidized in the presence of oxygen by the enzyme methane monooxygenase. Current evidence suggests that this same enzyme can nonspecifically catalyze the oxidation (to epoxides) of some chlorinated alkenes. The unstable epoxides degrade to biodegradable intermediates that the microorganisms can subsequently use as carbon sources. Ultimately, the chlorinated alkenes are degraded to CO_2, H_2O, and Cl^-.

A bench-scale trickle-filter bioreactor system is being operated to determine those factors which affect the rate and extent of TCE and DCE degradation. The bioreactor is about 5 cm ID, 110 cm tall, and packed with 0.6-cm ceramic berl saddles. The methanotrophic microbial population, which is maintained as a biofilm on the packing, was initially isolated by C. D. Little (ESD) from a TCE-contaminated well on the ORR. The bioreactor system has been maintained in continuous operation at a liquid flow rate of 10 mL/min and a gas (methane/air mixture) flow rate of 25 mL/min. Both the liquid and the gas flow downward in a concurrent stream over the packing.

The influent concentrations of TCE and DCE have been maintained at about 1 mg/L. With a methane concentration in the range of 2 to 4%, approximately 50% of the TCE was removed from the solution (presumably by biological action), approximately 10% appeared in the off-gas, and the remainder was in the liquid effluent. No DCE could be detected (detection limit ~10 μg/L) in the liquid effluent or off-gas.

The liquid residence time was estimated to be from 6 to 9 min for a single pass through the system. When the residence time in the bioreactor was extended using total recycle of the liquid phase, approximately 80% of the TCE was removed within two residence times (15 min). During the recycle of liquid with various influent concentrations of TCE (0.4 to 1.2 mg/L), the effluent concentration consistently approached 0.2 mg/L within 15 min, and further removal occurred at a much slower rate. Effluent TCE concentrations of 40 to 60 μg/L were reached after 2 to 4 h.

The reaction rate appears to be a function of the influent TCE concentration; it was lower at lower TCE concentrations and higher at higher TCE concentrations. This relationship may be the result of a direct competition of TCE with methane for the active site(s) on methane monooxygenase.

In separate batch experiments done in cooperation with A. V. Palumbo (ESD), it was shown that the microbial population from the bioreactor converted about 63% of ^{14}C-labeled TCE to CO_2. Twenty-six percent appeared in the cell mass, and 11% appeared as water-soluble products. In contrast, pure cultures of methanotrophs produced a greater amount of water-soluble products. Undoubtedly, nonmethanotrophic microorganisms that coexist in the biofilm were able to metabolize the water-soluble products from the methanotrophs. In the bioreactor system and in batch experiments, higher methane concentrations lowered the TCE degradation rate, again presumably because of a direct competition of TCE and methane for the enzyme site(s). The extent of TCE degradation, but not the rate, was dependent upon initial oxygen concentration. This behavior indicates that sufficient oxygen must be supplied, primarily to maintain the general health of the population.

Laboratory studies are planned to further define parameters for optimal reactor operation. Funding is being sought for construction and operation of a small pilot-scale bioreactor at a contaminated site to demonstrate the process using actual contaminated water.

Biodenitrification of Fernald Wastes

Design verification studies for the fluidized-bed bioreactors under construction at the FMPC in Fernald, Ohio, were conducted at ORNL from May 1985 through May 1986. These studies, using actual FMPC nitrate wastewaters and the pilot-scale bioreactors at ORNL, indicated that the design biodenitrification rate for the FMPC bioreactors could be achieved; however, softening of the feed water would be required, and pH adjustment within the bioreactors might be necessary.

In September 1986, the FMPC began development of a test plan for startup and demonstration of two of the four bioreactors at the FMPC. Several Chem Tech staff members participated actively in the design of the test plan. Meanwhile, one of the bioreactors was started, using bioparticles from the ORNL bioreactors. Only dilute nitrate wastewater was available at that time at the FMPC because the refinery was not operating. The single bioreactor was operated on this dilute wastewater to build up the biomass in preparation for later startup of a second bioreactor. Chem Tech staff participated on-site during this startup and shakedown phase.

The refinery at the FMPC was restarted in April 1987, and wastewater containing up to 4 g/L of nitrate became available. Subsequently, an official demonstration of the FMPC biodenitrification process was conducted from May 5 through June 24, 1987. Two bioreactors in series were used. Again,

Chem Tech staff members provided on-site process engineering assistance to help evaluate the data and guide the demonstration program.

During the demonstration test, the FMPC bioreactor performance was comparable to the performance of the ORNL pilot bioreactors. The biodenitrification rate at FMPC actually rose faster during the startup period than it did at ORNL. However, the maximum rate of the FMPC reached only about 75% of the design rate during the demonstration test. This result is believed to be due to carbon and nitrate limitations arising from inadequate process control equipment, insufficient bioparticles in the bioreactors, and the relatively short period of operation under favorable conditions.

The FMPC is continuing to use the biodenitrification plant to treat nitrate wastewaters, and it is now in regulatory compliance for nitrate discharge. Only two of the four bioreactors are needed to handle the present volume of wastewater. ORNL is not involved with the FMPC denitrification plant at the present time.

Microbial Accumulation of Neptunium

Neptunium, an activation product of ^{238}U, can occur in wastewaters from nuclear facilities. It was recently proposed that wastewater outfalls be required to meet the "derived concentration guide" for drinking water (DOE Draft Order 5480.XX, Radiation Protection of Public and Environment). A discharge limit of 3×10^{-5} μCi/L has been proposed for neptunium. There is concern within the nuclear industry that, using existing technologies, this requirement could be difficult to meet for neptunium. Our experience with the microbial uptake of other radionuclides prompted a preliminary investigation of whether microorganisms could accumulate neptunium and be of use in removing this radionuclide from nuclear processing wastewaters.

As shown in Table 2.8, a variety of microbial species accumulated ^{237}Np; several accumulated from 11 to 15 mg Np/g cells (dry weight). The distribution coefficients (g metal per g cells/g metal per g solution) ranged from a few hundred to ~1500. This is an order of magnitude less than that found for several other metals and microorganisms in previous studies.

The results shown in Table 2.8 were obtained at an initial pH of 4.0. Neptunium uptake by *Micrococcus luteus* was found to be greater at pHs 4 and 7 than at pH 5.4 (data not shown). A similar decrease in neptunium uptake at pH 5.4 occurred for *Streptomyces viridochromogenes*. The reason for this is not currently understood.

The rate of neptunium uptake was rapid. Maximum uptake (at pH 4) by *M. luteus* occurred within 10 min. The rate of uptake by *S. viridochromogenes* was somewhat slower.

Little, if any, accumulation of neptunium occurred (data not shown) when *M. luteus* and the denitrifying mixed culture were exposed to ^{237}Np for 3 h at an initial concentration of ~35 μg/L (3×10^{-2} μCi/L). This is approximately 1000 times the proposed effluent requirement. Thus, the utility of these particular species as biosorbents for removing neptunium from nuclear processing waste streams is not indicated. However, other microorganisms and biomass sources (e.g., sewage sludge) could be tested. If it is determined that microorganisms in general have a similar low affinity for neptunium, this information would be useful in understanding the distribution and fate of neptunium in nature and in waste treatment systems.

Water Management Technology Center Support

Current needs, future concerns, and problems associated with past practices are being addressed by the DOE Model for Waste Management, a cooperative effort of DOE/ORO and Energy Systems. The mission of WMTC is to play a leading technical role in the model. Under the DOE Model, waste management needs are identified and prioritized, applicable technologies are evaluated and developed, and selected waste management technologies involving innovative approaches are demonstrated. The results are translated into new and more efficient alternatives for solving problems of interest to DOE and other federal agencies.

The WMTC support group consists of a team of engineers and scientists from Energy Systems working with private industry and business, the academic sector, and government agencies to define solutions to waste management problems. The cornerstone of WMTC philosophy is that the proof of any waste management technology lies in demonstrated performance. Therefore, projects may be demonstrated at appropriate sites in the Oak Ridge system in cooperation with line organizations responsible for site-specific problems.

Table 2.8. Accumulation of neptunium by microbial cells

Organism (growth medium)[a]	Time (h)	Neptunium remaining in solution[b] (mg/L)	Cells (mg/L, dry wt)	Sorbed neptunium (mg/g cells)	Distribution coefficient[c]
Control[d]	1	36			
	3.5	35			
Saccharomyces cerevisiae NRRL Y2574 (YM)	1	33	1.7	2	40
	3.5	29	1.7	4	140
Pseudomonas aeruginosa NRRL B-4452 (YM)	1	15	1.8	11	710
	3.5	14	1.8	11	820
Streptomyces viridochromogenes ATCC 3356 (YM)	1	13	1.8	13	940
	3.5	10	1.8	15	1540
Micrococcus luteus ATCC 4698 (ENR)	1	9	2.0	14	1550
	3.5	10	2.0	13	1390
Rhizopus oryzae NRRL 395 (YM)	1	28	1.0	8	310
	3.5	30	1.0	6	200
Scenedesmus obliqus (Bold's)	1	31	0.3	16	500
	3.5	32	0.3	14	430
Denitrifying mixed culture[e]	1	22	1.6	8	380
	3.5	22	1.6	8	380

[a]YM = YM Broth (Difco, Detroit, Michigan). ENR = heart infusion broth, 1.25%; nutrient broth, 0.54%; yeast extract, 0.25%; tryptic soy broth, 1%; protease-peptone, 0.2% (All Difco). Bold's medium (see ref. 25). The bacteria and fungi were cultured for 48 h and *S. obliqus* for 5 d, all at 30°C.

[b] $$\frac{(\text{dpm})(237\ \text{mg/mol})}{(0.9\ \text{counting efficiency})(5 \times 10^{-4}\ \text{L})(3.709 \times 10^{8}\ \text{dpm/mmol})}$$

The initial Np concentration was 36 mg/L, and the initial pH was 4.0.

[c] $$\frac{\text{g Np/g cells (dry wt)}}{\text{g Np/g solution}}$$

[d]Control contained water in place of cell suspension.

[e]Obtained from a fluidized-bed denitrifying bioreactor (courtesy of J. B. Patton, Westinghouse Material Co. of Ohio, Fernald, Ohio).

Organizationally, the WMTC support group consists of technical experts drawn from Energy Systems, with Chem Tech at ORNL being assigned WMTC management responsibility. The team is complemented by a variety of subcontractors and consultants with expertise in specialized areas.

The WMTC's strategic objectives are:

1. Assisting sites and the Energy Systems Office of Environmental and Safety Activities in the development of strategies for managing the wastes at Energy Systems facilities.
2. Facilitating the demonstration of innovative waste treatment technologies at DOE/ORO facilities and evaluating the technical and economic feasibility of these technologies.
3. Facilitating waste management technology exchange among DOE facilities, other federal agencies, universities, and the private sector.
4. Promoting research and development (R&D) in the waste management area by matching the capabilities and interests of Energy Systems scientists and engineers with recognized R&D needs on DOE and other federal facilities. Energy Systems has assigned the WMTC technical support responsibility for several multisite waste management programs. These include the Low-Level Waste Disposal Development and Demonstration (LLWDDD) Program, which is funded through the ORNL Interim Waste Operations Program; and the Hazardous Waste Development, Demonstration, and Disposal (HAZWDDD) Program, which is funded by the Energy Systems Office of Environmental and Safety Activities. The WMTC is also conducting large-scale demonstrations on DOE/ORO facilities for HAZWRAP.

Low-Level Waste Disposal Development and Demonstration Program

The Waste Management Technology Support Group provides the program management and the management of major work elements of the LLWDDD Program. The purpose of the LLWDDD Program is to develop new and improved waste disposal facilities for the management of low-level solid wastes generated on the ORR. The task is needed both because current disposal capacities are being exhausted and because improved waste management practices are required to satisfy regulatory groups.

The scope of the LLWDDD Program encompasses the following:

1. developing and securing approvals for an LLW management strategy for the ORR;
2. assisting sites in the development of site-specific LLWDDD strategy implementation plans and assisting the Central Waste Management Office by reviewing these plans and integrating them into an overall LLWDDD plan;
3. defining the functional requirements for new disposal facilities (to include characteristics of waste streams or waste packages to be routed to the disposal facilities, a schedule for needed new disposal capacity for various waste streams or waste packages, and performance requirements for disposal facilities);
4. siting new disposal facilities and satisfying National Environmental Policy Act (NEPA) requirements for disposal facilities;
5. conducting demonstrations of treatment and packaging, storage, and disposal technologies to reduce technical and economic uncertainties associated with deployment of new disposal facilities;
6. supporting improved LLW management operations conducted by waste management organizations at plants on the ORR (to include assistance in the design of disposal facilities, development of monitoring plans, and evaluation of performance); and
7. preparing new sites and constructing new disposal facilities for operation by waste management organizations at plants on the ORR.

Current planning is based on beneficial occupancy of new prototype waste disposal facilities on the ORR by September 30, 1991. These facilities will encompass a complex of sites and disposal technologies representing best management practices for the different LLWs handled in the facilities. These disposal operations will be treated as demonstrations for an indefinite period of joint assessments by Energy Systems, DOE, and regulatory agencies.

The LLWDDD Program consists of four major work elements:

1. source term characterization and performance activiti ,
2. demonstration and assessment of waste management technologies,
3. development of environmental data package and support of the EIS process, and
4. development of new LLW disposal facilities.

Source Term Characterization and Performance Activities. One work element of the LLWDDD Program is the development of new regulator-approved, performance-based radioactive waste disposal systems for managing solid LLW on the ORR. A waste classification framework has been developed by the LLWDDD Program to help focus the development of waste disposal systems and to ensure compliance with current and future regulations. Three classes of on-site solid LLW disposal practices are included in this framework:

1. Class I—solid LLW that is suitable for unrestricted burial and has levels of contamination that will result in radiation doses below the regulatory limits,
2. Class II—solid LLW that is suitable for engineered disposal and that has contamination levels that will result in doses below the regulatory limits at the end of a period of institutional control, and
3. Class III—solid LLW that is suitable for restricted disposal and contains constituents with long half-lives that could result in doses to individuals or intruders that exceed the regulatory limits unless intruder protection is provided.

The Waste Facility Performance Model (WFPM) is a new initiative of the LLWDDD Program. It is being developed as a response to the need for analytical capabilities to help characterize and evaluate the performance and cost-effectiveness of solid LLW management systems. This capability will support waste acceptance processes and waste management decisions under the LLWDDD Program, as shown in Fig. 2.13. A fully developed WFPM will help ensure that the design and performance objectives for solid LLW disposal established under the LLWDDD Program are met in a cost-effective manner.

Central to the development of a waste acceptance process and waste management decisions is the ability to evaluate the performance of the disposal system and to determine the amount of radioactivity released from the disposal system into

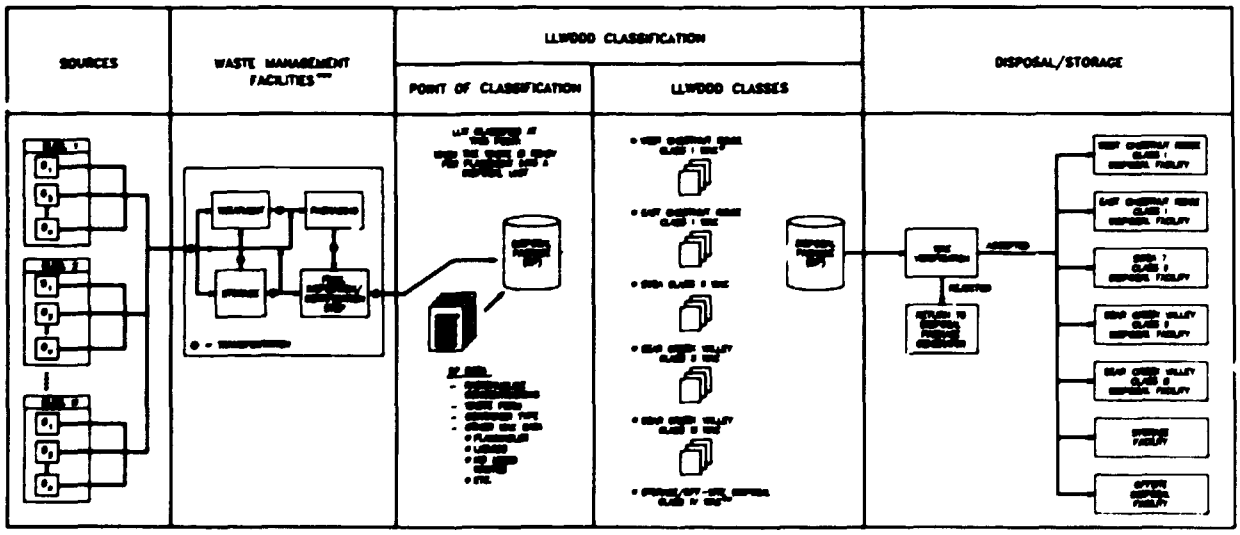


Fig. 2.13. Functional areas of solid LLW management systems. The WFPM is being developed for the LLWDDD program as a systems analysis tool to support the characterization and evaluation of performance and cost-effectiveness of treatment, packaging, and disposal technologies.

the environment (source terms). These source terms will then be used by pathway analysts in studies to calculate the resulting radiation doses. The pathway analysis studies will help ensure that the radiation doses resulting from the source terms are within the regulatory limits established under the LLWDDD Program. The WFPM helps ensure that the disposal system meets the performance objectives in a cost-effective manner.

Elements of the disposal system to be included in performance assessment studies using the WFPM are the waste disposal package (waste form plus container), the waste packaging method, and engineered barriers for leakage containment (e.g., liner, leachate collection system, and cover). In addition, this institutional control procedure will also be factored into the facility performance assessments.

Phase I of this initiative is the development of the methodologies and the design of the data bases needed to conduct performance assessments of the disposal systems to generate the source terms. Phase I is planned for completion by the end of FY 1988.

Phase II will develop a cost model and the supportive data bases for cost estimating the development of the waste disposal systems.

When the WFPM is complete, it will provide the LLWDDD Program with a capability to perform technology assessments (performance vs costs) and sensitivity studies for cost-effective disposal system designs. Also, the results will aid in defining additional data needs in support of improved and cost-effective solid LLW disposal practices.

LLWDDD Program Demonstrations. A number of demonstrations have been initiated and carried out to support the goal of the LLWDDD Program, which is defining satisfactory technology to treat and dispose of LLW. These demonstrations include: (1) the waste isolation technology project, (2) supercompaction of ORNL drums, (3) the ORNL tumulus disposal demonstration, (4) supercompaction of Y-12 baled waste, and (5) improved disposal operations at ORNL.

Evaluation of Vendor, Process, and Waste Form and Packaging of ORR Waste Streams—Waste Isolation Technology Project. The Waste Immobilization Technology Project (WIT) under the LLWDDD program is specifically set up for the conditioning, treatment, and immobilization of selected liquid waste streams on the ORR. The objective of this program is to find processes that will neutralize the hazardous and radioactive nature of the liquid wastes and put them in a form suitable for interim storage for a period of 10 to 15 years, followed by eventual disposal in a land facility. The major emphasis of the program is the identification and demonstration of innovative technologies for processing four specific waste streams. These liquid waste streams generated by operations at various facilities on the ORR, because of the nature and volume of the stream, pose the largest immediate problem for waste management operations. All of these waste streams contain hazardous, radioactive, or a combination of both hazardous and radioactive elements and compounds. The four waste streams specifically being addressed are:

1. Stream 1. Y-12 wastewater treatment sludge containing heavy metals and spent uranium.
2. Stream 2. ORNL wastewater treatment water softening sludges containing Cs, Sr, Co, and trace rare earths.
3. Stream 3. Y-12 metal plating sludges containing heavy metals and chlorinated phenolics.
4. Stream 4. ORNL wastewater treatment concentrates containing very high nitrates, Cs, Sr, and trace rare earths.

This project, which is broken into three phases, investigates and compares different processes using cement, bitumen, polymer, or another binder for immobilization of the waste streams.

1. Phase 1 is the development, by vendors, of specimen waste forms using surrogate waste formulas for testing and evaluation.
2. Phase 2 is the pilot-scale demonstrations of the processes that are acceptable in Phase 1 testing.
3. Phase 3 is the full-scale testing of the best technology as demonstrated in Phase 2.

In the Phase 1 program currently under way, nine contracts have been awarded to four vendors in the private sector for preparation of the waste form samples. All samples have been received and are in testing. There are two parts to the Phase 1 testing of the waste form samples: one performed by the vendor and the other performed by the WMTC. The vendors perform the widely accepted

standardized tests that require minimal interpretation, while the WMTC performs the tests that are more subjective and need interpretation. The tests are:

Vendor tests	WMTC tests
Biodegradation	Homogeneity
EP toxicity	Immersion/compression
Explosiveness	Flash and ignition point
Drainable liquid	Thermal stability
Compressive strength	ANS 16.1 leach test
Gamma irradiation	MCC-1 leach test
	Corrosion testing
	Site-specific biodegradation
	Thermal cycling

The Phase 1 test program for these four waste streams is scheduled to be completed in January 1989, and Phase 2 is scheduled to begin in FY 1989.

A second set of waste streams is currently being evaluated for acceptance into the program, which will begin Phase 1 procurement and testing when funding is made available.

Supercompaction and Grouting of ORNL Solid LLW Drums. The demonstration of supercompaction and grouting of ORNL, 55-gal drums that had been examined under real-time radiography was successfully completed between March 9 and March 27, 1987, at the ORNL Solid Waste Storage Area (SWSA). The subcontractor, U.S. Ecology of Louisville, Kentucky, used its mobile supercompaction system operating at 2200 tons of compressive force. During the demonstration, 300 drums were reduced in volume by a factor of approximately 6.5:1; following grouting of the supercompacted drums into 47 125-gal overpacks, the overall volume reduction was approximately 2.8:1. During the crushing of the drums, it was found that absorbed liquids undetected by the real-time-radiographic examinations were released and collected from 94 of the 300 drums in amounts varying from a fraction of a liter to ~10 L. However, contamination of the supercompaction unit was insignificant, and decontamination to meet Department of Transportation (DOT) standards was carried out at the conclusion of the processing to the satisfaction of Energy Systems and U.S. Ecology.

Pictures and video coverage of the demonstration are available, and an ORNL technical manuscript was prepared. There were no accidents, injuries, environmental releases, radiation releases, or worker exposures as a result of the demonstration. Favorable publicity was generated by the three local television news releases and in local newspaper coverage. In addition, the demonstration was witnessed on two occasions by representatives of the Tennessee Department of Health and Environment. Completion of the demonstration and issuance of the final report satisfied a DOE/HQ milestone.

ORNL Tumulus Disposal Demonstration. In cooperation with Energy Systems' Engineering and Waste Management Operations at ORNL, an above-grade disposal unit was designed and built to evaluate the concept of disposal of short-half-life radionuclides in a configuration that is capable of "zero-release" of any contaminants during a period of institutional control. The tumulus concept was adapted from a similar design now being used for LLW disposal in France. The major goal of the demonstration is to gain experience with above-grade, modular disposal techniques, particularly in areas of construction, operation, and monitoring.

The SWSA-6 Tumulus Disposal Demonstration consists of a grade-level concrete pad [20 × 32 m (65 × 105 ft)] on which approximately 793 m^3 (28,000 ft^3) of solid LLW containers in concrete vaults will be placed. The project includes:

1. a drained under-pad plastic liner to assist in evaluating the concrete pad performance,
2. a curbed and drained pad surface to collect any water that may come in contact with the waste to evaluate the waste form performance, and
3. a station where the liner and pad surface drains will be monitored.

Construction of the tumulus was completed in January 1987, meeting an award-fee milestone. Since that time, concrete disposal vaults for waste containment were designed and procured. Actual loading of wastes began in May 1988, following an extensive preoperational review period. Management and operation of the project have been turned over to Waste Management Operations at ORNL.

Supercompaction of Y-12 Baled Waste. The objectives of the demonstration included an evaluation of (1) volume reduction factors for the previously baled Y-12 solid waste, (2) cost for the off-site service, and (3) transportation and handling of

the wastes. The LLWDDD Program, working with Y-12 waste disposal personnel, prepared a statement of work and handled the procurement activities. Scientific Ecology Group, Inc., a local radioactive waste processing firm, was selected by competitive procurement and carried out the demonstration in May 1987 on approximately 6364 kg (14,000 lb) of material contained in ten bales.

Following initial shredding of the baled material, the waste was precompacted into interpacks [metal boxes about 1.2 × 1.2 × 0.9 m (4 × 4 × 3 ft)]. The interpacks were then subjected to compressive forces in excess of 4000 tons in the world's largest waste press, known as an "ultracompactor." The resulting wafers were overpacked in rectangular containers for return to Y-12.

As a result of the demonstration, the 15.9 m^3 (561 ft^3) of waste was reduced to about 5.2 m^3 (185 ft^3), for an average volume reduction factor of about 2.9:1. The demonstration verified that off-site processing of DOE waste is practical and that transportation and handling are workable. The overall volume reduction factor for the waste, including a factor of about 7 to 1 for the initial baling, was approximately 20 to 1, indicating that compaction before disposal has tremendous potential for reducing the volume of waste.

Demonstration of Improved Operations Disposal at ORNL SWSA-6. Following an agreement with the Tennessee Department of Health and Environment that disposal of wastes in the existing SWSA-6 would be carried out with a barrier between the wastes and the environment, the LLWDDD Program has worked with ORNL Waste Management Operations to design and build disposal units exhibiting improved isolation characteristics.

The majority of the units are based on the silo configuration, where a cylindrical concrete shaft with a concrete bottom is located vertically from just below the surface of the ground to a depth of about 6.1 m (20 ft). Variations of the concrete silo were also developed to contain high-activity wastes and to replace the former practice of depositing material in unlined auger holes. In the course of the development of improved operations, a number of readily available construction materials, such as corrugated drainage culverts, steel pipe, concrete culverts, steel reinforcing bar, and concrete mix, were successfully adapted for use in the construction of these units.

Environmental Impact Statement Support. This work element encompasses the identification and accumulation of all data and information necessary to prepare an environmental data package for the EIS and to provide technical support during the EIS process associated with the development of new disposal facilities for LLW on the ORR. There are currently four sites being considered in the EIS: East Chestnut Ridge, West Chestnut Ridge, SWSA-7, and Bear Creek Valley. This element of the LLWDDD Program will prepare an environmental data package for each of these sites and include extensive site characterization at the East Chestnut Ridge and Bear Creek Valley sites, where site data are currently lacking.

This effort will include the identification of disposal sites that are both compatible with the capacity needs implied by waste management strategies and defensible through pathways analysis that will be conducted to support EIS development.

Another major effort is the site characterization model validation work. The objective of this work is to provide a demonstration of the predictive accuracy of groundwater contaminant transport modeling in the saturated zone on the ORR. The predictive accuracy will be determined by using site characterization data to calibrate existing transport models. Field tracer tests will be performed, and the collected data will be compared with predicted results. Analysis of the results will resolve apparent differences between model predictions and the field test results. Modifications to the model will be made to improve the predictive accuracy for future applications.

Specific activities to be included are as follows:

1. Identification of most appropriate sites for field activities. These sites are West Chestnut Ridge and Bear Creek Valley.
2. Collection of site characterization data.
3. Calibration of contaminant transport models through hydraulic conductivity testing and monitoring of piezometric surfaces. Models being considered for calibration include SEFTRAN, USGS-MOC, MIGRAT, and FEMWATER/FEMWASTE.
4. Tracer testing on a small scale, conducted without disturbing the aquifer below the site being studied. Well installations and monitoring instrumentation will be required.

5. Evaluation of the effect of bedding of the rock strata on transport of contamination. The literature suggests that poor comparison of prediction and measurement may occur because of interference from the complex geology at these sites.
6. Sensitivity analysis of the results and comparative analysis of the contaminant transport model predictions.
7. Model modifications to improve accuracy.

New Facility Development. The major responsibility of this work element is to conduct the activities needed to support the design, construction, and operation of new LLW disposal facilities on the ORR so that beneficial occupancy may begin by the end of FY 1991. These facilities are expected to consist of a set of expense, General Plant Projects (GPP), or line-item projects, with several of these to be brought online in 1992 and beyond as the new disposal facilities expand. The traditional engineering approach to accomplishing these projects will be conceptual studies, detailed design activities, and construction work. These efforts include completing alternative evaluations, study and estimates, conceptual design reports, and design criteria documents, at a minimum.

Hazardous Waste Development, Demonstration, and Disposal Program Overview

The HAZWDDD Program was formed in late 1987 to integrate hazardous and mixed waste treatment, storage, and disposal needs. The program charter lists several functions of the program:

1. prepare a planning document at all five Energy Systems facilities, then prepare an overall corporate strategy plan covering hazardous and mixed wastes;
2. identify and coordinate needed development, demonstration, and technology transfer projects to address the needs identified in the HAZWDDD plan; and
3. serve as a communication link among the five Energy Systems facilities, the Environmental and Safety Activities (ESA) organization, and WMTC.

The HAZWDDD Program is a part of the WMTC and complements the LLWDDD Program, which is coordinated by the CSA.

Current activities of the HAZWDDD Program center on the need to prepare a HAZWDDD plan at each plant and to develop an integrated plan. Each of the plants has a HAZWDDD team that will finalize its plan during August 1988. The participants have met in joint planning sessions several times, and the documents being developed will have a common format. A HAZWDDD data base is being developed concurrently with the plan for each of the plants and is being coordinated by the program office.

The integrated corporate plan is being prepared by a team from the HAZWDDD Program, the Energy Systems engineering organization, and outside consultants. Waste minimization, internalization, consolidation, and disposal options are developed in this analysis, and recommendations are presented to address technology and facility needs. This document will be reviewed and approved by the plant management of the five Energy Systems installations and by the ESA before being submitted to DOE/ORO as an award-fee milestone.

HAZWDDD Demonstration. The five Energy Systems installations are jointly funding a technology demonstration to thermally decontaminate PCBs from soil. This project will treat contaminated soils, which came from Paducah, Portsmouth, and Y-12, in a calciner at IT Corporations' Oak Ridge facility. Should this technology prove viable, a treatment method with costs lower than the Toxic Substances Control Act (TSCA) incinerator will be available to handle this large multisite waste stream. This demonstration was conducted in August and September of 1988.

Y-12 Sludge Detoxification. As part of its responsibilities to assist the DOE/ORO in implementing the DOE Model, the WMTC is responsible for managing demonstration projects of innovative technologies for various programs, including HAZWRAP.

The first project selected by HAZWRAP for demonstration by a private sector contractor was the Y-12 Sludge Detoxification Demonstration. This project includes permitting and demonstration treatment of mixed-waste sludges.

The project was started late in 1986 with the preparation of a statement of work, followed by the request for proposal (RFP), bid evaluation, contractor selection, contractor review, and approval and contract placement in June 1987.

The process to be demonstrated involves thermal separation of water and organics from the sludge, leaving a powder-like residue and further separation of the organics from the water. Detoxification of the residue is also to be demonstrated by mixing the residue with water and a grout to form a solid. The solidified material will be subjected to the detox test. Disposal of the organics is not part of the demonstration.

The project was started with a DOE-approved action plan, followed by preparation of a statement of work and issuance of an RFP to private industry. Proposals were received, evaluated, and a contractor was selected. A contract was executed in June 1987, and Phase I, "Permitting" of the contract, is proceeding. Preparations are being made to move into Phase II, "On-Site Demonstration," at the completion of Phase I.

Demonstrations

Old Hydrofracture Facility Surface Impoundment—Technical Assessment, Closure Plan, and Stabilization and Fixation. The Old Hydrofracture Facility (OHF) is located ~1.8 km (1.1 mi) southwest of the main plant area. It was operative from 1964 through 1976 as a disposal facility for liquid radioactive waste generated at ORNL. The system included a cased borehole that was used to develop bedding plane fractures for waste injections in the subsurface at depths of 229 to 305 m (750 to 1000 ft). The liquid wastes were mixed with a cement-based grout to form a slurry that was then injected under high pressure into the fracture zones to form eliptical sheets of solidified, radioactive wastes. As part of this operation, a shallow impoundment pit was constructed at the site to contain certain contaminated sludges, liquids, and soils. To stabilize these materials, commercial vendors were invited to submit proposals for the demonstration of in situ or implaced fixation of the contaminated soils and sediment in the impoundment. The work is being carried out in two distinct phases. The first phase, which has been initiated, consists of a comprehensive technical assessment of the OHF pit to determine the compatibility of the waste constituents with commercially available in situ stabilization and fixation technology. A site closure plan with the recommended technology and equipment is also being developed as a part of Phase I. Three in situ stabilization processes, developed by two separate commercial vendors, were selected for this work. Phase II of this work will consist of the implementation of the site closure plan.

Westinghouse Pyroplasma Demonstration Project. In late February 1987, Westinghouse Plasma Systems (WPS), a subsidiary of Westinghouse Electric Corporation, sought DOE/ORO assistance to conduct a demonstration of their innovative Pyroplasma waste destruction process. The process basically destroys the liquid waste by injecting it into a high-temperature (5000 to 15,000°C) thermal-plasma field.

WPS's objective for the demonstration was to conduct an EPA-approved trial burn test of their 3-gpm mobile Pyroplasma unit for destroying PCB-contaminated oils in conformance with the TSCA regulations. This demonstration was conducted in April 1987, outside Portal 8 at the Oak Ridge Gaseous Diffusion Plant. Figure 2.14 is a photograph of the demonstration setup at Portal 8.

At DOE/ORO's request, the WMTC orchestrated Energy Systems support for conducting the demonstration. Under terms of the contract between WPS and Energy Systems, Energy Systems provided certain support services, utilities, and the PCB-contaminated oil for the demonstration and then disposed of the liquid effluent. WPS, in turn, paid Energy Systems certain DOE/ORO-approved costs for these services.

The demonstration indicated that the Pyroplasma process was not ready for commercial use. As a part of its function under the DOE Model, the WMTC's technical assessment of the process concluded that:

1. There was insufficient understanding of the process chemistry.
2. The process technology had deficiencies.
3. Effluent management had not been fully developed.

However, the Pyroplasma demonstration was noteworthy in that it was the first demonstration in which a private U.S. organization of its own volition approached DOE/ORO for assistance in conducting a demonstration (under the DOE Model) of an innovative waste management process. Also

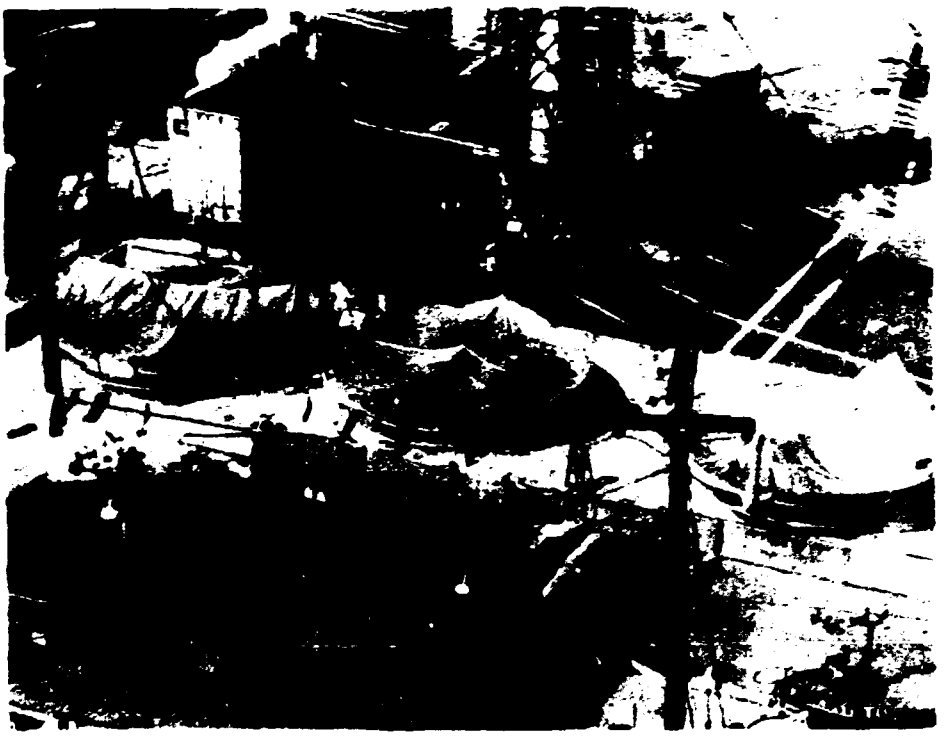


Fig. 2.14. Pyroplasma demonstration unit outside portal 8 at the Oak Ridge Gaseous Diffusion Plant.

noteworthy was the WMTC's role in marshaling the requisite resources in a timely manner to bring forth the demonstration, in keeping with its mission under the DOE Model.

Workshops

In addition to technology transfer accomplished through demonstrations, transfer and information exchange is being accomplished by conducting a series of workshops. The following four workshops are currently planned and are being coordinated: leaching test series, uranium-bearing waste series, contaminated soil series, and the Resource Conservation and Recovery Act/Comprehensive Environmental Response, Compensation, and Liability Act (RCRA/CERCLA) series.

Uranium-Bearing Waste Workshops. The objective of the workshop series dealing with management of uranium-bearing wastes is to examine the current treatment, storage, and disposal practices and the regulatory concerns affecting such practices. The contaminated-soil workshops will deal with similar issues that affect the TSD of contaminated soils.

A one-day workshop on the Management of Uranium-Bearing Wastes was held in Oak Ridge on May 5, 1988. The meeting was hosted and directed by the WMTC for DOE. One hundred thirty-one scientists and engineers representing government, industry, and academia participated in the workshop. Discussions were focused on technical issues related to (1) processing and recycling, (2) treatment, (3) storage and disposal, and (4) regulatory and environmental protection.

The imposing quantities of long-lived uranium-bearing wastes coupled with the complexities of current and pending regulations make the management of these wastes one of the most perplexing problems facing the nuclear industry today. Both government and private industry are faced with increasing quantities of wastes that are mainly generated at DOE-owned facilities related to the development and manufacture of weapons and at commercial plants engaged in the fabrication of fuel elements. Because of the long-lived nature of uranium wastes, the disposition of current and future generated wastes is at issue, as is uranium waste produced over the past several decades. Currently, several technologies, including chemical

and biological means, are used to condition and treat uranium-bearing waste. Shallow-land burial, using lined trenches and the tumulus concept, is commonly practiced for disposition of a large fraction of these wastes, while much is also confined to temporary storage in a variety of containers and structures. Regulations that govern the management of uranium-bearing wastes include those of DOE, NRC, EPA, and affected states. The complex and somewhat overlapping body of current regulations may be clarified and rectified by pending regulations. However, even with more quantitative standards, the implementation of these regulations will undoubtedly prove to be a formidable management task.

RCRA/CERCLA Workshops. A workshop on RCRA/CERCLA Treatment Alternatives for Hazardous Wastes was held May 10–11, 1988, at Oak Ridge Associated Universities in Oak Ridge, Tennessee. It was presented by EPA in cooperation with ORO, DOE, and Energy Systems. Local arrangements were made by the WMTC and HAZWRAP.

This two-day seminar presented an overview of and introduction to a wide range of hazardous waste management technologies that are entering the application stage or are under development. These technologies are in accordance with both the RCRA and the CERCLA. The seminar was intended principally for entry-level personnel who are unfamiliar with the broad range of treatment technologies available today. However, it also provided the most recent information on problem areas and waste management technologies. The seminar focused on descriptions of the available technologies, their suitability for differing waste streams and waste management problems, and their probable costs.

A second workshop is being planned and will include detailed discussions of specific technologies and their applications.

Leaching Test Workshop Series. The leaching test series is designed to provide the scientific basis and applicability of the several leaching tests currently used. Information will be developed concerning the suitability of solid waste forms for disposal of low-level and hazardous waste. The ultimate objective of the workshop series is to provide information concerning the long-term durability of solid waste forms.

Because of the importance of leaching tests in waste management, the WMTC is sponsoring a series of four workshops on leaching tests and their relation to disposal conditions and waste form durability. Most of the currently viable tests were considered in the first (July 21–24, 1987) workshop on scientific basis and applicability of leaching tests. The second (October 25–26, 1988) workshop continued to develop the scientific basis and applicability of leaching tests, with primary emphasis on the significance and meaning of accelerated leaching tests.

The third and fourth workshops will address leaching under disposal conditions and durability of waste forms, respectively. The ultimate goal of the workshop series is to provide information and data on the durability of waste forms and how to prove or predict the durability of stabilized or solidified waste forms.

Support Activities

The WMTC support group provides management and technical assistance to several other programmatic areas, for example, HAZWRAP and the Historically Black College and University Program.

Strategic Alternatives Studies (SAS). HAZWRAP recently conducted an SAS study for developing a set of waste management options applicable to DOE-Defense Program hazardous and mixed waste. WMTC staff members participated in the successful SAS effort by developing methodologies to collect, analyze, and summarize waste generation data and by assisting in the development of several options for management of DOE/DP hazardous and mixed waste.

Several thousand varied and unique waste streams exist in the large DOE industrial complex studied for this report. To facilitate the use of waste stream data and the integration of such information into collective generalizations useful from a system-wide waste management viewpoint, waste stream data collected by the DOE Waste Information Network were abstracted and organized into a form easily usable on a PC. Because of the relative ease of access and flexibility in data manipulation, commercially available spreadsheet software (Lotus 1-2-3 and dBASE III) was selected for use, and spreadsheets were prepared for each geographical site. The approximately 50-column spreadsheet categorized essential waste management data such as chemical and physical characteristics, concentrations, amounts or current

inventories, and generation rates. Based on the chemical information, candidate EPA hazardous waste numbers were assigned to waste streams. Candidate EPA TSD numbers were also assigned for the current TSD methods. Other information included TSD locations, future projections, and problem areas.

In classifying the waste streams, assumptions were made that reflect a generally conservative approach to the management of mixed waste:

1. Any chemically hazardous waste streams also containing radioactivity were categorized as mixed wastes.
2. Wastes containing several hazardous constituents were classified according to the components considered most hazardous.

Waste stream data in the spreadsheet format were submitted to each generation site for corroboration and evaluation. This QA step was of considerable importance for verification of data value.

The treatment, storage, and disposal of hazardous and mixed wastes are dependent upon the nature and composition as well as the volume of the waste. Collectively, the waste streams were generalized into the following categories: (1) wastes that are treated as wastewaters that produce sludges or residues requiring possible further treatment (e.g., detoxification or solidification); (2) wastes that may be burned and produce ash requiring possible further treatment (e.g., solidification); (3) solids or noncombustibles that require fixation, solidification, or packaging before disposal (e.g., asbestos); (4) chemicals requiring special detoxification or treatment (e.g., sodium, cyanides, etc.); and (5) historic wastes requiring remedial action. This categorization of wastes into treatment groups has been conceptualized into a flowchart (Fig. 2.15). The conceptualization incorporates a hierarchy of steps leading from generation to disposal. In general, it was assumed that all wastes were treated before disposal. Treatment residues may require fixation or solidification, and some wastes may be packaged before disposal. Following QA by the individual sites, the waste stream data were sorted and grouped into the treatment categories: incineration, neutralization, precipitation, physicochemical treatment, wastewater treatment, and fixation and solidification. During the course of the study, the unit operations neutralization, precipitation, and physicochemical treatment were determined to be subsets of general wastewater treatment.

These methods enabled the identification and characterization of individual waste streams with appropriate QA, the subsequent step-wise integration of the data through treatment flowsheets at the site level, and the final integration of the data at the national level through generic flowcharts.

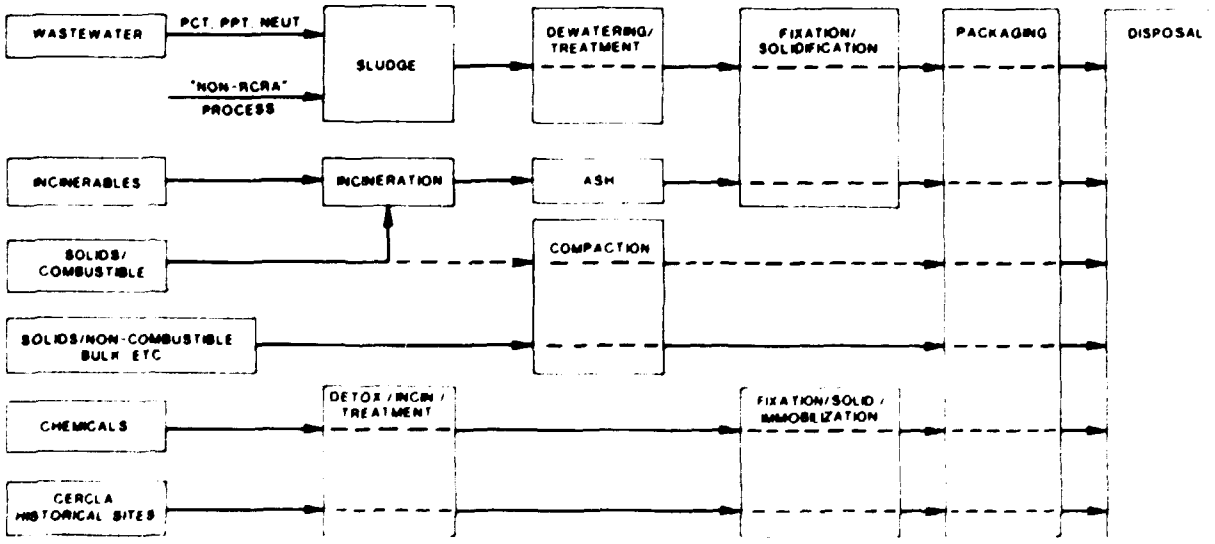


Fig. 2.15. Waste treatment categories and conceptual flow chart.

The data summaries and analysis permitted a comprehensive examination of corporate waste management practices.

ORNL/HAZWRAP Interface (OHI) Activity. At the start of FY 1988, the WMTC was asked to serve as the interface between HAZWRAP and ORNL when HAZWRAP was transferred out of ORNL. In this role, the WMTC serves the following functions:

1. be a facilitator to match ORNL capabilities with the diverse HAZWRAP needs that range from project planning to conducting waste management technology demonstrations,
2. be a point of contact for HAZWRAP to tap ORNL capabilities and expertise in performing their projects, and
3. serve as a monitor to gauge ORNL support provided to HAZWRAP.

In FY 1988, ORNL is supporting HAZWRAP in conducting over 40 projects with a total project funding level exceeding $18 million. These projects use a wide variety of ORNL capabilities ranging from conducting environmental assessments to performing technology demonstrations.

Historically Black College and University Interactions. The WMTC support group functions as a focal point involving industries and universities in the definition and resolution of waste management problems on the ORR. One component of this function is the recruitment of staff members from Minority Educational Institutions (MEIs) for involvement in significant waste management problems on the ORR. The WMTC has identified and proposed cooperative work in several significant areas:

1. Development of Systems Optimization Techniques as a Planning and Analysis Capability for the LLWDDD Program, North Carolina Agricultural and Technical University, Greensboro, North Carolina. Work is in progress.
2. An Assessment of Economies of Scale vs Operating Subcontracts in the Development of Waste Management Systems, North Carolina Agricultural and Technical University, Greensboro, North Carolina. Work is in progress.
3. Evaluation of Air Stripping for Removal of Volatile Organic Pollutants from Soil and Groundwater, Southern University, Baton Rouge, Louisiana. Work is completed.
4. The regeneration of zeolites used to remove radionuclides (^{90}Sr, ^{137}Cs, ^{60}Co) from wastewaters through ion-exchange processes, Southern University, Baton Rouge, Louisiana. Work is completed.
5. Characterization of exhausted High Efficiency Particulate Air (HEPA) filters to determine whether hazardous materials or transuranics at concentrations greater than 100 nCi/g are present, Tuskegee University, Tuskegee, Alabama. Work is in progress.

Support for the ORNL Environmental Projects Program

The Environmental Projects (EP) Program was established at ORNL to provide the Laboratory with the capability for complying with existing and future environmental regulations and requirements. This program is directed toward establishing the improvements needed for continued protection of the environment and the health and safety of onsite workers and the public and for compliance with current and future environmental regulations. It has also been involved in improving or developing waste collection, treatment, storage, and disposal capabilities for all ORNL-generated waste and effluent. The Chem Tech Division supports EP in two major areas: (1) management of the Air Pollution Control Projects and (2) management of the Water Pollution Control Projects.

Air Pollution Control Projects

In the air pollution control area, Chem Tech personnel support the EP program through the management of projects designed to upgrade the air pollution control systems. The air emission systems at ORNL are being upgraded to ensure (1) compliance with environmental, health, and safety regulations and (2) the long-term reliability of the system. Because radioactive air emissions from ORNL facilities are in compliance with existing regulations, the primary emphasis of the upgrade program is to improve the long-term reliability of the air emissions systems and to upgrade the monitoring systems. Projects are under way to provide additional, backup HEPA filters for isotope production facilities; to upgrade filter enclosures for the main ventilation stacks; to replace worn fans, motors, and emergency generators; and to upgrade the monitoring and sampling systems for the main stacks. The capital projects that are under way or

are scheduled for upgrading the air pollution control systems are as follows:

1. Upgrade Cell Filtration, Building 3517;
2. 3039 Emergency Power System;
3. Upgrade 3047 Filter House;
4. Upgrade 3108 and 7913 Filter Pits;
5. Install Emergency Generator, 7025;
6. Upgrade 3039 Stack Fans;
7. Stack Monitoring Improvements;
8. Ventilation System Upgrade; and
9. Improvements to ORNL Air Monitors.

Water Pollution Control Projects

In the water pollution control area, Chem Tech provides management support for a number of the ORNL water pollution control projects. These projects are directed at collection, treatment, and disposal of a wide variety of liquid wastes (LWs) generated by the diverse activities at ORNL. The most noteworthy projects being managed in this area are three Congressional line-item projects:

1. Nonradiological Wastewater Treatment Projects (NRWTP),
2. Bethel Valley Liquid Low-Level Waste Collection and Transfer System Upgrade (BV-CAT), and
3. Isotopes Area LLW-CAT Upgrade.

The Nonradiological Wastewater Treatment Facility (NRWTP), which has a total estimated cost of $18 million, will provide wastewater collection tankage (6800 m^3) to replace seven active surface impoundments and a 2700-m^3/d treatment facility. The process flow diagram for the treatment facility is shown in Fig. 2.16. Trace metals will be precipitated by chemical treatment with sodium hydroxide, followed by dual-media filtration to remove the suspended solids. Volatile organics are to be removed by air stripping, and final polishing is to be accomplished by passage through activated carbon columns. The carbon columns will remove semivolatile organics and trace quantities of mercury. Construction of the NRWTP is under way. The collection tankage construction was completed in September 1988; the treatment facility is scheduled for completion in September 1989.

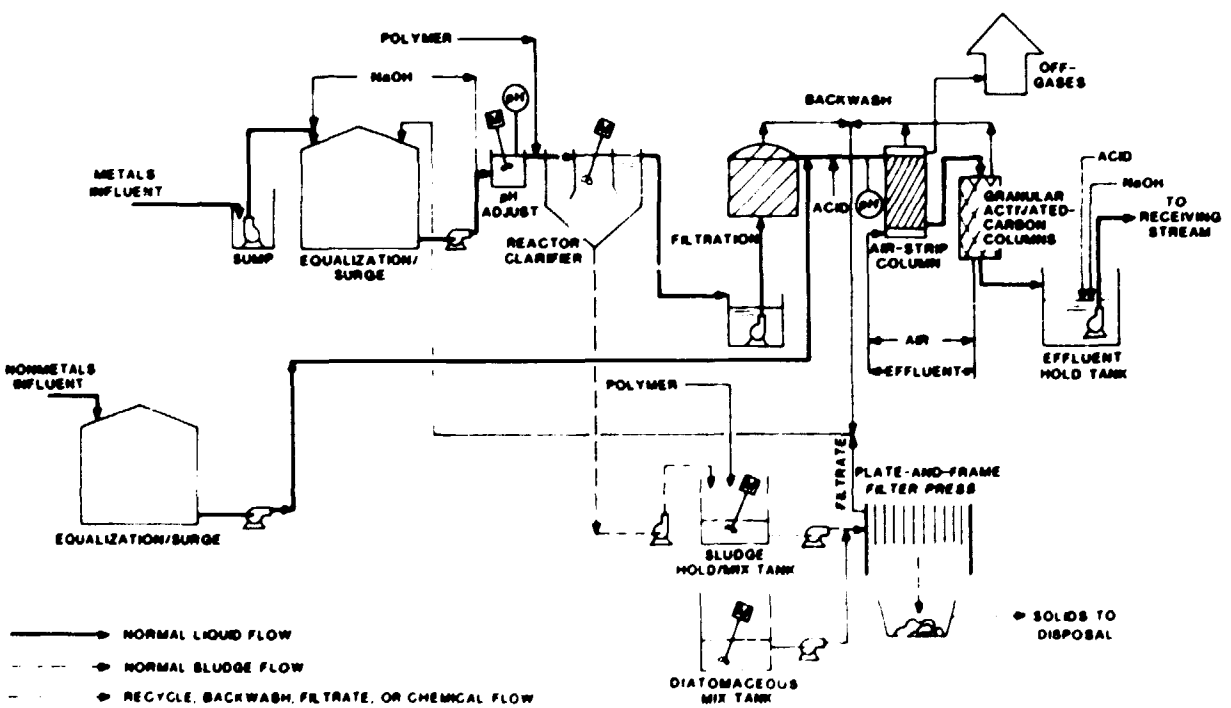


Fig. 2.16. Process flow diagram for the Nonradiological Wastewater Treatment Facility.

The BV-CAT and the Isotopes Area LLW-CAT projects are currently planned to upgrade the aged ORNL LLW-CAT system in BV. The BV CAT System (exclusive of the Isotopes Area) is an FY 1988 project with a total estimated cost of $35 million. The proposed FY 1991 project will upgrade the CAT system in the Isotopes Area with a TEC of $33 million.

These two projects have a number of common features, and management of the projects is being coordinated to take advantage of these commonalities. Both projects will provide systems that use best available technology (BAT) for satisfying regulatory requirements and will include double containment, active leak detection, and corrosion protection.

New underground pipelines, serving major source facilities to the LLW system, are to be provided. These pipelines will be double-contained (pipe within pipe) and will be cathodically protected against corrosion. The annulus of the double-contained pipelines will be pressurized with nitrogen and monitored for detection leaks (in either the primary inner pipe or the outer containment pipe).

New Monitoring and Control Stations (MCSs), which are associated with source facilities to be served by the projects, will be provided. The MCSs will consist of a below-grade, vaulted collection tank; monitoring instrumentation, including indication of pH, temperature, pressure, and tank level; a caustic addition system; and an above-grade control room that contains a sampling system. An architectural rendering of a typical MCS is shown in Fig. 2.17. Generally, waste will flow from the source facility to the MCS by gravity. At the MCS, tank contents will be monitored, sampled, and pH adjusted as necessary. Periodically, tank contents will be discharged to the downstream LLW system by means of steam-jet ejectors.

The BV-CAT project will provide underground piped service to five facilities and a new central facility for receiving and discharging liquid waste that is transported in small volumes in bottles and

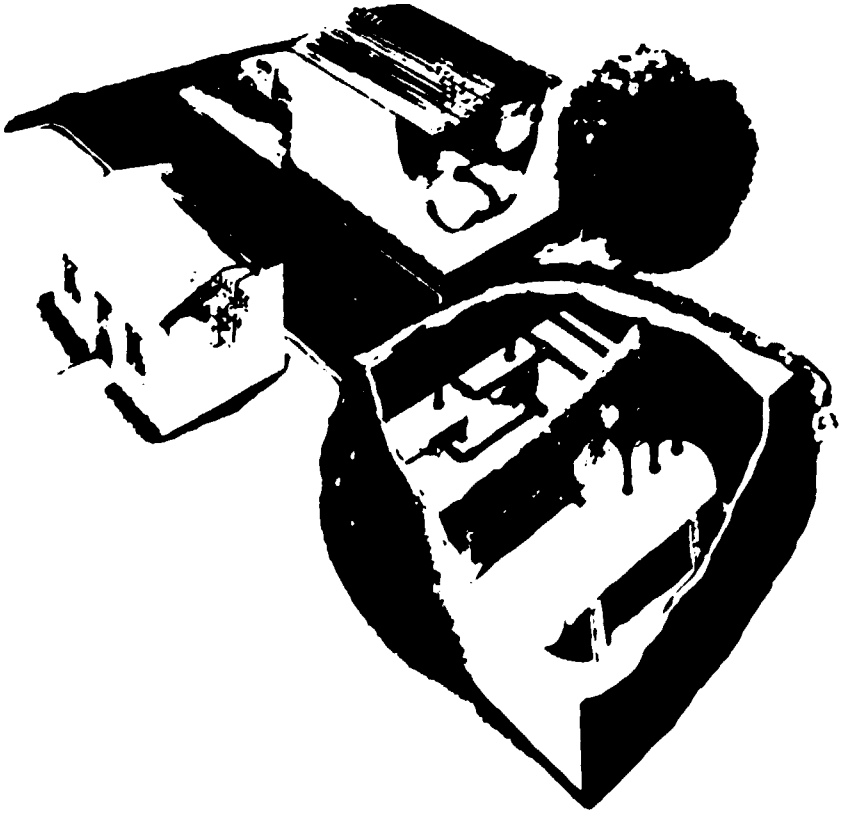


Fig. 2.17. Architectural rendering of monitoring and control station for LLW-CAT project.

larger volumes using a tank truck. The facility will include diked floors, hoods, and necessary ventilation and handling equipment to ensure operator safety and environmental compliance. The bottles and trucks used for this effort will meet ORNL and DOE regulations for transporting regulated substances. The upgrade project includes ~1 mile of double-contained stainless steel pipeline and five MCSs.

The Isotopes Area LLW-CAT project will provide underground piped service to six facilities, which generate and process approximately 9.46 m^3 (2500 gal) of radioactive and hazardous liquid waste per month, and will also have the capability to service two facilities where wastes will be collected in bottles for transport to treatment facilities. The upgrade project includes ~2000 m (~7000 linear ft) of double-wall stainless steel pipeline, three 0.95-m^3 (250-gal) stainless steel intermediate collection tanks, and one MCS.

Liquid and Gaseous Waste Treatment

Significant improvements are being made in the liquid and gaseous waste systems at ORNL, and Chem Tech has a major role in conducting process R&D and program management. A close working relationship with Environmental and Health Protection Division personnel is maintained to effectively conduct the programs in the two major work areas of (1) Liquid Low-Level Waste and Transuranic Sludge Treatment Research and Development and (2) ORNL Waste Operations Support. In addition, On-Site Activity Support, a new research initiative for the Fossil Energy Program, has been started.

Liquid Low-Level Waste and Transuranic Sludge Treatment R&D

Studies are being performed to support treatment and eventual disposal of the contaminated liquid waste that is generated at ORNL, as well as the inventory of contaminated liquid waste concentrate currently being stored at ORNL. Subtasks include Near-Term Support Tests, Waste-Handling and Packaging Plant Support, and Long-Term Process Selection.

Near-Term Support Tests. Tests are being conducted to support the Emergency Avoidance Solidification Campaign (EASC). The EASC process will solidify supernate from two Melton Valley Storage Tanks (MVSTs) using solidification agents and equipment supplied by the LN Technologies Company, of Columbia, South Carolina. The leachabilities of radionuclides and chemical species are used as waste acceptance criteria for disposal and storage. Leach-test specimens were prepared with actual radioactive waste so that the validity of results obtained by vendors could be verified. The leachability of RCRA metals was tested using the Extraction Procedure Toxicity (EP Tox) Test, and the leachabilities of nitrate, chloride, ^{137}Cs, ^{90}Sr, and ^{60}Co were tested using the ANS 16.1 90-d immersion procedure. Samples of the leachate have been submitted to the Analytical Chemistry Division for analysis. A report summarizing the leach-test data will be prepared.

The volume of waste stored in the MVSTs will be reduced by the in situ evaporation of liquid; the evaporation rate will be increased by augmenting the flow of air through the waste in the tanks. Results of bench-scale tests, conducted to simulate the in-tank evaporation process, showed evidence of carbonate formation on the vessel walls shortly after air was bubbled through the liquid. In addition, crystals started to form after the waste volume had been reduced by 50 vol %, and the pH decreased from 11.5 to 6.8 during the evaporation process. Experiments will be conducted with actual waste in a hot-cell facility to determine (1) relative effects of temperature, salt concentration, and sparge rate on the rate of evaporation; (2) the waste concentration at which solid formation is initiated and the characteristics of the solids formed; and (3) the quantity of radioactive material in the off-gas.

Support to the Waste-Handling and Packaging Plant (WHPP). Extensive R&D is being conducted to support the design of a system that will remove, treat, and solidify contaminated MVST liquid waste to prepare it for shipment to the WIPP located near Carlsbad, New Mexico. A schematic of the planned liquid waste processing steps is shown in Fig. 2.18. Simulated waste has been evaporated via radiant heat and microwave energy using bench-scale equipment. The waste forms a molten mass that solidifies into a monolith upon cooling. Equipment that will be used during extensive bench- and pilot-scale studies (scheduled to begin in FY 1989) is being designed and procured. A comprehensive program plan, which has already

been initiated, will serve as a guide for R&D planned through 1992. This project has high priority because each processing step must be proven prior to finalization of the design criteria (scheduled for 1991).

Long-Term Process Selection Support. Centralized treatment of liquid waste is needed in addition to the capability for solidification of RH TRU waste that will be provided by the WHPP. R&D is being conducted on long-term options for liquid waste treatment that prepares it for disposal. The goal of the long-term processing is to render the bulk of the ORNL-contaminated liquid waste acceptable for final disposal. This work has a high priority because planned near-term actions and processing provided by the WHPP will not prepare all ORNL-contaminated liquid waste for disposal.

Decontamination of dilute low-level liquid waste (LLLW) and supernate is an important step in long-term process selection. Decontamination by filtration and ion exchange has been investigated, and cross-flow filters have been extensively tested. A 0.5-μm sintered stainless steel filter (manufactured by Mott) was found to be effective in separating TRU solids from the supernate. Test results indicate that one filter element can potentially be used to treat 950 m^3 (250,000 gal) of waste before flux reduction due to solids plugging warrants filter cleaning and replacement.

Decontamination of LLLW by two types of ion-exchange media [Savannah River Laboratory resorcinol-formaldehyde (SRL-RES) and CS-100] was assessed by evaluating data from batch beaker tests in which distribution coefficients were measured. The SRL-RES exhibited excellent performance for the removal of ^{137}Cs (cesium is the primary contributor to radiation dose); therefore, column testing of this resin is planned.

ORNL Waste Operations Support

The Environmental Control Technology Group supplies programmatic management for the Air and Water Pollution Control Programs, which are

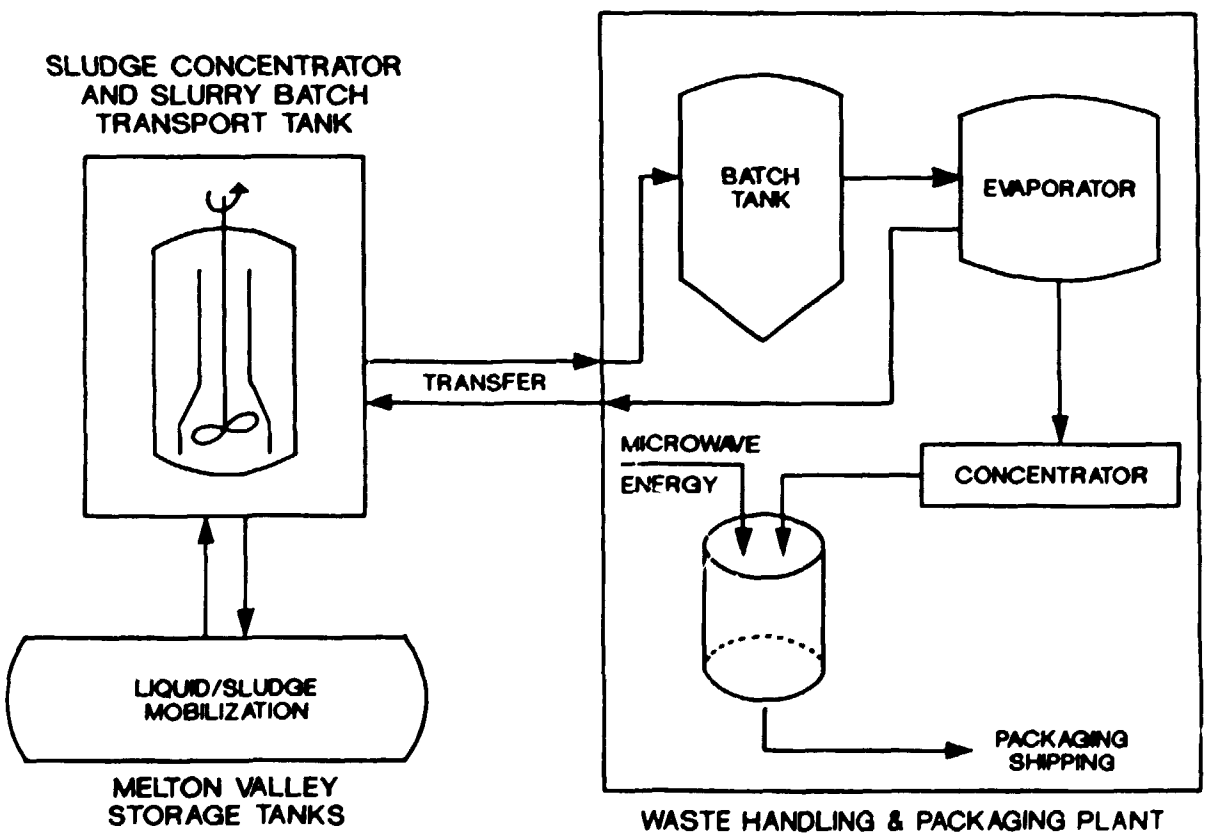


Fig. 2.18. Waste-Handling and Packaging Plant flowsheet for liquids/sludges.

directed at minimizing waste generation at ORNL and keeping the Laboratory in compliance with regulatory requirements. In this capacity, the group provides long-range planning and strategy development necessary to define the future direction of the program and performs technical studies and analyses needed for waste treatment and minimization projects.

Gaseous Waste Programs. The air pollution control projects include a project to establish a preventive maintenance program for fans and motors in main stacks that discharge radioactive emissions so that problems in these units can be detected before failure occurs. To date, base-line data have been taken to determine the condition of the equipment, and a routine surveillance program is being implemented. The group is also coordinating inspections of ORNL ventilation ducts that are not accessible for visual inspection and, thus, require the use of remote video inspection equipment. Inspections are scheduled to begin in the fall of 1988. In addition, an engineering evaluation of the main hazardous waste emissions sources is being performed to determine where upgrades are needed.

Liquid Waste Programs. The water pollution control projects are aimed at upgrading the nonradiological and process wastewater treatment facilities to ensure that ORNL meets regulatory requirements while minimizing secondary LLW generation. Technical development efforts have been focused in three areas: (1) development of on-line monitoring systems and pretreatment processes for influent waste streams to the NRWTF, (2) development of improved methods to treat slightly contaminated wastewaters to meet stricter discharge limits, and (3) evaluation of alternative treatment methods for various process waste streams to reduce the generation of LLLW.

NRWTF Support Studies. Treatability studies, including chemical precipitation, filtration, ion exchange, and carbon adsorption, are being performed to develop methods for removing ^{60}Co from High-Flux Isotope Reactor (HFIR) wastewater to meet the NRWTF acceptance criteria. A potentiometric metals monitor is being developed for continuous monitoring of metals at the parts-per-billion concentration level. The on-line metals monitor could be used for diverting streams to the NRWTF and for monitoring the effluent from this facility.

Improved Treatment Methods. Ion-exchange systems utilizing zeolites (inorganic molecular-sieve exchange media) are being developed to treat wastewaters that are slightly contaminated with ^{137}Cs and ^{90}Sr. This process is being developed for implementation at the Process Waste Treatment Plant (PWTP). Tests indicate that this process can be used to meet stricter discharge limits while reducing both the operating costs and the secondary waste generation rate by one-third.

A continuous ion-exchange column suitable for pilot-scale studies is being procured, installed, and tested for potential use in treating wastewaters that are slightly contaminated with ^{90}Sr. Initial testing at the PWTP will establish system design parameters necessary for full-scale treatment units and provide data needed to compare this system with other alternative processes.

LLLW Minimization Studies. Process evaluations are being performed to identify the most appropriate method for treating waste streams that are significant contributors to the LLLW. These evaluations include tasks such as characterizing the stream to determine if alternative treatment is warranted and performing treatability studies to permit decisions to be made as to whether the process can be altered to reduce generation or the stream can be treated at an existing facility or at the generation site.

More extensive evaporation of the liquid waste produced by the regeneration of organic ion-exchange resin is being evaluated at the PWTP. Solidification of the resulting evaporator bottoms is also being investigated as a means of decoupling the PWTP from the LLLW system. On-site evaporation of resin regeneration wastes or direct disposal of the loaded resin is being evaluated for application at the HFIR. Characterization studies of the 3039 Stack Scrubber waste stream indicated that this stream generates less than 200 L of LLLW annually and should not be considered for alternative treatment.

Study of Effluent Reduction at Tritium-handling Facilities

At the instigation of the National Academy of Sciences and the National Research Council, ORNL has begun a reevaluation of atmospheric effluents from tritium-handling facilities with the objective of reducing their levels to as low as reasonably achievable (ALARA). Tritium airborne effluents are the largest single source of radionuclide emissions from ORNL, even though the dose equivalent of tritium therefrom represents 2% of

the maximum permissible dose according to the regulations of the Clean Air Act.[26] The objective of this study was the identification, in the literature and from discussions with people at other DOE facilities that handle tritium, of available technology to reduce the already small emissions to ALARA levels in a cost-effective manner.

The study identified two broad areas where improvements may be made: (1) reduction of tritium losses from fume hoods during ORNL processing and purification operations and (2) reduction of emissions of tritium in stack flue gases by using a catalytic oxidation and sorption technique. The first area involves changes to the operation and maintenance of the uranium-bed reactor, which is used to reversibly purify the incoming tritium cylinders of the helium decay daughter. In this area, the prevention of tritium losses means the saving of valuable tritium gas that can be shipped to clients. The second area involves the addition of equipment to react tritium gas with oxygen, reaction of the resulting tritiated water with molecular sieve sorbents, and ultimate disposal (or recycle) of the sorbed tritiated water. In the latter area, the tritium is removed from the stack effluents directly and is lost as a salable product.

Unfortunately, operating data that would identify the relative importance of the two source areas were unavailable; however, specific recommendations for improvements can be summarized as follows. Reduction in tritium losses for the first area, the uranium-bed fume hood, would be effected by increasing the recirculation time of the helium-contaminated tritium across the uranium beds, regenerating the uranium beds periodically to remove helium that is present in the uranium lattice, cooling the uranium beds during the exothermic bed reaction, and adding an oil-less "scroll pump" that would permit circulation of lower-pressure tritium gas as well as eliminating the oil-sealed pumps with their oil contamination problems. For the second area, the stack flue gases, the addition of a small-scale effluent removal system (ERS) is preferred. Although laboratories throughout the country use different versions of the ERS of widely varying capacities and thoroughness, it appears that a simple, small-scale version of an apparatus such as that designed at Lawrence Livermore National Laboratory (LLNL) for portable applications would be most appropriate at ORNL.[27] The apparatus at LLNL uses a conventional platinum-palladium catalyst to oxidize the tritium to tritiated water and sorbs it on a zeolite molecular sieve that has a useful lifetime of ~1 year. This apparatus could be transported from one tritium facility to another at ORNL to control stack emissions as the need arises.

Gas Separations Using Inorganic Membranes

The objective of this new project is to explore the applicability of inorganic membranes for gas separations at high temperatures or in hostile process environments encountered in fossil energy conversion processes such as coal gasification. The program will apply porous membrane technology to the separation of various gases to improve the economics of fossil energy conversion processes by reducing gas cleanup and separation costs.

The overall R&D program consists of the development of membrane separation systems for the recovery of H_2 and the separation of acid gases (H_2S, CO_2) from synthesis gas. The program is composed of two major parts that are closely interlinked. Part A consists of four elements: (1) identification of the candidate membrane materials based on the chemistry of the separations environment, (2) evaluation of the separations capability of the fabricated membranes in terms of permeabilities and fluxes of gases, (3) assessment of the worldwide R&D activity in this field, and (4) preparation of reports on the project. Part B involves fabricating and physically characterizing the candidate membranes using the specialized techniques available at the Oak Ridge Gaseous Diffusion Plant.

A simplified block flow diagram of a typical entrained-bed coal gasification–combined cycle (IGCC) process is shown in Fig. 2.19(a). Present technology requires that product gases be cooled from 816 to 37.8°C (1500 to 100°F) to permit removal of CO_2, H_2S, and other contaminant gases. The cleaned fuel gas (i.e., CO and H_2) must then be reheated to 500–600°F for downstream combustion in a gas turbine to generate power. The efficiency of the process could be increased substantially if the contaminant gases could be separated at the higher downstream operating temperature. A block flow diagram for a conceptual membrane type of gas separation system for this application is shown in Fig. 2.19(b). Briefly stated, the gas cooling and the gas cleanup facilities would be replaced with a membrane separation system operating at high temperature.

Several materials, such as alumina, zirconia, and titania, have been identified as potential membrane media. The final selection will be based on several

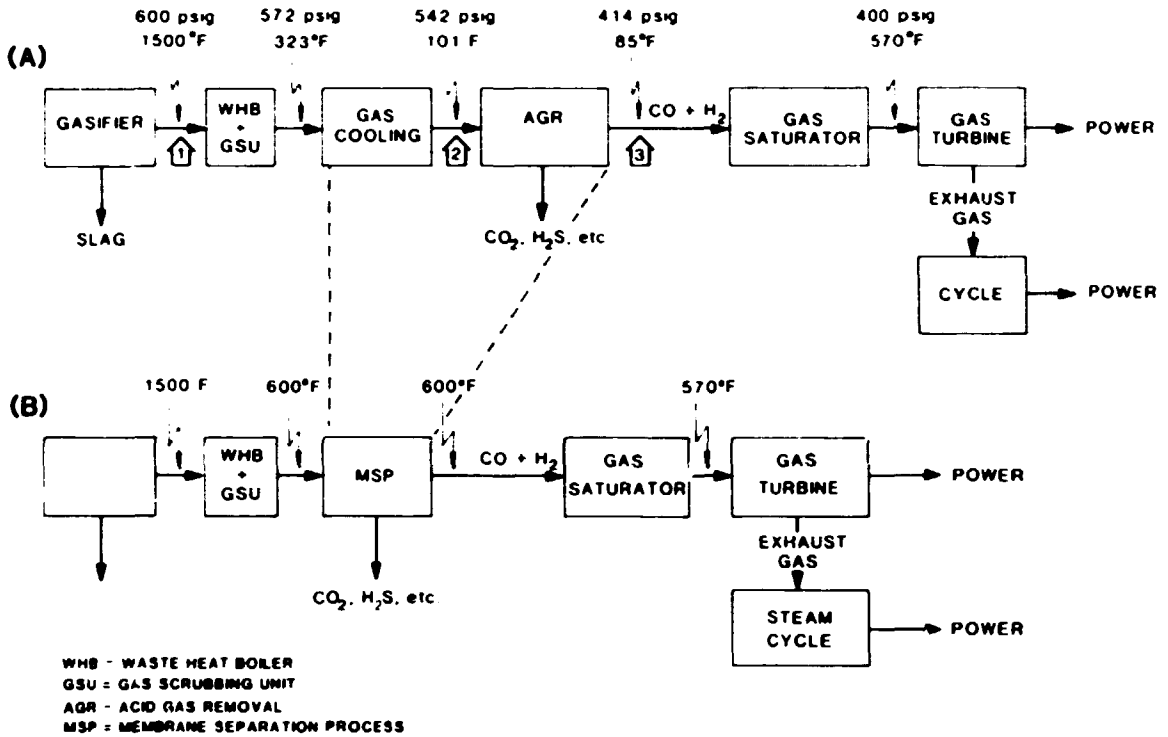


Fig. 2.19. (a) Schematic of a typical entrained-bed IGCC process; (b) schematic of a conceptual IGCC using a membrane-separation system.

factors, including mechanical and chemical stability of the membrane at the process operating conditions, availability and cost of raw materials, and the types of gas transport mechanisms that might be utilized to achieve the performance targets.

Preliminary evaluations suggest that an inorganic membrane could be developed with an H_2 permeability of 0.01 $cm^3(STP)/cm^2 \cdot cm$ Hg. This membrane would have a very high separation factor for H_2 relative to the other gases present in the raw synthesis gas. Based on the above permeability and using the typical raw gas composition of the gas produced in an entrained-bed coal gasifier, preliminary calculations were made to estimate the size of a conceptual commercial membrane separation unit to recover, for example, 90% of the H_2 in the gas. The calculation showed that recovery of 18 kg·mol/s (14,000 lb·mol/h) would require 1580 m^2 (17,000 ft^2) of membrane surface area. This would translate into a membrane separation unit similar in design to a conventional shell-and-tube heat exchanger that is 3 m (10 ft) in diameter by 3.7 m (12 ft) long.

Facility Restoration and Deactivations

Several projects were conducted to upgrade radiochemical facilities at the Laboratory to prepare them for safe standby status or for continued use in a changing regulatory environment.

Radiochemical Processing Plant

The Radiochemical Processing Plant (RPP), which includes the ^{233}U Processing Facility, the CEUSP Facility, and the High-Radiation-Level Analytical Facility (HRLAF), underwent major renovation of its ventilation, liquid waste drains, and instrumentation systems to facilitate completion of processing activities and to promote effective surveillance during safe standby status. Activities that will continue in this complex include operation of the Solids Storage Facility as a national repository for ^{233}U, operation of the radiochemical hood and glove box laboratories, and surveillance of the inactive areas.

The upgrades to the complex can be summarized as follows:

1. improving the security features of the Solids Storage Facility;
2. isolating the contaminated processing areas from routine access;
3. improving the laboratories, offices, and shops for use, with limited restrictions;
4. removing and replacing aged liquid waste drains and adding new drains, as needed, to complete processing activities and to leave the laboratories in usable condition;
5. replacing almost all of the containment ventilation ductwork and fans with new equipment to correct chronic system integrity and reliability problems; and
6. improving the emergency power system and adding remote alarms for critical equipment to provide upgraded power supplies and improved surveillance features.

Demolition activities involved removing highly contaminated, corroded ducts from the roof of the RPP and removing plutonium- and RCRA-contaminated drain lines from occupied and operating glove box laboratories. A unique method of classifying, segregating, and documenting eight distinct classes of wastes was developed to provide auditable records for each waste item. All construction activities were accomplished without adverse effect on ongoing processing operations or release of contamination to the environment.

Molten Salt Reactor (MSR) Surveillance and Maintenance Project

In response to tightening environmental standards at ORNL facilities, the aging containment features of the MSR Facility were evaluated and upgrades were planned for sustained surveillance and maintenance with the solidified fuel salt remaining in the drain tanks. The improvements to be made were divided into the following six work elements:

1. replace the stack fans, which have exceeded their design life;
2. provide ventilation for the reactor-drain tank (RDT) cell;
3. provide a means for leak-testing the RDT cell under vacuum rather than at an elevated pressure, as is the current practice;
4. empty the liquid waste tank;
5. upgrade the surveillance instrumentation by grouping signals and adding alarms; and
6. install a monitor to determine the F_2 concentration in the gas in contact with the fuel salt.

Conceptual planning for each work element has been accomplished, and control documentation (QA plan, Safety Assessment, etc.) for the project is in the approval process.

Hot Cells Revitalization Project

The hot cells located in the High-Radiation-Level Examination Laboratory (HRLEL), Building 3525, are being upgraded to permit their continued use and to permit consolidation of metallurgical examination activities so that the aging 3026D Facility and the Oak Ridge Research Reactor's South Cell can be taken out of service. Improvements to the HRLEL include replacement of selected ventilation ductwork to eliminate a radiation hazard and reduce operating personnel radiation exposures; preparation of the West Cell for resumption of examination activities in support of existing programmatic commitments; and preparations for, and construction of, East Cell and North Cell upgrades (FY 1989 and 1990 GPPs, respectively) to correct facility design deficiencies and to meet upgraded building codes. During this period, ventilation ducting, located in the basement, was demolished and preparations were completed for cleaning the remaining ductwork that is located in the ground around the building foundation. The West Cell of HRLEL has been emptied of old, inoperable equipment, and cleaning operations have begun to reduce the impact of this cell's residual radiation on future construction work in the adjacent North Cell. Preparation of control documents (including Preliminary proposals, QA Plan, Safety Assessment, Action Description memorandum, Waste Management Plan, and Risk Assessment) were initiated for the upcoming GPP items.

Decontamination and Decommissioning Technology

The decommissioning and decontamination of nuclear facilities are topics of significant interest to

the nuclear community. To date, more than 140 nuclear facilities, including research, test, and prototype reactors, have been or are being decommissioned. In addition to reactors, a wide variety of other nuclear-fuel-cycle and non-nuclear-fuel-cycle facilities will have to be decommissioned or have remedial actions performed.

The overall objective of Chem Tech's efforts in the fields of decommissioning, decontamination, and refurbishment of nuclear facilities is to assist the nuclear community in performing these tasks in a manner that is safe, timely, and cost-effective and keeps the total radiation exposure to the workers and the public, both at present and in the future, below permissible and within ALARA levels.

This objective is met by organizing a multidisciplinary team to fulfill the following criteria:

1. collect, review, and disseminate the most up-to-date information on technical, scientific, economic, and regulatory aspects of these activities;
2. develop acceptable guidelines, standards, codes of practice, and recommendations for use by sponsors;
3. coordinate and perform research work, data development, and technology transfer; and
4. provide technical assistance and advice.

During this report period, the Chem Tech D&D activities focused on DOE and DOD nuclear facilities.

Technical and Economic Assessment for Decommissioning the Air Force Nuclear Engineering Center, Wright-Patterson Air Force Base, Ohio

The DOD initiated a four-phase Installation Restoration Program (IRP) to identify and evaluate suspected problems associated with past hazardous waste sites on DOD installations. The deactivated nuclear reactor at the Wright-Patterson Air Force Base (AFB), Facility 20470, was included as an IRP site. Concurrently, the Terrestrial Nuclear Reactor Safety Study Group (TNRSSG) reviewed the deactivated status of Facility 20470 and directed that a new decommissioning plan or remedial action plan (RAP) be developed. Chem Tech was selected to prepare the decommissioning option study.

This study presented the results of a technical and economic assessment of the disposition options for decommissioning the deactivated and entombed nuclear reactor at Wright-Patterson AFB, Ohio. Each option was screened on the bases of technical feasibility, environmental impact, economics, and fulfillment of the goals of the IRP.

The four options for study were:

Option 1: Facility upgrade and surveillance program

Option 2: Removal of radioactive material inside the entombment and adjacent facilities

Option 3: Implementation of Option 2 plus removal of concrete monolithic structure

Option 4: Raze all structures and restore site to allow unrestricted use

Option 1 is a continuation of the current program for the operation of Building 20470 with the implementation of various measures to upgrade the facility, improve monitoring, and ensure a safe working environment. Implementation of this option will only ensure better control over the environment than now exists; the facility has deteriorated noticeably since entombment. Option 2 consists of removing the radioactive structures and components inside the entombment and adjacent facilities. Completion of Option 2 will eliminate the containment requirements for radioactivity associated with the reactor, but the utility of the building will not be significantly enhanced. Option 3 is a continuation of Option 2 and provides for the removal of any concrete structures and associated piping left standing in Option 2. In Option 3, the interior of the containment shell is cleared to the 272-m (893-ft) elevation, thereby maximizing the utility of the building for future use. In Option 4, all structures on-site are razed and the site is then restored to unrestricted conditions.

The technical feasibility of each option was weighed. All options are technically feasible, but the handling of radioactive metal and concrete materials will require careful planning. The total dismantling of nuclear reactors is a relatively new frontier; however, past decommissioning efforts at the Elk River Project and Shippingport Station Reactor were successful.

The environmental impact for each option was evaluated. Option 1 has the potential for the

highest environmental impact because no radioactive materials will be removed. Some atomic species will not decay to acceptable levels to allow unrestricted access for centuries. In Options 2, 3, and 4, the environmental impacts are short term and focus on the decommissioning activities. The major environmental impacts for Options 2, 3, and 4 are waste disposal, occupational exposure, and waste transportation.

Economic assessment of the four options was based on past experience with similar decontamination and decommissioning projects. The general tasks for each option were identified, which was considered sufficient for an economic comparison of the four options. However, a detailed work plan and schedule will be necessary for determining a more definitive cost. The estimated implementation and annual operating costs in 1987 dollars for the four options are given in Table 2.9.

A direct comparison between the implementation costs of the four options can be misleading. The cost for Option 1 ($1.0 million) is only for improvements to make the facility safer and more manageable, whereas Options 2, 3, and 4 implement final decommissioning. The character of the entombment in Option 1 is not altered, and surveillance must be continued for many years. Furthermore, the site can be expected to require dismantling sometime in the future, with an expenditure comparable to the estimate for Option 2, 3, or 4.

Implementation costs for Options 2, 3, and 4 are comparable because the options differ only in the extent to which decommissioning (or demolition) is carried out. Option 2 was analyzed in considerable detail to define individual tasks and costs, after which the additional requirements of Options 3 and 4 were treated as add-ons to Option 2. The costs of the latter options were found by determining the incremental costs and adding the base cost of Option 2. The estimated cost of Option 2 is $26.4 million, and the additional cost of extending decommissioning to the removal of all interior structures in the containment shell (Option 3) is $1.5 million. Hence, Option 3 can be implemented for about $27.9 million. Option 4 requires the removal of all buildings and other structures from the site and restoration of the site to its original condition. The additional cost between the estimates of Options 3 and 4 is approximately $14.3 million, for a total Option 4 cost of $42.2 million.

Surveillance, maintenance, and utility costs for Option 1 are estimated at $530,000/year, which includes air conditioning the containment shell. The annual operating cost ($353,000) for Option 2 or 3 is less than that of Option 1 because no surveillance is required. There is no annual operating cost for Option 4.

The operating costs for the facility can be expected to escalate over its lifetime because of economic factors. These costs were calculated for Option 1 using annual escalation rates of 3, 4, and 5%, and the accumulated costs to 2020 were $30.6, $37.0, and $45.0 million, respectively. The year 2020 was chosen to given an understanding of the economics for maintaining the facility for an indefinite period. The lifetime stipulated in the original decommissioning plan (i.e., 2121) no longer applies because the regulations passed since entombment extend the year 2121 to an indefinite date in the future.

Table 2.9. Cost comparison of decommissioning alternatives Basis: 1987 dollars

Option	Estimated cost ($10³)	
	Implementation	Annual operating[a]
1	1,000	530[b]
2	26,400	353
3	27,900	353
4	42,200	

[a]Includes manpower, maintenance, and utilities.
[b]Includes programmed surveillance and environmental monitoring.

Oak Ridge Research Reactor Shutdown Program

On July 20, 1987, DOE made the decision to shut down the ORR permanently. The ORR was the last DOE general-purpose high-flux test reactor remaining in operation. The prime purpose of the 30-MW reactor was to provide a facility in which a variety of irradiation experiments could be performed. The decision to shut down the ORR necessitated that the reactor be placed in a safe storage mode while awaiting final decommissioning. Chem Tech has assisted the Research Reactors Division in determining what constitutes safe shutdown for the ORR and has developed the program plan for implementation.

The program plan for safe shutdown of the ORR is defined in three work phases:

Phase I - Fuel disposition and minimal disposition of reactor support systems

Phase II - Placing the ORR facility in a safe condition for routine maintenance and surveillance

Phase III - Decommissioning planning

The guidelines for determining shutdown tasks were:

1. The reactor is completely defueled.
2. The heat transport and moderator system fluids, which are readily removable contaminated materials, are disposed of properly.
3. The first containment barrier (e.g., the reactor and associated piping) is kept as it was during operation, but with all mechanical openings (valves, piping, etc.) blocked and sealed.
4. The building and containment systems are maintained intact in a state appropriate to the remaining hazard.
5. The atmosphere inside the containment building (and in all areas containing radioactivity) is controlled, and the ventilation systems are operated as required.
6. Access to the inside of the containment building is controlled by physical barriers and administrative procedures.
7. The facility is kept under continuous on-site surveillance, and equipment necessary for monitoring radioactivity both inside the facility and in the surrounding area is kept operable and used as required. Surveillance is maintained in accordance with applicable regulatory requirements. Periodic measurements and visual checks are carried out to ensure that the contamination control systems (i.e., ventilation systems and containment barriers) continue to function.
8. Dismantling of structures, systems, or components does not, in general, take place.
9. Quantities of LLWs will be shipped from the facility for disposal.
10. Hazardous, chemical, and mixed wastes are shipped from the facility for disposal.

Table 2.10 lists the activities to be performed during the three work phases. The ORR is currently implementing Phase I of the ORR Shutdown Program Plan.

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 (TMI-2) Nuclear Power Station has continued since shortly after the accident in 1979. In addition to the active participation of several of its staff members, Chem Tech has managed all TMI-2 assistance provided by personnel at DOE facilities in Oak Ridge, Tennessee.

Several projects have been carried out to analyze the potential for criticality during various phases of the reactor defueling operations. These analyses have been made by staff members of the Nuclear Engineering Applications Group in the Reactor and Fuel Cycle Section of the ORNL Computing and Telecommunications Division.

A significant project was carried out in Chem Tech to (1) perform leaching tests on eight concrete samples taken from the TMI-2 reactor building basement, where radiation sources in some areas still exceed 100 R/h; (2) measure pertinent characteristics of the concrete, such as density, porosity, and permeability; (3) derive diffusion models from the leaching data; and (4) predict leaching times required to decontaminate the various types of concrete in the basement.

The leaching data were fitted to equations describing diffusion from the region between two parallel planes.[28] For example, for a fixed-surface concentration of zero, the equation used was

$$F_T = \left[\frac{A_{ow} - W}{A_{ow}}\right]\left[1 - \frac{8}{\pi^2}\right]\sum_{n=0}^{\infty}\frac{1}{(2n+1)^2} \times \exp\left[-(2n+1)^2\pi^2 Dt/4\ell^2\right] + \frac{W}{A_{ow}}, \quad (1)$$

where

A_{ow} = total amount of material in the specimen and loosely bound to the surface,

Table 2.10. Oak Ridge Reactor (ORR) shutdown activities

Phase I	Phase II	Phase III
Preparation for fuel removal	Remove miscellaneous equipment from pool	Decommissioning option study
Ship fuel off-site	Resin dewatering/disposal	Select decommissioning option
Fuel to BSF	Tower removal	Prepare detailed decommissioning
Fuel to SRP	Study/characterization	Prepare environmental assessment
Pool study	Removal/disposal	Transfer of ORR to SFMP for
Waste disposal on-site	Spring water diversion system	implementation of detailed
Piping changes/winterization	Pool demineralizer system study	decommissioning plan
Drain secondary tower basins/	Hot cell shutdown	
primary water line (pump area)	ORR Systems documentation	
and decay tank	verification	
Preparation and approval safe	Remove beam tube shields/seal	
shutdown plan	beam tube plugs	
	I.D. Tag verification for all pipes,	
	valves, etc.	
	Seal exterior pool wall penetrations	
	Identify/drain stagnant lines	
	Establish ORR archives	
	Waste disposal	
	ORR control panel consolidation	
	Alarm system for emergency	
	generator	
	ORR House cleaning	
	Operations/maintenance plan	
	ORR site characterization	
	Environmental considerations	
	Quality assurance document	
	Revised ORR technical specifications	
	document	
	Phase II final report	
	Application for ORR transfer to	
	SFMP and beginning of legal	
	transition period	

D = apparent diffusion coefficient,
F_T = total fraction leached by washoff of the surface and diffusion,
ℓ = length (specimen volume/exposed surface),
t = leaching time,
W = amount of loosely bound surface material.

Estimates of values for D and W were obtained by fitting the equations through use of a computer program based on a direct-search method of constrained optimization.[29] These values were then used with the appropriate equation to predict the percentages of ^{137}Cs and ^{90}Sr leached at extended times (Table 2.11).

Further, a model was developed to predict potential enhancement in decontamination of concrete block walls by forcing leachant through the walls. This model used a numerical algorithm that describes convection/adsorption phenomena in packed-bed systems[30] and assumed that (1) the center of the block was filled with leachant, (2) the block behaved as a packed bed containing 1-mm-diam spheres, and (3) the leachant traversed the block in plug flow. To utilize the model, the previously obtained values of D and the measured void-free density, ρ (2.5 g/cm^3), and porosity, ϵ (0.35), were used to estimate the cesium and strontium distribution coefficients, K [(μCi/g of solid)/(μCi/mL of liquid)], between the liquid and solid phases at equilibrium, using the simplified relationship[31]

$$D = \frac{D_e}{g\left[1 + \frac{(1-\epsilon)}{\epsilon}\rho K\right]} \quad (2)$$

Table 2.11. Predicted percentages of ^{137}Cs and ^{90}Sr leached from concrete samples as a function of time without forced flow[a]

Time (months)	Unpainted concrete block[b]		Painted concrete block[c]		3000-psi unpainted concrete[d,e]		3000-psi painted concrete[d,f]		5000-psi painted concrete[d,g]	
	Cs	Sr	Cs	Sr	Cs	Sr	Cs	Sr	Cs	Sr
6	28	70	26	77	46	87	36	73	35	68
12	39	88	35	93	63	98	48	92	49	88
18	48	96	43	98	75	99	61	98	60	95
24	55	98	49	99	83	100	73	99	68	98
36	67	100	59	100	92		88	100	80	100
48	75		67		97		95		87	
60	82		74		98		98		92	
120	96		91		100		100		99	

[a]Predictions were made with distribution coefficients (D) obtained by fitting the leaching data to established mass transport equations.
[b]The value of D for Cs = 3.4×10^{-9} cm^2/s and for Sr = 2.2×10^{-8} cm^2/s.
[c]The value of D for Cs = 6.4×10^{-10} cm^2/s and for Sr = 6.8×10^{-9} cm^2/s.
[d]Poured concrete having the indicated compressive strength.
[e]The value of D for Cs = 1.0×10^{-9} cm^2/s and for Sr = 4.4×10^{-9} cm^2/s.
[f]The value of D for Cs = 2.2×20^{-9} cm^2/s and for Sr = 1.2×10^{-8} cm^2/s.
[g]The value of D for Cs = 5.9×10^{-10} cm^2/s and for Sr = 2.4×10^{-9} cm^2/s.

Values for the dimensionless geometry factor, g (20 for cesium and 8.9 for strontium), and for the diffusion coefficient at infinite dilution, D_s (2.1×10^{-5} cm^2/s for cesium and 7.9×10^{-6} cm^2/s for strontium), were taken from the available literature.[31-33] Estimates of K for unpainted block (66 for cesium and 8.4 for strontium) and for painted block (350 for cesium and 28 for strontium) were made using Eq. (2).

Using the forced-flow model, the time required to decontaminate unpainted block was calculated to be several days. This is several orders of magnitude less than that required when using only diffusive flow.

Characterization of Y-12 Plant Waste

The purpose of this activity (a task of the Uranium Lysimeter Project) was to develop uranium leaching data to support the (1) evaluation of disposal options and (2) analysis of disposal scenarios for wastes that are radiologically contaminated with depleted uranium and are generated by production operations at the Y-12 Plant. The task supported field-scale activities by characterizing the wastes that may be placed in the lysimeters and by aiding in the prediction and analysis of waste performance in the lysimeters as a function of time. The primary task milestone, which was completed on schedule (June 30, 1988), was the contribution of uranium leaching information for the LLWDDD Environmental Data Package.

The task was primarily a laboratory-scale activity. Work on a relatively small scale (compared with field lysimeters or actual disposal operations) was essential in maintaining the task schedule and in allowing control of test parameters that could simulate accelerated time (i.e., allow the laboratory study in a few days of leaching events that will take longer times in the lysimeters). Most of the leaching was done in 30-gal drums. Two leachants (synthetic groundwater and synthetic landfill leachate) were used, and two test protocols (batch and sequential contact) were followed.

Five wastes that contained depleted uranium were identified by the Y-12 Plant Waste Transportation, Storage, and Disposal Department as priority materials for investigation, and samples of these were used in the leaching tests. These priority wastes were:

1. *Production trash*—waste generated by the procedures used for cleaning and protecting the floors, resulting from efforts to minimize the spread of contamination, and general trash found on the floors. Production-trash samples consisted of a wide variety of components. Samples frequently contained floor sweepings, paper, metal scrap or parts, Hot Hogs (an adsorbent in a cloth tube), oily cloths, plastics, and so forth.
2. *Mixed-metal chips*—a mixture composed of metal turnings of aluminum, iron, stainless steel, copper, or brass. Mixed-metal chip samples were primarily turnings and machining chips of various nonuranium metals. Most samples were quite oily.
3. *Composite waste*—an assortment of materials consisting of one-third production trash, one-third mixed-metal chips, and one-third general trash from highly contaminated areas. This composite waste is representative of the overall mixture of Y-12 Plant wastes for disposal. Composite waste samples were a mixture of the components in the first two waste types plus a wider variety of cloth, leather, and plastic materials. Sometimes these components were coated with a red oily liquid.
4. *Air filters*—contaminated air filters from building ventilation systems throughout the Y-12 Plant. These samples were standard HEPA air filters. The filter medium, which is constructed of fiberglass with aluminum separators, is supported on both sides by galvanized-steel wire guards inside a steel frame [0.6 × 0.6 × 0.3 m (2 × 2 × 1 ft)]. Appreciable amounts of an easily dispersed, yellow-green U_3O_8-containing powder were observed on all filters.
5. *Uranium oxide powder*—powder from the uranium chip oxidation facility (UCOF) where uranium metal turnings and chips are burned to a mixture of uranium oxides. One sample was obtained and homogenized for use in all uranium oxide tests. The sample was primarily UO_2, with a small amount of U_3O_8.

Perhaps the single most significant observation is the finding of bimodal uranium release curves as a function of time for the batch-contact tests. In some tests, the uranium concentration in the leachate or the fraction of the initial uranium leached increased over time and did not reach a steady-state limit or constant value in 7 d (the last time point). Such behavior could be consistent with slow leaching kinetics, for example, reactions such as slow oxidation of uranium metal or reduced uranium oxides. In the other tests, the uranium concentration or fraction released maximized on day 1 (the first data point) and then decreased steadily to very low values by day 7. Such behavior could be consistent with the adsorption or precipitation of initially solubilized uranium.

It is possible that the redox state of the system controls the leaching mode observed and is responsible for the different leaching modes. The decreasing-mode observations are consistent with the development of reducing redox conditions in the leaching vessel. The most likely reductant for these tests is the reactive metal chips (aluminum, brass, iron, etc.) in the various wastes, as well as any uranium metal chips or turnings. These metals can react with water to form strongly reducing conditions. Under such reducing conditions, any solubilized uranium would be reduced to the quadrivalent state; and it is well known that the corresponding uranium(IV) oxide, UO_2, has a very low solubility in aqueous solutions. Therefore, if strongly reducing redox conditions developed during the leach test, any uranium initially solubilized as U(VI) species would be precipitated (decreasing leach mode); and, of course, any undissolved uranium (in the form of the metal or lower-valence oxide) could not be solubilized because no oxidant remains in the mixture to convert the U(IV) to the soluble U(VI) species.

The best correlation of the uranium leaching data with experimental parameters was obtained by plotting the uranium concentration in the leachate vs leachate pH (Fig. 2.20). The log of the uranium concentration is plotted vs sample pH for the two leachants used, two test protocols, and the 1- to 7-d samples for each of the five waste types. The figure reveals clusters of data points for the different waste types; however, the initial leachant employed had only a minor or second-level effect on the uranium concentration. It is not clear why the sample pH values for the buffered synthetic landfill solution cover essentially as wide a range as the unbuffered synthetic groundwater samples. Uranium oxide powder and air filter wastes gave the highest uranium concentrations, and the data points are clustered in the more acidic pH range.

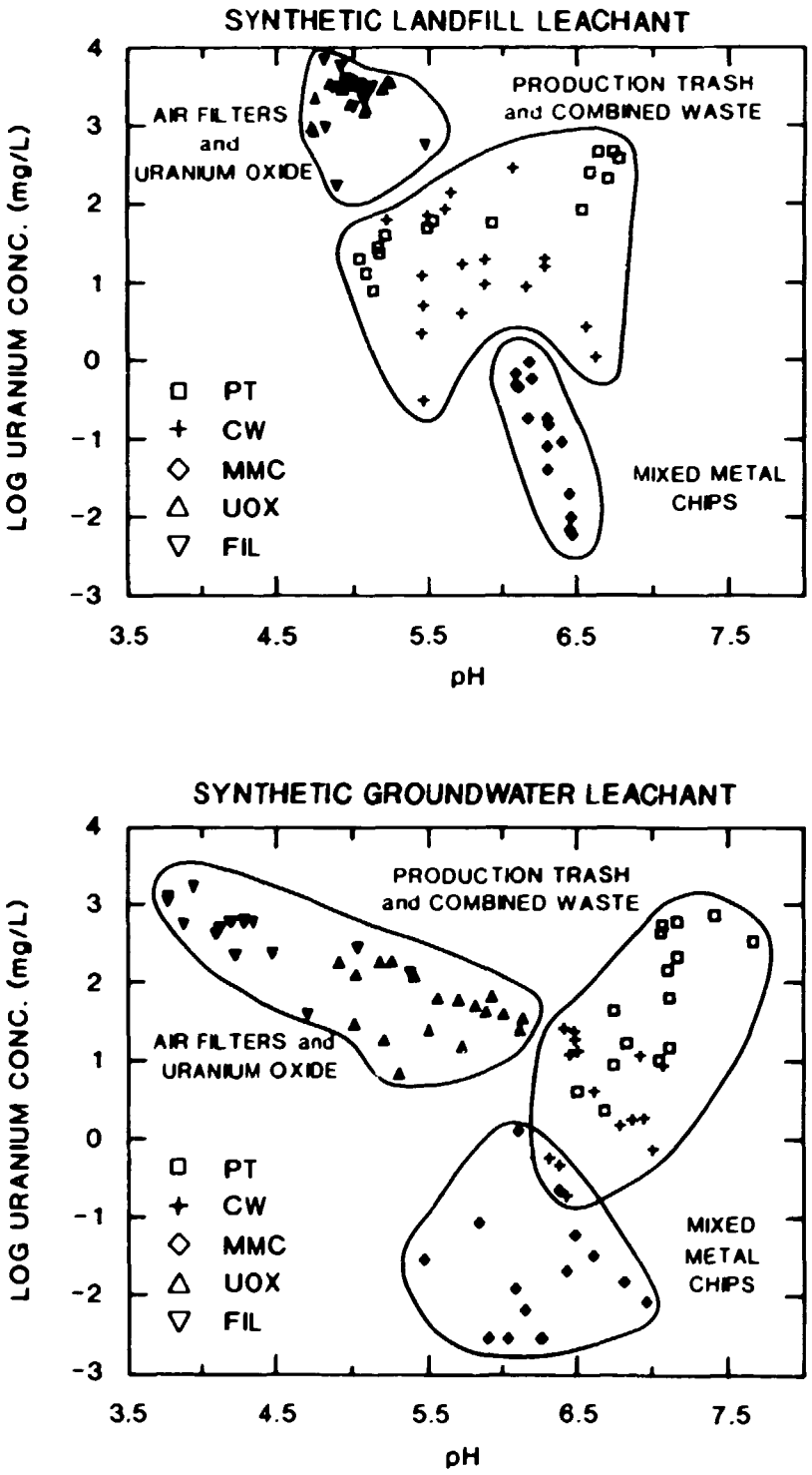


Fig. 2.20. Uranium concentration in the leachate as a function of leachate pH. □, production trash; +, composite waste; ○, mixed-metal chips; Δ, uranium oxide powder; and ▽, air filters.

This is a logical result for leaching into the buffered landfill leachate; however, because the dissolution reactions consume acid, unbuffered solutions such as the synthetic groundwater were expected to become more basic, instead of more acidic, during the test. The mixed-metal chip data occupy a region in the plot at very low uranium concentrations. The data points for production trash and combined waste overlap to a considerable degree and generally show more scattering. A possible conclusion that can be drawn from this plot is that waste components other than the uranium contaminant or the leachant employed control the chemistry of the leaching system and, in turn, the uranium concentration in the leachate.

Waste Immobilization Studies

Transportable Grout Facility

Since FY 1982, Chem Tech has provided technical support to Westinghouse Hanford Operations (formerly Rockwell Hanford Operations) in their efforts to dispose of radioactive LLLW by immobilization. Initial efforts focused on the transfer of technology developed during the operation of the hydrofracture facility to the Hanford site. As the program progressed, our role expanded to include support in the areas of plant design, process development, and waste form development. This program has led to the design, construction, and operation of a Transportable Grout Facility (TGF) at the Hanford site. The major components of this facility are: a 3785-m^3 (1×10^6 gal) waste feed tank, a dry materials receiving and handling facility, the transportable grout equipment, and a near-surface disposal vault in compliance with RCRA requirements. The TGF is scheduled to begin operation in the summer of 1988 and will operate on a campaign basis. As currently envisioned, each campaign will consist of grouting 3785 m^3 (1×10^6 gal) of waste; four to six campaigns are expected to be carried out annually.

In addition to providing technical support for facility startup, efforts have been directed during this report period toward the development of cement-based grout formulas for a wide variety of LLLWs to be processed in the TGF. The formulation criteria can be summarized as follows:

1. *Expansion*
   - No increase in volume while grout is in the nonplastic state
2. *Heat Generation*
   - Maximum adiabatic temperature rise of 30°C heat of hydration, with a simulated feed starting temperature of 60°C
   - Thermal conductivity of 28-d-cured grout must be ≥0.45 Btu/h·ft^2·°F (≥2.56 W/m^2·K)
3. *Dry Blend*
   - No more than four dry materials
   - Formulation must pass all criteria for ±5% relative variation in dry blend
   - Mix ratio of ≤1018 kg/m^3 (≤8.5 lb/gal) liquid waste
   - Formulation must pass all criteria ±599 kg/m^3 (±5 lb/gal)
4. *Additives*
   - No more than three additions per single formulation
   - Additive flow rate shall not exceed 3.15×10^{-5} m^3/s (0.5 gal/min)
5. *Processing Parameter*
   - Frictional pressure drop of ≤2533 Pa/m (≤11.2 psi per 100 ft) of pipe
   - Critical flow rate of ≤3.79×10^{-3} m^3/s (≤60 gal/min)
   - 10-min (600-s) gel strength of ≤47.9 Pa (≤100 lb_f per 100 ft^2)

   [Note: all parameters to be calculated on 0.05-m (2-in.) schedule 40 pipe]
6. *Regulatory Criteria*
   - ≥413-kPa (≥60-psi) compressive strength using ASTM Test C-39 [483 kPa (≥70-psi) compressive strength using ASTM C-109-80]
   - ≤3 vol % bleed water after 28 d
   - ANS 16.1 leach index ≥7.0 for ^{99}Tc, ^{129}I, ^{79}Se, NO_3, NO_2, ^{14}C, and total uranium
   - Pass EP Tox nonhazardous criteria for heavy-metal priority pollutants

Solidification of Melton Valley Storage Tank Supernate

At ORNL, eight 190,000-L waste storage tanks containing alkaline, sodium nitrate-based (30 wt % nitrate) LLW are located in an area designated as Melton Valley. Most of this waste has originated from ion-exchange processes involving the use of nitric acid as an eluent. The radioactivity associated with the alkaline sodium nitrate-based solution comes, for the most part, from ^{137}Cs, ^{90}Sr, and some trace rare earths. In addition to radioelements, low levels of trace metals and nonmetals of current concern to the EPA are present in the solutions.

Concern over the rate at which the storage tanks were filling led to the implementation of an Emergency Avoidance Solidification Program, which has the overall objective of immobilizing 192,000 L of the tank supernate. Cement-based grout was chosen as the immobilization medium, and a number of commercial vendors were requested to bid on the process. Two of them, LN Technologies Corporation of Columbia, South Carolina, and Chem-Nuclear Systems, Inc., also of Columbia, were selected as the primary and backup vendors, respectively.

Both vendors were requested to develop cement-based formulas for laboratory testing with a surrogate waste formula representing the MVST waste. They were also requested to perform the relevant laboratory performance testing at their facilities or to find a contractor to do it for them. The waste forms were required to meet all the testing requirements set forth in the Nuclear Regulatory Commission's 10 CFR 61 Branch Technical Position Paper and those of the EPA. In addition to the vendors' tests (once the vendors decided on an acceptable cement-based formula), experiments were also performed at ORNL using the actual waste to verify compliance with established objectives.

As a part of our verification effort, ORNL personnel traveled to the vendors' facilities to review all tests in progress, methods, and quality assurance procedures. The results confirmed that each vendor was able to meet the requirements as specified in the NRC and EPA regulations. As a result, LN Technologies Corporation plans to have its equipment on-site in Melton Valley in August 1988 to immobilize the MVST supernate.

In the field, an in-container mixing mode will be used in which the supernate is pumped into a mild steel liner (3- by 3-m right-circular cylinder), and the dry-solid components will be added and blended by using a dc-motor power head attachment as shown in Fig. 2.21. The container already has built-in paddle mixing blades and baffles that remain inside the waste form after the power head has been removed. The waste-form solid will contain 6.2 kg of dry solid per liter of waste, and each container will hold 3400 L of waste. The immobilization campaign will yield about 60 containers, and each will be inside its own special concrete cask to minimize radiation exposure and facilitate optimum storage conditions. The containers and casks will be stored on gravel and concrete pads in Melton Valley until final disposition of the containers is determined.

Evaluation of Grouts Under Pressure

For more than 20 years, ORNL successfully disposed of radioactive liquids and sludges with a unique process that combines horizontal hydrofracturing of a competent shale with a grout slurry and in situ waste solidification technologies. When operations ceased in 1984, studies were initiated to support the closure of the hydrofracture site. One of these studies was the laboratory characterization of physical properties of grouts representative of those produced during the operation of the facility. Characterization measurements included leachability, compressive strength, density, porosity, permeability, internal surface area, and phase separation. The characteristic of greatest interest proved to be phase separation.

Test results have shown that curing the grouts under pressures representative of those found in hydrofracture shale beds (500 to 600 psi) would result in no increase in drainable water volume over what would be expected for grouts cured at atmospheric pressure. The only place that such conditions would exist in the field is probably at the leading edge of the grout sheet. At that point, the grout would cure under uniform overburden pressure, and it would not be possible for water to escape because of the relatively impermeable shale and the bulk of the grout beginning to set behind the front. However, the bulk of the grout would be subjected to a pressure gradient from the injection pressure during the injection process. An outlet for

Fig. 2.21. Photograph of the LN Technologies Corporation steel liner with the fill and mixing head attachment above (left) and hydraulic skid with dust collector (right). (Photograph used by permission of LN Technologies Corporation)

expressed water would exist via the shale fracture. These conditions were simulated in a study performed at the University of Illinois. In this study, consolidation was defined as the degree, or percentage, to which the density of the grout is increased under pressure as a result of the expression of water from the grout.

Grouts representative of those used during hydrofracture operations were prepared at mix ratios of 716, 842, and 947 kg of dry solids blend per m^3 of waste and subjected to pressure gradients ranging from 2068 to 10,548 kPa. Both the compressive strength and the percentage of consolidation were determined for these grouts at various times during the curing period and at pressure gradients of 2068, 4136, 6205, 10,341, and 10,548 kPa. The resulting data demonstrated a dependence of consolidation potential of a grout sample on its compressive strength at a particular stage of the curing period. This relationship between consolidation and compressive strength, which proved to be relatively independent of both consolidation pressure and mix ratio, can be expressed by the following:

$$C = -10.1\,[\log(S/6.894)] + 34.5\,, \qquad (3)$$

where C is percent consolidation and S is compressive strength in kPa.

The results of the study allow prediction of the degree to which consolidation will occur in any particular hydrofracture grout sample at a given pressure if the compressive strength of that sample is known. The study also predicts that grouts that were injected into the underground shale beds in the hydrofracture process will undergo some degree of consolidation during cure, and the result will be a denser, less permeable product with a higher

effective dry solids-to-liquid ratio than is reflected by the original formulation.

Process Safety Studies Initiative

Chem Tech has a long history of safe and successful operation of radiochemical facilities. Although handling radioactive materials without significant problems requires careful planning, intensive training, and extraordinary controls, the necessary design concepts and operating procedures have been developed and are reviewed periodically to ensure a high degree of confidence in safe operation. With the worldwide concern about the safety of chemical process plants, as stimulated by the tragic accident at Bhopal, India, for example, it is clear that some of the design and operating principles used for radioactive work at ORNL could be transferred to the nonradioactive arena as well. This extension represents a new initiative for Chem Tech in chemical process safety.

Familiarization training in safety assessments such as Hazard and Operability Analysis was provided as part of this new initiative. This training was applied to an existing program, the EASC, as a demonstration of the strength of the procedure and our ability to provide the service on-site. In addition, the nonreactor nuclear facilities (TPP, RPP, and the 4501 Hot Cells) in Chem Tech were subjected to an exhaustive internal safety review. The results proved useful enough that we were requested to provide a similar review for the Transuranium Research Laboratory in the Chemistry Division.

The process was continued with a project within HAZWRAP for the U.S. Air Force. The objective of this project was to determine the source terms of chemicals from a fire-fighting training facility where jet-fuel fires are set and extinguished.

Hazardous Waste Technology Program Overview

The HWTP is the focal point of hazardous waste technology R&D for the Energy Systems' research divisions. This program represents the inherent technical strengths that ORNL can contribute to various sponsors, such as the U.S. Air Force, the U.S. Navy, and EPA. The DOE national HAZWRAP funds RD&D projects at many DOE sites, and the HWTP serves as liaison between the technical community at Oak Ridge and HAZWRAP.

The HWTP offers assistance to the sponsors by building technical teams to work on projects that they bring to ORNL. The program is responsible for ensuring that the results are responsive to the sponsor, are of high quality, and are integrated across organizational boundaries. Since hazardous waste problems tend to be complex, the expertise of researchers from several divisions is often required. The HWTP serves as a business agent for the projects, helping to prepare the necessary documentation to allow the sponsored work to be done.

Several projects aimed at developing technology useful to the federal sponsor and to cleanup efforts at Oak Ridge have been initiated or completed. The Air Force Engineering and Services Center is sponsoring two multiyear studies to remediate soil and water containing volatile organics and has obtained support in an evaluation of fire-training pit design. ORNL's expertise in nuclear reactor D&D was employed by the Air Force Logistics Command (AFLC) to develop options for the shutdown of a test reactor in Dayton, Ohio (described earlier). The AFLC is using our waste immobilization experience to develop and demonstrate solidification of oil-contaminated soil and sludges at Robins AFB, Georgia. The Naval Engineering and Energy Support Activities sponsored wastewater cleanup and remediation studies and demonstrations at a naval facility in Louisville, Kentucky. The Tennessee Valley Authority (TVA) utilized our water treatment capabilities in a study on hyperfiltration process development. These projects represent Chem Tech's initial efforts in the exciting program area that is known as "Work for Others."

Remediation of Soil and Water Containing Volatile Organics

Air Stripping with Emissions Control

This project, which is being conducted for the Department of the Air Force, will field test innovative air-stripping techniques (packed-tower and rotary air strippers) and materials in concert with emissions control technologies (catalytic incineration and granular activated carbon). The ultimate goal of the project is to provide managers and engineers responsible for groundwater cleanup with design information for full-scale implementation and to permit a direct comparison with technologies and materials tested in previous Air Force

studies. A secondary objective is to develop a manual to aid the major commands (MAJCOM) and base-level personnel in the selection of an appropriate remedial action. This manual will focus on comparing pump-and-treat technologies but will briefly describe other viable alternatives.

A packed-tower type of air-stripping system, which includes an activated-carbon adsorption unit, has been designed, fabricated, and installed on a portable skid at ORNL. Also, a trailer has been outfitted with analytical equipment and a data acquisition and analysis system. The skid and trailer, along with a purchased rotary air stripper and a leased catalytic incineration unit, will be shipped to Eglin AFB, Florida, during the first part of August 1988.

After startup operations have been completed in early September, an experimental program will be conducted through January 1989. The various objectives of the air-stripper tests involve evaluation and development of performance correlations, evaluation of new packings, and demonstration of long-term operability. In the carbon adsorption and catalytic incineration tests, the objectives include performance correlation evaluation, evaluation of different types of catalysts, and determination of the behavior of a multicomponent feed to these emissions control units. A final technical report, with an expected date of issue of April 1989, will be prepared after completion of the experimental test program.

A questionnaire has been sent to MAJCOM and base-level personnel responsible for groundwater cleanup. The objectives of this questionnaire are to outline the scope of the user's manual, to solicit information concerning problems that face decision makers, and to determine how the manual can best serve the users' needs.

In Situ Soil Venting

A promising technology for the remediation of unsaturated zone soils contaminated with volatile organic compounds (VOCs) is in situ soil venting. In this technique, a large volume of air is passed through the soil. The air flow sweeps out the gas present in the soil, disrupting the equilibrium that exists between hydrocarbons that are sorbed onto soil particles and those that are dissolved in soil pore space water as free liquid or are present as vapor. This leads to volatilization of the contaminants and subsequent removal in the air stream. In situ soil venting, which is most applicable to soils of high permeability and contaminants of high volatility (vapor pressure >70 Pa), may be a cost-effective decontamination technology.

Chem Tech's Environmental Control Technology Group is working in conjunction with ESD on a project for the Air Force Engineering and Services Center to demonstrate in situ soil venting at a fuel spill site. The major aim of the test is to determine cleanup efficiencies attainable by soil venting and to define operating, cost, and system design parameters for application of soil venting at other sites.

The site selected for the demonstration is a fuel-storage area at Hill AFB, Utah, where 102,000 L of JP-4 was spilled in January 1985. Because of remedial action by the base, including tank excavation and reconstruction of the tank enclosures, the site provides an opportunity for testing three venting geometries: (1) conventional vertical vents, (2) lateral vents, and (3) vents in the excavated soil pile.

Site characterization by soil gas survey and soil sampling has been conducted by ORNL's Health and Safety Research Division to determine both lateral and vertical distribution of contaminants Hydrocarbons are present within a 30- by 60-m area and are limited to a depth of 18 m.

A single-vent pilot test was conducted to determine site-specific information necessary for design of the full-scale demonstration system. A single extraction vent with a slotted section from 3 to 15 m deep was installed, surrounded by an array of nine pressure-monitoring points. The system was operated to reach steady state at four extraction rates that ranged from 0.033 to 0.12 std m^3/s, during which pressure distribution in the soil and extraction gas hydrocarbon concentration were measured. The results of the pilot test indicate very high initial removal rates, with 607 kg of hydrocarbons being removed during 20 h of operation at an average flow rate of 5.7 std m^3/s. The soil in the area is highly permeable, allowing high extraction rates at relatively low vacuum (0.12 std m^3/s at a vacuum of 4980 Pa below atmospheric pressure).

Presently, the test plan for the full-scale demonstration at Hill AFB is under revision by the Air Force, EPA, and the state of Utah. The plan for the full-scale demonstration system includes 15 vertical extraction vents, 6 lateral vents, and 8

vents in the excavated soil pile. The extracted soil gas will be fed through a common header to a catalytic incineration unit for emissions control.

The full-scale system vents will be installed beginning in August 1988. System operation, which is scheduled to begin in October 1988, has a projected length of at least 1 year.

Air Stripping at Eglin Air Force Base

Two groundwater contamination sites at Eglin AFB, Florida, are being remediated via a pump-and-treat technique. At the first site (denoted as "A-20" by the Air Force), where a diesel fuel spill had occurred, the remediation treatment involves a packed-tower type of air-stripping operation followed by aqueous-phase granular activated carbon adsorption. The second site (Seventh Street BX) is a gasoline spill site, and the method of treatment is packed-tower air stripping. At the A-20 site, the system effluent is returned to the groundwater via exfiltration galleries. At Seventh Street BX, the air-stripper effluent is discharged to the local sewage treatment plant. The Air Force requested assistance from ORNL via HAZWRAP to analyze the operation of each system for a 1-year period during which data on efficiency and operability would be collected.

Additional instrumentation and sample points were installed on the air-stripping systems by ORNL in August 1988. After maintenance and operational testing procedures have been completed by the CH2M HILL, Inc., ORNL will be responsible for operation of the strippers for 1 year. ORNL will also be responsible for the sampling and analyses required by the state of Florida for the remediation of the A-20 and Seventh Street BX sites.

Hazardous Waste Minimization at the Naval Ordnance Station at Louisville, Kentucky

The Naval Ordnance Station (NOS) is an industrial facility that produces new ordnance hardware and reconditions hardware already placed in service. Processes in the NOS include: (1) machine and tool shops, (2) a complete plating facility, (3) a variety of painting and stripping areas, (4) assembly areas, and (5) test and evaluation areas. Approximately 2300 m^3 (600,000 gal) of wastewater per day is produced by these processes within the NOS. This wastewater is discharged to the local sanitary sewer, which is regulated by a local Metropolitan Sewer District (MSD). The MSD has established discharge limits for the NOS, and reports have indicated that the major source of wastewater (and primary area of concern) is the discharge from the industrial plating shop within the NOS. This plating shop generates $\sim$1100 m^3 (300,000 gal) of wastewater per day.

Because construction of a new plating shop is currently planned, complete renovation of the existing plating shop is not economically feasible. As a result, the Naval Energy and Environmental Support Activity (NEESA) has been given the task of demonstrating the use of innovative wastewater treatment technologies that will minimize the volume of wastewater generated from the plating processes and maintain compliance with MSD discharge until the new plating shop has been constructed.

The problems at the NOS plating shops have been analyzed, and a characterization study of the effluent from the plating shop indicated that the effluent contained chromium concentrations that were, at times, in violation of the permit discharge limits. Several improvements in the operation of the plating shop were made to reduce the quantity of chromium-contaminated wastewater generated. These improvements included: (1) installation of a closed-loop rinsing system with zero aqueous discharge on the chrome plating line, (2) utilization of a "new bright-dip formula" that contains $\sim$75% less chromium than the previous formula, and (3) replacement of the dichromate deoxidizer on the chromate conversion line with a chromium-free deoxidizer. A new ventilation system, which is in the process of being installed, should eliminate the chromium that collects in the defunct ventilation scrubbers. These improvements in the system should reduce the quantity of chromium contamination by as much as 85%.

With the improvements noted above, the remaining major sources of chromium contamination are the rinse waters from the gun-barrel line and the bright-dip line. A system consisting of reverse osmosis (RO) coupled with concentrate evaporation was recommended to treat these streams. The RO unit will meet NEESA's objective of demonstrating innovative wastewater treatment technology that will produce information useful for treating similar wastewaters at other naval facilities. Evaporation of the RO concentrate will maximize

waste volume reduction and process flexibility. Pilot-scale RO tests currently being conducted at the NOS indicate that the gun-line rinse water can be successfully treated by RO. Further tests are being conducted on the bright-dip rinse water to determine the best pretreatment scheme for this waste stream. The installation of a full-scale unit at the NOS is dependent on the successful completion of pilot studies currently in progress.

Hyperfiltration Process Development

Hyperfiltration technology will be utilized by TVA to concentrate radionuclides present in reactor waste streams such as floor drains, tritiated water drains, and demineralizer regeneration overflows to reduce the load to a downstream evaporator. The hyperfiltration membranes have a limited life and, unlike other osmotic membranes, can be regenerated remotely by flowing chemical solutions through the porous frit pipes of the apparatus. TVA requires that operating procedures for the membrane stripping and re-formation processes be on file. Since this information is not readily available to TVA, ORNL was requested to prepare a work plan to supply the needed information. The work plan discussed the equipment and materials required to develop detailed procedures to clean used membranes, strip permanently degraded membranes, and re-form new hyperfiltration membranes based on information from published ORNL sources and the open literature.

Preliminary work was begun on the laboratory-scale development of the process by using some ultrafiltration equipment available from earlier tests at ORNL. The membrane re-formation is a one- or two-step process that involves the formation of a first (ultrafiltration) layer of hydrous zirconium oxide and a second (hyperfiltration) layer of polyacrylic acid. The principal difficulty is believed to be the formation of the second layer, especially on the long-tube apparatus that TVA has purchased. High-speed, high-pressure circulating pumps are necessary to achieve the high velocity flows necessary to generate effective hyperfiltration membranes. The project is expected to be completed during late FY 1988 or early FY 1989.

Environmental Evaluation of Air Force Fire Training Pits

The Martin Marietta Energy Systems, Inc., Engineering Organization at ORNL has undertaken the design, construction, and testing of a new Environmentally Acceptable Fire-Training Facility (EAFTF) for the purpose of providing live fire-training capabilities without harming the environment. At present, prototype fire-training facilities are planned for the Tyndall, Davis Montham, Grand Forks, and Chanute AFBs. The specific goals of these facilities are (1) to eliminate contamination of area soil, groundwater, and surface water by the loss of JP-4 and fire suppressants; (2) to provide enhanced training and improved safety for fire-fighting personnel; (3) to reduce air emissions associated with conventional fire training; and (4) to minimize air and water pollution impact by treatment, recycling, and reuse of resulting effluent and wastewater and recovered fuel.

Construction of a prototype EAFTF is currently nearing completion at the Tyndall AFB. The EAFTF at Tyndall AFB is expected to (1) demonstrate that the design concepts incorporated into the EAFTF are technically sound, environmentally acceptable, and economically feasible; (2) demonstrate that the EAFTF design provides a safe and adequate basis for fire-fighter training to support the Air Force mission; (3) provide enhanced research opportunities for Air Force personnel; (4) evaluate the performance of individual components of the facility (e.g., fuel/water separator, effluent/wastewater holding pond, smoke abatement system, reburn system, washout system, liners); and (5) determine the ability of the facility to enhance training by accommodating multiple training events.

To assist in this effort, a test plan was prepared to detail the tests to be conducted, the procedures to be used, and the data to be collected during startup and initial operation of the prototype EAFTF at Tyndall AFB. The test plan includes analyses required for preoperational characterization of the possible areas of contamination at the Tyndall AFB facility, initiation of a long-term monitoring program to detect contamination in the area soil or groundwater, and specification of data required to evaluate the wastewater treatment system at the Tyndall EAFTF. This information will be used to (1) evaluate the individual components of the wastewater treatment system (oil/water separator, holding pond, etc.); (2) assess various wastewater treatment scenarios (reuse, recycle, partial recycle, pretreatment); (3) compare results of various treatment scenarios with local/federal environmental regulations; and (4) identify potential wastewater problem areas. The data collected

from the Tyndall AFB prototype will also be used to assess each of the potential EAFTF sites from an environmental standpoint by taking into account differences in the type of wastewater systems planned, the climate, the type of discharge planned, and the environmental regulations for each site. The data from the Tyndall AFB prototype can be compared with similar data that will be taken from the three additional planned prototypes when they are completed.

Closure of Sludge Lagoon at Robins AFB

Technical support is being provided for the closure of a sludge lagoon associated with the Superfund site landfill at Robins AFB in Georgia. A recent feasibility study by CH2M HILL, Inc., identified exhumation and incineration, at a cost of $20 million, as the only permanent solution to close this site. The study identified less costly technologies but could not recommend them because of the lack of development data concerning their direct application to the control of volatile organic compounds, the principal site contaminants of concern.

Technical support is being provided by ORNL to obtain laboratory data required to develop these other technologies to the point that they may be considered as an option to incineration. The technology under study at present involves in situ immobilization. It is estimated that successful implementation of this technology would realize a cost savings of approximately $15 million as compared with incineration.

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3. Radiochemical Engineering Programs

The earliest roots of Chem Tech were formed to provide research and development for radiochemical engineering programs, primarily those associated with nuclear fuel reprocessing. Through these past efforts, well-known separation processes such as Purex, Thorex, and fluoride volatility were developed. Closely related programs involving synthesis (via irradiation), separation, and purification of uranium, plutonium, and the transplutonium elements continue today as a major part of Chem Tech's efforts. National supply and distribution programs for ^{233}U and the transuranium elements were begun in the mid-1960s. Recently, the needs for ^{233}U have diminished, and the processing equipment used for purification and preparation of oxide products is being placed in standby condition; however, the need for the transuranium element processing program remains strong. In addition, various isotopes of transuranium and other elements are recovered and purified for special applications.

Work on developing and applying chemical processes for the decontamination of waste streams from radiochemical operations has been a major effort throughout these programs. This effort has been intensified recently to improve existing processes and to develop new and better methods for meeting environmental regulations and disposal criteria. An example is a recent project to assess the application of the TRUEX process, developed at Argonne National Laboratory, to the processing of waste liquids at Hanford.

Radiochemical engineering activities are also carried out to support nuclear reactor development programs, such as the High Temperature Gas-Cooled Reactor (HTGR) Program. In addition, a new engineering study has begun to determine the feasibility of creating a Developmental Light-Water Reactor, with major improvements in economics and radical improvements in passive, resilient, and inherent safety.

3.1 Plutonium Chemistry and Purex Flowsheet Development

Research and development activities continued to provide a better understanding of the chemistry of plutonium and certain fission products in the Purex solvent extraction process. Improvements for various aspects of this process are being developed.

The detailed results of these studies cannot be presented here because DOE regulations stipulate that such information must be distributed only as applied technology (category UC-526T).

Plutonium Chemistry and Uranium/Plutonium Partitioning

The conventional flowsheet for Purex processing of reactor fuels separates plutonium and uranium by adding a reducing agent to convert the plutonium from the tetravalent state to the trivalent state. This agent causes some problems in the overall process and increases the amount of residual waste. Therefore, a flowsheet that would partition the plutonium and uranium without the need of a reducing agent would be a significant advantage. The acid-split flowsheet, which is based on the differences in the extraction coefficients of plutonium and uranium in the Purex separation system at low temperatures and with low-acid conditions, has been proposed for this purpose.

Evaluation of the acid-split flowsheet requires detailed distribution data for Pu, U, and Np in the Purex system under conditions of low acid concentration and low (<25°C) temperature. The disproportionation of Pu(IV) in low-acid systems affects the total plutonium distribution, and this plutonium chemistry must also be defined. An experimental program is under way to establish the needed data base.

The Pu(IV) disproportionation studies are essentially complete, including the effects of high uranium concentrations over the 5 to 25°C temperature range. Distribution coefficients for Pu(IV) and U(VI), both independently and combined, have been determined for many of the conditions that support an acid-split flowsheet.

Purex Solvent Treatment

Purex solvent is degraded by both radiation and nitric acid. Standard solvent treatment, typically

with a basic solution, removes the major portion of the degradation products. Some degradation products, formed by degradation of the diluent, are not soluble in aqueous solutions and slowly build up in the solvent until they adversely affect the operation of the solvent extraction system. Earlier work has shown that treatment of the solvent with activated alumina, particularly if the solvent is pretreated to remove water, is very effective in returning the degraded solvent to near-pristine condition. Additional studies are currently under way on the development of methods for removing water and ruthenium from the solvent and on improving the efficiency and effectiveness of the activated alumina.

3.2 ^{233}U Program

This program has existed at ORNL for more than two decades and has provided the DOE with (1) a national repository for ^{233}U, (2) uranium purification (for removal of ^{232}U decay daughters) and oxide preparation equipment, and (3) distribution services to various users of ^{233}U. In recent years, receipts and shipments have diminished. Because of this decreased demand and because the 40-year-old equipment and facilities at the Radiochemical Processing Plant (RPP) are obsolete, Chem Tech recommended to DOE's Office of Nuclear Materials Production that (1) the existing equipment be placed in long-term standby condition by FY 1989 and (2) modern equipment be installed in better hot-cell facilities at Building 7930 if needs for ^{233}U are renewed.

Current Operations

Work during the period between January 1987 and June 1988 was performed to (1) convert all ^{233}U to a solid oxide form for storage in the shielded and safeguarded storage facilities at the RPP and (2) clean out the ^{233}U processing equipment to prepare it for long-term standby condition. Also, a significant number of facility upgrading projects were carried out to enable surveillance and maintenance of the facility in the future. The latter work is described in Sect. 2.2 of this report.

The residual liquid solutions contained 36 kg of ^{233}U and a variety of chemical impurities. A special extraction chromatography process was developed and used to purify the uranium. Batches containing ~1 kg of uranium were processed using a large (9-L) column that contained a stationary-phase extractant (di-*sec*-butylphenyl phosphonate) impregnated at 100% (by weight) onto a hydrophobicized diatomaceous earth packing. The process was used successfully even though difficulties were encountered because of a gradual loss of the extractant from the column and subsequent plugging problems in the product evaporator. Following the uranium purification and solidification operations, the processing equipment was cleaned by extensive flushing with dilute nitric acid and water.

Facility Cleanout

A major part of the facility cleanout was the removal of ~300 m^3 of contaminated solid wastes. Extensive monitoring, documenting, and packaging were required for disposal of the transuranic, low-level, hazardous, and mixed wastes. A special project was carried out to solidify (by grouting) and dispose of ~26 kg of uranium-thorium oxide microspheres that had been used in the KEMA (Netherlands) Suspension Test Reactor.

3.3 Transuranium-Element Processing Program

This program is carried out at the Radiochemical Engineering Development Center (REDC), which includes the hot cells and laboratories in Buildings 7920 and 7930. The REDC, located in the Melton Valley area of ORNL, is the processing, storage, and distribution center for materials used in the heavy-element research program in the United States. The isotopes produced here 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, neutron-rich isotopes of the heavier elements. Activities at the REDC are carried out in collaboration with those in other divisions at ORNL and at other national laboratories where research on actinide and transactinide elements is performed. The REDC is the sole source of the ^{252}Cf that is distributed worldwide through the DOE's ^{252}Cf Industrial Sales/Loan Program, which was recently transferred from the Savannah River Laboratory to ORNL. That program involves loans of ^{252}Cf neutron sources to agencies of the U.S. government and sales of ^{252}Cf in the form of bulk oxide and palladium-californium alloy pellets or wires.

Typical Processing Steps

Target rods containing americium and curium are remotely fabricated at the REDC, irradiated in the High Flux Isotope Reactor (HFIR), and then processed at the REDC for the separation, recovery, and purification of the heavy actinide elements, which are distributed to laboratories throughout the country for use in research. The isotopes recovered directly from the mainline chemical processing of irradiated HFIR targets are ^{249}Bk, ^{252}Cf, ^{253}Es, and ^{257}Fm. Other products are separated decay daughters. Each of these is recovered by purifying the radioactive parent, allowing the parent to decay, and then processing to recover the daughter. The separated daughters include ^{248}Cm, ^{249}Cf, and high-isotopic-purity ^{253}Es. 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. All of the ^{252}Cf feed material for the DOE ^{252}Cf Industrial Sales/Loan Program is recovered in the REDC.

Processing of the irradiated HFIR targets is accomplished by a long sequence of individual steps that begins in the heavily shielded, remotely operated hot cells in Building 7920. The processing steps generally tend to separate the transuranium elements as a group from aluminum, metallic impurities, activation products, and fission products before partitioning and purifying the individual elements.

The sequence of processing steps is called a "campaign." All of the target rods for a single campaign (usually a group of 10 to 15) are put into a dissolver vessel and solubilized in two steps. The aluminum is preferentially dissolved in caustic nitrate solution, which is decanted through a filter. Then the residual actinide and fission product oxides are dissolved in 5 to 6 *M* HNO_3. Various metallic impurities are removed by means of the Cleanex batch extraction process, which uses di-(2-ethylhexyl)-phosphoric acid as the extractant. The Cleanex product solution is subsequently treated in two steps to remove rare-earth fission products and separate the curium and americium from the transcurium elements: (1) a rough separation is made, using the Tramex batch extraction process, in which the extractant is a tertiary amine; and (2) more complete decontamination from fission products and separation of americium and curium from the transcurium elements are accomplished by means of the LiCl-based anion-exchange process. The transcurium elements are then separated from each other by means of a pressurized ion-exchange process using chromatographic elution with α-hydroxyisobutyrate.

The curium is converted to curium oxide microspheres for fabrication into HFIR targets. The transcurium products are transferred from the main cell bank to other cells and glove boxes for final purification. Product purification operations for a particular actinide element may include cation-exchange, anion-exchange, solvent extraction, and precipitation techniques in various combinations to provide the purity and chemical form required by the experimenter.

Current Operations

During this report period, 13 HFIR targets were processed in one campaign (No. 67) to recover the transplutonium elements. Table 3.1 presents data on the amounts of key isotopes obtained during this 18-month period, as well as the total production of each key isotope at the REDC in 22 years of operation.

Sixty-two shipments were made during the report period. This total includes all of the ^{252}Cf shipments made under the auspices of the ^{252}Cf Sales/Loan Program. The total quantities of each type of product shipped are listed in Table 3.2.

Flowsheets and process equipment used for transuranium-element processing remained unchanged during this report period.

Purification and Shipment of ^{248}Cm

The recovery of ^{248}Cm is accomplished by purifying the parent ^{252}Cf to remove all curium isotopes, storing the ^{252}Cf ~2 years to allow ingrowth of the ^{248}Cm daughter, and chemical processing to separate the ^{248}Cm from the ^{252}Cf parent. The separation and final purification of ~115 mg of ^{248}Cm were completed during this report period. The curium had been stored in storage/shipping packages since its initial purification from californium in 1984. The processing steps included (1) elution of the actinides from the storage packages, (2) concentration and purification of the actinides by means of cation-exchange techniques, (3) removal of residual ^{252}Cf from ^{248}Cm by selective

Table 3.1. Materials recovered in the Radiochemical Engineering Development Center

	January 1987 through June 1988	Total through June 1988
Number of years	1.5	22
Number of target campaigns	1	48
Number of targets processed	13	565[a]
Amounts of key isotopes recovered		
^{242}Pu	0	141 g
^{243}Am	1.1 g	295 g
^{244}Cm	22(65) g[b]	2324 g
^{249}Bk	42 mg	810 mg
^{252}Cf	429 mg	7691 mg
^{253}Es	1 mg	33 mg
^{257}Fm	~1 pg	19 pg[c]

[a]Includes 368 HFIR targets, 176 SRP slugs and targets, and 21 SRP Pu-Al tubes.
[b]The value in parentheses is the total mass of all curium isotopes.
[c]Estimated amount.

Table 3.2. Shipments made during the period January 1, 1987, through June 30, 1988

Major isotope	Total quantity	Number of shipments
^{22}Na	21 μCi	7
^{240}Pu (99%)	24 g	1
^{243}Am	1.2 g	4
^{248}Cm (97%)	114 mg	5
^{249}Bk	30 mg	6
^{249}Cf	43 mg	7
^{252}Cf	239 mg	22
^{253}Es (mixed)	6 μg	2
^{253}Es (milked)	60 μg	2
^{254}Es	4.5 μg	1
^{255}Fm	7 ng	4
^{257}Fm	1 pg	1

chromatographic elution from cation-exchange resin using α-hydroxyisobutyric acid, and (4) final removal of cationic and anionic impurities. The final product contained 114 mg of ^{248}Cm with <100 ppb of ^{252}Cf. The ^{248}Cm product was divided for shipment as follows: 53 mg to Argonne National Laboratory, 46 mg to the Transuranium Research Laboratory at ORNL, 10 mg to the Solid State Division at ORNL, 5 mg to Westinghouse Idaho Nuclear Company, and 5 μg to the ORNL Isotope Distribution Office for sale to the Federal Health Office of West Germany.

Upgrading of Target Fabrication Equipment

An extensive program has been initiated to upgrade the equipment in the Building 7920 hot cells for remote fabrication of HFIR targets. Primary emphasis has been on the target pellet fabrication equipment in Cell 3, which had become very unreliable. The entire sequence of operational steps required for pellet fabrication was reviewed. For some of the steps, it was determined that the existing equipment design was adequate; but the equipment was worn out. For those items, it was only necessary to fabricate new equipment with few, or no, design changes. Equipment for other fabrication steps had to be redesigned in order to meet functional requirements. A major change involved the use of precision positioning equipment that has become available commercially since the original pellet fabrication equipment was designed. Detailed engineering design has been completed. Commercially available equipment and controls have been ordered, custom-designed equipment is being fabricated, and a mockup of the in-cell equipment support base has been fabricated for out-of-cell testing.

Advanced Neutron Source

The Advanced Neutron Source (ANS) is a proposed multipurpose reactor to be sited at ORNL. The primary purpose for the ANS is to provide substantially enhanced capabilities for neutron scattering research. Since current plans are for the HFIR to be shut down when the ANS begins operation, the ANS must also be capable of producing transuranium elements. Unfortunately, the ANS reactor design cannot be optimized for both purposes simultaneously, and compromises must be made.

The REDC staff has taken a leading role in evaluating transuranium-element production capability for proposed ANS reactor designs. A number of transmutation calculations have been made based on unperturbed, region-specific, two-group fluxes. Preliminary indications were that the unperturbed fluxes were of sufficient magnitude to produce the required quantities of ^{252}Cf and ^{254}Es. However, questions regarding number of targets, target loading, reactivity effects, flux perturbations, and other reactor physics concerns have not been totally resolved. In addition, the ANS core

design has recently been changed, and the impact of the change on the production rate has not been evaluated due to a current lack of basic reactor physics data. Interaction with the ANS staff will continue; and as the reactor design matures, additional calculations will be made in order to evaluate the capability of the ANS to produce transplutonium elements.

3.4 Recovery and Purification of Isotopes for Special Applications

The facilities at the REDC are used for a variety of purposes in addition to those associated with the mainline processing and distribution of transuranium elements and the fabrication and distribution of ^{252}Cf neutron sources. These particular projects may include special irradiations in the HFIR, as well as the nonroutine processing and purification of special isotopes and product forms.

Preparation of ^{24}Na

When the HFIR and the Oak Ridge Research Reactor were shut down, the ORNL Physics Division was without a source of supply for 15-h ^{24}Na, which is used in the calibration of various accelerator experiments. The ^{24}Na is produced by neutron irradiation of aluminum in the $^{27}Al(n,\alpha)^{24}Na$ reaction. A portion (300 to 400 mg) of the ^{252}Cf inventory at the REDC was arranged in a suitable array in the Building 7920 hot cells to provide a large neutron source for irradiating aluminum disks to produce the needed ^{24}Na. A total of seven samples, each containing 1 to 3 μCi of ^{24}Na, was produced to provide a continuing supply for the Physics Division.

Electroplated ^{252}Cf Sources

Two special ^{252}Cf sources, containing 6.2 and 1.7 μg of ^{252}Cf, respectively, were prepared during this report period by electroplating the desired amount of purified californium from dry isopropanol solutions onto platinum disks and then thermally affixing the deposit at ~750°C.

One source (6.2 μg of ^{252}Cf) was prepared for the ORNL Isotopes Research Materials Laboratory for use in the fabrication by self-transfer of "mass-free" fission sources in different geometries as required for the various experiments. The other source (1.7 μg of ^{252}Cf) was assembled into a fission chamber for use at ORNL in the development and demonstration of a technique for measuring the effective multiplication constant of a fissile array that is slightly subcritical. This technique has application in the startup of new reactors and in places where criticality must not be reached, such as fuel fabrication plants, fuel storage basins, and fuel reprocessing plants.

Preparation of ^{255}Fm

Fermium-255 (half-life, 20 h) is the decay daughter of ^{255}Es (half-life, 40 d), which is present in small quantities in the einsteinium product from irradiated HFIR targets. Following separation of the primary fermium product, which contains ^{257}Fm, the einsteinium product (0.88 mg) from REDC Campaign 67 was stored to allow ^{255}Fm to grow in by radioactive decay. The high-purity ^{255}Fm, which resulted following each of four successive decay intervals, was recovered and purified by a series of pressurized ion-exchange operations. Approximately 6.6 ng of ^{255}Fm was recovered from the einsteinium product available during this report period. Two shipments, totaling 4.3 ng of ^{255}Fm, were transferred to the Transuranium Research Laboratory of ORNL's Chemistry Division for inorganic chemistry studies. The remaining 2.3 ng of ^{255}Fm was sent, in two shipments, to the Oak Ridge Associated Universities' Medical Division for studies of radiolabeled monoclonal antibodies as a possible diagnostic and/or therapeutic tool in the treatment of colorectal cancer.

Preparation of ^{240}Pu

A batch of 23.8 g of ^{240}Pu was prepared for final distribution. This plutonium was initially recovered during Campaign 65 from a batch of aged "Curium II" curium that was obtained from the Isotope Distribution Office to augment the feed for HFIR targets. Since the original Curium II material was primarily ^{244}Cm, the plutonium that has grown into it is very high purity ^{240}Pu. The initial separation from the curium was accomplished using the Pubex batch extraction process. The plutonium product was further purified with one cycle of anion exchange and then converted to an oxide form by oxalate precipitation and calcination of the precipitate. Table 3.3 lists the isotopic composition of this product.

Table 3.3. Isotopic composition of plutonium from Campaign 65

Nuclide	Distribution (atom %)
^{238}Pu	0.010
^{239}Pu	0.017
^{240}Pu	99.949
^{241}Pu	0.0015
^{242}Pu	0.022

3.5 Californium Neutron Sources

Californium-252 is an ideal material for making portable neutron source capsules. The half-life of ^{252}Cf is 2.65 years, and the primary decay mode is by alpha emission to ^{248}Cm; however, ~3% of the decay occurs by spontaneous fission whereby 2.3×10^{12} neutrons/s · g^{-1} are released, thus making a very intense neutron source. Compared with conventional (α,n) neutron sources, ^{252}Cf neutron sources have smaller dimensions. As with all isotopic neutron sources, ^{252}Cf sources do not require elaborate power supplies, control systems, or maintenance programs.

Californium-252 neutron sources have many applications, including cancer therapy, reactor startup sources, and nuclear fuel rod scanners. There are also many applications in the fields of neutron radiography and neutron activation analysis, as well as some special applications.

^{252}Cf Industrial Sales/Loans Program

Beginning in 1986, ORNL assumed responsibilities for the DOE ^{252}Cf Industrial Sales/Loans Program, which involves loans of ^{252}Cf neutron sources to agencies of the U.S. government and sales of ^{252}Cf in the form of bulk oxide and palladium-californium alloy pellets or wires. The program had been operated from 1968 through 1986 in temporary facilities at the Savannah River Laboratory (SRL). These activities were then transferred to the REDC Californium Facility in Building 7930. The Californium Facility has been operated since 1970 by the staff at the REDC as part of the DOE Heavy-Element Research Program. Additional equipment and facilities were provided in the Californium Facility to accommodate the Sales/Loan Program activities while continuing the Transuranium Element Processing Program work.

The expanded Californium Facility has the capabilities for purifying and packaging bulk californium, as well as fabricating neutrons sources, in all of the physical forms required by both the research and industrial sales/loans programs.

As of April 1, 1988, ORNL assumed responsibility for administering the contracts for all of the loans of ^{252}Cf neutron sources that had previously been administered by SRL. At that time, 76 contracts covering 179 different items were in force with 73 different institutions. Concurrently, ORNL had 43 loan contracts with 28 institutions covering 83 items.

REDC Californium Facility Operations

Californium Facility restart operations included (1) the "hot" testing of the newly installed equipment, (2) in-cell testing to refine and modify the operating procedures to adapt to the equipment, and (3) recalibration of the californium assay counters and other processing instrumentation.

Storage packages containing ~81 mg of ^{252}Cf were transferred from the Building 7920 hot cells for the ion-exchange purification. The main product fraction, containing 72 mg of ^{252}Cf, was concentrated, and the nitric acid concentration was adjusted to 0.75 *M*. A portion of this californium product was then precipitated as the oxalate, filtered into the primary source capsule, and calcined to convert the oxalate to the oxide (Cf_2O_3), the chemical form used in the SRL-type sources. Following encapsulation of the primary capsule in the secondary capsule components, the completed source, OR-Cf-3016, was assayed to contain 50.01 mg of ^{252}Cf, which was the largest quantity ever loaded in a source of this type. Three additional SRL-type sources, designated OR-Cf-3017, -3018, and -3019 and containing 44, 43, and 48 mg of ^{252}Cf, respectively, were fabricated and shipped along with OR-Cf-3016 to the Naval Ocean Systems Center to be used for studies in "environmental testing" (neutron bombardment) of infrared devices.

A special NBS-type (NBS Mark III) neutron source containing 3.03 mg of ^{252}Cf was fabricated using the resin-loading/calcination procedures developed for ORNL-type sources. The californium in this type of source is in the oxysulfate form and

is confined to a very small volume within a source capsule 0.3 in. (8 mm) long and 0.3 in. (8 mm) in diameter. This source was transferred to the National Bureau of Standards for use in their program of developing calibration techniques for neutron fields and radiation detectors and to serve as an interlaboratory reference source.

Another source form developed at SRL and incorporated at the Californium Facility for the Industrial Sales/Loans Program is palladium-californium oxide cermet/alloy pellets and wires. The preparation of Pd-Cf_2O_3 pellets and wires includes (1) precipitation of ^{252}Cf as the oxalate, (2) addition of palladium tetramine dinitrate, (3) reduction of palladium to the metal with hydrazine hydrate, (4) drying and calcination to produce Pd-Cf_2O_3, (5) pressing the oxide to pellet form, (6) melting the Pd-Cf_2O_3 pellet at 1600°C, and (7) processing the melted pellet through a rolling mill to form wire. Wires are prepared at concentrations of 5, 50, and 500 μg of ^{252}Cf per inch (25 mm). Cutting these wires to specified lengths provides wire segments containing the desired ^{252}Cf content. Wires are then encapsulated into welded capsules for shipment. Five shipments of Pd-Cf_2O_3 wires were made during this report period. These shipments, which were requested to be at 500 μg of ^{252}Cf per inch (25 mm), contained a total of 17,564 μg of ^{252}Cf in a total length of 35.04 in. (89 cm), that is, 501.2 μg/in. or 197.3 μg/cm.

A large part of the californium inventory in the Building 7920 hot cells has been transferred to Building 7930 for storage in the underwater Californium Storage Facility. All items must be welded, leak-free capsules, which have been thoroughly decontaminated before storage in the pool is permitted. Many of the transferred items have required welding into intermediate capsules, helium-leak checking, and decontamination prior to storage. Other items, such as neutron sources, require only decontamination prior to storage. To date, 46 items containing a total of 232.5 mg of ^{252}Cf have been transferred and stored in the Californium Storage Facility.

Allocation of Neutron Sources

Neutron sources are returned to the REDC when the projects for which they were requested are completed or when replacements are ordered following decay of the ^{252}Cf. These sources are available for reassignment until the timing is appropriate for reprocessing to recover the ^{248}Cm daughter. During the current report period, 17 sources were returned and 11 were reassigned; 2 were withdrawn from the loan program; 262 are currently on loan; and 111 (containing from 53 ng to 7.6 mg of ^{252}Cf) are available for reassignment. These numbers were increased not only by the new loans transferred from Savannah River, but also by the receipt at ORNL of 75 sources that had been returned to SRL from prior loans and are now in our inventory.

^{252}Cf Workshop

A ^{252}Cf Workshop was held in Oak Ridge on April 13-14, 1988, to inform the ^{252}Cf user community of recent developments in the production and distribution of ^{252}Cf and ^{252}Cf neutron sources. The workshop provided an opportunity for the current and potential users of ^{252}Cf to meet the producers and distributors. It also allowed users to be informed of changes in administrative requirements and procedures for arranging californium sales and loans from ORNL.

Total attendance at the workshop was 117. The current interest in a wide variety of applications of ^{252}Cf was demonstrated in 27 presentations in 5 technical sessions.

3.6 TRUEX Development for Deployment at Hanford

The TRUEX process for separating actinides from waste streams was developed at Argonne National Laboratory and is being deployed at Hanford in the plutonium reclamation facility (PRF). We have assisted this TRUEX deployment by flowsheet review[1] and assessment and by carrying out a study to determine the best management of the waste americium by-product.[2] After a thorough review, we recommended that the americium be loaded onto a cation-exchange resin and that the loaded resin be dried and calcined to produce a small volume of solid material that could be conveniently stored until proper methods for long-term storage and management could be selected. This concept was then laboratory-tested, using europium as a surrogate for americium; the results indicated that 1 L of resin has the capacity to remove the americium from the process stream to an acceptable level for an operating period of 4 d.[3]

3.7 HTGR Fission Product Behavior

The Chemical Technology Division's activities under the HTGR Program include the behavior of volatile fission products in the helium circuit of a gas-cooled reactor and, in particular, interactions with the metallic parts of the circuit. This work is not reported here this year, in compliance with regulations which stipulate that such information must be distributed solely as applied technology.

3.8 Developmental Light-Water Reactor Program

The Developmental Light Water Reactor (DLWR) program represents a new activity in the Chem Tech Division. Its objectives are to determine the feasibility of creating an LWR with major improvements in economics and radical improvements in safety, including meeting PRIME (*P*assive, *R*esilient, *I*nherent, *M*alevolent resistant, *E*xtended time) safety goals. The program was initiated within Chem Tech following a series of inventions by its staff.

A developmental reactor, by definition, refers to a reactor whose characteristics are advanced enough that a test or demonstration reactor may be necessary to prove feasibility. This implies initial application in the 2000 to 2020 time frame. The term PRIME refers to a set of safety goals:

1. **Passive Safety**—The DLWR should be designed to have passive safety systems with no moving parts (motors, pumps, valves). This eliminates the potential for mechanical failure and operator error.
2. **Resilient Safety**—The DLWR safety and operating systems must be resilient (i.e., activate when required, but not during normal plant transients). Safety systems should not interfere with normal operations or create other incentives to be bypassed or disabled by operating and maintenance staffs.
3. **Inherent Safety**—The DLWR safety systems should have inherent safety whenever possible; that is, materials and structural configurations should be selected to eliminate classes of accidents (such as those caused by chemical reactions) and their associated safety systems.
4. **Malevolence Resistance** The DLWR should be capable of passively withstanding malevolent acts of man (conventional off-the-shelf munitions and short-term power plant takeover by terrorists) without significant release of radionuclides to the environment. Plant security for public health and safety should depend primarily on passive rather than active (guards, security checks, etc.) techniques. In practice, this design objective also covers all operator errors and inaction.
5. **Extended Safety**—DLWR safety should be ensured under the above conditions for extended time periods (>1 week) after an accident without human intervention.

The DLWR is not presently a single reactor concept but, rather, is defined by a set of goals.

Program Strategy

Any DLWR will be composed of systems, structures, and components (SSCs) that meet PRIME safety goals and are the building blocks for a power reactor. Various building blocks (SSCs) are designed to meet the functional requirements for plant operation and/or to prevent, stop, or mitigate a reactor accident. To achieve these goals, the program has three components:

1. identify, invent, and develop SSCs that meet PRIME safety goals;
2. integrate SSCs into several mainline DLWR concepts; and
3. evaluate SSCs and reactor concepts to define preferred options.

Example DLWR

An example of a single DLWR concept (Fig. 3.1) can provide: (1) an understanding of SSCs, (2) an example to clarify directions of research, and (3) a definition of how this activity relates to other programs.

The PIUS/BWR,[4-6] which was invented by members of the Chem Tech staff, has a conventional nuclear reactor core and steam cycle (750 MWe) but a radically different set of safety systems. Three of these systems are described below: (1) the Prestressed Concrete Reactor Vessel (PCRV), (2) the Fluidic In-Vessel Emergency Core Cooling System (FIVES), and (3) the Core Melt Source Reduction System (COMSORS).

In the PIUS/BWR, all reactor safety systems are located within a large PCRV which protects

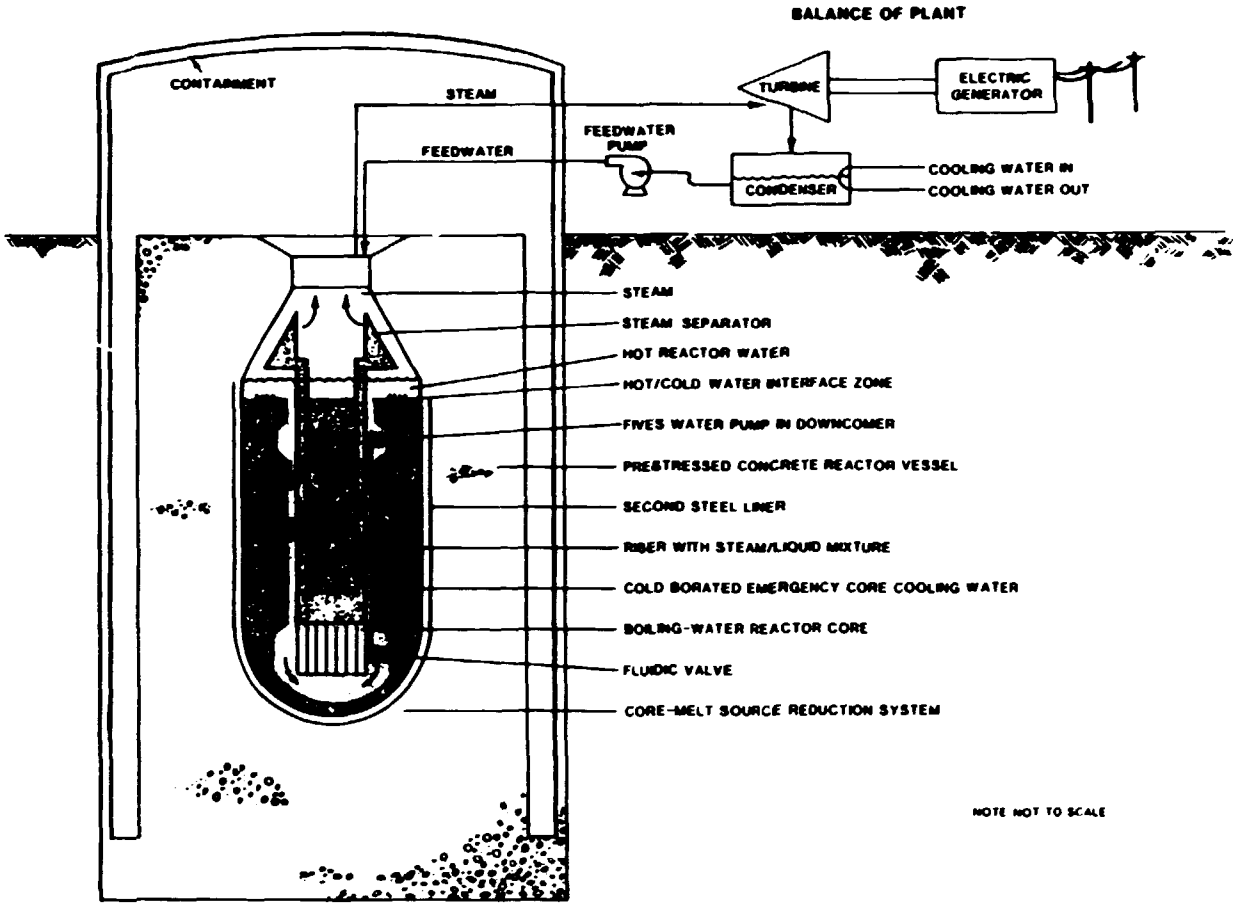

Fig. 3.1. Schematic of a Process Inherent Ultimate Safety boiling-water reactor (PIUS/BWR).

the reactor from violent internal and external threats. This particular PCRV design is based on work in Sweden for another type of DLWR.[7] The interior of the reactor vessel has a shape similar to that of a soft drink bottle with a diameter of 13.5 m and a height of 35 m. Vessel walls are 7 m thick. The PCRV is held together with both prestressed steel cable and steel rebar, but it is designed so the loss of either cables or rebar will not result in vessel failure. There are multiple design features to ensure leak-tight integrity from the vessel. All pressure vessel penetrations are at the top of the vessel, and the vessel has multiple interior liners. From inside to outside the PCRV, the vessel wall includes (1) a steel liner, (2) 1 m of concrete, (3) a second steel liner, and (4) 6 m of concrete. Under severe accident conditions, the vessel is designed to depressurize (but not to leak water) by seal failure at the top of the PCRV.

The primary safety concern in an LWR is to prevent a reactor core meltdown. This can be done by maintaining the reactor core underwater at all times. The PIUS/BWR uses an emergency core cooling system, called FIVES, which is, in part, based on earlier work on nuclear fuel reprocessing plants.

The inside of the PCRV is divided into two zones. The first contains the BWR primary system (clean primary coolant water, reactor core, downcomer, and steam separator). The second contains FIVES, which includes a fluidic valve, a hot/cold water interface zone, a small water pump, and sufficient borated water to shut down the reactor and cool it for 1 week by water boiloff.

If the reactor core is short of water, the borated water enters the reactor core, shuts down the reactor, and removes heat by boiloff of water. The borated water during normal operations is in con-

tact with the reactor primary coolant at two locations. Near the top of the PCRV, the two water zones are in direct contact with each other through a hot/cold water interface; the cold, high-density, borated water is below the low-density, clean, hot water. Appropriate design reduces the diffusion of boron across this water interface zone. Near the bottom of the PCRV, a fluidic value (a valve with no moving parts) separates cold borated water from hot reactor water. The valve remains closed only if it receives high-pressure water from a water pump high in the downcomer. If the reactor water is low or a power failure occurs, this pump fails; the result, then, is that no water is sent to the fluidic valve, the valve opens, and the reactor core is shut down and flooded with FIVES cool, borated water.

The central component of FIVES is the vortex fluidic valve assembly (Fig. 3.2), which is a modified vortex fluidic amplifier operated as a valve. This arrangement is similar to a conventional centrifugal pump with a blocked exit line. The incom-

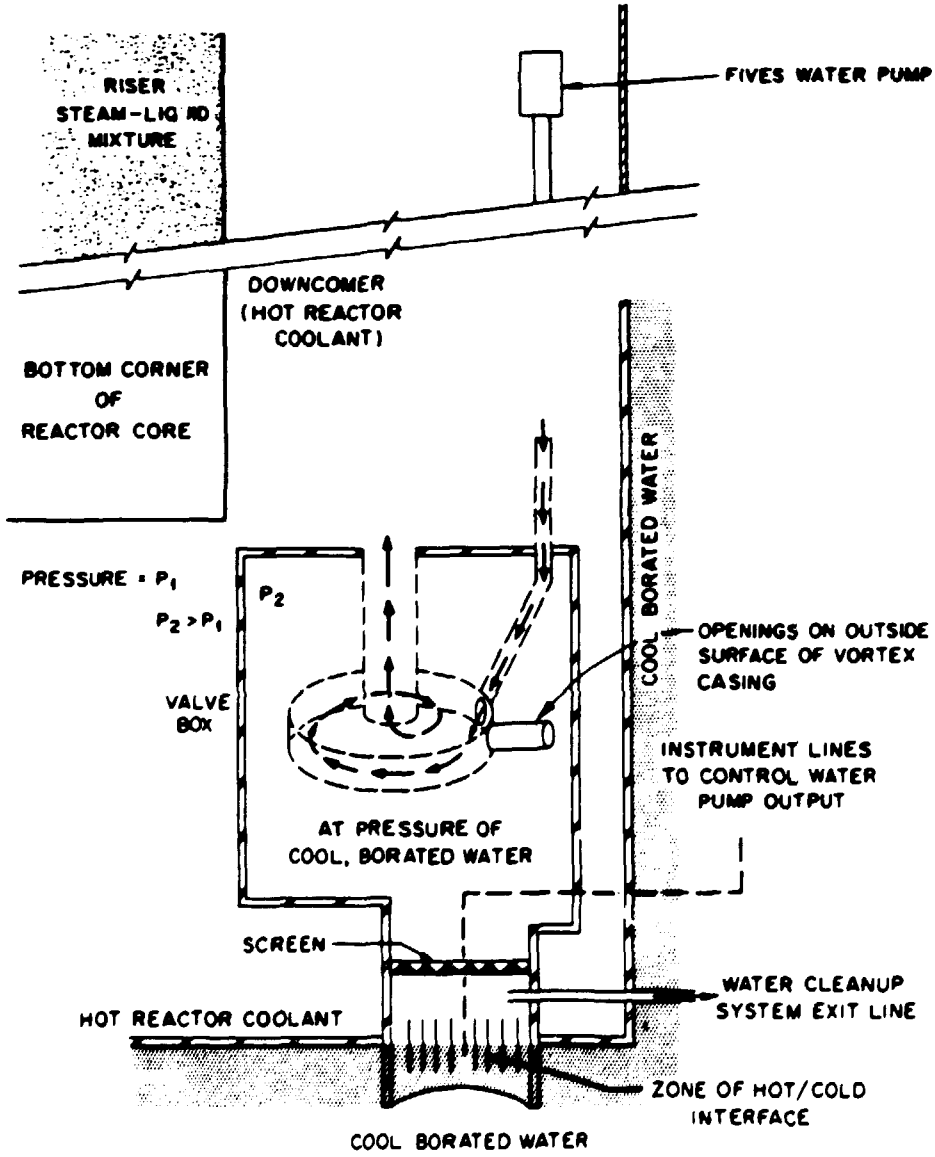

Fig. 3.2. A vortex fluidic valve assembly.

ing FIVES water is injected tangentially at high velocities into the vortex casing, causing the water to move in a circle. The centrifugal forces create higher pressures near the outside surface of the vortex valve casing and lower pressures near the inside. The outside surface has holes (short lengths of tubing) that connect it to a zone of clean, higher-pressure reactor water, which, in turn, is in contact with the borated water zone at the hot/cold water interface zone. The center of the vortex casing is connected to the downcomer and exhausts FIVES water to it. By adjusting FIVES water-pump output, pressures across the vortex valve can be made to match those of the two water zones. In effect, a valve exists that uses the dynamic forces of water rather than pieces of metal to prevent flow through the valve.

The hot/cold water interface that separates borated water from reactor coolant is located below the fluidic valve. Water temperature and boron concentration sensors determine the location of the interface zone. The sensors are used to control the speed of the FIVES water pump system during normal operation and, hence, the interface position.

A third proposed feature (SSC) of the PIUS/BWR is the Core Melt Source Reduction System, which is designed to limit the maximum potential release of radioactivity to the environment after an accident by control of core meltdown chemistry. It provides a form of inherent safety by limiting maximum accident size. The system consists of carefully selected concrete aggregate under the reactor core. It is based on two observations:

1. Experiments and theoretical calculations in the Chem Tech Division and at Sandia National Laboratory (NRC program) indicate that the quantity of radioactive vapor and aerosols released after a reactor accident depends on the chemical reactions between molten core and concrete. For example, if the concrete contains limestone aggregate, the molten core/concrete interactions generate carbon dioxide, which bubbles through the molten mixture, creating aerosols and stripping volatile fission products from the melt to the atmosphere.
2. Research on solidification of radioactive wastes from nuclear fuel reprocessing plants indicates that control of waste chemistry can encourage fission products to remain in the melt and minimize aerosols in the off-gas systems.

These diverse lines of research suggest that proper selection of aggregate (chemical composition) for concrete under the reactor core can significantly reduce the release of radionuclides to the containment after an accident. This reduces the challenge to containment, hence decreasing the probability of containment failure. If containment failure occurs, it reduces radionuclide releases to the environment. It may also reduce containment costs if maximum accident pressures and temperatures in containment after an accident are decreased.

Activities During FY 1988

The specific activities during this report period follow:

1. Completed a long-range program plan with specific activities defined through 1993.
2. Defined DLWR goals. This activity included (a) evaluation and understanding of safety philosophies of the AIChE Institute for Process Safety[8] and of the Institution of Chemical Engineering;[9] (b) assistance in organizing and presenting papers[10] and working with the International Atomic Energy Agency (IAEA) Technical Committee Meeting on Definitions and Understandings of Engineered Safety, Passive Safety, and Related Terms (Västerås, Sweden); and (c) analysis of foreign reactor safety goals.
3. Prepared three notices of invention and disclosure for new SSCs that met PRIME safety goals.
4. Initiated preparation of a document to identify existing and proposed SSCs for DLWRs that would meet PRIME safety goals.

Observations

The DLWR program is a new exploratory research effort with aggressive long-term goals. Program success will depend on cooperation with many divisions within ORNL, various national laboratories, universities, and private industry. The radical technical approaches being investigated for safety systems do not fit within traditional disciplines or divisions.

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4. Basic Science and Technology

The Chemical Technology Division's research and development efforts cover a rather broad spectrum. They include both fundamental and applied studies in the area of biotechnology, focusing on such areas as microbial solubilization of coal, the biological conversion of light into energy, and the kinetics of enzyme processes. Fundamental investigations into the chemistry of the actinide elements and their fission products build upon the historical expertise of this division in separations research in the nuclear field. Chemical separations is also the focus of a fossil energy program directed toward the upgrading of condensible products from coal gasification. Engineering research generally serves as a bridge between fundamental studies and their application to meaningful processes. Improvement in solvent-extraction-induced mass transfer is the goal of studies on the effects of external electrical fields on multiphase systems. Other focuses include the development of a fundamental understanding of phenomena in the critical region to foster a predictive capability to aid chemical separations and continuous annular chromatography. Finally, materials research efforts in the division are concerned with basic thermodynamic studies of energy-related materials, including one of the new superconducting ceramics, particle growth and nucleation, and measurement of transport phenomena of fluids at high temperatures and pressures.

4.1 Biotechnology Research

Research and development in biotechnology is carried out in four major areas: (1) biological interactions with coal in which microbiological and enzymatic methods to solubilize coal are studied; (2) biophotochemical research involving studies on electron transport mechanisms in living and nonliving systems for the conversion of solar energy into chemical energy; (3) enzyme kinetics, in which the interaction of enzymes with solid surfaces is examined, illustrated by the cellulase/cellulose systems; and (4) bioengineering research that provides the bridge between the more fundamental work and processing systems.

Biological Interactions with Coal

The use of biocatalysts in the treatment of lower-ranked coals has been the focus of considerable scientific attention in recent years. The ability of some microorganisms to transform and/or to degrade coal was first described in the mid-1970s. The work reported here is aimed at exploiting the potential of these biological agents, or of catalysts derived from them, in the design of new technologies for coal processing.

Microbial Coal Solubilization

Certain microorganisms have been shown to modify lignites and subbituminous coals, transforming them into a water-miscible liquid product. The biocatalytic agent involved in this activity has not been identified. The reaction chemistry of coal biosolubilization and possible product utility are unknown.

A number of organisms have been screened for their ability to solubilize coal. Two strains, including a filamentous fungus isolated from coal in this laboratory, were identified as superior performers. They were therefore chosen as model organisms for both basic studies and process design.

Developmental work for the microbial system resulted in the design of a superior system in which to study cell growth and coal solubilization (Fig. 4.1). The technology developed here results in good solubilization rates with minimal product contamination. Product recovery has been facilitated. Successful use of this bench-scale technology suggests excellent potential for the design of a viable process and its eventual scaleup.

Substantial progress has been made in the accompanying fundamental scientific investigations into the mechanism of coal solubilization by microorganisms. An improved assay for coal solubilization, based on spectroscopy rather than gravimetry, was designed and validated. Use of this new methodology facilitated the detection and characterization of certain coal degradation products. These products are similar to those produced by the nonbiological action of alkali on coal. Their presence in the biological system suggests that the organisms use alkaline catalysis as well. Other studies suggest that the biological alkaline catalyst is produced in specific response to the presence of

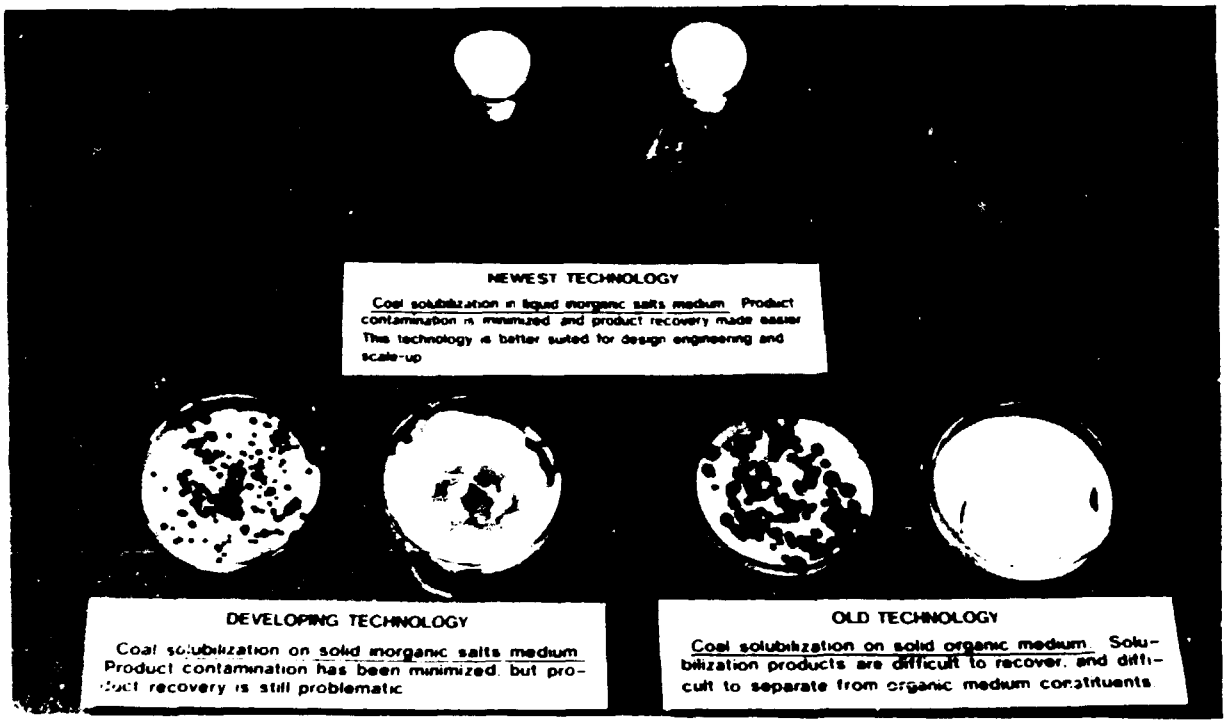


Fig. 4.1. An improved microbial system designed to study cell growth and coal solubilization.

coal. Further investigation revealed that production of coal-solubilizing activity is under endogenous control (i.e., the microorganisms possess specific metabolic controls which govern expression of the biological activity). The activity is stimulated in the presence of coal degradation products (e.g., aromatic acids). This intricate feedback control mechanism for microbial coal solubilization will be investigated further.

The liquid coal product generated by microbial action has been tested as a feed stream for biological methane production. A mixed culture of anaerobic bacteria has been developed that generates methane in medium containing the coal liquid as sole carbon source. Current efforts focus on improving the yield and rate of methane production. Interesting and potentially useful compounds were detected as intermediates in the biogasification process. These compounds include acetate and possibly other liquid oxychemicals. The potential for production of fuel alcohols methanol and ethanol from the liquid coal product is also being explored

Enzymatic Coal Solubilization

Organic biological catalysts (i.e., enzymes) have been shown elsewhere to function in the modification and/or degradation of polymeric substrates structurally related to coal. These reactions may take place in aqueous or nonaqueous (organic) environments. The potential for enzymatic processing of coal in organic media is particularly attractive. This technology is predicted to yield reduced products similar to those generated during conventional thermochemical processing. The use of enzymes would guarantee control over the reactions catalyzed and thus product specificity. The potential for use of enzyme technology in the treatment of coal is now being explored.

Preliminary tests at ORNL have indicated that coal could be treated with isolated enzymes in vitro to yield some degree of solubilization. These enzymes included those with nonspecific oxidizing activity (e.g., peroxidase and alcohol dehydrogenase) that are active against other complex organic materials. Some activity was seen in

organic media; however, rates were low. Current efforts focus on achieving better coal solubilization rates in these systems. The major obstacle has been the poor solubility of enzymes in organic solvents and their resultant low activity. Developmental work has therefore focused on the problem of enzyme solubility and behavior.

Extensive studies on enzyme derivatization have been carried out with some success. Acylation of complexation of enzyme with nonpolar reagents has improved enzyme solubility while retaining catalytic activity. Initial tests showed that enzyme thus modified is active against coal: alcohol dehydrogenase from yeast, complexed to a triazine dye and dissolved in benzene, catalyzed a 5% weight loss from untreated subbituminous coal in 48 h under anaerobic conditions. Future studies will concentrate on improvement of rate and yield as well as on product characterization.

Biophotochemistry

The conversion of light energy into chemical energy by the photosynthetic apparatus occurs in and across membranes that contain specialized reaction centers where the primary photo-induced electron transfer reactions take place. The prevailing model of the molecular structure of photosynthetic membranes indicates that the photochemistry associated with photosynthesis is of a vectorial nature. Oriented reaction centers are embedded in the photosynthetic membranes that are composed of grana stacks and stroma lamellae, structures that are topologically equivalent to spheres. Therefore, irrespective of the rotational orientation of the thylakoid membranes, photosynthetic electrons emerge from the membrane and enter the stroma region of the chloroplast. The ability to observe sustained photocurrents and photovoltages from chloroplast-based systems depends on the capability of the reducing end of photosynthesis to electrically communicate with an electrode.

The key result of this work is the observation of oriented photocurrents from platinized chloroplast membranes that have been entrapped on fiberglass filter paper. What distinguishes these experiments from previous research is the strict symmetric electrode structure that is used for the construction of the photobioelectrochemical cell and the control experiments, which exclude the effect of light gradients in the photosynthetic tissue.

These results provide a simple demonstration of the inherent vectorial properties of intact photosynthetic thylakoid membranes and show that the directionality of the flow of photocurrent can only be explained by the orientation of the photosynthetic reaction centers within the membranes. For this purpose, platinized chloroplasts were entrapped on fiberglass filter paper and contacted with a platinum gauze electrode. Simultaneous experiments were performed in which counterelectrodes of either platinum or silver/silver chloride were used.

First, as indicated in Fig. 4.2, the direction of the flow of photocurrent from the platinized chloroplast assembly is the same for both the platinum/platinum and platinum/silver electrodes. Second, the orientation of the photocurrent is independent of the direction of incident light. Control experiments were performed in which the

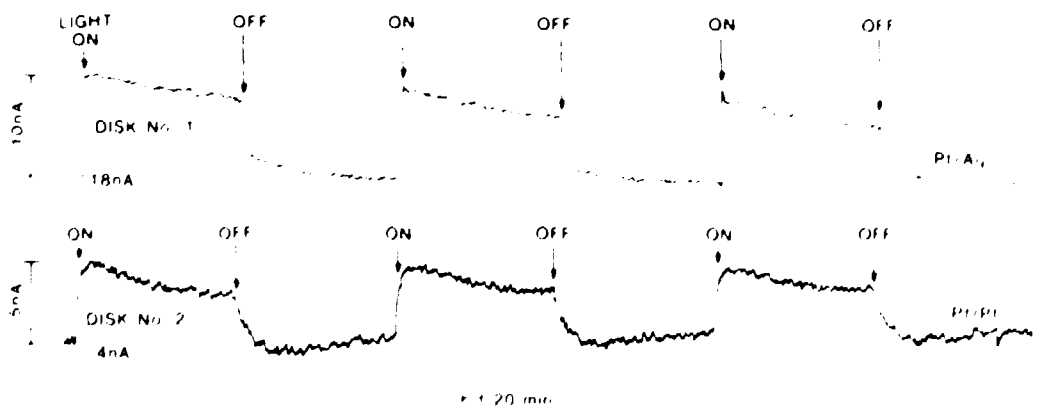


Fig. 4.2. Simultaneous photocurrents as a function of counterelectrode.

sandwich-like cell assembly was irradiated from either direction. Of course, when the cell was irradiated from the "back" side, the light was somewhat attenuated. However, the electrolyte-impregnated filter paper is by no means opaque, and photocurrents were easily seen in this mode of irradiation.

The key result of this control experiment was that the direction of the flow of photocurrent was the same, irrespective of the direction of incident light. This observation eliminated Dember-like effects that are associated with light gradients as being the origin of the orientation of the photocurrents. Third, the orientation of the measured photocurrent is consistent with the prevailing vectorial model of photosynthesis. In this model, the orientation of the photosynthetic reaction centers in the chloroplast membranes is such that electrons move from the inner (lumenal) surface to the outer (stromal) surface under the influence of light. Therefore, the electrical potential of the external surface of chloroplast membranes swings negative, with respect to the internal surface, upon illumination. The polarity of the flow of photocurrent is such that the platinum gauze electrode in contact with the platinized chloroplasts is negative with respect to the counterelectrode. From the preparation method of the platinized chloroplasts, it is clear that the gauze electrode which makes pressure contact with the platinum colloid particles also makes contact with at least some of the reducing ends of Photosystem I.

In conclusion, a composite metal-photosynthetic membrane material has been developed and entrapped on electrolyte-impregnated fiberglass filter paper. Oriented photocurrents, which are consistent with the prevailing vectorial model of photosynthesis, have been observed and provide a simple macroscopic demonstration of the validity of this molecular model.

Research directed at several aspects of the biological water-splitting process, using *Chlamydomonas* hydrogen-producing algae, has provided several significant advances, including (1) construction of an apparatus which allowed automated screening of individual microalgal colonies for sustained ability to photoevolve hydrogen during anaerobic photosynthesis; (2) measurement of photosynthetically generated oxygen from single algal colonies in a helium atmosphere, using an enhanced Hersch galvanic cell; and (3) isolation of a new strain of algae which had greatly increased tolerance to anaerobiosis, a prerequisite condition for hydrogen photoevolution due to the oxygen lability of hydrogenase.

A wild-type strain of *Chlamydomonas moewusii* was subjected to repeated periods of anaerobiosis, CO_2 starvation, and irradiation. Following 144-h periods of anaerobic stress, surviving cells were recovered. The frequency of viable cells that were recovered following the first stress period indicated the isolation of a true genetic mutant rather than the selection of a subpopulation of anaerotolerant cells. Upon exposing the new strain to further cycles of anaerobic stress selections, complete cell viability was observed by the third stress cycle. The anaerotolerant phenotype was also stable in the absence of selection pressure. Table 4.1 summarizes the strain development process. When the H_2- and O_2-evolving abilities of the new strain were compared to the parent, the anaerotolerant strain displayed greater stability in H_2 evolution without significant enhancement of yield. These results suggest that appropriate application of physiological stress is an effective means of isolating a genetic mutant with a specific phenotype.

Enzyme Kinetics

This research has focused on the measurement of the dissociation constant (K_d) and the maximum amount of enzyme (N) that can be bound onto a given quantity of microcrystalline cellulose (Avicel) for the purified cellulase components, cellobiohydrolase (CBH) I and II and endoglucanase (EG) I and II. Based on these measurements, hydrolysis of cellulose was carried out using a combination of components at saturating or nonsaturating concentrations. A key finding was that synergism between components was greater when nonsaturating concentrations of components were used to hydrolyze cellulose.

The binding of saturating (N) or nonsaturating (<N) amounts of a given purified cellulase protein to Avicel was achieved by incubating the latter with a given concentration of protein, which is determined from the saturation binding data. Synergism between EG I and CBH I as well as between CBH I and CBH II was determined by measuring the amount of soluble reducing sugar or glucose produced by the action of the components alone or in combination with each other on a fixed concentration of substrate. If the amount of reducing sugar or glucose produced by the combination

Table 4.1. Long-term stress selection of anaerotolerant *Chlamydomonas reinhardtii*

Period	Condition	CFU/mL[a]		pH		Chl(μg/mL)	
		Start	End	Start	End	Start	End
Cycle 1	Stressed	8.00E+6	<10	6.17	6	50	58.2
Day 0	Unstressed	8.00E+6	<10	6.17	6.13	50	37.2
Cycle 2	Stressed	6.00E+5	4.10E+2	5.49	6.02	50	27.2
Day 42	Unstressed	6.60E+5	<10	5.75	6.02	50	34.9
Cycle 3	Stressed	2.50E+5	4.80E+5	6.06	6.02	50	41.4
Day 72	Unstressed	8.30E+5	—	6.34	—	50	—
Cycle 4	Stressed	9.60E+5	2.30E+6	6.3	6.32	50	62.5
Day 86	Unstressed	2.30E+6	<10	6.2	6.28	50	37.3
Retest[b]	Stressed	6.50E+5	6.35E+5	—	—	50	47.7
Day 146	Unstressed	5.40E+6	<10	—	—	50	36.4

[a]CFU, colony-forming unit. Cell viability after stress selection was measured by determining the CFUs/mL of recovered cultures. 10 CFU/mL was the limit of detection.

[b]Retest refers to the reexamination of the stress-selected strain after three cycles of growth under aerobic, photoautotrophic conditions.

of components (measured curve, Fig. 4.3) is greater than that produced by the components acting alone (theoretical curve, Fig. 4.3), then synergism exists between them.

Synergism between EG I and CBH I was observed at both saturating and nonsaturating concentrations (~50% saturations of Avicel) over a 24-h period. However, the greater synergistic effect was observed with nonsaturating concentrations of EG I and CBH I (Fig. 4.3). A similar result was obtained with CBH I and CBH II (data not shown). The relationship between the level of saturation of Avicel with cellulase components and the synergistic effect was seen emphatically when Avicel was hydrolyzed by a mixture of EG I with CBH I and CBH II (Table 4.2).

Other studies investigated the effect of different concentrations of EG II, CBH I, and CBH II acting in combination on Avicel on the degree of synergism. At the highest concentration of cellulase used, 360 μg/mL (160 μg/mL EG II, 100 μg/mL CBH I, and 100 μg/mL CBH II), little synergism was observed (Fig. 4.4). However, as the concentration of each component decreased, the degree of synergism increased, reaching a maximum at a cellulase concentration of 20 μg/mL (5 μg/mL EG II, 10 μg/mL CBH I, and 5 μg/mL CBH II). At lower concentrations than these, the degree of synergism declined, suggesting that an optimum concentration of cellulase components exists for a maximum synergistic effect on Avicel. At concentrations of enzyme components above those that

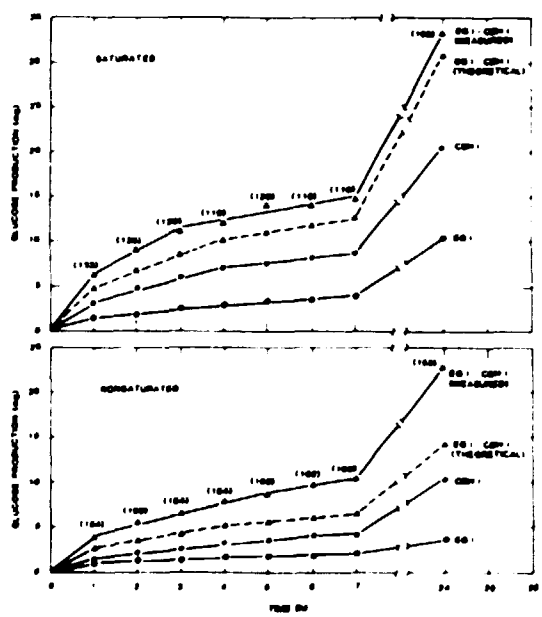


Fig. 4.3. Hydrolysis of Avicel by *T. reesei* EG I and CBH I in combination at saturating and nonsaturating enzyme concentrations.

result in maximum synergism, the lower degree of synergism could be explained by overcrowding of the substrate binding sites. This would then hinder the migration of components from one site to another, which is necessary for their cooperative action. Conversely, as the concentration of components becomes limiting, cooperation between them is reduced.

Table 4.2. Hydrolysis of Avicel by a mixture of EG I, CBH I, and CBH II by saturating and nonsaturating concentrations of each enzyme

Time of reaction (h)	Enzyme concentration					
	Nonsaturating			Saturating		
	Theor. (A)	Meas. (B)	B/A (%)	Theor. (A)	Meas. (B)	B/A (%)
1	3.6	5.6	155	7.4	7.6	103
2	5.0	10.3	206	10.6	12.6	119
3	6.0	10.9	182	13.5	16.3	121
4	7.3	13.9	190	16.0	18.4	115
5	7.6	15.4	203	17.5	20.6	118
6	8.1	17.5	216	19.3	23.5	122
7	8.8	19.3	219	20.3	24.4	120
24	18.4	31.8	173	41.2	43.4	105

[a]Avicel (10 mg/mL) was incubated with EG I (20 μg/mL), CBH I (20 μg/mL), and CBH II (10 μg/mL) under nonsaturating conditions and with EG I (100 μg/mL), CBH I (80 μg/mL), and CBH II (67 μg/mL) under saturating conditions. For other details see experimental protocol.

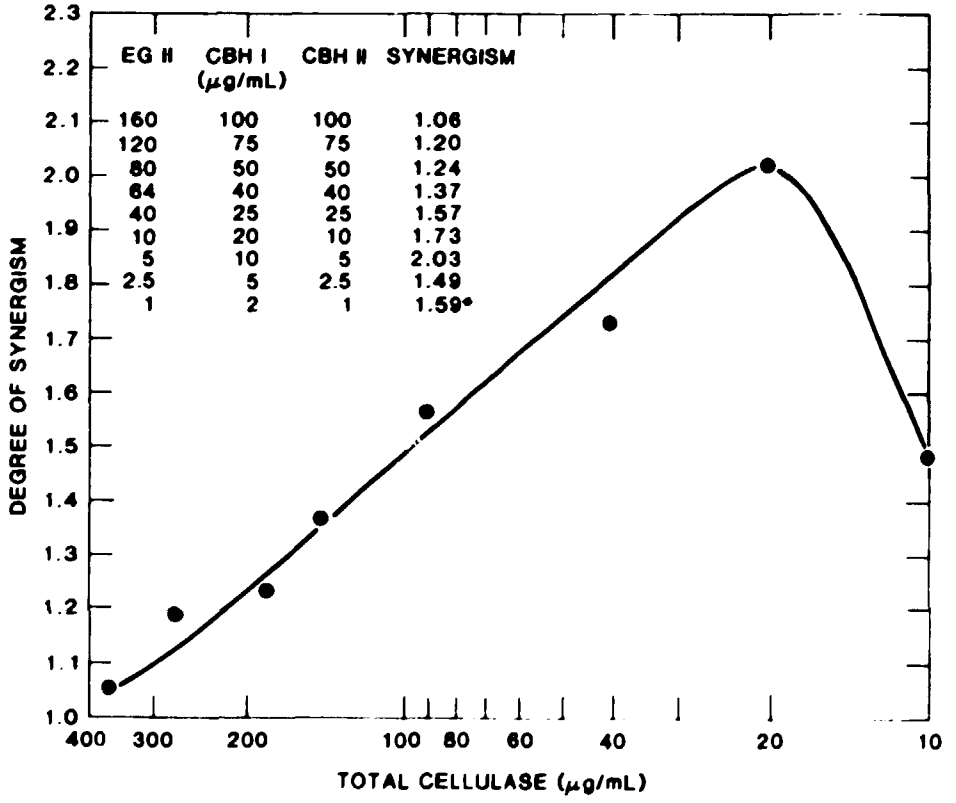


Fig. 4.4. Hydrolysis of Avicel by different concentrations of *T. reesei* EG II, CBH I, and CBH II acting in combination. *Data not shown on graph

The reason why synergism is minimal when the substrate is saturated with each enzyme component is not understood. Perhaps under nonsaturating conditions, cellulase could hydrolyze one glycosidic bond after another because of the ease with which the enzyme could migrate from one productive binding site to another without hindrance. In a process for the enzymatic hydrolysis of cellulose in which an initial saturating concentration of cellulase is used, the synergistic effect is minimal. When EG I or EG II was mixed with CBH I and CBH II, the glucose yield obtained with nonsaturating (50% saturating) concentrations of these components was ~70 to 80% of that obtained with saturating concentrations (Table 4.3). Yet only 20% of the total amount of protein needed in the latter case was required. These data could, therefore, have a real impact on the economics of cellulase utilization. In the future, when "inexpensive" cellulase components (e.g., CBH I, CBH II, EG I, or EG II) are produced through genetic/protein engineering techniques, they may be more efficient if they are used under nonsaturating conditions, where the phenomenon of synergism can be utilized effectively in bioconversion processes.

Bioengineering Research

These projects have continued work in the study of immobilized biocatalysts and of columnar bioreactors.

Biocatalysts

Production and Characterization of Gel-Entrapped Biocatalysts

Natural hydrocolloidal gels can be used to entrap biocatalysts such as microorganisms into beads. The forced flow of these gels through a small nozzle under an imposed vibration can be used to produce small uniform monodispersed beads of <1 mm in diam.[1] These biocatalyst beads can be successfully used in fixed and fluidized columnar reactors as described below. Since mass transfer of substrates and products within the gel matrix can affect the overall kinetics of the biological process, these effects must be measured and correlated to allow prediction and scaleup.

A transient equilibration technique was used to measure solute diffusion coefficients within the gel beads of different compositions. The beads were placed in a well-mixed, isothermal vessel in which the decrease (diffusion into the beads) or increase (diffusion out of the beads) of various solutes in the bulk liquid was measured as a function of time (Fig. 4.5). A computer program was written to determine the diffusion coefficients from these curves. The diffusion coefficients of low-molecular-weight solutes, such as glucose, in beads of carrageenan without any additives approach those in pure water. (Table 4.4) The effect on the diffusion coefficient of the concentration of the gelling material was negligible for 1 to 4%

Table 4.3. Hydrolysis of Avicel by a mixture of EG II, CBH I, and CBH II by saturating and nonsaturating concentrations of each enzyme

Time of reaction (h)	Enzyme concentration					
	Nonsaturating			Saturating		
	Theor. (A)	Meas. (B)	B/A (%)	Theor. (A)	Meas. (B)	B/A (%)
1	3.5	5.5	157	7.5	6.9	92
2	4.9	9.0	184	11.0	10.7	97
3	6.0	9.7	162	13.6	14.8	109
4	7.3	11.4	156	16.3	16.2	99
5	7.6	13.3	175	17.7	19.3	109
6	8.1	16.0	197	19.7	21.2	108
7	8.7	17.4	200	20.8	22.9	110

[a]For details see Table 4.2 Note that EG I (10 μg/mL) was used instead of EG I

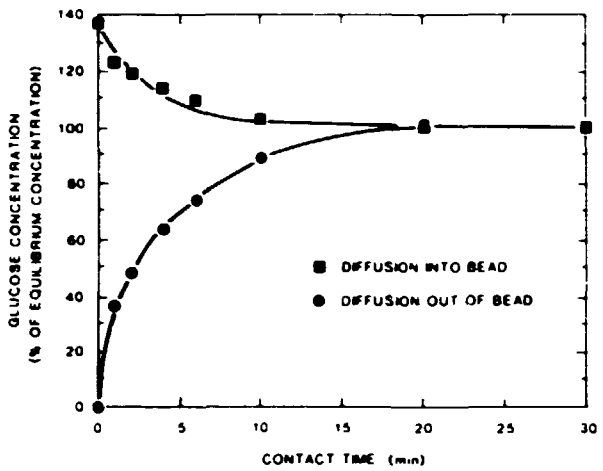


Fig. 4.5. Diffusion of various solutes into and out of biocatalyst beads as a function of time.

Table 4.4. Effect of gel material on glucose diffusion in nominal 2-mm-diam gel beads at 30°C

Gel material	Average diffusion coefficient x 10^6 (cm^2/s)[a]
Water	6.8
1% k-Carrageenan	6.7
2% k-Carrageenan	6.6
4% k-Carrageenan	6.7
1% Alginate	6.6
2% Alginate	5.5
3% Alginate	5.0

[a]Each value is the average of the diffusion coefficients of glucose into and out of the same gel beads with a standard deviation of 0.3 × 10^{-6} cm^2/s.

k-carrageenan but was significant for 1 to 3% alginate, another common gel. The bead size between 1 to 4 mm in diam was found to have no effect on the diffusion coefficient. High concentrations of glucose (>100 g/L) can lower the measured diffusion coefficient slightly, apparently due to hindered diffusion.

Additives to the gel matrix also affect the diffusion coefficient. Iron oxide powder has been added as an inert material to control the bead density. There was a measurable decrease in the glucose D value with the increase in Fe_2O_3. More important is the effect of entrapped microorganisms, as high levels of biocatalyst are expected in operating systems. Tests were performed with 20 to 60 vol % (or 1 to 8% dry weight) of inactive cells in carrageenan gel. A linear relationship was found between the diffusion coefficient and the cell loading, with the diffusivity at the highest concentration being about half of that in the gel alone.

Metal Uptake by Immobilized Microorganisms

Wastewaters from industrial and laboratory operations can contain toxic or undesirable metal ions, which must be removed before discharge. Selected microorganisms have been shown to be potentially useful adsorbents for this purpose because they can be inexpensive, have high selectivities, and exhibit high capacities for adsorption of heavy metals. Nuclear wastewaters contain low concentrations of radionuclides such as strontium and cesium.

While previous work demonstrated the adsorption of uranium by cells, these studies explored the removal of strontium. Twenty microorganisms were tested for their ability to concentrate strontium in batch equilibration experiments. A partial list of these microorganisms is presented in Table 4.5. From these results, *Micrococcus* was chosen for further studies in immobilized gel beads. The gel is also able to slowly adsorb some strontium and enhance the removal process. The cells adsorb strontium rapidly (within minutes) and then slowly release the ion. This release is significantly decreased when the cells are immobilized. This may be explained by strontium adsorption to a

Table 4.5. Rank of strontium uptake by 14-d distribution coefficient values

Organism	Distibution coefficient 14 d	2 d	7 d	pH
1. *Rhizopus*	26,240	1,470	85,533	7
2. *Micrococcus*	9,983	24,943	12,010	7
3. *Anabena*	8,198	5,554	10,304	7
4. *Streptococcus*	5,488	297	2,173	7
5. *Bacillus*	5,240	62,996	5,085	4
6. *Chlamydomonas*	3,750	1,639	1,785	7
7. *Coelastrum*	2,993	3,518	1,925	4
8. *Penicillium*	2,914	7,181	3,625	7
9. *Scenedesmus*	2,175	452	1,215	7
10. *Citrobacter*	1,804	771	1,507	4

high-molecular-weight material that is released by the inactivated cells but retained in the gel matrix. Current experiments are exploring the potential interference by other metal ions.

Advanced Bioreactor Systems

Ethanol Production in a Fluidized-Bed Bioreactor (FBR)

The production of ethanol from dextrose by immobilized cells of *Zymomonas mobilis* continues to be studied in an FBR. This project has demonstrated the feasibility of this concept and its potential advantages over the conventional fed-batch operation with yeast. Some optimization of the operating parameters has also been investigated.

Studies were carried out using immobilized *Z. mobilis* in FBRs, emphasizing operation during high productivity and conversion.[2] The bacteria were immobilized within small uniform gel beads (~1 mm diam) at high cell loadings. Volumetric productivities of 50 to 100 g EtOH/L h^{-1} have been achieved with residual glucose concentrations of <0.1%. The ethanol yield was shown to be nearly constant at 0.49 g EtOH/g glucose, or 97% of the theoretical yield under a variety of conditions and transients. This yield represents a major improvement over that of the traditional species, *S. cerevisiae*, which produces a yield of 85 to 94% of the stoichiometric limit. The biocatalyst beads have been shown to remain active for >2 months during operation with nonsterile feed.

After the successful demonstration of the feasibility of this process, preliminary optimization of environmental conditions was performed. Temperature, pH, and supplemental nutrient concentrations were varied in the larger FBR. A temperature of 30°C and a pH of ~5 were found to be optimum for this system.

The immobilized *Z. mobilis* FBR was shown to have definite advantages over the typical yeast fed-batch reactor. First, there was a more than tenfold increase in the ethanol productivity with the same high (>99%) conversion of a 15% dextrose feed and with ethanol concentrations >7 w/v %. This is due to the combination of a high biocatalyst concentration from immobilization, improved kinetics from *Z. mobilis*, and the multistage character and improved gas disengagement of an FBR. Therefore, a much smaller reactor, with lower capital costs, could be used for the same alcohol output. In addition, the current industrial feedstocks have been shown to be fully utilizable by this new process.

Contamination is a serious problem in the long-term operation of many continuous bioreactors. Here, a second advantage is seen in the use of nonsterile feed for weeks without detrimental contamination. The nonsterile operation was successful, as the high flow rate and mixing removed the contaminants. The ability of an immobilized-cell FBR to wash out contaminants while maintaining the desired pure culture within the beads has been demonstrated. This capability counterbalances the absence of the low pH that is used in yeast fed-batch reactors to maintain sterility. Such an advantage should be applicable to most immobilized-cell FBRs, resulting in definite benefits in performance and in operating costs.

The third major advantage offered by this FBR system is the improved ethanol yield per gram dextrose due to the use of *Z. mobilis*. Under current economic conditions, the cost of the raw materials (i.e., dextrose from corn or other sources) is the largest single production expense; therefore, even a small but consistent increase in the yield can result in appreciable savings. Industrial interest has been expressed in this process by several ethanol fermentors. In addition, A. E. Staley & Co. provided the industrial feedstocks used in the above experiments.

Anaerobic Beads for the Acetone-Butanol Fermentation

Considerable interest has focused on the anaerobic fermentation of sugars into organic solvents, such as acetone and butanol, by *Clostridium acetobutylicum*. Establishing the strict anaerobic environment required by these microorganisms adds yet another dimension of difficulty to this bioconversion process. Adler at Oak Ridge Associated Universities has developed cell membrane fragments that will reduce oxygen to water.[3] These fragments can be immobilized in gel beads along with microorganisms to create a completely anaerobic microenvironment within the biocatalyst, despite the external conditions. This will allow the use of high biocatalyst loadings in a three-phase FBR for solvent fermentation, eliminating the need for special treatment to remove oxygen from the feed.

Preliminary experimental results demonstrated the successful use of oxygen-reducing biocatalytic

cell fragments for anaerobic solvent fermentation. The anaerobic cell fragments were coimmobilized with *C. acetobutylicum* into gel beads. This system could achieve anaerobic conditions in a batch system within a few minutes despite repeated challenges. Solvent production by *C. acetobutylicum* with the membrane fragments in both batch and continuous systems was superior to that obtained with nitrogen sparging to maintain anaerobiosis.

Solvent production was also demonstrated for a continuous columnar FBR with coimmobilized cells and membranes that used an air-saturated feed. A continuous three-phase columnar reactor was used in two runs, with monitoring at three ports along the reactor. The entire bed, from the first port on, became anaerobic within 1 h while using an oxygenated feed. Glucose conversion was low, due to the high flow rate necessary for fluidization, but continued to increase throughout the 165-h experiment, as seen in Fig. 4.6. The production of the desired solvents, such as butanol and acetone, can be sustained in this immobilized columnar reactor using only the membrane fragments, in spite of the use of oxygenated feed; continuous sparging of the feed is not required.

FBR Hydrodynamics

In designing an FBR, it is necessary to understand the distribution of the fluidized biocatalyst within the FBR, as well as the gas and liquid phases. Electroconductivity is being used to measure the axial dispersion and phase holdup in a fermenting FBR and in a model nonfermenting, three-phase FBR. Multiple axial conductivity probes were used to nonintrusively monitor the bed conductivity. In essence, both the broth and the beads are equally conductive due to the salts present, while the gas is not. Therefore, the conductivity will decrease linearly with the amount of gas present, or

$$\gamma/\gamma_0 = \epsilon_L + \epsilon_S = 1 - \epsilon_G ,$$

where

γ = the overall measured bed conductivity,
γ_0 = the bulk liquid conductivity,
ϵ_G = the phase fraction for the gas,
ϵ_L = the phase fraction for the liquid,
ϵ_S = the phase fraction for the solid.

The solid fraction was estimated from the visually observed bed height. It was found to be a weak function of the gas flow rate and can be reasonably estimated from the two-phase system. The system successfully measured the gas holdups directly, but more work is needed to correlate the results.

An additional advantage of using conductivity measurements is that conductivity has long been used in dispersion experiments to monitor the response of a system to an impulse or step change of salt tracer. The axial dispersion of a solute in a nonreactive system can be modeled as

$$dC/dt + u_L dC/dz = D\, d^2C/dz^2 ,$$

where

C = concentration,
D = liquid axial dispersion coefficient,
u_L = liquid velocity.

After nondimensionalization, the Peclet number, which is a ratio of convective to dispersive forces, is defined as

$$Pe = \frac{u_L d_p}{D} ,$$

where d_p is the particle diameter. The method of moments was then used to calculate the residence times and the Peclet number from the response to an arbitrary tracer impulse that moves between two probes.

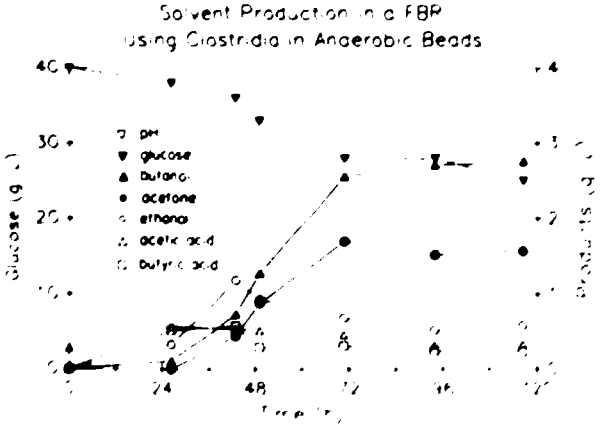


Fig. 4.6. Glucose and products concentration at the effluent of the columnar reactor with coimmobilized cells and membrane fragments during a continuous fermentation run. Flow rate = 30 mL/h.

Dispersion experiments were performed in both the inert and fermenting FBRs as described above. Particle Peclet numbers on the order of 10^{-2} were estimated as a function of flow rates and compared to three semiempirical literature correlations. Peclet numbers at other flow rates and from the fermenting FBR are comparable to those reported here. Figure 4.7 shows the effect of gas flow rate on the Peclet number at a constant liquid flow rate. As expected, the dispersion increases with increasing gas flow. Although the differences among the correlations are comparable to the difference obtained with the experimental data, the experimental data still appear to be lower than the correlations. This effect may be related to the fact that these correlations were developed in systems with high density differences and Reynolds numbers from 10 to 500 or more, whereas the gel biocatalysts typically have a density near 1 g/L and Reynolds numbers near 1. Further work is planned on the correlation of the gas fraction and dispersion results and on incorporating these measurements into an overall three-phase fermenting FBR model.

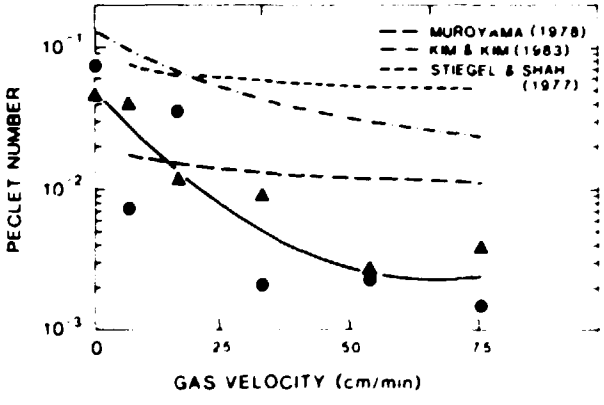


Fig. 4.7. Experimental Peclet numbers as a function of gas flow rate with F_L = 5 L/h. Peclet numbers compared with literature correlations from: K. Muroyama et al., *Kagaku Kogaku Ronbunshu* 4(n), 662 (1978); S. D. Kim and C. H. Kim, *J. Chem Eng. Japan* 16, 172 (1983); and G. J. Stiegel and Y.T. Shah, *Ind. Eng. Chem.* 16, 37–43 (1977).

4.2 Chemistry Research

A primary focus of this work is the fundamental chemistry of the actinide elements and their fission products. Investigators are concentrating their efforts in six areas:

1. small-angle X-ray scattering studies of hydrous polymers,
2. low-temperature infrared studies of matrix-isolated actinides,
3. development and characterization of synergistic solvent extraction systems for the separation of lanthanides and actinides,
4. redox and coordination chemistry of actinides and lanthanides in concentrated electrolytes,
5. separation chemistry of the transpultonium elements in alkaline media, and
6. use of sol-gel technology to produce ion-exchange materials for actinide and fission product separations.

A small effort in the fossil energy area is concerned with the development of chemical separations methods, such as solvent extraction, as a means of upgrading the quality of condensible products that are derived from coal gasification at mild temperatures.

Chemistry of Actinides and Fission Products

The primary purpose of this program is to explore the fundamental chemistry of the actinide elements and their fission products. Emphasis is placed on the photochemistry, coordination chemistry, and separation chemistry of these elements, especially as they relate to problems in waste management, environmental control, and the nuclear fuel cycle. The chemistry of other elements is often examined in parallel with that of the actinides and fission products in order to develop a better understanding of the periodic behavior of the elements. Special efforts are made to explore areas that cannot or will not be examined elsewhere and to use the unique facilities that are available at ORNL for handling hazardous and radioactive materials.

The program is divided into two broad areas: spectrophotometric studies and chemical studies. Specific areas of interest include:

1. examination of spectroscopy and photochemistry of actinides in low-temperature matrices, with particular emphasis on isotope effects;
2. small-angle scattering studies of hydrous polymers in aqueous and nonaqueous solvents;

3. characterization of the structures and properties of pure and mixed actinide polymers;
4. photochemical studies of U, Np, and Pu in aqueous solution;
5. kinetic and thermodynamic studies of complexation and redox reactions in aqueous and nonaqueous solutions;
6. the determination of solvation numbers in aqueous and nonaqueous solutions;
7. investigation of the properties of complexes with macrocyclic ligands;
8. the development of inorganic ion-exchange materials for actinide and lanthanide separations; and
9. studies of fundamental properties of aqueous and nonaqueous solvent systems in support of these primary fields of investigation.

Currently, there are six areas of active investigation:

1. small-angle scattering studies of hydrous polymers,
2. spectroscopic studies of matrix-isolated actinide compounds,
3. synergistic effects of macrocycles on the solvent extraction of actinides and lanthanides,
4. coordination chemistry of actinides and lanthanides in nonaqueous systems,
5. extraction chemistry of actinides and lanthanides in alkaline solution, and
6. inorganic ion-exchange materials for actinide and fission product separations.

Progress in each of these areas is summarized here.

Photochemical Studies

Small-Angle Scattering Experiments

The first stage of the small-angle X-ray scattering (SAXS) experiments on Th(IV) hydrous polymers has been completed. These studies have been conducted on 0.01 *M* $Th(NO_3)_4$ aqueous suspensions and organic extractants that were equilibrated with the aqueous phases. Assuming spherical geometries, fresh Th(IV) polymer suspensions consist of particles that vary in size from 25 to 84 Å, whereas aged (by reflux) Th(IV) polymer suspensions consist of particles that range in size from 16 to 174 Å. The decrease in size of the small extreme on aging (i.e., the 25-Å particles becoming 16 Å) is consistent with a dehydration and shrinkage process that occurs during conversion of hydroxyl bridges to oxygen bridges between thorium atoms.

These suspensions have been shown to be extracted by 0.5 *M* dibutylphosphate in *n*-dodecane, *nDD*, but not by 0.1 *M* trioctylphosphine oxide in *nDD* or 0.1 *M* dihexyl-[(diethylcarbamoyl)-methyl]phosphonate in *nDD*. The particle size distribution in the organic extractant was similar to that in the aqueous phase, showing that there was no size segregation as a result of extraction.

Attempts to alter the size of the polymers by growing them in the presence of UO_2^{2+} produced no differences in particle size distribution; it has since been realized, though, that the 0.5/1 ratio of UO_2^{2+}/Th(IV) would probably have to be increased by 10X before measurable effects could be obtained, if at all. It is probable that the tendency of the UO_2^{2+} and Th^{4+} ions to hydrolyze in the same pH region, namely at a pH of 3 to 4, and the subsequent precipitation of UO_2^{2+} interfere with the extensive interaction of UO_2^{2+} with Th^{4+}. (It should be recalled that the significant chain inhibition reaction was noted with the UO_2^{2+}/Pu^{4+} system in which Pu^{4+} begins to hydrolyze in the region where pH = 1 to 2.)

With the development of modeling routines at Argonne National Laboratory (ANL), the above SAXS data for Th(IV) have been analyzed with respect to geometry and particle size by fitting the scattering data, using Fournet functions for various geometries. A nonlinear regression analysis was used for obtaining diameters and/or length-to-diameter ratios (when less symmetrical geometries were tested). As a result of these analyses, it was apparent that the complications caused by the polydisperse nature of the colloid (i.e., the polymer suspension consists of particles of widely varying sizes) would not permit a satisfactory fit of the SAXS data to a given model which assumed a particular geometry of uniform-size particles. Improvements in the model are currently being developed that should permit inclusion of the polydispersity of the system.

In collaboration with ANL staff, small-angle neutron scattering experiments (SANS) have been

initiated on Pu(IV) polymer suspensions in nitric acid solutions. These experiments are being performed on the Intense Pulsed Neutron Source (IPNS) at ANL. At this stage of the analysis, the polymers appear to be long, thin rods of 14-Å diam. If the polymer is grown in the presence of UO_2^{2+}, where U/Pu = 5, the length of the rod is considerably shortened. Extraction of the polymer into a variety of organic extractants appears to be accompanied by some depolymerization.

Spectroscopic Studies of Matrix-Isolated Actinide Compounds

The focus of our low-temperature studies of matrix-isolated actinides has been the production of a uranyl species, which could subsequently be photoreduced, in the inert gas matrix. Although the technique for producing matrix-isolated uranyl has not been perfected, two sets of experiments have yielded very promising results. In both procedures, gas samples were condensed on an infrared-transmitting cold window, as shown schematically in Fig. 4.8. Reactions were monitored spectroscopically.

During the first group of experiments, pure UF_6 and H_2O were deposited on the cold window, which was then warmed to effect hydrolysis of UF_6 to uranyl fluoride (UO_2F_2). In addition to reactants and products, at least two distinct species were observed. The first species was formed in substantial quantity during the deposition and was characterized by a strong absorption at 550 cm^{-1}. because the peak position did not shift upon isotopic substitution (^{18}O-labeled H_2O), we attributed the absorption to a UF_6-H_2O adduct. A small amount of the second species was also formed during deposition. This species was characterized by a strong absorption at 850 cm^{-1}, which shifted to 800 cm^{-1} upon ^{18}O substitution. The frequency and isotopic shift indicated that this peak was the U-O stretching vibration of a hydrolysis intermediate. As the sample temperature was increased, the absorption due to the adduct decreased in intensity while that due to the intermediate increased. Most of the adduct had reacted to form the intermediate before the temperature reached 100 K. A small amount of UO_2F_2 was also detected below 100 K. These experiments indicated that the formation of uranyl by hydrolysis is thermodynamically possible at low temperatures.

During the second group of experiments, UF_6 and H_2O were codeposited with an excess of argon matrix gas, as indicated in Fig. 4.8. Spectra

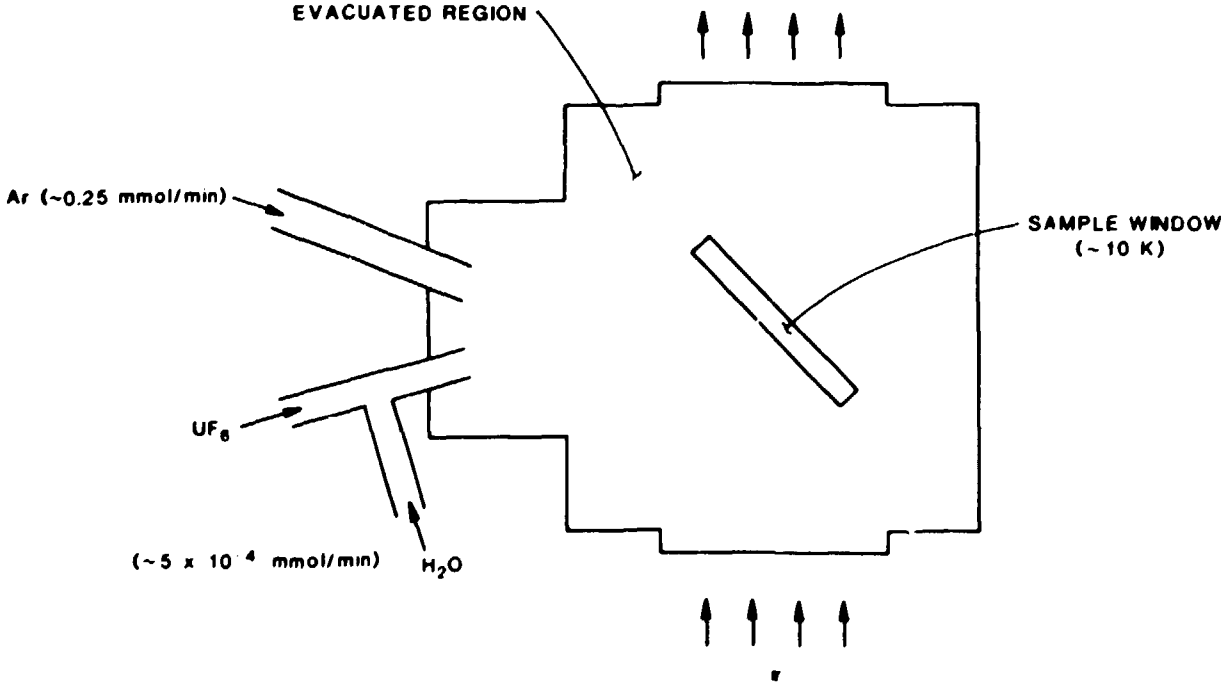


Fig. 4.8. Schematic drawing of matrix-isolation apparatus.

obtained during these experiments exhibited the sharpness and intensity characteristic of matrix-isolated species. Again, we observed a band which we attributed to an adduct. Warming to effect hydrolysis has been limited to 30 K (due to boiloff of the argon), which has not been sufficient to induce reaction. Warming combined with 254-nm photolysis, however, produced the results shown in Fig. 4.9. A pair of bands, again assigned to a hydrolysis intermediate, appeared at 866 and 855 cm^{-1}. Isotopic substitution demonstrated that the species contains only one U-O bond. Also shown in the figure are the U-F stretching absorptions. Those at 600 to 620 cm^{-1} are due to UF_6. The absorption at 589 cm^{-1} (greatly reduced after reaction) is due to the adduct. The bands that appear at 586 and 563 cm^{-1} are due to UF_5 (product of UF_6 photolysis), while the other U-F absorptions must be associated with the intermediate.

Because the earlier experiments indicated that hydrolysis to UO_2F_2 can occur without photolysis below 100 K, we are currently experimenting with matrix gases (Xe) which can be warmed to temperatures substantially above 30 K. In addition to producing matrix-isolated uranyl, we will attempt to establish the identities of the adduct and intermediate species.

Chemical Studies

Synergistic Effects of Macrocycles on the Solvent Extraction of Actinides and Lanthanides

The goal of this task is the development and characterization of synergistic solvent extraction

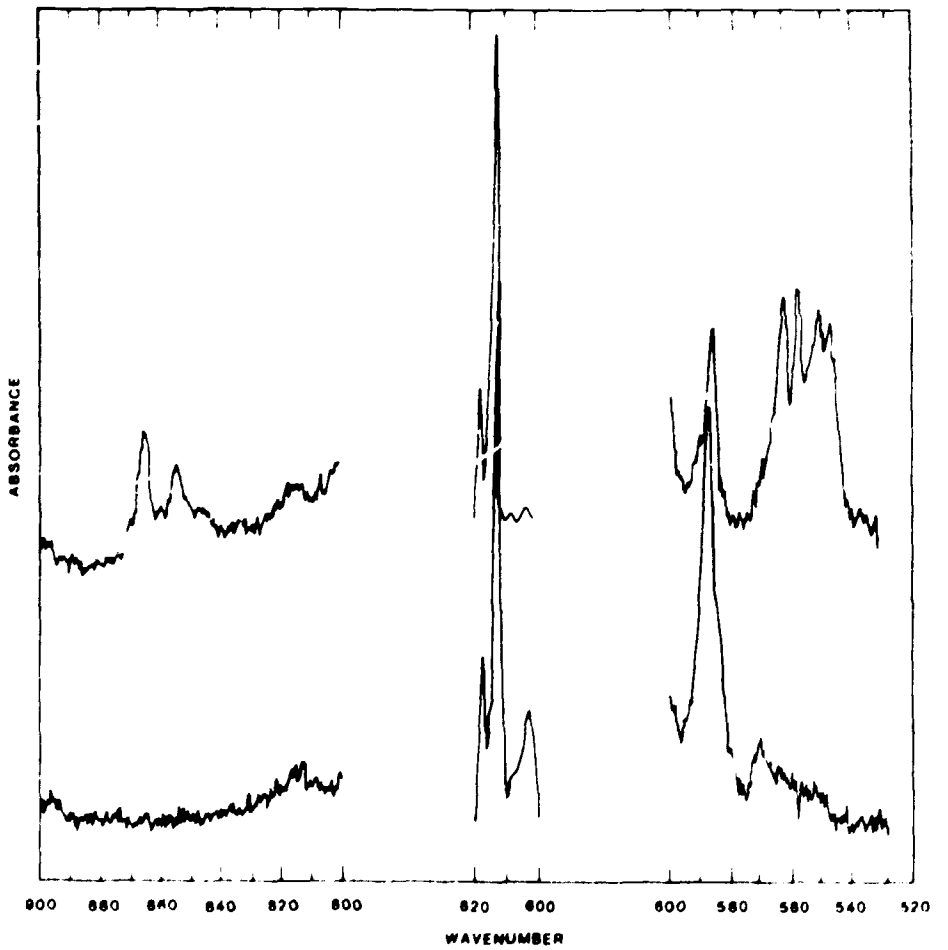
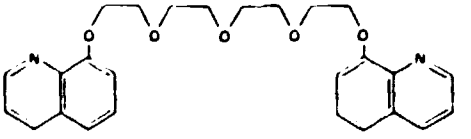


Fig. 4.9. Infrared spectra of matrix-isolated uranyl photolysis products.

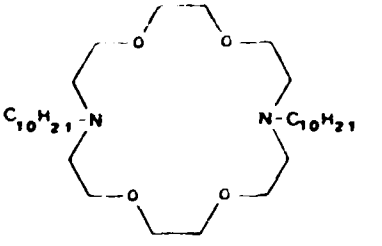
systems using macrocyclic ligands for the separation of lanthanides and actinides. This work is being carried out in collaboration with D. D. Ensor of Tennessee Technological University.

Initial studies of the mechanism of extraction of U(VI) and several trivalent actinide and lanthanide elements using thenoyltrifluoroacetone (TTA) in combination with a linear polyether, an aza-crown ether, or a crown ether (Fig. 4.10) were completed. Two papers describing these studies have been accepted for publication. The addition of K-5 or K22DD caused a significant synergistic effect on the extraction of UO_2^{2+} by TTA (Fig. 4.11). The extraction constants and the organic-phase stability constants (B_{org}) in the formation of uranyl and several actinide and lanthanide synergistic complexes are reported in Table 4.5.

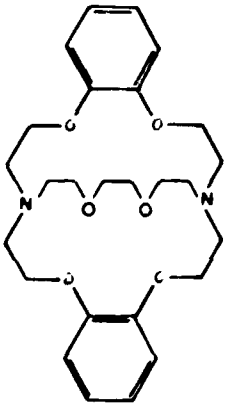
The two nitrogen-containing synergists show similar values for B_{org} in the extraction of each metal, despite the different sizes, shapes, and complexing geometries in the ligands. The synergistic effect obtained using 15-C-5 was too small to measure under the conditions examined. These results suggest that the nitrogen atoms in the macrocycle are largely responsible for the synergistic effect. It can further be seen that the synergistic effect is largest for the ions with the greatest ionic radius and decreases as the radius decreases.



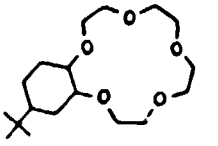
(1,13-bis-quinolyl)-1,4,7,10,13 pentaoxatridecane (K-5)



4,13,-didecyl-1,7,10,16-tetraoxa-4,13-diazacyclooctadecane (K22DD)



5,6,14,15-dibenzo-4,7,13,16,21,24-hexaoxa-1,10-diazabicyclo-[8,8,8] hexacosane (22BB)



4-tertbutylcyclohexo-15-crown-5 (15-C-5)

Fig. 4.10. Extractants used in synergistic studies.

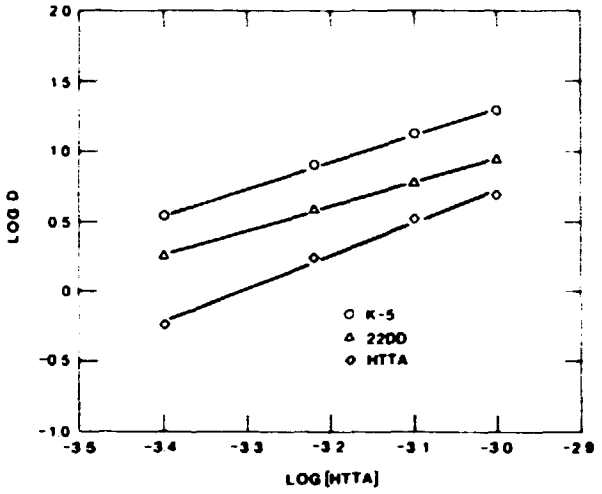


Fig. 4.11. Effects of K-5 and K22DD on the extraction of U(VI) by TTA.

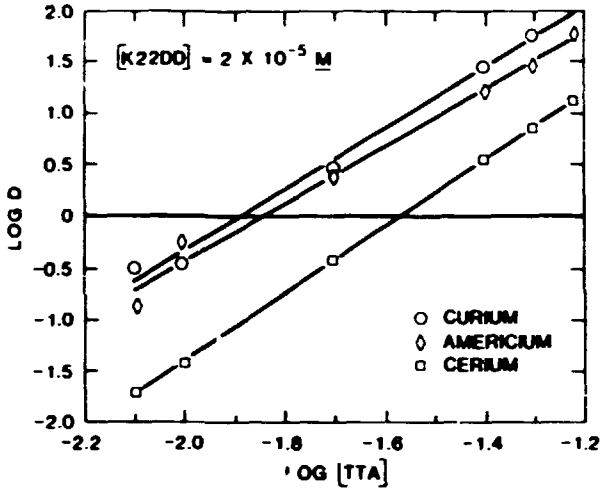


Fig. 4.12. Extraction of Cm(III), Am(III), and Ce(III) by K22DD-TTA mixtures.

An excellent group separation could be achieved, but there was no significant separation of the individual elements (Fig. 4.12).

Coordination Chemistry of Actinides and Lanthanides in Nonaqueous Systems

Current studies emphasize actinide and lanthanide redox and coordination chemistry in concentrated electrolytes, low-melting fused salts, and other nonaqueous systems. The phosphoryl trichloride-tin(IV) chloride system, the aluminum chloride-*n*-butyl pyridinium chloride system, and aluminum chloride-1-ethyl-3-methyl-1H-imidozolium chloride (AC-EMIC) system have been examined in the last year.

Attempts to measure acetate, sulfate, thiocyanate, and nitrate complexation of neodymium and uranium in $POCl_3$:$SnCl_4$ were unsuccessful. It was found that the solvent attacks all of these common anions very quickly, producing a variety of products. No further studies of this system are planned.

The aluminum chloride-*n*-butyl pyridinium chloride system (a salt mixture that melts at or slightly above room temperature) was also examined. The reagents were successfully synthesized and purified. However, the reagents were expensive and time-consuming to prepare. Furthermore, the resulting melts did not have sufficient optical purity to allow spectroscopic studies. The AC-EMIC system proved to be much less expensive to prepare, has better spectroscopic and electrochemical properties, and is not photosensitive. Hence, the pyridine system was abandoned, and a graduate student spent 2 months at ORNL synthesizing

Table 4.6. Equilibrium values for the extraction of metal ions by synergistic mixtures[a]

Synergist	U(IV)	Ce(III)	Pm(III)	Eu(III)	Am(III)	Cm(III)
			Log K			
TTA only	−3.00	−9.95	−9.28	−8.58	−9.10	−9.10
TTA + K22DD	−2.85	−4.70	−5.12	−5.03	−5.32	−4.00
TTA + K-5	−3.12					
			Log β_{org}			
TTA + K22DD	3.47	4.93	4.54	4.61	5.06	4.97
TTA + K-5	3.64	4.15	3.97	3.62	4.35	4.2₁

[a]Organic phase: chloroform; aqueous phase: 0.50 *M* NaNO3, pH = 4.7, acetate buffer; 25°C.

~2 kg of the AC-EMIC salts. Studies of *(III)-U(IV) complexation and redox chemistry are just beginning.

These studies are a continuation of an ongoing collaboration with G. R. Choppin of Florida State University. One postdoctoral student and one undergraduate are supported by a $48K subcontract.

Extraction Chemistry of Actinides and Lanthanides in Alkaline Solution

This task is aimed at exploring the separation chemistry of the transplutonium elements in alkaline media. Initial studies have focused on the chemistry of berklium(IV) and californium(III) and their lanthanide analogs, cerium(IV) and europium(III).

Preliminary work showed that *t*-butylcatechol could be used to preferentially extract trivalent actinides/lanthanides from a mixture of trivalent and tetravalent ions (Fig. 4.13). However, the catechol was quite soluble in the aqueous phase, reducing its usefulness as a solvent extraction reagent.

Recent results have shown that 3,5-di-*t*-butylcatechol is equally effective at extracting the trivalent ions, and there is little or

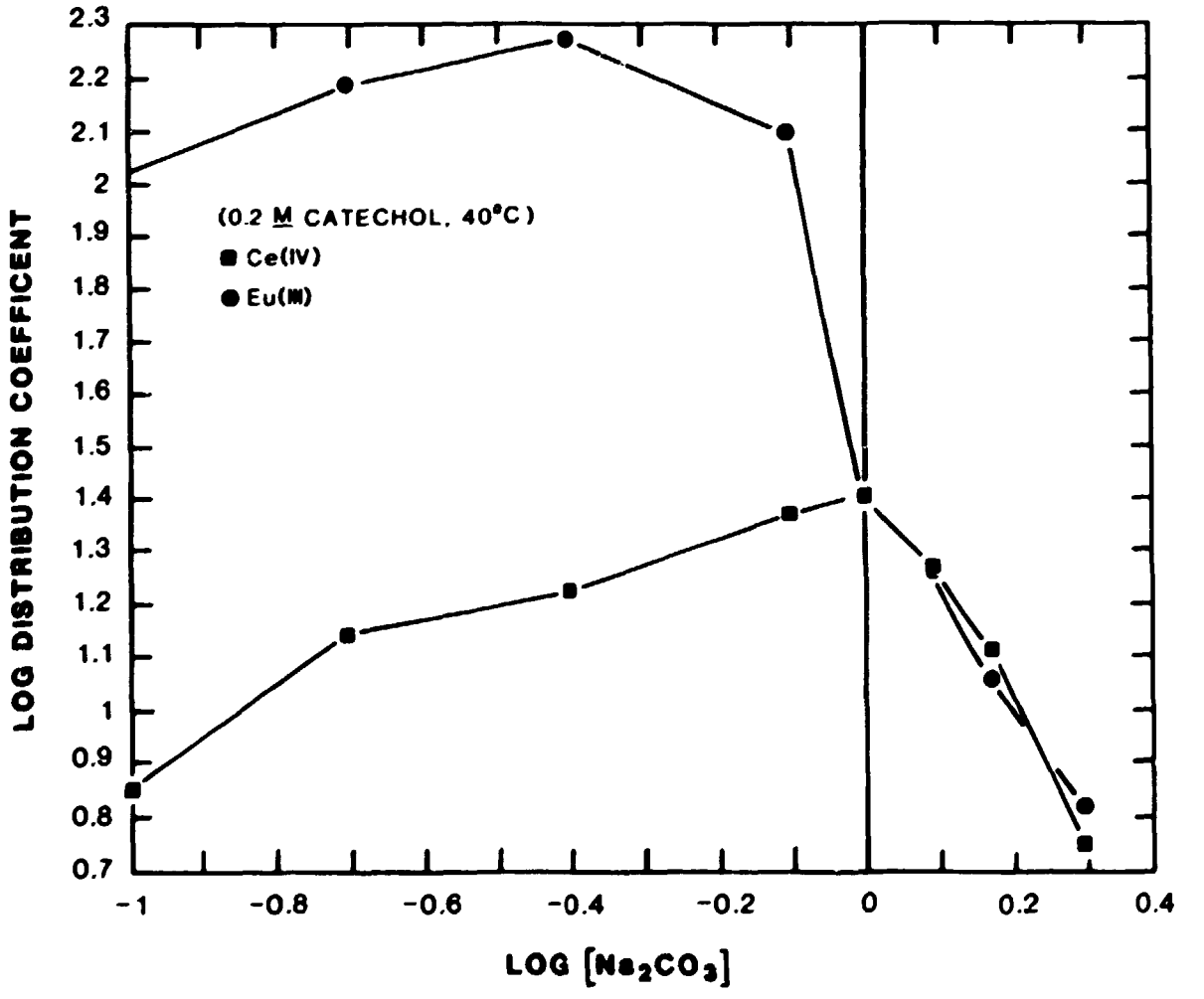


Fig. 4.13. Extraction of Ce(IV) and Eu(III) by *t*-butylcatechol from aqueous carbonate solution.

no loss of this reagent to the aqueous phase. However, the compound is easily oxidized, and the extractions must be carried out under an inert atmosphere. In collaboration with J. R. Petersen of the University of Tennessee, efforts are now under way to find an extraction reagent that is water insoluble and stable to air.

Inorganic Ion-Exchange Materials for Actinide and Fission Product Separations

Initial studies of inorganic ion exchangers made from heteropolyacids have been shown to have some selectivity for lanthanide and actinide elements, but the synthesis and properties of the materials are irreproducible. The use of sol-gel technology to produce these materials is being explored.

Removal of Heteroatoms from Coal Gasification Condensibles

Coal gasification is a process that essentially converts a solid fuel (coal) into gaseous and liquid fuels by reacting the coal with oxygen and steam. The conversion of coal from a solid to a liquid or a gas has several advantages: (1) direct use in conventional heat engines; (2) removal of contaminants such as ash, sulfur, and nitrogen; and (3) generation of coproducts which can be processed further into other fuels or chemicals. Reaction temperatures can vary from ~1000 to 3000° F, depending on the type of gasifier and its configuration.

Mild gasification of coal is a process which is gaining prominence because its reaction temperatures, in the range of 900 to 1200°F, enhance the yield of liquid products. These liquid products, or condensibles, have the potential to improve the economics of mild gasification processes through the generation of value-added products such as octane enhancers, chemical feedstocks, and some forms of transportation fuels. However, in order to achieve this value-added status, the condensible products must be upgraded by some cost-effective process, removing heteroatom-containing "contaminants" such as oxygen, nitrogen, and sulfur. The conventional process employed to upgrade petroleum products is catalytic hydrotreating. This is a high-temperature, high-pressure process, which makes it costly and not very energy efficient. In addition, hydrotreating of liquids derived from coal tends to cause rapid degradation of the catalysts used in the process.

The primary purpose of this project is to develop alternative or adjunct methodologies to hydrotreating for upgrading condensible liquids derived from mild-temperature devolatilization of coal. Some form of hydrotreatment will continue to be an important processing tool for high-quality transportation fuels, but an alternative, or, perhaps, joint, processing step may incorporate a separation stage in which solvent extraction and/or solid-phase adsorption is employed to remove heteroatom-containing "contaminants." Depending on the aromatic content of the condensibles and the type of transportation fuel desired (high-quality jet and diesel fuels have strict limitations on aromatic content), reduction in aromatics can also be accomplished by solvent extraction.

The emphasis of this work is on the development of cost-effective upgrading methodologies that are based on solvent extraction using high-efficiency contactors. Initial efforts will address the removal of organo-nitrogen compounds, as these materials tend to poison catalysts, are hazardous to human health, and are generally the most difficult to remove by more conventional upgrading methods.

Coal condensibles of primary concern to this project have been those derived from the mild devolatilization process being tested by the United Coal Company Research Corporation (UCCRC) in Bristol, Virginia. Difficulties in obtaining sufficient quantities of representative condensible liquids from this process have necessitated the use of samples from other devolatilization processes, including the KILnGAS process being tested by the Allis-Chalmers Coal Gas Corporation in East Alton, Illinois, and that being tested by SGI International at the Salem Furnace Company in Pittsburgh, Pennsylvania. The UCCRC Mild Gasification process development unit is capable of handling up to 1500 lb/d of coal at temperatures up to 1500°F under pressure conditions ranging from atmospheric to a mild vacuum. The KILnGAS commercial module is a 600-ton/d plant and uses a ported and pressurized rotary kiln to produce fuel gas from coal. The SGI International system is a relatively small, batch-fed, atmospheric pyrolyzer with a nominal operating temperature of 1000°F, a residence time of 20 min, and a capability of feeding 50 lb of coal per batch.

Extraction Studies on a UCCRC Liquid

Upgrading studies initially have emphasized the removal of nitrogen-containing compounds from raw condensible products of the UCCRC process by solvent extraction using acidic reagents. The high viscosities of the initial samples and their tendency to retain water during two-phase contacting with aqueous acidic reagents suggested that distillation would be a necessary step in the upgrading process. Low-pressure distillation (<1 torr) of a UCCRC liquid was performed to produce a <650°F fraction for further solvent extraction studies (see ultimate analyses in Table 4.7).

Primary reagents that were employed to remove basic nitrogen compounds by solvent extraction were acetic and formic acids, at concentrations ranging from 25 to 90 vol %; however, monochloroacetic, trichloroacetic, and phosphoric acids were also examined. The chlorinated acetic acids were utilized to determine if acid strength (p*K*a) influences the degree of nitrogen removal. Results that were obtained indicated that acetic acid, the weakest acid, was the most effective extractant of the nitrogenous compounds. Economically, acetic acid would be favored because it is the cheapest reagent on a strictly weight basis; however, on an equivalent molar basis, the bulk cost of formic acid, a somewhat stronger acid (p*K*a = 3.75, compared with that of acetic acid, which has a p*K*a = 4.75), is only 3.6% higher than acetic acid.

Solvent extraction studies were performed on UCC-2 with both formic and acetic acids in concentrations ranging from 50 to 90 vol % (for 90 vol % acetic acid extraction, insufficient sample remained for analytical workup). The results, illustrated in Table 4.8, show that maximum removal of nitrogen was 51% using 75 vol % acetic acid. Previous work on a UCCRC condensible sample of somewhat higher viscosity (barely pourable at ambient temperature) showed incomplete separation of the aqueous phase and high water content of the organic phase. The analytical results obtained for extractions of UCC-2 show no tendency for the organic phase to retain water, as indicated by the oxygen assays. This was most probably due to the lower viscosity of UCC-2 compared with that of the previous sample.

A patent describing the extraction of nitrogen-containing contaminants from shale oil[4] with phosphoric acid was used as the basis for solvent extraction studies on the raw UCC-2 condensible sample using this acid. Acid concentrations of 25, 50, and 75 vol % were tested, utilizing the same contacting procedure employed with acetic and formic acids. Unextracted nitrogen assays were 0.46, 0.44, and 0.37 wt %, respectively, with the highest acid concentration (75 vol %) providing the greatest removal—66% of the original nitrogen. A third phase, which made clean separations difficult to achieve, was formed in these experiments.

Extraction studies on the <650°F distillate of UCC-2 using 50 and 75 vol % formic and acetic acids are shown in Table 4.9. There was little difference between nitrogen removal by 75 vol %

Table 4.7. Ultimate analysis of a UCCRC condensible liquid and its <650°F distillate

Element	UCC-29[a] (wt %)	Distillate[b] (wt %)
Carbon	86.56	86.41
Hydrogen	6.32	7.71
Oxygen[c]	5.24	4.37
Nitrogen	1.08	0.40
Sulfur	0.84	0.52
H/C ratio	0.88	1.07

[a]Sample from UCCRC test run 2-8'0 (6/11/87).
[b]<650°F distillate at 0.3 torr from UCC-2.
[c]Determined by neutron activation analysis.

Table 4.8. Extraction studies on UCC-2 with aqueous formic and acetic acids[a]

Element	Raw UCC-2	Organic-phase concentration (wt %)					
		Formic acid[b]			Acetic acid[b]		
		502	752	902	502	752	902
Nitrogen	1.08	0.85	0.74	0.59	0.56	0.53	c
Oxygen	5.24	4.70	4.64	7.96	4.54	4.79	c

[a]Three extractions with fresh acid; water washed.
[b]Concentrations in vol %.
[c]Insufficient sample for analytical workup.

Table 4.9. Extraction studies on a <650°F distillate of UCC-2

Acid concentration (vol %)	Nitrogen remaining[a] (wt %)	
	Formic acid	Acetic acid
50	0.13	0.17
75	0.10	0.09

[a]Original distillate contained 0.40 wt % nitrogen.

concentrations of either of these two acids: ~75% removal of nitrogen was demonstrated in each case. Separations were clean, and no experimental difficulties were encountered. This degree of removal probably can be improved in an actual process using more efficient contactors.

Extraction Studies on an SGI Condensible

The condensible product from SGI International was described as a second-stage product, using coal mined from the Rosebud mine in Montana, which was obtained through the courtesy of R. L. Graves of the Energy Technology Division of the Oak Ridge National Laboratory. Little detail is known about this particular sample; however, its appearance and analysis suggested that, at the very least, it was a type of distillate (see Table 4.10). Both the aromatic content, as determined by nuclear magnetic resonance, and the oxygen and sulfur assays were unusually low for a coal condensible liquid.

Solvent extraction studies with both acetic and formic acid showed nearly complete removal of nitrogen (98 to 99%) with all concentrations tested (see Table 4.11). Clean separations were again observed with this extremely light organic liquid, as indicated by the oxygen assays on the organic phase. The nitrogen removal demonstrated for this product, coupled with the already low oxygen, sulfur, and aromatic assays, is probably enough upgrading to enable it to be used directly as a fuel in some diesel engines; however, its true origin is still in question.

Table 4.10. Analysis of a second-stage condensible product[a] of SGI's gasification process at the Salem Furnace Company

Element	Analysis (wt %)
Carbon	87.22
Hydrogen	12.60
Oxygen	0.16
Nitrogen	0.40
Sulfur	0.07
H/C ratio	1.73

[a]Run No. 1003, 5/13/86.

Extraction Studies on a KILnGAS Condensible

A relatively large sample (~5 gal) of a KILnGAS condensible, described as a "high-naphthalene-content" product, was received through the courtesy of the Allis-Chalmers Coal Gas Corporation. This product appeared to be intermediate in viscosity between the UCC-2 and SGI samples. Although made difficult by the relatively high naphthalene content, a vacuum distillation was performed and a small amount of material prepared for extraction studies. As indicated in the analysis of the raw and distilled KILnGAS products (see Table 4.12), the sulfur content was very high and the product was highly aromatic, as indicated by the H/C ratios of 0.76 and 0.83. Naphthalene precipitated in the distillate and was removed by filtration prior to the extraction studies.

Solvent extraction studies were performed on both of these products with acetic acid only. The results, shown in Table 4.13, indicate that 67% and 58% of the nitrogen were removed from the raw and distilled KILnGAS products, respectively, with 75 vol % acetic acid. The usual improvement in

Table 4.11. Extraction studies on an SGI liquid with aqueous formic and acetic acids[a]

		Organic-phase concentration (wt %)							
	SGI Raw SGI	Formic acid[b]				Acetic acid[b]			
		25	50	75	90	25	50	75	90
Nitrogen	0.40	<0.01	<0.01	0.01	<0.01	<0.01	<0.01	0.02	<0.01
Oxygen	0.16	0.12	0.13	0.11	0.10	0.14	0.11	0.10	0.08

[a]Three passes with fresh acid; water washed.
[b]Concentrations in vol %.

Table 4.12. Analysis of a raw KILnGAS condensible and its <650°F distillate

Element	Raw KILnGAS sample (wt %)	<650°F distillate (wt %)
Carbon	88.56	87.42
Hydrogen	5.60	6.03
Oxygen	1.43	1.87
Nitrogen	0.66	0.31
Sulfur	3.38	3.93
H/C ratio	0.76	0.83

Table 4.13. Extraction of nitrogen from raw and distilled KILnGAS products using acetic acid[a]

Acetic acid (vol %)	Remaining nitrogen (wt %)	
	Raw KILnGAS	<650°F distillate
25	0.31	0.22
50	0.32	0.17
75	0.22	0.13
90	0.21	*b*

[a]Three extractions with fresh acid; water washed.
[b]No sample prepared.

nitrogen removal on increasing the concentration of acid from 50 to 75 vol % was observed for the raw product but not in the case of the distillate. Based on the starting nitrogen concentration of 0.31 wt %, a higher removal than 58% was expected; thus, these results will have to be rechecked.

Conclusions

The conclusions that can be drawn from solvent extraction studies on limited amounts of three condensible liquids must be tentative. However, it appears that incomplete separation of the aqueous phase and poor extraction of the nitrogen-containing contaminants from the condensible liquids produced by UCCRC can be alleviated by distillation of the raw condensible liquids. Distillation is recommended as the first step in an upgrading process that incorporates solvent extraction for nitrogen removal. The present carload prices for three of the acids that were evaluated during the course of this study are (in cents per pound): acetic acid—27; formic acid—36.5; and phosphoric acid (85%)—25.5. Based on our present state of knowledge concerning cost, efficiency, availability, and construction materials, 75 vol % acetic acid is recommended as the extraction medium. However, to be economically feasible, a solvent extraction upgrading process must incorporate solvent stripping and reagent regeneration stages, and these have not been defined at the present stage of this project.

4.3 Engineering Research

Fundamental studies are under way in two areas of engineering research, with the long-term objective being improved and predictable chemical separations. Improved mass transfer performance is sought in solvent extraction by utilizing the theoretical insight gained from an in-depth analysis of the effects of electrical fields in dispersed liquid systems. In addition, high-intensity, high-gradient magnetic field effects are being studied for their potential beneficial effects in macromolecular separations. The supercritical region is the focus of mathematical modeling studies designed to improve their predictive capabilities in chemical separations within this unique and important solubility-enhancing region for gases/liquids. In addition, the solubility of inorganic fluorides is being investigated in this experimentally difficult supercritical region. Finally, more applied studies concerned with technology transfer are being pursued, employing a previous Chemical Technology Division invention, the continuous annular chromatograph. Pilot-scale sugar separations studies are under way, together with more fundamental investigations into continuous gradient elution chromatography and continuous displacement chromatography.

Effects of External Fields on Multiphase Systems

This program is comprised of several fundamental studies that address the use of electromagnetic fields to enhance the efficiency of multiphase separation processes. The primary focus lies in improving mass transfer performance in solvent extraction by utilizing theoretical insight gained from an in-depth analysis of electric field effects in dispersed liquid systems. The goal of this portion of the program is to explore effects on transport processes through a basic understanding of the electrohydrodynamical system. A secondary thrust has been initiated to pursue investigation of poten-

tial applications of high-intensity, high-gradient magnetic field interactions in separation schemes. This project involves investigation of electric and magnetic field effects as they pertain to macromolecular separations processes.

Droplet Formation in Electric Fields

Current work is investigating the use of an electric field to control the formation of small drops at an orifice in a circular disk. During the past reporting period, water drops were formed in a pure cyclohexane continuous phase in the prejetting regime using four different orifice diameters. Because of the application of high-potential gradients (up to 1000 V/cm) and because the orifice plate produced a nonuniform field, drop volumes were as much as 20 times smaller than drop volumes for the zero-field case. The drops were virtually ellipsoidal in shape, and droplet eccentricity increased dramatically with field intensity.

An approximate mathematical model was developed, based on the potential field between a disk (orifice plate) and an infinite plate (a grounded screen). The electric force on the drop was calculated from the field intensity at discrete points around the periphery. An empirical correlation for the electrical force for this particular plate diameter, spacing, and organic fluid is

$$F_e = 9.80 \times 10^{-5}(V/1000)^2 v^{0.54},$$

where V is the applied voltage, and v is the drop volume. Using this model, the values that were obtained for predicted drop volumes agreed well with those for experimental drop volumes, up to voltages of 2500, as shown in Fig. 4.14.

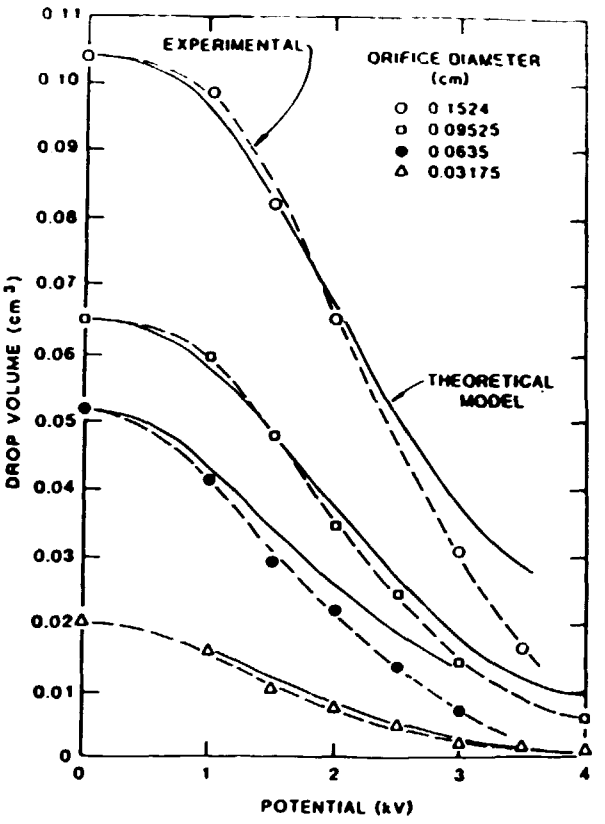


Fig. 4.14. Effect of electric field strength on droplet volume.

Mass Transfer Enhancement by External Electric Fields

The finite-difference method previously used to model circulating and oscillating-circulating mass transfer has been extended to include stagnant and oscillating-stagnant droplets. A preliminary comparison of results for circulating and stagnant droplets reveals that the rate of mass transfer is a complex function of the hydrodynamic state of the system as well as of the interfacial distribution properties of the transporting chemical species. These results suggest that with certain combinations of distribution coefficients, the transient separation factor between two chemical species may be enhanced by utilizing a stagnant droplet rather than one which undergoes internal circulation.

Improvement on and continued use of the fluorescent dye technique has provided a large set of precise, single-component mass transfer data for circulating, oscillating-circulating, and oscillating-stagnant droplets. Use of the fluorescence technique along with the video camera system allows accurate determination of values of the flux rate below 3×10^{-11} g cm^{-2}s^{-1}.

Figure 4.15 contains a plot of a portion of the mass transfer results for circulating and oscillating-circulating droplets. For the circulating case, a plot of dye flux vs continuous-phase concentration yields a straight line, which is indicative of the presence of an overall mass transfer coefficient relationship. Additional data on the graph indicate increases in the rate of mass transfer due to droplet oscillation at oscillation amplitudes of

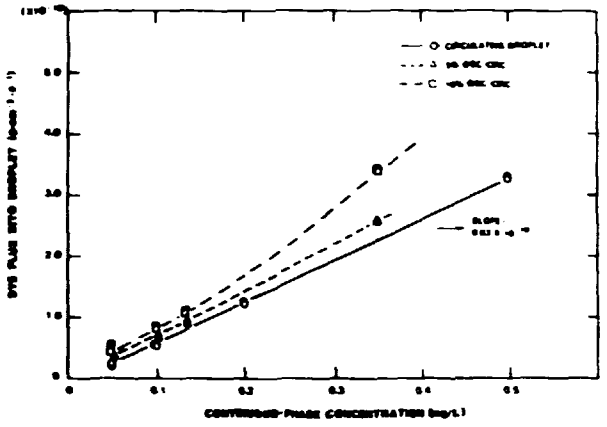


Fig. 4.15. Mass transfer results for circulating and oscillating-circulating droplets.

5% and 10% of the droplet radius. The magnitude of the increase is a function of the oscillation amplitude as well as of the dye concentration in the continuous phase, with higher oscillation amplitudes and continuous-phase concentrations resulting in the greatest increases. Enhancement ranges from a 10% increase, where the amplitude is 5% of the droplet radius and the continuous-phase concentration is 0.05 mg/L of dye, up to approximately a 50% increase for a droplet with 10% amplitude and a 0.35-mg/L concentration.

An electrically driven, high-surface-area solvent extraction system has been developed and tested. In this system, the dispersed phase is pumped into the top of a vessel through an inlet nozzle which is centered between two electrodes. Imposition of a high-intensity electric field across the electrodes causes the formation of an emulsion via electric-field-stress-induced droplet rupture. The emulsion is comprised of micron-sized droplets in the continuous phase. Coalescence of the emulsion occurs at a point below the area of droplet rupture. The resulting large droplets fall through the lighter continuous phase and form a separate phase at the bottom section of the vessel. Simultaneous to emulsification and coalescence of the dispersed phase, the (lighter) continuous phase enters near the bottom of the vessel, flows upward, and is withdrawn at the top of the column (see Fig. 4.16). The emulsion remains in the vicinity of the electrodes and therefore is separated from the upflowing organic phase. As the emulsion behaves as a separate phase, the name of the device employed in this process is the emulsion-phase contactor (EPC). The mass transport capability of this system has been analyzed by observing the transfer of acetic acid from water (dispersed phase) into methyl-isobutyl-ketone. The EPC, with four theoretical stages per inch, outperforms laboratory-scale versions of the York-Scheibel column (0.25 stages) and the Podbielniak centrifugal contactor (0.4 stages) by factors of 16 and 10, respectively. The mass transfer performance, combined with the minimal energy requirements for this system, makes this an attractive possibility for greatly enhanced solvent extraction operations.

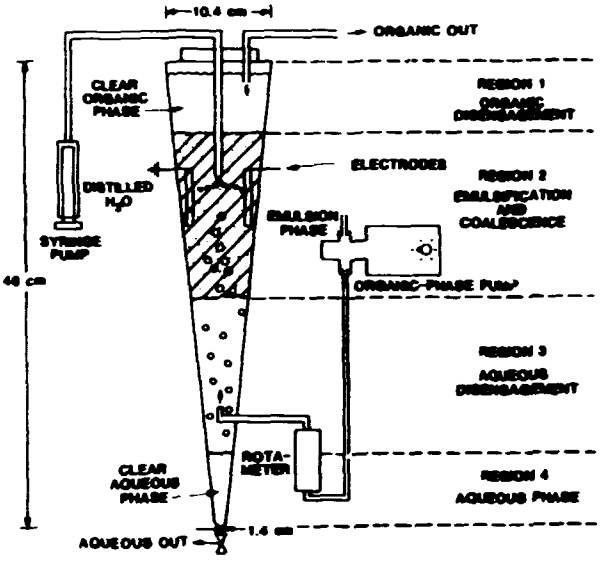


Fig. 4.16. The emulsion-phase contactor (EPC).

Phase Stability and Coalescence in External Fields

The oscillations of drops play a central role in areas as diverse as mass transfer, separations, and meteorology. Mathematical analysis of this problem leads to a dispersion equation that governs the frequency, β_I, and the rate of damping (or decay factor), β_R, of oscillations of a drop of one viscous fluid immersed in another. The dispersion equation is expressed by the vanishing of the determinant of a 7×7 matrix, solutions to which were generally heretofore known only in limiting cases: for example, when both fluids are inviscid, when a drop of a viscous fluid is oscillating in a vacuum or a low-density gas, or when the viscosities of both fluids

are "low." The original determinant has first been reduced to a 3 × 3 determinant:

$$\Delta = \begin{vmatrix} \omega_1 R\, Q^{l} & -1 - 2\cdot & C \\ \dfrac{2\mu_1\omega_1 Q^{l}}{R} - \dfrac{\beta\rho_1}{\iota} & \dfrac{\beta\Gamma}{d(\iota+1)} - \dfrac{2}{R^2}[\mu_1(\iota-1) + \mu_0(\iota+2)] + \dfrac{\beta^{*2}\Gamma}{\beta d(\iota+1)} - \dfrac{\beta\rho_0}{\iota+1} & + \dfrac{2\mu_0 C}{R^2} \\ \mu_1 G & 2[\mu_1(\iota^2-1) - \mu_0(\iota+2)] & \mu_0 F \end{vmatrix} = 0$$

The resulting nonlinear equation has been solved numerically in the complex plane to determine the exact character of drop oscillations for arbitrary values of drop size, physical properties of the fluids, and interfacial tension.

High-Gradient Magnetic Field Separations

Latex beads of 0.1 μm diam have been captured by high-intensity, high-gradient magnetic fields that are located in a packed column of ferromagnetic spheres. In order to initiate capture, it was necessary to utilize a carrier solution containing $MnCl_2$ to increase the magnetic susceptibility of the beads in solution. Preliminary results with the iron-storage protein, ferritin, indicate that interactions suitable for separation may exist between the magnetic system and the protein.

Interactions of Solvents, Solutes, and Surfaces: Adsorption and Supercritical Extraction

Fundamental experimental and theoretical studies focus on relating macroscopic properties of mixtures to intermolecular interactions and molecular correlation functions for the highly asymmetric systems characteristic of extraction by supercritical fluids and adsorption from supercritical fluids in order to improve understanding of the molecular interactions and their influence on basic phenomena important in practical separation processes.

Experimental studies of solubility of transition metal fluorides in supercritical carbon tetrafluoride (CF_4) are under way. The choice of metal fluorides as solutes will substantially broaden the data base for supercritical solubility because virtually all previous data are for large organic solutes. The current solute is molybdenum hexafluoride (MoF_6), and future studies are planned for uranium hexafluoride (UF_6) and sulfur hexafluoride (SF_6). These solutes exhibit a range of volatility appropriate for our measurements, are considerably smaller in molecular size than previously studied solutes, and are of some importance in the nuclear energy programs of DOE. The molecular size should be of benefit in testing models because this parameter appears to be of primary importance, and the range of molecular size of previously studied solutes is rather narrow. Carbon tetrafluoride is an interesting solvent, not previously studied as a supercritical solvent, and, in its critical point properties, is appropriate for the chosen solutes.

Solubility of MoF_4 in supercritical CF_4 has been measured. This system provides information of an unusually dense solute with appropriate volatility. Measurements (see Fig. 4.17) have been made over a range of pressures from well below the critical point to pressures well above the critical point. Temperature has been varied over a more limited range of ~20 C°. The experimental apparatus has been substantially improved during the review period to ensure that maximum solute purity is maintained (see Fig. 4.18). All of the surfaces that could contact MoF_6 are treated with fluorine gas prior to experimentation. This effectively removes all water, hydrocarbons, and oxygen from the surfaces, preventing reaction and contamination of the MoF_6. The solute is transferred as a gas to a cooled extraction cell where it is deposited as a high-surface-area solid. The extraction cell is contained in an air bath with accurate temperature control. The supercritical CF_4 solvent passes through the extraction cell at the desired temperature, pressure, and flow rate. The solution is then flashed to atmospheric pressure, heated, and analyzed for its molybdenum content. A new mass spectrometer has been installed for efficient and accurate on-line analysis.

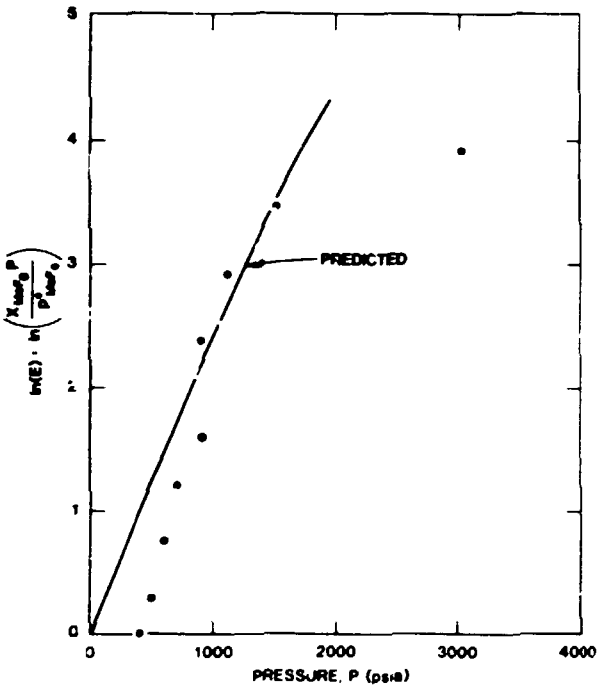


Fig. 4.17. Solubility of MoF_6 in CF_4 at 243 K.

Complementary theoretical studies based on distribution function theories from statistical mechanics are continuing; these efforts have already led to new models for solubility and other equilibrium properties. Through the formalism of the Kirkwood-Buff theory, bulk equilibrium properties are related via the Kirkwood fluctuation integral to the molecular distribution functions. One approach to modeling, applicable to dilute supercritical solutions, relates the solute-solvent fluctuation integral to that for the pure solvent through molecular solution approximations. The formalism for dealing with solute-solute interactions is available so that models for more concentrated solutions may be developed. This approach is also suitable for treating the effects of added cosolvent or entrainer, as may be used in practical separations. Other routes to relate molecular interactions to bulk properties that are being explored include scaled particle theory (particularly well-suited to modeling the effects of solute size and shape), integral equation theories (accurate integral equation theories for mixtures have recently been developed), and molecular

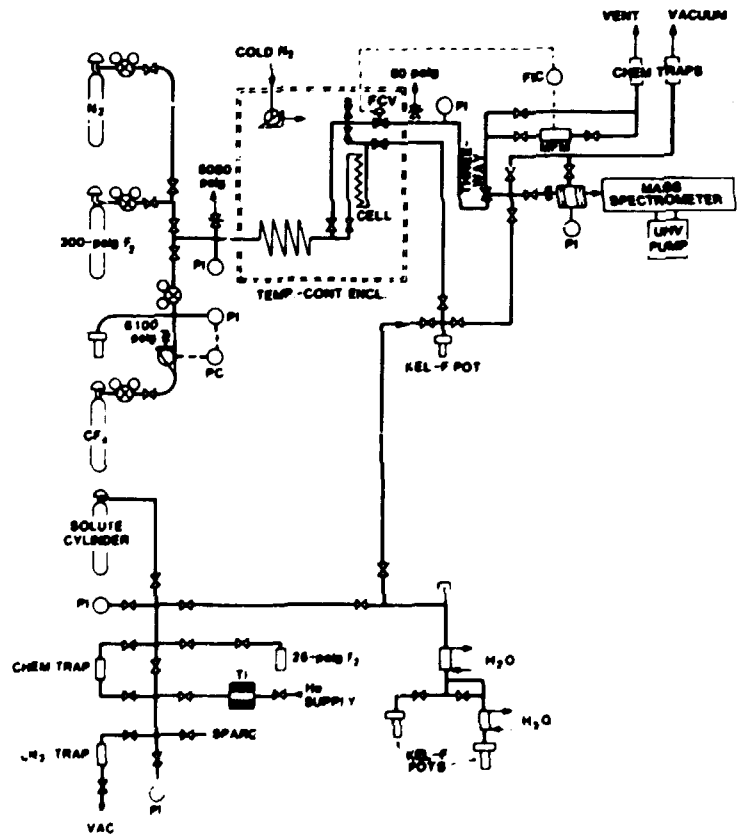


Fig. 4.18. Schematic drawing of supercritical solubility apparatus.

simulations (a grand canonical Monte Carlo code is being adapted for this application).

Further theoretical developments from our Kirkwood-Buff approach to understanding molecular interactions in supercritical solutions have been successfully tested.[5-8] This work has produced practical models that can predict the solubility and partial molar volume of solutes in supercritical fluids from fundamental molecular theory (e.g., Fig. 4.19). The theory provides the basis for planned models explaining the effect of increased solute concentration and solute-solute interactions which are also important in systems with added entrainers or cosolvents.

A related distribution function theory has been developed to better describe the effects of solute size and shape. This work employs scaled particle theory to determine reference state chemical potentials and then uses a Kirkwood coupling parameter technique.[9] Distribution functions have been obtained[10] from an accurate integral equation theory, which has been applied for the first time to supercritical solutions (e.g, Fig. 4.20). The robust and efficient algorithm of Labik has been adapted to make these theoretical calculations possible.

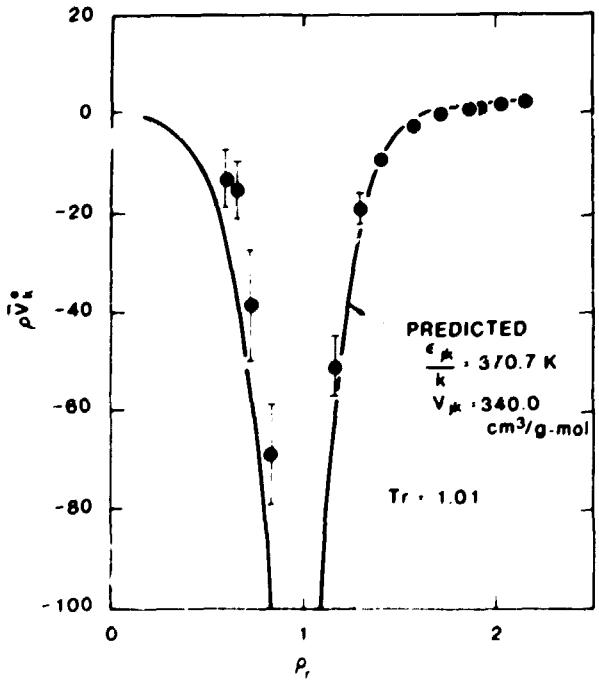


Fig. 4.19. Partial molar volume of naphtalene in supercritical CO_2 predicted by model.

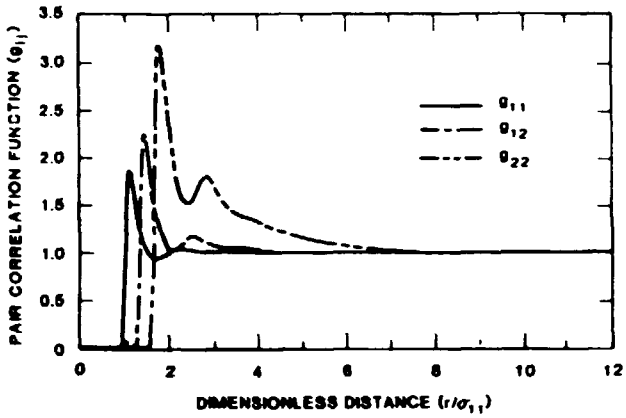


Fig. 4.20. Pair correlation functions of naphthalene in supercritical CO calculated by HMSA integral equation using Labik algorithm.

A new technique for measuring adsorption rates from supercritical mixtures, based on a surface acoustic wave transducer, is being evaluated. It has been shown that the frequency of surface acoustic waves on a piezoelectric substrate can be used as an extremely sensitive monitor for the mass adsorbed on the surface (100 to 1000X as sensitive as bulk wave devices). This suggests a technique for measuring the mass adsorbed that may be amenable to operation at elevated pressures (in contrast to almost all other surface physics techniques which operate only at vacuum conditions) and which may be used to measure rates of adsorption/desorption. Experiments are under way to test the feasibility of this experimental approach because of the opportunities for research that could be introduced by such a technique.

Experimental studies of interactions with adsorbed molecules have concentrated on testing the feasibility of a new and powerful experimental technique for direct, dynamic measurement of the mass of adsorbed gas on a surface.[11] After demonstrating that piezoelectric silica crystals could detect even small changes in the mass of adsorbed molecules by measuring the change in frequency of acoustic waves across the surface, we have designed a research apparatus (Fig. 4.21) suitable for quantitative measurement of adsorption phenomena. This has involved design of new electronic circuitry as well as an improved experimental adsorption cell with associated flow systems. Using this improved apparatus, the initial goal is to demonstrate that quantitative and reproducible

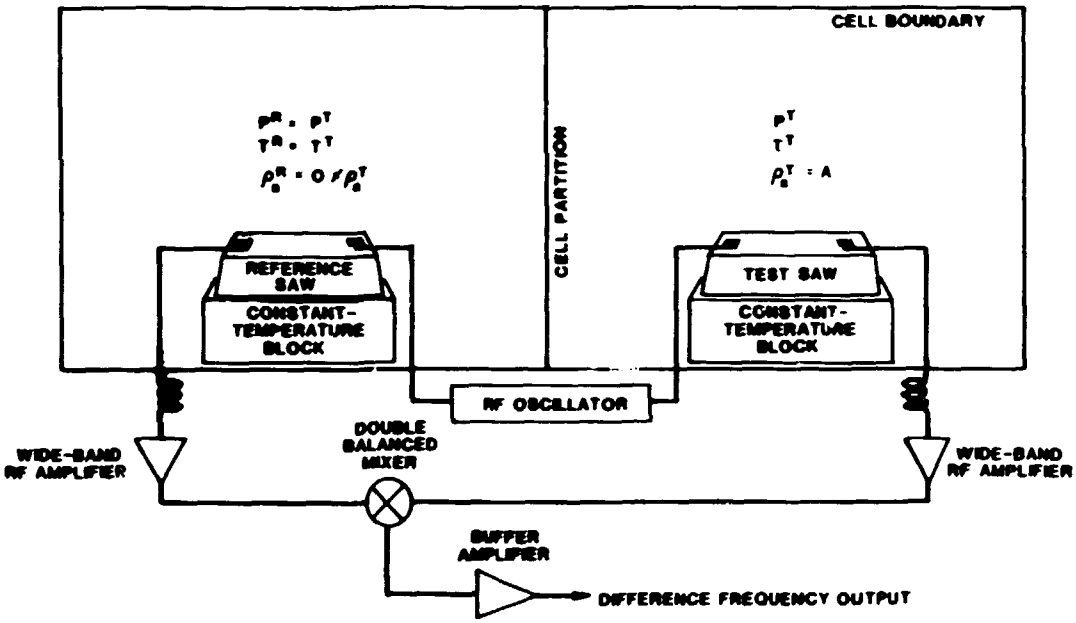


Fig. 4.21. Schematic drawing of SAW apparatus for adsorption dynamics.

static adsorption-desorption results can be obtained, first in vacuum conditions and then at high pressure, including necessary calibration factors for pressure and temperature effects. The second goal is to determine the capability of the approach to make dynamic measurements; transient measurements of periods <1 s appear attainable, and 1 ms may be achieved. If this apparatus realizes its potential, it should be very useful for measuring diffusion and mass transfer in supercritical solutions and as a detector for supercritical fluid chromatography.

Future experiments are under consideration for direct measurement of molecular correlation functions of supercritical mixtures using neutron scattering.

Multicomponent Separations by Continuous Annular Chromatography

The development of the continuous chromatograph at ORNL made possible industrial-scale separations that were heretofore performed only on a laboratory scale.[12–14] Applications are legion—from pharmaceuticals and bioseparations to the heavy metals industries. The new technology has been shown to offer excellent opportunities for energy conservation. Unlike most other separation techniques, this is a multicomponent separation concept: that is, several species of product are purified simultaneously, conserving space, energy, and costly equipment.

The program in continuous chromatography is divided into a research phase and a series of energy comparison and economic evaluation studies. The primary thrusts of the research program involve dilution reduction and capacity increases, as well as an expension of the method to more sectors of the process industries. Three specific projects are addressing these goals: (1) pilot-scale sugar separation studies with high loading, (2) continuous gradient elution chromatography, and (3) continuous displacement chromatography.

Pilot-Scale Sugar Separations

The feasibility of the separation of sucrose, glucose, and fructose in an aqueous solution by continuous chromatography was established earlier.[15–17] High-concentration mixtures (up to 650 g/L solids) were successfully separated, initially using a batch chromatograph and more recently using a bench-scale unit. The system used was the calcium form of Dowex 50W-X8 resin

(50-μm average diameter), with water as the eluent. A stainless steel pilot-scale continuous chromatograph (45 cm in OD, 100 cm deep) has been developed for optimization studies and scaling factor evaluations. Earlier fixed-bed and bench-scale experiments scaled almost perfectly to the pilot unit when loading was low and the feed mixture had low viscosity. Based upon these and other results, recommendations have been made for optimizing the scaling of the process. Using a feed band 72° wide, the separation shown in Fig. 4.22 may be anticipated.[18] An industrial-scale unit, 3 m in diameter and 1.25 m deep, will produce 22,500 kg/d of purified fructose. Work aimed at resolving angular dispersion caused by high-viscosity feeds is continuing, with help from an industrial concern.

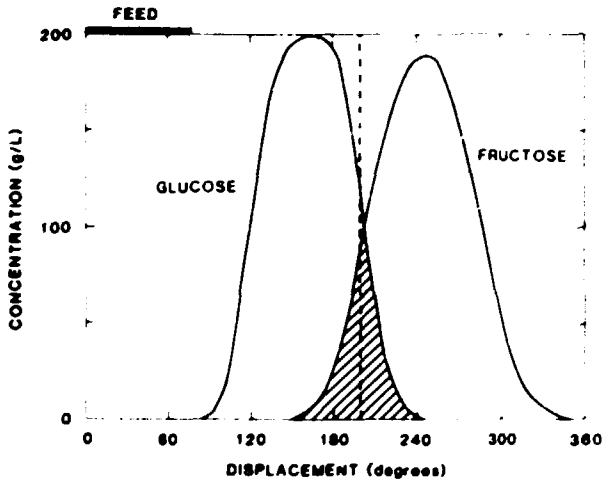


Fig. 4.22. High-loading sugar separation: feed, 300 g/L in each component; feed section, 72°; fructose, 90% recovered; concentration, 90 wt %.

Continuous Gradient Elution Chromatography

Typically, separation processes, including chromatography, dilute streams by a significant factor, leading to subsequent energy-intensive product reconcentration. Gradient elution involves the variation of the concentration of species in the eluent, with a goal of improving the separation. Many analytic separations use this technique to improve and, in some cases, to make possible some separations. These have not yet found their way into industrial practice. Continuous gradient elution chromatography is conceptually shown in Fig. 4.23. Our first application is to the separation of

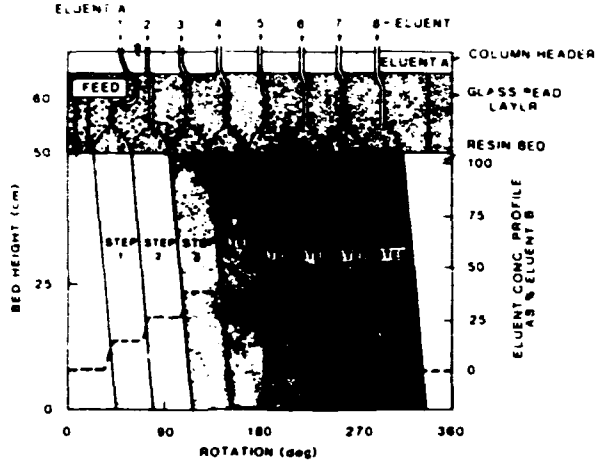


Fig. 4.23. Typical implementation of continuous gradient elution chromatography.

iron and chromium, using varying concentrations of ammonium sulfate as the continuous gradient eluent. The method selected provides an excellent example of the improvement in efficiency one can experience using this procedure.[19,20] In a typical comparison, dilution of the gradient elution run has almost been eliminated, while the conventional run is diluted by a factor of ~10. In an optimized case, it is possible to obtain exit concentrations above the feed concentration. Research is continuing in this area.

Continuous Displacement Chromatography

A second means of eliminating dilution is the use of displacement chromatography.[21] In this case, the process is significantly different in that the feed band is added until a significant portion of the bed's sites are taken up. Then, an eluent that replaced the feed components on the bed (displacer) is used to remove the feed components. Finally, the displacer is removed by a regenerator and the cycle resumes. As a sample system, we have adsorbed a mixture of amino acids on the acid form of Dowex 50-X8 resin and displaced it with 0.1 *N* NaOH. The regenerator is dilute H_2SO_4.

We have developed a concept for continuous displacement chromatography, for which a patent disclosure has been filed. It has been applied here to the separation of amino acids, but it also has wide applicability to metals and many other separations. The separation illustrated in Fig. 4.24 shows elimi-

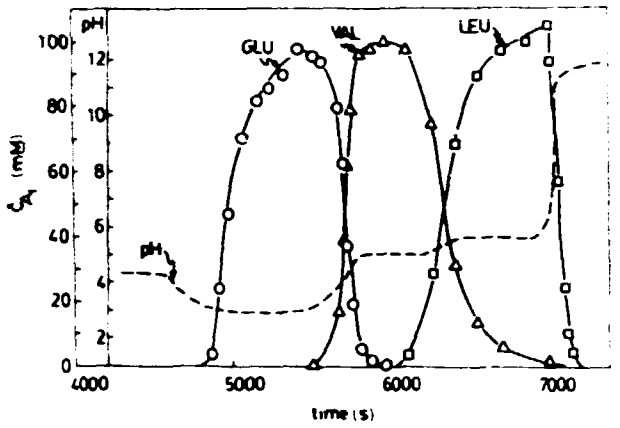


Fig. 4.24. Continuous displacement chromatography of amino acids.

nation of dilution; indeed, the product is *concentrated* by a factor of 6 over the feed. This represents a very significant energy saving. The optimization and scaling of this separation are being explored.

Dump Leaching Technology

Low-grade copper is recovered by dump and in situ leaching.[22] Large volumes of leach liquor are contacted with the dumped ore. For example, during operation at Kennecott's Bingham mine (Bingham, Utah), ~250,000 tons of waste rock are placed on the leaching dumps each day. Fifty million gallons of leach solution is applied daily, producing effluent copper solution grades of ~0.5 g/L. The copper waste going to leaching dumps is 0.2% copper, opposed to an average head grade of 0.6% copper. Copper is then removed from the leach liquor by solvent extraction in which an organic solvent (LIX) is used. The copper is subsequently desorbed into an aqueous phase and recovered as a pure metal by electrowinning. It is the solvent extraction unit that we are proposing to replace with annular chromatography.

Several advantages would accrue with this change: (1) no organics are involved, decreasing solvent costs and increasing electrowinning efficiency; (2) the equipment is less expensive, and there is a very significantly increased energy efficiency; and (3) crud formation is eliminated. Still of concern is the dilution, which is characteristic of both processes and the subject of the research efforts of this program. Recently, new ion-exchange resins have been developed that may be used in either gradient elution or continuous displacement mode. Although, to date, no direct experiments have been done on this technology, there is reason to believe that they will eliminate the dilution problem. Based on the new technology, preliminary plant design was proposed which indicated that one could build a CAC-based recovery unit with a throughput of 6000 gal/min for ~$12M, or 10% less than the equivalent solvent extraction unit. The savings realized with the new technology will yield an initial year's investment return of 16.2%. The major improvements that are realized in the electrowinning performance are difficult to evaluate but are definite positive features of the new technology.

Energy Savings

The savings analysis is being performed with the aid of the ASPEN process simulator, which allows ready access to a consistent set of costing factors. Our first task, which was recently completed, was the writing of a chromatograph costing module. More recently, an overall analysis of dump leaching of ore by conventional technology was put in to place. It is estimated that there is the potential for saving ~0.1 quad of energy in the copper industry as a whole, assuming that the metal values, currently not used, are recovered simultaneously by chromatography rather than by the conventional means currently practiced for these species. The use of gradient elution chromatography must be developed before these savings are feasible.

4.4 Materials Research

The DOE Office of Basic Energy Sciences (BES), through the Division of Materials Sciences, supports Chem Tech research programs that are concerned with therodynamics and kinetics of energy-related materials, as well as basic chemical engineering studies.

Thermodynamics and Kinetics of Energy-Related Materials

The objective of this program is measurement and interpretation of chemical thermodynamics and chemical kinetics in high-technology ceramic systems.

One major study in the field of structural ceramics concerns the kinetics of silicon nitride decomposition in the presence of graphite under controlled oxygen, nitrogen, and hydrogen potentials. This is being done from 1575 to 1975 K and is still in progress. The decomposition follows a first-order rate equation and seems relatively insensitive to the experimental atmosphere.

A second study under way concerns the thermodynamics of the Y-Ba-Cu-O system, with emphasis on the $YBa_2Cu_3O_{7-x}$ "1-2-3" superconducting phase. One primary result to date is the determination of the oxygen-to-metal ratio as a function of temperature and oxygen chemical potential. A second result is a chemical thermodynamic representation of these data.

Previous experimental work on the $U_{1-z}Gd_zO_{2\pm x}$ phase has been completed and has been published.[23] Here, the extensive nonstoichiometry in the $U_{1-z}Gd_zO_{2\pm x}$ phase was investigated experimentally and the data represented by a chemical thermodynamic method. The experimental ranges of temperature, oxygen potential, and z were 1273 to 1773 K, 0 to −600 kJ/mol, and 0.1 to 0.8, respectively. For hypostoichiometry, ideal-solution thermodynamics for the equilibrium $3Gd_{4/3}O_2 + 4UO_2 + O_2 = 6U_{2/3}Gd_{2/3}O_{8/3}$ were used to represent the experimental data, while for hyperstoichiometry, a nonideal solution was used for the equilibrium $4UO_2 + O_2 = 2U_2O_5$. The wide ranges in x and z led to an improvement of the previous analysis of literature data and led to partial molal Gibbs free energy values that are useful for any thermodynamic calculation involving the phase.

Nucleation, Growth, and Transport Phenomena

Two interdependent research programs in the Chemical Technology Division utilize laser-light-scattering spectroscopy to investigate the phenomena of homogeneous precipitation, coagulative nucleation, and particle growth. The first is a series of fundamental studies of nucleation and growth in systems that are of interest as precursor materials in ultrafine processing for a new generation of ceramic materials. The second area of interest is the measurement and theoretical interpretation of transport properties of fluids in the critical region. The transport properties, such as viscosity and diffusion coefficient, provide important clues to the behavior of the fluid media in which the precipitation occurs.

Particle Growth Kinetics in the Synthesis of Pure Component and Composite Ceramic Powders by Metal Alkoxide Hydrolysis

The synthesis of powders that consist of high-purity metal oxide particles is of primary interest in the development of new ceramics and in the improvement of existing ceramics. Furthermore, the shape, size, degree of monodispersity, and, in the case of composite powders, the homogeneity within these particles are found to significantly affect the microstructure of the final ceramic material.

The hydrolysis of metal alkoxides at low temperatures is an attractive method for synthesizing high-purity monodisperse metal oxide particles.[24] This method has been successfully used to synthesize monodisperse particles of silica, titania, zirconia, and composite materials.[24−27] The parameters that affect the monodispersity and morphology of the final powder include the reactant concentrations (i.e., water, alkoxide, and catalyst), temperature, solvent, and type of alkoxide group that is attached to the central metal. Several studies have been conducted that determined the overall effect of these parameters on the characteristics of the particles. However, there remains a need for additional fundamental studies on the effect of important parameters on the formation of these particles. These fundamental studies will result in a better understanding of the effects that diffusion and statistical interactions have on the early stages of nucleation and particle growth. The ultimate goal is to eventually model, from microscopic properties and Brownian coagulation, the formation of submicron particles by metal alkoxide hydrolysis.

This program has included several series of fundamental investigations of homogeneous nucleation, coagulative nucleation, and growth in synthesis of ceramic powders. The hydrolysis of metal alkoxides and subsequent condensation reactions result in a precipitation of oxide powders, such as silica, titania, and zirconia, and titania-zirconia and yttria-zirconia composite powders. These are studied in a variable-angle laser-light-scattering facility, using a thermally controlled quartz cell as a crystallization vessel. Rapid, nonintrusive moni-

toring of particle growth in the 0.01- to 1-μm range is routinely achieved at a scattering angle of 90°. A study of the particle growth and powder characteristics of metal oxides indicates that a major contributing factor which determines the ultimate particle size and morphology is the nature of the solvent used as a medium for the reaction. Metal oxide particles were synthesized by the hydrolysis of titanium ethoxide (TIE), zirconium butoxide (ZBB), and yttrium isopropoxide (YIP) in ethanol, 1-butanol, 2-butanol, and *t*-butanol solvents.

Before evaluating particle growth kinetics, it is important to study the alcoholysis (i.e., replacement of alkoxy groups by the host medium) and hydrolysis kinetics. Fourier transform infrared spectroscopic measurements have shown that alcoholysis occurs rapidly in the case of titanium ethoxide in *t*-butanol. Other systems are being investigated. It is also found that the hydrolysis reactions are rapid, and particle growth is controlled by the rate of precipitation of the hydrolysis products.

Particle growth from 0.01 to 0.9 μm is monitored by dynamic laser-light scattering. Figure 4.25 shows typical particle growth behavior in normal and branched chained alcohol solvents. A linear growth behavior (i.e., constant growth rate) is observed for particles grown in normal alcohols, while particles that are synthesized in branched alcohols showed an increase in the rate of particle growth with time. Futhermore, the plots show that the first observable particle size (i.e., within the first tens of seconds of nucleation) is an order of magnitude smaller in the branched alcohol (~0.01 μm) than in the normal alcohols (~0.2 μm). Tramission Electron Microscopy (TEM) analysis showed that particles grown in normal alcohols were more monodisperse and exhibited a more uniform spherical shape than those particles produced in branched alcohols.

Based on thermodynamic considerations and chemical analysis, the first observable particle sizes in normal and branched alcohols are larger than the theoretical critical nuclei by one to two orders of magnitude. It is postulated that during this initial period (within the first tens of seconds of nucleation), the very small nuclei are not stable and tend to flocculate very rapidly. The nuclei continue to aggregate until larger and more stable colloids are formed. These stable colloids may continue to grow by collecting smaller, less stable

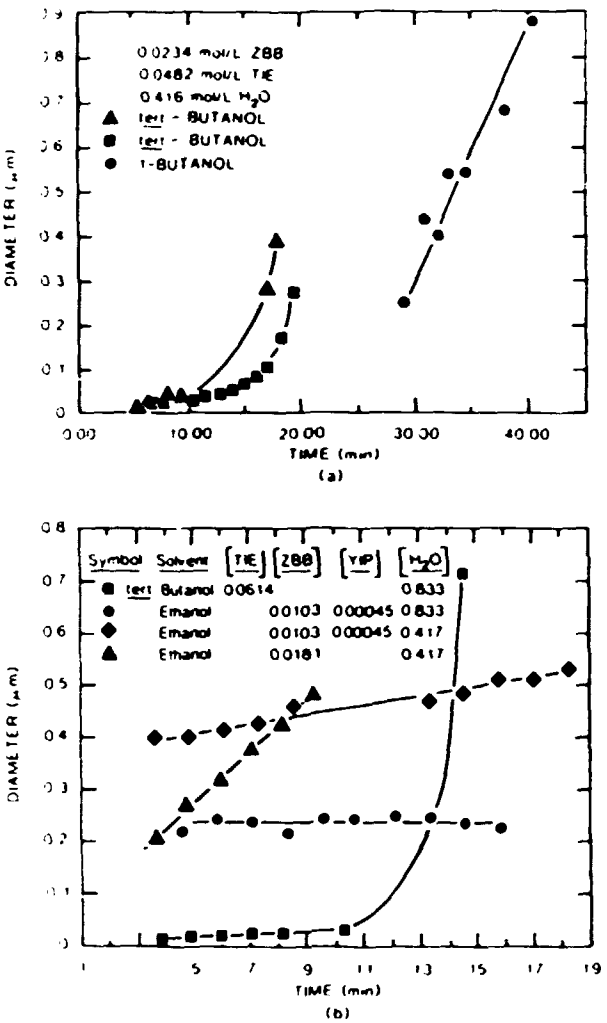


Fig. 4.25. The effects of solvent and [H_2O] on particle growth: (a) growth of "titania-zirconia" particles in 1-butanol and *tert*-butyl alcohol, and (b) growth of "titania," "zirconia-yttria," and "zirconia" particles in ethanol and *tert*-butyl alcohol.

nuclei and/or by monomer addition. A nucleation/coagulation model has, therefore, been developed to simulate continuous nucleation with coagulation (i.e., coagulative nucleation) during the first tens of seconds of the precipitation process. The model assumes that electric double-layer repulsive forces (DLVO theory) and van der Waals attraction forces determine the coagulation efficiency of the coagulating particles. Preliminary results have suggested that under certain circumstances, monodisperse particles may be formed by a coagulative nucleation mechanism. Additional simulations are needed to better define conditions where monodispersity and polydispersity are

achieved and to compare theoretical values with experimental data.

As an extension to the above research on the effect of solvent on particle morphology, samples of the 1-2-3 (Y-Ba-Cu) superconductive precursor powders were synthesized at room temperature using alkoxide hydrolysis in an organic salt matrix. A very fine powder was produced, which, according to TEM analysis, had a characteristic size of ~0.01 μm. Further processing of the powder resulted in a superconductive material. Several batches were made to show the reproducibility of the powder.

Transport Properties of Fluids at Extreme Conditions

This program applies dynamic laser-light scattering to the study of high-temperature and/or high-pressure fluid properties. Small sample volumes (<5 mL) are required, which simplifies the temperature and pressure control. Dynamic light-scattering techniques permit the extraction of information concerning the physical properties of the liquid containing a dilute suspension of colloidal particles. The mobility of the colloidal species is characterized as a diffusion coefficient, while an application of the Stokes-Einstein relationship to the model colloid system allows calculation of the fluid's viscosity. The present work focused on measuring the viscosities of pure alcohols and hydrocarbons as well as of binary mixtures of these compounds. The viscosities were measured from room temperature to the critical region using laser-light scattering. Specially prepared monodisperse submicron silica particles were developed and used to achieve suspension stability into the critical region. The viscosity at low temperatures was verified by a Ubbelohde viscometer.

Figure 4.26 shows the viscosity of ethanol at temperatures ranging from 20 to 230°C.[28] The critical point for ethanol is 243°C. The data agree very well with those of the Ubbelohde viscometer at low temperatures and with literature data at higher temperatures. An attempt to fit the data with an empirical Andrade correlation proved inadequate for the wide temperature range of the study. Therefore, the correlation shown in Fig. 4.27 includes only the data below the normal boiling point. The curvature of the relationship near the critical point indicates that a more complex relationship is necessary to describe the data. Reid, Sherwood, and Prausnitz[29] recommend a three-constant equation of the Antoine equation form; however, this correlation gave physically unreasonable constants. The limited success encountered in predicting liquid viscosity over the temperature range between the melting point and the critical point is the direct result of inadequacies in the theory of the liquid state. Figure 4.27 shows the viscosity of a mixture of ethanol (x=0.8) in heptane ranging from 20 to 220°C.[28] The Andrade equation gave a good fit over the entire temperature range.

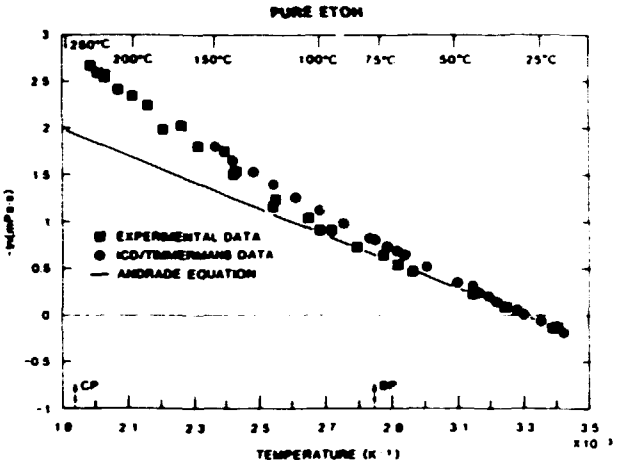


Fig. 4.26. Viscosity of pure ethanol for the temperature range 20 to 230°C.

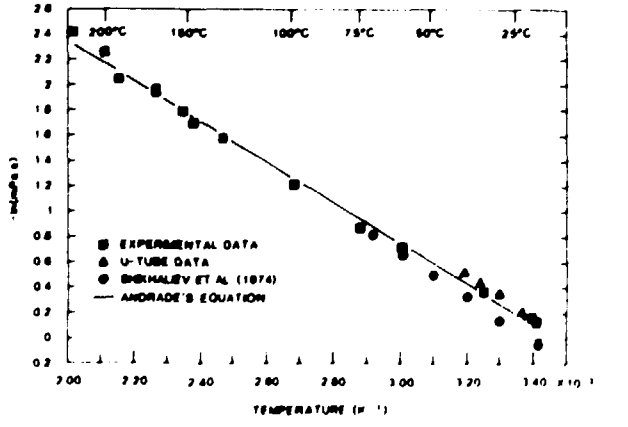


Fig. 4.27. Mixture viscosity data for a typical ethanol (x=0.8) – n-heptane mixture over the temperature range of the study.

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5. Nuclear Regulatory Commission Programs

The U.S. Nuclear Regulatory Commission (NRC) programs in Chem Tech are focused 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.

5.1 Nuclear Reactor Safety

Current NRC reactor safety programs emphasize research mechanisms and pathways by which fission products are released to the environment from failed fuel elements under hypothetical reactor accident conditions. The following sections discuss programs for the investigation of fission product release from LWR fuel, postaccident iodine and tellurium chemistry, the evaluation of iodine transport from a failed steam generator tube, and support for the LWR Aerosol Containment Experiment (LACE).

Fission Product Release from LWR Fuel under Accident Conditions

In the continuing study of the release and behavior of fission products under severe accident conditions, specimens of LWR reactor fuel have been heated to very high temperatures in steam. These tests, which are sponsored by the NRC, have provided release data for the more volatile fission products as functions of the principal variables—temperature, steam flow rate, fuel type, time, and fraction of fission products remaining in the fuel. Such information is needed as input for computer programs that analyze the accident sequences and calculate the consequences of hypothetical reactor accidents. Reviews of the available data and assessments of the needs for additional information have been published by the NRC.[1,2] Earlier work at ORNL has been reported, but recent experiments in a revised test apparatus have extended the range of our test conditions.[3-8]

The tests have been conducted in the hot cell facility illustrated in Fig. 5.1, which includes an induction furnace capable of operation in steam and/or hydrogen at temperatures up to 2700 K. Three parallel trains of fission product collectors, each of which included a thermal gradient tube, filters, and both heated and cooled charcoal, were used to isolate different fission product forms for subsequent analysis. The inventories of the gamma-emitting fission products in the fuel specimens were determined, before and after testing, by

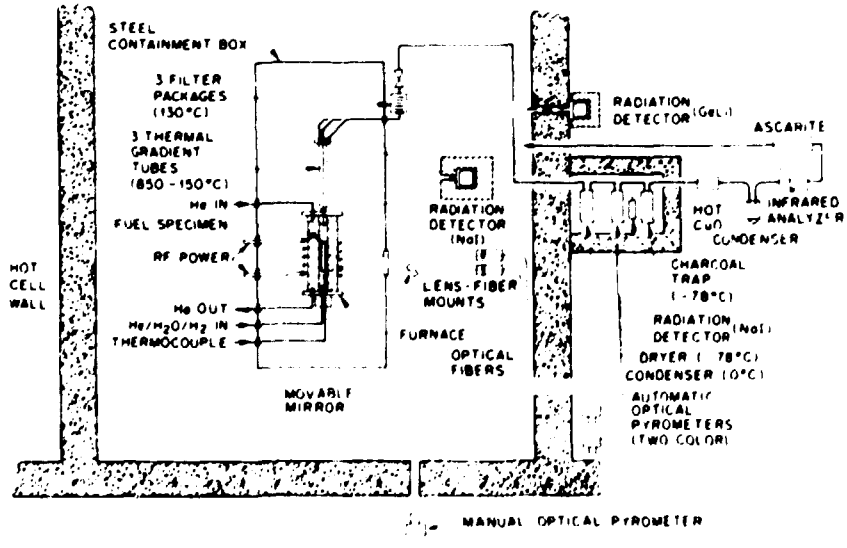

Fig. 5.1. Vertical fission product release apparatus.

gamma spectrometry, and the inventories of other fission products were calculated by ORIGEN2.[9] The accumulations of ^{85}Kr and ^{137}Cs were monitored at 1-min intervals during the test, and the deposits on all apparatus components were measured after the test by various methods: gamma and mass spectrometry, neutron activation, weight, and scanning electron microscopy.

The irradiation history of the fuel was found to significantly affect release behavior. In 20-min tests in flowing steam, the fractional releases of krypton and cesium (which were similar in most tests) varied from 20–50% at 2000 K to 40–70% at 2300 K. In a single test at 2700 K, 99% of the krypton and cesium was released. Also, varying fractional releases for many other fission products have been observed. The available data indicate that the fractional releases for Br, I, and Rb have been similar to those for the more easily measured krypton and cesium. Some of the other fission product elements, such as tellurium and antimony, appear to be readily released from the UO_2 but are effectively retained in the Zircaloy cladding until it becomes almost completely oxidized. Large fractions (up to 30%) of the silver have been released in some tests, and it appears to be transported as aerosol. Also, very small releases (<1%) of the Mo, Ru, Ba, Ce, and Eu have been detected; these elements are expected to become more significant in future higher-temperature tests.

Comparison of the release rates for cesium, based on the fraction remaining in the fuel, showed a general decline with time at constant temperature. In a test at 2300 K, the release rate for cesium varied from >0.05 min^{-1} initially to <0.004 min^{-1} after 60 min, as indicated in Fig. 5.2.[10] Release rates at 2000 K were much lower and varied with fuel type, but declined with time. At 2700 K, the release of cesium was too rapid for accurate measurement of the rates as a function of time, but a similar decrease in rate was apparent. This varying behavior with fuels of different irradiation histories suggests that a difference in fuel-fission product morphology affects the release path in subsequent temperature transients.

In the analysis of our release data, classical single-gas atom diffusion, as in the Booth diffusion model, has been shown to apply reasonably well.[11,12] This treatment appears to fit our data much better than the NRC-developed CORSOR-M model, which has been used widely in reactor safety analysis.[13] The irradiation and test conditions of the fuel specimens suggest that the release behavior of the volatile species should correspond closely to the type IIB diffusion described by Matzke.[14] At temperatures of 2000 K or greater, we calculate effective diffusion coefficients for both krypton and cesium that agree well with the Matzke IIB criteria. At lower temperatures, however, the effective diffusion coefficients diverge,

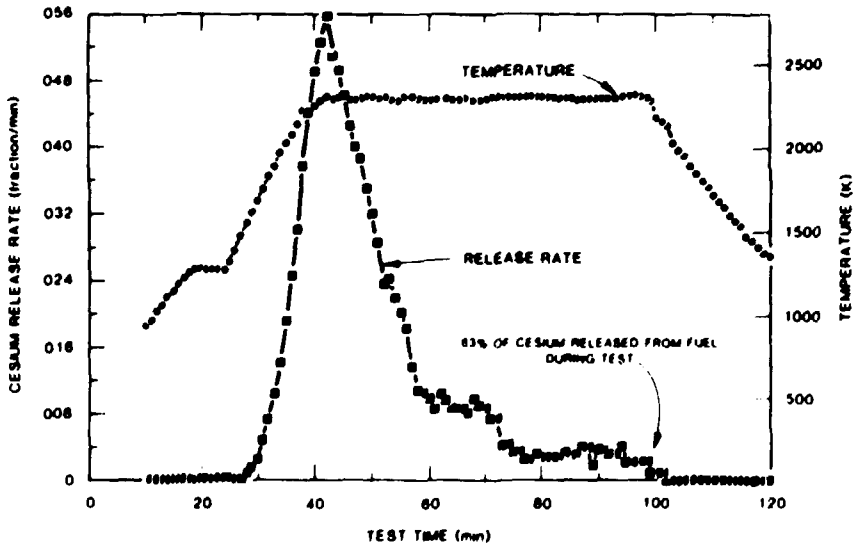

Fig. 5.2. Cesium release rate in test VI-2.

with a tendency toward higher-than-predicted values, probably because of more rapid release occurring from grain boundaries and edges.

Postaccident Fission Product Chemistry

Assessment of the chemistry and mass transport of iodine in containment during severe accidents in LWRs is an important component in determining the "source term," or fission product release from containment.

The TRENDS code for calculation of iodine behavior and release in containment is a system of FORTRAN routines whose objective is the quantitative determination of fission product transport and retention characteristics during severe reactor accidents. It represents a best estimate of nuclide behavior under the hypothetical conditions of various severe accident sequences.

Every attempt has been made to account for all significant processes. Reaction rate constants for iodine hydrolysis and radiolysis were obtained by a variable algorithm that gives values closely modeling our experimental data.

Five iodine species are explicitly considered, as shown in the first column of Table 5.1. The phases in which each form can exist are also listed. The release of iodine from either the reactor coolant system or fuel rubble can occur in any of the species listed. Subsequent interconversions between different species are modeled as follows:

$$I_2(g) \leftrightarrow CH_3I(g), \quad (1a)$$

$$I^-(aq) \leftrightarrow CH_3I(aq), \quad (1b)$$

$$I^-(aq) \leftrightarrow I_2(aq), \quad (1c)$$

$$I^-(aq) \leftrightarrow AgI(s), \quad (1d)$$

$$I^-(aq) \rightarrow AgI(s), \quad (1e)$$

$$CH_3I(aq) \rightarrow AgI(s). \quad (1f)$$

Table 5.1. Iodine chemical forms

Species	Phases considered
I_2	Gas, aerosol, liquid, surfaces (paint, steel, concrete), deposited aerosol
CH_3I	Gas, liquid, surface (paint)
CsI or I	Gas, aerosol, liquid, surfaces, deposited aerosol
HI	Gas, aerosol, surface (steel), deposited aerosol
AgI	Precipitate

TRENDS output provides the distribution of iodine in containment and release from containment as a function of time during a severe accident sequence. Initial calculations with TRENDS have shown that the amount of volatile iodine released from containment is sensitive to the value of the liquid-gas (evaporation) mass transport coefficient for I_2.

We are now in the process of making TRENDS "portable" so that it may be run outside of ORNL. To accomplish this, we are installing routines to internally calculate iodine hydrolysis, iodine radiolysis, and solution pH during a severe accident sequence.

Experimental Determination of Iodine Transport from a Failed Steam Generator Tube

Pressurized water reactors are designed to withstand certain postulated accidents, called design-basis accidents. One such accident would be the release of radioactive fission products to the environment through a path opened by a rupture of one tube in the steam generator. The release doses depend directly on the concentration levels of fission products in the primary coolant; therefore, these levels are kept below those that could result in exceeding the allowable release doses given in the regulatory document 10 CFR 100.

Measurements of iodine speciation in aqueous solution at 285°C and 1000 psig (6.9 MPa) show a higher percentage of I_2 in solution at tracer concentrations than at higher concentrations. A 0.1 *M* I^- solution resulted in 2% I_2, whereas 0.1 m*M* I^- solution had only 0.1% I_2. These tests indicate that iodine at tracer concentrations can have a species distribution in solution that is not expected or observed at higher concentrations.

Partition coefficients are a convenient way of expressing volatility. They can be given in terms of concentrations per unit volume or in terms of mass. In this work, the iodine partition coefficient (PC) is defined as

$$PC = \frac{\text{concentration of iodine species in aqueous solution}}{\text{concentration of iodine species in gas}}.$$

Table 5.2 gives the results of simulated steam generator tests in terms of PC and the percentage of iodine in aqueous solution as I_2 and organic iodide. Hydrolysis of I_2 and oxidation of I^- are the

Table 5.2. Summary of steam generator iodine experiments

Conditions: 286°C, 1000 psi, 0.2 *M* borate, 1.0E-9 *M* I^-

Atmosphere	pH at 25°C	% I_2 in liquid	% Organic I in liquid	Partition coefficient
Argon	5	2.04	0.11	687
	7	0.44	0.07	5,180
	9	0.02	0.00	47,500
Air	5	22.00	3.95	350
	7	1.20	0.15	888
	9	0.12	0.01	7,160

two processes that may be cited as important in determining the iodine speciation and thereby the partition coefficients in these tests. Results given in Table 5.2 show that, in either argon/steam or air/steam, tests run at pH 9 gave a lower I_2 percentage than those run at pH 5. The partition coefficients at the higher pHs were higher than those at low pH, and this is the behavior that would be expected in solutions with a lower percentage of I_2.

LACE Aerosol Code-Comparison Project

The LWR Aerosol Containment Experiments (LACE) were performed at the Westinghouse Hanford Engineering Development Laboratory under the leadership of an overall project board and the Electric Power Research Institute. These tests were performed to investigate, at large scale, aerosol retention behavior in containment under simulated severe LWR accident conditions. As part of this project, an aerosol code-comparison exercise was performed under ORNL guidance. In this exercise, pretest and blind posttest aerosol transport code calculations were performed by LACE participants to model LACE tests LA1, LA2, LA3, LA4, and LA6. The pretest code calculations were performed to permit code-to-code comparisons; the blind posttest calculations were performed to permit comparisons of code results with the test data.

Table 5.3 lists the 16 aerosol transport codes used in this study and the six countries that used them. A major result of the pretest calculations performed to model tests LA1 through LA4 was the discovery of a number of coding errors, which were corrected. The improved coding methods were used to calculate the aerosol size distribution parameters such as the aerodynamic mass-median diameter (AMMD) and the geometric standard deviation (GSD).

Table 5.3. Aerosol transport codes used in the LACE aerosol code-comparison exercise

Aerosol code	Country
AEROSIM-M	United Kingdom
AUX 2.9	Sweden
CONTAIN	United Kingdom, United States
HAA4	United States
MAAP.3	Sweden
MCT-2	United States
NAUA-4	United States
NAUA-5	Finland, Italy
NAUA4-HYGROS	Finland, United States
QUICK-M	United States
RAFT	Finland
REMOVAL/2G	Japan
RETAIN-2C	Finland
SWNAUA-HYGRO	United States
TRAP-MELT2	Italy, Japan, United Kingdom, United States
TRAP-MELT2.2	United States

Tests LA1 and LA3 (LA3 consisted of three tests noted as LA3A, LA3B, and LA3C) evaluated aerosol transport through a 0.063-m-diam, ~30-m-long test pipe that had six 90° bends in it. Variables in the LA1 and LA3 tests included gas-flow velocities through the test pipe and the mass ratio of the MnO/CsOH aerosol source used in the tests. Comparisons with measured aerosol deposition in the pipe and data for deposited MnO/CsOH mass ratio data illustrated the importance of correct modeling of bend deposition and the potential influence of aerosol resuspension on the test results.

Tests LA2, LA4, and LA6 investigated aerosol behavior in the 852-m^3 Containment System Test Facility vessel under saturated-steam conditions and for different vessel leakage rates and histories. Comparisons of airborne aerosol concentrations, aerosol mass leaked, airborne MnO/CsOH mass ratio, and AMMD and GSD data were made. The comparative results illustrated the importance of correctly modeling the "hydroscopic" effect of steam on the growth of the CsOH aerosol and showed that the codes did not do an adequate job of predicting the "multicomponent" aerosol behavior observed in the tests.

As a result of the code-comparison project, the codes and the ability of the code users to correctly apply the codes were significantly improved.

5.2 High-Level-Waste Repository Licensing Program

The objectives of this activity are to provide the NRC with technical assistance in

- reviewing the geochemical data, information, plans, and conclusions relative to DOE high-level-waste repository site characterization activities or repository design;
- reviewing the DOE geochemical research and development programs related to site characterization activities or repository design; and
- identifying the geochemical information needed and the sensitivity of the resulting application or use of the data and information to the precision and accuracy of the information.

The requirements of the NRC regulation 10 CFR 60, Subpart E, deal with a number of geochemical conditions that need to be considered in order to provide reasonable assurance of satisfactory waste isolation. For example, the long-term performance of a high-level-waste repository is affected by the geochemical environment of the site and the changing geochemical conditions near the waste packages in the engineered facility. In addition, the rates at which radionuclides are transported from a repository to the accessible environment depend on the rates of groundwater flow and flux as well as on how much the rates are retarded by geochemical reactions. Thus, geochemical properties and processes are some of the most important components of the natural and engineered systems that will be required to contain and isolate radionuclides from the accessible environment. Often, new and unique problems must be addressed in the NRC evaluations and analyses. This project provides detailed, state-of-the-art support to the NRC staff in geochemical-related subjects concerning the DOE geologic repository program.

Assistance has been provided to the NRC to support their evaluation of geochemical information about the DOE site characterization and repository development activities. Assistance has included: (1) review of technical documents, including environmental assessments; (2) participation in site visits and data reviews; (3) analysis of the site characterization report; (4) organization of technical workshops; and (5) preparation of technical positions. Assistance has been provided at the direction of the NRC project manager, and reporting has consisted of journal articles, NUREG/CR reports, informal letter reports, letters, workshops, and consultations.

A major task during this report period has been the preparation of a draft NUREG/CR report entitled *Geochemistry Issues for the Yucca Mountain Candidate High-Level Waste Repository Site.* It describes geochemical technical issues for this site being characterized by DOE. These issues are derived from DOE guidelines, NRC rules, and EPA standards that indicate a need for geochemical information relevant to site characterization and selection and construction, operation, and closure of a repository. The ambient geochemical conditions and processes and the changes that may occur over time are analyzed, as are geochemical conditions and processes that may help determine the radionuclide source term at the boundary of the waste package or engineered barrier system and that may control radionuclide transport and release. The report identifies (1) the regulatory rationale for the geochemical issues; (2) the data and information needed to analyze the issues; (3) the methods, procedures, and approaches for obtaining the needed data and information; and (4) the uncertainties associated with the methods and strategies.

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6. Administrative Resources and Facilities

Chem Tech's facilities range across three plant sites and include numerous buildings as well as advanced equipment. Some of these facilities have been improved as a result of General Plant Projects and through the efforts of the Engineering Division.

6.1 Facilities

As one of the largest and most diverse divisions of ORNL, Chem Tech operates research programs in a number of buildings at the ORNL site and has recently occupied additional space at the Oak Ridge Gaseous Diffusion Plant (ORGDP) site, principally for activities in the Waste Management Technology Center. Chem Tech's facilities consist largely of general-purpose laboratory and office space, but some facilities are unique or rare. The Radiochemical Engineering Development Center (REDC) contains some of the most advanced radiochemical "hot cells" capable of containing alpha activity while providing shielding from gamma rays.

The Division operates two user facilities used for tests by scientists and engineers from other government, industrial, or academic organizations. In the Drop Test Facility, the ability of shielded shipment containers to survive transportation accidents can be tested, and in the Bioprocessing User Facility, a unique variety of bioprocessing equipment has been assembled. Chem Tech also plays a dominant role in the Waste Management Technology Center, where outside organizations can test and demonstrate innovative approaches and equipment for treatment of a variety of wastes and waste forms.

The Division's fundamental research programs also utilize advanced equipment. As part of the effort to keep its research and development (R&D) among the best in the world, Chem Tech strives for laboratories and equipment that are well maintained and are at or near state-of-the-art technology.

Most of the buildings are more than 30 years old, and some of them have undergone one or more stages of modification or renovation. Some facilities no longer meet the needs of the Division, and efforts are being made to decommission them or convert them to types needed for current programs. Still in the planning stages, the most notable change is the placing of Building 3019 into standby. This building has served as our principal facility for pilot plant studies in fuel reprocessing. With the completion of most R&D in this area, it has become appropriate to consolidate the remaining related studies in the transuranium processing facilities (REDC) and thus reduce the costs associated with utilities and maintenance of a large and underused facility. A number of similar renovation efforts are expected in coming years as more of our aging facilities become either uneconomical or ineffective for Chem Tech's newer activities.

New or upgraded facilities will be needed to replace those which must be removed from service. Even structurally sound facilities often serve inadequately, either because they were originally designed for other uses or because of degradation through use (such as corrosion of drain pipes). The increased activity in waste management and handling of hazardous materials is increasing the need for facilities capable of testing large equipment and providing adequate containment to protect both the environment and personnel working with the equipment.

6.2 Capital Projects Involving Engineering Division Assistance

From January 1, 1987, to June 30, 1988, Chem Tech participated in seven General Plant Projects (GPPs), which provide capital improvements. Progress on these was at various stages ranging from study and estimate, to design criteria, to design, through various stages of construction, and to completion. The amount of allocated capital funds involved for all these GPPs was ~$2,750,000. The completed projects were accomplished safely, in a timely manner, within budget, and in such a way that the conceptual design and system requirements were satisfied. Services and facilities obtained by these GPPs included renovation and expansion of normal and emergency electrical circuits, upgrade of ventilation systems, and replacement of low-level-waste drains in critical facilities.

Also, old facilities were demolished, cleaned up, and renovated to produce contemporary engineering laboratory spaces (Table 6.1). During the same time frame, numerous work orders (10 to 20) were processed through the Engineering Division to obtain engineering expertise for expense-funded projects totaling between $1,000,000 and $2,000,000.

Chem Tech enjoys a unique relationship with the Engineering Division. A staff engineer is assigned as a liaison to the Engineering Division, thus directly representing Chem Tech's interests and ensuring that the Division acquires from Engineering, in a timely fashion, the services (estimates, design, fabrication, and construction) that it requests according to appropriate ORNL and DOE health, safety, and environmental regulations and in compliance with all relevant quality and engineering standards. Acquisition of Engineering Division expertise becomes increasingly important in the contemporary atmosphere of "critical facilities" that must be operated in compliance with ever-increasing stringent nuclear quality assurance Contemporary Configuration Management and Engineering Design Criteria to avoid potential health, safety, or environmental insults.

Table 6.1. Capital projects for Chem Tech

Budget year (FY)	Project title	Cost of construction ($)		Completion date	
		Estimated	Actual	Scheduled	Actual
1989	Install operational and heating, ventilating, and air-conditioning improvements, Building 4501	850,000	In design	5/90	Unknown
1987	Replace ductwork, West Roof, Building 3019	520,000	498,500	12/87	5/88
1987	Upgrade utilities, Laboratory 16, Building 4501	141,000	In design	11/88	Unknown
1986	Upgrade emergency power, Radiochemical Processing Plant	176,000	176,000	11/86	8/87
1986	Update egress controls, Building 7930	126,000	126,000	12/86	4/87
1985	Renovate process and low-level-waste drains, Radiochemical Processing Plant	750,000	701,000	11/87	5/88
1989	Filter house over Building 3108	200,000	In design	Unknown	Unknown

Administrative Summary

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Berry, J. B., McNeese, L. E., Rohwer, P. S., Youngblood, E. L., Hill, L. G., Myrick, T. E., Smith, J. H., Hollenbeck, P. E., Pudelek, R. E., and Yook, H. R., *Preliminary ORNL Long-Range Environmental Management Plan—Program Overview and Summary*, ORNL-6344, July, 1987

Patents

E. Greenbaum

"Method of Producing Metallized Chloroplasts and Use Thereof in the Photochemical Production of Hydrogen and Oxygen," U.S. Patent 4,657,646 (April 14, 1987)

V. L. Fowler, M. H. Lloyd, and P. A. Haas

"Preparation of Nuclear Fuel Spheres by Flotation-Internal Gelation," U.S. Patent 4,663,093 (May 5, 1987)

C. W. Forsberg

"Boiling Water Neutronic Reactor Incorporating a Process Inherent Safety Design," U.S. Patent 4,666,654 (May 19, 1987)

C. D. Scott

"Method and Apparatus for Continuous Annular Electrochromatography," U.S. Patent 4,683,042 (July 28, 1987)

C. A. Burtis, W. F. Johnson, and W. A. Walker

"Method and Apparatus for Automated Processing and Aliquoting of Whole Blood Samples for Analysis in a Centrifugal Fast Analyzer," U.S. Patent 4,740,472 (April 26, 1988)

Awards, Honors, and Recognitions

H. L. Adair

1986–1990 President of the International Nuclear Target Development Society (INTDS)

R. T. Barnett

1987 Tennessee Society of Professional Engineers, Oak Ridge Section, Outstanding Engineering Achievement Award, Consolidated Edison Uranium Solidification Program

E. C. Beahm

1987 Technical Communication Award of Merit (Third Place) in Technical Reports Category, East Tennessee Chapter of the Society for Technical Communication

J. T. Bell

1988 Co-Chairman, Fifth International Symposium on Separations Science and Technology for Energy Applications

L. H. Bell

1988 Technical Communication Award of Excellence (Second Place) in Promotional Materials Category, East Tennessee Chapter of the Society for Technical Communication

J. B. Berry

1987 Martin Marietta Energy Systems President's Award for the performance improvement project "Optimize Management of Liquid Low-Level Waste in Chemical Technology Division"

Waste Minimization Award, U. S. Department of Energy, Oak Ridge Operations

1988 Secretary, American Society of Testing and Materials subcommittee on Waste Minimization

C. A. Burtis

1987 Elected Fellow, National Academy of Clinical Biochemistry

1987–1990 President, American Association for Clinical Chemistry

J. M. Butler

1987 Tennessee Society of Professional Engineers, Oak Ridge Section, Outstanding Engineering Achievement Award, Consolidated Edison Uranium Solidification Program

D. T. Brooksbank

1987 Technical Communication Award of Distinction (First Place) in Scholarly and Professional Articles Category, East Tennessee Chapter of the Society for Technical Communication

Technical Communication Award of Achievement (Fourth Place) in Scholarly and Professional Articles Category, International Society for Technical Communication

D. O. Campbell

1988 Martin Marietta Energy Systems Technical Achievement Award for Fuel Cycle Work

M. C. Clark

1987 Tennessee Society of Professional Engineers, Oak Ridge Section, Outstanding Engineering Achievement Award, Consolidated Edison Uranium Solidification Program

H. D. Cochran, Jr.

1987 Who's Who in Technology Today

E. D. Collins

1987 Chairman, Oak Ridge–Knoxville Section, American Institute of Chemical Engineers

Tennessee Society of Professional Engineers, Oak Ridge Section, Outstanding Engineering Achievement Award, Consolidated Edison Uranium Solidification Program

J. L. Collins

1987 Co-Chairman, Workshop on Chemical Reactivity of Oxide Fuel and Fission Product Rele ·, Berkeley Nuclear Laboratories, Great Britain

1988 Technical Communication Award of Distinction (First Place) in Scholarly and Professional Articles Category, East Tennessee Chapter of the Society for Technical Communication

Technical Communication Award of Achievement (Fourth Place) in Scholarly and Professional Articles Category, East Tennessee Chapter of the Society for Technical Communication

J. M. Dalley

1988 Martin Marietta Energy Systems Significant Event Award for "Packaging of ^{238}Pu Oxide"

B. H. Davison

1987 Martin Marietta Energy Syster·s Significant Event Award for "High Productivity Ethanol Fermentation"

J. R. Devore

Martin Marietta Energy Systems Monetary Award for Sale of Technology on Uranium-Tritium Trap

T. L. Donaldson

1988 Chairman, American Institute of Chemical Engineers Personnel Supply and Demand Committee

B. C. Drake

1987 Technical Communication Award of Merit (Third Place) in Technical Reports Category, East Tennessee Chapter of the Society for Technical Communication

M. J. Emmett

1988 Certified Professional Secretary

A. J. Farmer

1987 Tennessee Society of Professional Engineers, Oak Ridge Section, Outstanding Engineering Achievement Award, Consolidated Edison Uranium Solidification Program

C. W. Forsberg

1987 Technical Communication Award of Distinction (First Place) in Scholarly and Professional Articles Category, East Tennessee Chapter of the Society for Technical Communication

Technical Communication Award of Achievement (Fourth Place) in Scholarly and Professional Articles Category, International Society for Technical Communication

1988 Martin Marietta Energy Systems Inventor's Award

S. E. Gheesling

1988 Performance Improvement Process Award

J. R. Gibson

1988 Martin Marietta Energy Systems Significant Event Award for "Packaging of ^{238}Pu Oxide"

T. M. Gilliam

1987 Chairman, Fourth International Hazardous Waste Symposium on Environmental Aspects of Stabilization/Solidification of Hazardous and Radioactive Waste

1988 Secretary, American Institute of Chemical Engineers Environmental Division

E. Greenbaum

1987 National Academy of Sciences Exchange Scholar with the People's Republic of China

1988 Chairman, Eleventh Symposium on Biotechnology for Fuels and Chemicals

Plenary lecturer at the Seventh International Conference on Photochemical Conversion and Storage of Solar Energy

K. W. Haff

1987 Special Award for Excellence for outstanding contribution in transferring technology, Federal Laboratory Consortium

R. Hall

1987 Tennessee Society of Professional Engineers, Oak Ridge Section, Outstanding Engineering Achievement Award, Consolidated Edison Uranium Solidification Program

J. R. Hightower

1988 Fellow, American Institute of Chemical Engineers

R. L. Jolley

1987 Distinguished Service Award, American Chemical Society

Alternate Councilor, American Chemical Society

J. M. Kennerly

1986–1987 Secretary, Knoxville–Oak Ridge Section of the American Institute of Chemical Engineers

1987 1989 Director, Knoxville-Oak Ridge Section of the American Institute of Chemical Engineers

V. M. King

1988 Certified Professional Secretary

P. F. Kirk

1987 Tennessee Society of Professional Engineers, Oak Ridge Section, Outstanding Engineering Achievement Award, Consolidated Edison Uranium Solidification Program

A. M. Krichinsky

1987 Tennessee Society of Professional Engineers, Oak Ridge Section, Outstanding Engineering Achievement Award, Consolidated Edison Uranium Solidification Program

N. E. Lee

1988 Elected Fellow, American Institute of Chemists

R. A. Lorenz

1988 Technical Communication Award of Distinction (First Place) in Scholarly and Professional Articles Category, East Tennessee Chapter of the Society for Technical Communication

Technical Communication Award of Achievement (Fourth Place) in Scholarly and Professional Articles Category, East Tennessee Chapter of the Society for Technical Communication

C. P. McGinnis

1987 Tennessee Society of Professional Engineers, Oak Ridge Section, Outstanding Engineering Achievement Award, Consolidated Edison Uranium Solidification Program

1988 Vice-Chairman, American Institute of Chemical Engineers Nuclear Engineering Development

Chairman, American Institute of Chemical Engineers Nuclear Engineering Division

M. F. Osborne

1988 Technical Communication Award of Distinction (First Place) in Scholarly and Professional Articles Category, East Tennessee Chapter of the Society for Technical Communication

Technical Communication Award of Achievement (Fourth Place) in Scholarly and Professional Articles Category, East Tennessee Chapter of the Society for Technical Communication

M. M. Osborne

1987 Tennessee Society of Professional Engineers, Oak Ridge Section, Outstanding Engineering Achievement Award, Consolidated Edison Uranium Solidification Program

I. W. Osborne-Lee

1987–1988 President, East Tennessee Chapter of the National Organization for the Professional Advancement of Black Chemists and Chemical Engineers

1988 Chairman, Oak Ridge National Laboratory Seed Money Committee

B. D. Patton

1987 Treasurer, Knoxville–Oak Ridge Section, American Institute of Chemical Engineers

Director, Great Smoky Mountains Alumnus Chapter, Tau Beta Pi

1988 President-Elect, Great Smoky Mountains Alumnus Chapter, Tau Beta Pi

W. W. Pitt, Jr.

1988 Fellow, American Institute of Chemical Engineers

1987–1988 Vice-Chairman, Long-Range Planning, American Society for Testing and Materials Committee D-34 on Waste Management

R. B. Pope

1988 International Committee Chairman, Ninth International Symposium on Packaging and Transportation of Radioactive Materials

C. E. Porter

1987 Tennessee Society of Professional Engineers, Oak Ridge Section, Outstanding Engineering Achievement Award, Consolidated Edison Uranium Solidification Program

D. J. Pruett

1988 Treasurer, Divisions of Nuclear Chemistry and Technology and of Industrial and Engineering Chemistry, American Chemical Society

Chairman-Elect, Subdivision of Separation Science and Technology of the Industrial and Engineering Chemistry Division, American Chemical Society

Appointed to the Committee on Nuclear and Radiochemistry of the National Research Council

Associate Editor of Solvent Extraction and Ion Exchange

D. W. Ramey

1987 Federal Laboratory Consortium Special Award for Excellence in Technology Transfer for Radioluminescent Light Technology Transferred to Private Industry

J. E. Ratledge

1988 General Chairman, ^{252}Cf Workshop presented by Oak Ridge National Laboratory

R. R. Rawl

1986–1988 American National Standards Institute Committee N14

D. R. Reichle

1987 Technical Communication Award of Merit (Third Place) in Technical Reports Category, East Tennessee Chapter of the Society for Technical Communication

1988 Technical Communication Award of Achievement (Fourth Place) in Scholarly and Professional Articles Category, East Tennessee Chapter of the Society for Technical Communication

Technical Communication Award of Distinction (First Place) in Scholarly and Professional Articles Category, East Tennessee Chapter of the Society for Technical Communication

B. R. Rodgers

1985–1987 Director, Fuels and Petrochemicals Division, American Institute of Chemical Engineers

1986–1988 Chairman of Program Committee, Fuels and Petrochemicals Division, American Institute of Chemical Engineers

O. W. Scates

1987 Tennessee Society of Professional Engineers, Oak Ridge Section, Outstanding Engineering Achievement Award, Consolidated Edison Uranium Solidification Program

C. D. Scott

1987 Fellow, American Association for the Advancement of Science

Senior Corporate Fellow, Martin Marietta Energy Systems

Martin Marietta Energy Systems Publication Award

Nathan W. Dougherty Engineering Award, University of Tennessee

1987–1990 Director, American Institute of Chemical Engineers

1988 Member, Visiting Committee of the Department of Chemical Engineering, University of Virginia

Missouri Honor Award for Distinguished Service in Engineering, University of Missouri

C. H. Shappert

1987 Technical Communication Award of Distinction (First Place) in Scholarly and Professional Articles Category, East Tennessee Chapter of the Society for Technical Communication

Technical Communication Award of Achievement (Fourth Place) in Scholarly and Professional Articles Category, International Society for Technical Communication

S. P. N. Singh

1984–1990 National Laboratories Consortium Representative to the Emissions and Control Technologies Task Group, National Acid Precipitation Assessment Program

1987–1989 Alternate observer for Chemical Process Research on Project Advisor Group, Gas Research Institute

R. D. Spence

1987 Performance Improvement Process Award, Martin Marietta Energy Systems

1988 Publications Award, Martin Marietta Energy Systems

B. W. Starnes

1987 Tennessee Society of Professional Engineers, Oak Ridge Section, Outstanding Engineering Achievement Award, Consolidated Edison Uranium Solidification Program

G. W. Strandberg

1987 Publications Award, Martin Marietta Energy Systems

J. A. Tompkins

1987 Federal Laboratory Consortium Special Award for Excellence in Technology Transfer for Radioluminescent Light Technology Transferred to Private Industry

V. C. A. Vaughen

1982–1987 Co-Chairman, Ethics Committee, Knoxville–Oak Ridge Section of the American Nuclear Society

1983–1987 Ad Hoc Committees on Socio-Technological (or Ethical) Issues for the American Nuclear Society

1988 Steering Committee of the National Society of Professional Engineers to establish a National Institute for Engineering Ethics

Board of Governors, National Institute of Engineering Ethics, National Society of Professional Engineers

R. J. Vedder

1987 Tennessee Society of Professional Engineers, Oak Ridge Section, Outstanding Engineering Achievement Award, Consolidated Edison Uranium Solidification Program

Martin Marietta Energy Systems President's Award for the performance improvement project "Optimize Management of Liquid Low-Level Waste in Chemical Technology Division"

J. Woodward

1987 Librarian, Division of Microbial and Biochemical Technology, American Chemical Society

A. L. Wright

1988 Publications Award, Martin Marietta Energy Systems

R. G. Wymer

1987 Robert E. Wilson Award in Nuclear Chemical Engineering, American Institute of Chemical Engineers

Consultants

J. R. Benemann
Affiliation: Georgia Institute of Technology
Expertise: Chemical Sciences/Advisory Committee
Duration: 2 d
Section/Program: Chemical Technology Division

T. Beresovski
Affiliation: Independent
Expertise: Chemical Sciences/Quality Assurance
Duration: 50 d
Section/Program: Department of Defense

P. R. Bienkowski
Affiliation: University of Tennessee
Expertise: Chemical Engineering
Duration: 12 months
Section/Program: Energy Research

L. B. Cobb
Affiliation: Independent
Expertise: Geologist
Duration: 60 d
Section/Program: Waste Management

C. F. Coleman
Affiliation: ORNL Retiree
Expertise: Chemical Sciences
Duration: 1 year
Section/Program: Chemical Development

J. C. Crittenden
Affiliation: Michigan Technological University
Expertise: Civil Engineering
Duration: 3 d
Section/Program: Department of Defense

P. A. Domenico
Affiliation: Texas A&M
Expertise: Geology/Hydrology
Duration: 25 d
Section/Program: Waste Repository

D. D. Ensor
Affiliation: Tennessee Technology University
Expertise: Chemistry
Duration: 20 d
Section/Program: Chemical Development

J. E. Gale
Affiliation: Memorial University
Expertise: Hydrological Engineering
Duration: 45 d
Section/Program: Waste Repository

H. E. Goeller
Affiliation: ORNL Retiree
Expertise: Chemical Engineering
Duration: 12 months
Section/Program: Chemical Technology Division

J. M. Gossett
Affiliation: Cornell University
Expertise: Chemical and Civil Engineering
Duration: 11 d
Section/Program: Department of Defense

J. M. Holmes
Affiliation: JMH Associates
Expertise: Industrial Chemistry
Duration: 12 months
Section/Program: Energy Research

T. A. Hueckel
Affiliation: Duke University
Expertise: Geology
Duration: 45 d
Section/Program: Waste Management

A. E. Humphrey
Affiliation: Lehigh University
Expertise: Biotechnology
Duration: 4 d
Section/Program: Energy Research

R. H. Jones
Affiliation: Independent
Expertise: Transportation
Duration: 60 d
Section/Program: Waste Management

B. Kanehiro
Affiliation: Independent
Expertise: Chemical Engineering
Duration: 45 d
Section/Program: Waste Management

R. Narayan
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Expertise: Coal Chemistry
Duration: 4 d
Section/Program: Energy Research

J. A. Phillips
Affiliation: Lehigh University
Expertise: Biotechnology
Duration: 4 d
Section/Program: Energy Research

P. E. Potter
Affiliation: Cincinnati University
Expertise: Geology/Statistics
Duration: 20 d
Section/Program: Waste Management

J. D. Rollins
Affiliation: Independent
Expertise: Nuclear Engineering
Duration: 12 d
Section/Program: Waste Management

H. C. Savage
Affiliation: Independent
Expertise: Quality Assurance
Duration: 10 d
Section/Program: Process Development

E. H. Snider
Affiliation: Independent
Expertise: Hazardous Waste
Duration: 2 d
Section/Program: Engineering Development

D. I. C. Wang
Affiliation: Massachusetts Institute of Technology
Expertise: Chemical Engineering
Duration: 4 d
Section/Program: Energy Research

W. A. Weigand
Affiliation: Illinois Institute of Technology
Expertise: Chemical Engineering
Duration: 4 d
Section/Program: Energy Research

Seminars

Chem Tech staff members invite specialists from around the world and from various fields to present seminars at ORNL.

January 8, 1987	"Bacterial Degradation of Pentachlorophenol," J. G. Steiert, University of Minnesota, Minneapolis-St. Paul, Minnesota
March 23, 1987	"Utilization of Lime Sinter Process Residue for the Manufacture of a Low Alumina Portland Cement," Dr. Jason Allen Chesley, Iowa State University of Science and Technology, Ames, Iowa
April 13, 1987	"A Graphical Determination of the Possibility of Multiple Steady States in Complex Isothermal CFSTRs," P. M. Schlosser, University of Rochester, Rochester, New York
July 23, 1987	"Electrohydrodynamics of Drops and Bubbles," O. A. Basaran, Air Products and Chemicals, Inc., Allentown, Pennsylvania
September 8, 1987	"Substituted 2-Nitrophenols: Kinetic Characterization for the Biodegradation of Single Compounds and Mixtures," Dr. Brian Folson, Eidgenossische Technische, Hochschulen, Switzerland
September 14, 1987	"Is There a Better Way to Develop Project Planning Documents?" T. I. McSweeney, Battelle Project Management Division, Columbus, Ohio
November 3, 1987	"Large-Scale, Continuous Culture of Mammalian for Production of Medical Proteins," Robert C. Dean, Jr., Synosis, Lebanon, New Hampshire
November 5, 1987	"Use of Immobilized Microorganisms in Bioprocessing Systems," F. C. Godia, Universitat Autonoma Barcelona, Bellaterra, Barcelona, Spain
November 18, 1987	"Determination of Biodegradation Kinetic through the Use of Electrolytic Respirometry," Les Grady, Clemson University, Clemson, South Carolina
November 18, 1987	"Bioprocessing Concepts Utilizing Advanced Bioreactor Systems," Carlos Sola, Universitat Autonoma Barcelona, Bellaterra, Barcelona, Spain
January 18, 1988	"An Investigation of the Microscopic Structure and Dynamics of Binary Liquid Mixtures," T. Michael Bender, Stanford University, Stanford, California
February 12, 1988	"Development of Large-Scale Chromatography," Phillip Barker, University of Aston, Birmingham, England
March 14, 1988	"Effects of Electric Fields on Liquid-Liquid Solvent Extraction," Tom Carlson, University of Idaho, Moscow, Idaho
April 8, 1988	"Salt Repository Waste Package Strategy and Design Concepts," Dr. James R. Schornhorst, Battelle Memorial Institute, Office of Nuclear Isolation, Hereford, Texas, and Columbus, Ohio
June 13, 1988	"Conversion of Municipal Solid Waste to Chemicals," Dr. Thomas J. Abraham, Jr., Kingsport, Tennessee

Internal Program Reviews

Date	Program	Coordinator
February 6, 1987	HTGR	J. C. Mailen
February 27, 1987	LLWDDD	R. K. Genung/ B. R. Rodgers
March 6, 1987	Actinide/Lanthanide Separation Chemistry	D. J. Pruett
March 13, 1987	WSDD	A. G. Croff
May 8, 1987	IDB	J. A. Klein
June 19, 1987	Waste Isolation Technology	E. W. McDaniel
July 10, 1987	Environmental Control Technology	C. H. Brown
August 7, 1987	^{252}Cf Sales/Loan Program	L. J. King
August 21, 1987	Biotechnology Research	E. Greenbaum
September 25, 1987	Waste (Transportation)	A. G. Croff/ L. B. Shappert
October 2, 1987	Support Activities (Drafting Room)	E. K. Johnson
November 6, 1987	Advanced Technology Development	T. L. Donaldson
November 20, 1987	Geochemistry Program	A. D. Kelmers
April 22, 1988	NRC Source Term Programs	A. L. Wright
April 29, 1988	HTGR Program	J. C. Mailen
May 20, 1988	CFRP and AVLIS	J. T. Bell
May 27, 1988	Waste Geochemistry	A. D. Kelmers

Chemical Technology Division Staffing Level and Financial Summary, FY 1988

	Funding (in thousands of dollars)	Chem Tech Scientific/Technical[a] (person-years)	Total Scientific/Technical[b] (person-years)
DOE Programs			
Coal	407	2.2	2.2
Gas	1	0.0	0.0
Petroleum	39	0.2	0.2
Breeder Reactor Systems	1,537	7.3	7.9
Remedial Action	463	2.4	2.6
Nuclear Fuel Cycle	6	0.0	0.0
Defense Waste and By-Products Management	3,058	15.3	15.8
Nuclear Waste Fund	6,828	17.8	25.4
Transportation	17	0.1	0.1
Industrial	135	0.4	0.4
Multisector	268	1.9	1.9
Materials Production	5,168	17.5	20.2
Environmental R&D	90	0.5	0.5
Basic Energy Sciences	9,555	45.9	51.2
Environmental Compliance	895	2.9	3.3
Subtotal	**28,467**	**114.4**	**131.7**
DOE-related Programs			
Nuclear Regulatory Commission	1,318	6.2	6.3
Other DOE and Contractors	748	4.0	4.6
DOE-ORO Contractors	33	0.0	0.0
State	2	0.0	0.0
Other Energy Systems Plants	459	3.1	3.4
Subtotal DOE-Related Programs	**2,560**	**13.3**	**14.3**
Total DOE Programs	**31,027**	**127.7**	**146.0**
Work for Others			
State and Local Government	239	1.0	1.0
Other Federal (Excluding NRC)	128	0.7	0.7
Department of Defense (Waste Management)	96	0.5	0.5
Other Department of Defense	2,339	9.4	12.9
Total Work For Others	**2,802**	**11.6**	**15.1**
Total	**33,829**	**139.3**	**161.1**

[a]Chem Tech research staff supported by the Division in FY 1988.

[b]Total research staff supported by Chem Tech funding in FY 1988, including personnel on loan to Chem Tech from other divisions.

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