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MASTER

Consolidated Fuel
Reprocessing Program
Progress Report for
Period July 1 to
September 30, 1981

ORNL/TM--8030

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**CONSOLIDATED FUEL REPROCESSING PROGRAM
PROGRESS REPORT
FOR PERIOD JULY 1 TO SEPTEMBER 30, 1981**

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NOTICE: This document contains information of a preliminary nature. It is subject to revision or correction and therefore does not represent a final report.

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DEPARTMENT OF ENERGY

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Foreword

The DOE has concentrated all U.S. research and development on fuel reprocessing into one major program — the Consolidated Fuel Reprocessing Program (CFRP) — under the management of the Oak Ridge National Laboratory and the Oak Ridge Operations Office. Other major program participants are the Savannah River Laboratory and General Atomic, where reprocessing research and development on the HTGR fuel cycle is done.

The coverage is generally overview in nature. Experimental details and data have been limited (1) to make the report more concise and (2) to meet the requirements which would qualify the report for unrestricted distribution in the open literature.

1. Highlights

All research and development on fuel reprocessing in the United States is managed under the Consolidated Fuel Reprocessing Program centered at Oak Ridge National Laboratory. Technical progress is reported in overview fashion in this series of quarterly progress reports.

1.1 PROCESS DEVELOPMENT

W. S. Groenier

Fast breeder reactor fuel (average burnup of about 30 000 MWd/t) was processed for the first time in the ORNL Solvent Extraction Test Facility. Operation was satisfactory with low heavy-metal losses and high decontamination factors (>1000) for all isotopes except ^{95}Zr , which exhibited moderate decontamination factors (180 and 750, respectively, in two runs). The recovered plutonium was further purified by anion exchange, precipitated with oxalic acid, and calcined to PuO_2 .

A small batch of PuO_2 was prepared in a small rotary calciner by the modified thermal denitration process used previously to prepare U-Pu mixed oxide. The PuO_2 powder yielded pellets that were $\sim 90\%$ of theoretical density. Cerium oxide was also successfully prepared by the modified process.

1.2 LABORATORY R&D

W. S. Groenier

Studies on plutonium polymerization under reflux conditions were successfully completed. An electrochemical method was used to suppress the oxidation of Pu(IV) to Pu(VI) . The method maintained 20 to 25% Pu(IV) in the solution.

The investigation of the degradation of normal paraffin hydrocarbon diluent by nitration with HNO_3 solutions or NO_2 gas has shown that the principal degradation compounds are produced by nitration of

the alkane constituents and not the hydrocarbon impurities in the diluent.

Measurements were made of the density and particle size of solids remaining after dissolving irradiated mixed oxide and light-water reactor fuels in nitric acid.

Fabrication of an optical system was completed for the proof-of-principle x-ray fluorescence spectrometer for the analysis of highly gamma-active samples.

Dissolver solutions and residues are being analyzed by the new high-gamma-level, spark-source mass spectrometry facility.

1.3 ENGINEERING RESEARCH

W. S. Groenier

The solids recirculation system recently installed on the full-scale experimental voloxidizer is functioning satisfactorily; this system will be used to transfer fine material that falls into the inlet breeching section to the feed chute.

In the dissolver digester tank studies, the presence of simulated fuel spacer wires has not prevented the suspension of prototypical solids when using a modified pulsed lift tube in the digester.

Individual stage efficiencies for multistaged centrifugal contactor operation are comparable to those obtained in single-stage tests at the Argonne National Laboratory.

Tests at the General Atomic Company have shown that extraction column flooding (at low pulsed volume

velocities) occurs at higher frequencies with 51% tri(2-ethylhexyl) phosphate than with 30% tributyl phosphate. Flooding at higher pulsed volume velocities (>1.7 cm/s), however, occurs at lower frequencies.

In solid sorbent studies for iodine retention, silver utilization has remained high ($\sim 100\%$) in continued tests with low silver-exchanged mordenites.

Decontamination factors for radon of up to 100 have been obtained using delay beds packed with hydrogen mordenite at the General Atomic Company.

1.4 ENGINEERING SYSTEMS

M. J. Feldman

The follow-on study on the potential utilization of the Barnwell Nuclear Fuel Plant has been completed, and the report is in preparation. The study concluded that a demonstration breeder fuel reprocessing plant could be colocated at Barnwell.

The mechanical design of an 8-pack centrifugal contactor has been completed, and fabrication of the first system is well under way.

The first phase of the remote disassembly system consisting of a review of the system design requirements and an evaluation of the conceptual design has been completed by the vendor. Detail design has been initiated.

A two-year program to evaluate sodium removal processes for fuel assemblies and develop process parameters has been completed by the contractor.

The conceptual design of an advanced servomanipulator system has been completed. Plans are to build and test one set of arms as a development unit during the next two years.

The conceptual design for the Hot Experimental Facility has been completed, and the report has been issued.

1.5 INTEGRATED EQUIPMENT TEST (IET) FACILITY OPERATIONS

O. O. Yarbrow

Major process equipment items in the IET line-item contract have been installed, and substantial progress

has been made on the installation of process and service piping. The DDACS (Distributed Data Acquisition and Control System) delivery date has been rescheduled to mid-February 1982 because of software and equipment delays. The Remote Operation and Maintenance Demonstration (ROMD) hoists and manipulators and the control console have been installed, and the wiring is essentially complete. New gears have been installed in the crane drives, and satisfactory operation has been attained. Approximately 90% of the Iodex and acid concentration system drawings have been reviewed and approved.

Work has continued in connection with detailing the equipment location and installation in the ROMD area. A special task force has been organized to identify the interface requirements of the IET equipment and prepare an overall report projecting the resource allocations required by IET operations between FY 1982 and FY 1985. This work is expected to be completed next quarter.

Preparations for delivery, installation, and initial operation of the acid fractionator process module mock-up have been made. A draft of the test plan and a list of the equipment operating procedures required for the module remote maintenance demonstration have been completed. Flowsheets for the IET dissolver and chilled water systems have also been completed, and Program approval has been obtained on the dissolver drawings.

1.6 HTGR FUEL REPROCESSING

W. S. Groenier

The below-bed fines recycle system was verified in recent burner tests with graphite fuel at the General Atomic Company in preparation for the low-enriched uranium fuel processing demonstration with the Federal Republic of Germany. Operating conditions were selected that permit the recycle of fines at rates that create optimum burner residence times for complete burning.

2. Process Development

W. S. Groenier and D. J. Crouse

The process development activities provide processing parameters and recommended materials of construction for both the Integrated Equipment Test (IET) facility and the Hot Experimental Facility (HEF). The use of partial partitioning in solvent extraction flow-sheets is being evaluated at ORNL in the Solvent Extraction Test Facility (SETF), which has an irradiated fuel capacity in the kilogram-per-day range. The product conversion program is establishing methods for converting U-Pu nitrate solutions to mixed oxide products that are suitable for pellet fuel fabrication. Candidate materials of construction are evaluated in experimental corrosion studies. Alternate extractants to tributyl phosphate (TBP) are being evaluated.

2.1 SOLVENT EXTRACTION TEST FACILITY

L. J. King, D. E. Benker, J. E. Bigelow, E. W. Brown, E. B. Cagle, F. R. Chattin, E. D. Collins, D. B. Owsley, R. G. Ross, H. C. Savage, R. C. Shipwash, R. G. Stacy, C. E. Waddell, A. V. Wilder, and K. Wilson

Campaign 5 at the Solvent Extraction Test Facility (SETF) was completed during this reporting period. The heavy metal (~2 kg) in 16 intact FBR fuel pins (plus pieces of three other pins) was dissolved and processed to recover and purify ~400 g of plutonium which was then converted to the oxide form and packaged for shipment to Hanford Engineering Development Laboratory (HEDL) where it will be used for reirradiation studies.

The fuel pins had originally contained ~25% plutonium and ~75% natural uranium. Nine pins (PNL-3 and PNL-15) had been irradiated in the EBR-2 and seven (PNL-59) in the General Electric Test Reactor (GETR); all had been discharged for six years or more. The burnups ranged from 70 GJ/kg to 5.7

TJ/kg (810 to 66 000 MWd/t). The pins were sheared into 2.5-cm-long pieces, mixed, and packaged into two batches. The average burnups of the sheared pins in the two batches were ~2.3 TJ/kg (~27 000 MWd/t) and ~3.0 TJ/kg (~35 000 MWd/t).

Each batch of fuel pins was separately processed because of safeguards limitations. The processing steps included dissolution, one cycle of solvent extraction, anion exchange, and oxalate precipitation of the plutonium.

The procedure used previously for dissolving LWR fuels was not satisfactory for FBR fuels and left a residue containing ~15% of the heavy metals. The residue was dissolved successfully in a 4-h digestion in 7 M HNO₃ at 90°C. Laboratory tests indicated that complete dissolution could have been obtained in the primary dissolution step by raising the acidity to provide a final HNO₃ concentration of about 3 M.

The dissolver solutions were clarified by filtration using a 1-μm etched-disk filter without filter aid.

Insoluble material retained on the filter was later recovered and concentrated by backflushing, settling, and decanting.

The clarified dissolver solutions were spiked with $^{95}\text{Zr-Nb}$ so that zirconium decontamination measurements could be made in the subsequent solvent extraction tests.

One condition was changed during the coextraction-coscrubbing part of the tests. In the first test, a split scrub (0.5 M HNO_3 to the top and 3 M HNO_3 to an intermediate point of the scrub section) was used. In the second test, only a single scrub (3 M HNO_3) was used. The split scrub gave a zirconium decontamination factor (DF) of 750 compared with 180 when using the single high-acid scrub. In both cases, the ^{95}Nb DF was 2000. Other measured DFs were ^{137}Cs , $>10^5$; ^{155}Eu , $>10^4$; and ^{125}Sb , $>10^4$. Losses of plutonium to the raffinate were $<0.02\%$.

In the stripping bank, part of the uranium was partitioned from the plutonium so that the final U-Pu product would contain $\sim 35\%$ plutonium. Partitioning was relatively easy to accomplish because of the small degree of uranium separation required to raise the plutonium concentration from $\sim 22\%$ (original value) to $\sim 35\%$. The amount of uranium stripped with the plutonium was controlled simply by maintaining the temperature at $\sim 30^\circ\text{C}$ and adjusting the aqueous-to-organic flow ratio.

In the first test a 0.009 M HNO_3 – 0.009 M hydroxylamine nitrate (HAN) solution was fed to the end of the stripping section, and 3 M HNO_3 was fed to an intermediate stage. A single stripping solution (0.1 M HNO_3) was used in the second test. In both tests the plutonium loss to the stripped organic solutions was $<0.005\%$, and the U-Pu product solution contained $\sim 32\%$ plutonium. The U-Pu solution was processed by anion exchange to separate and purify the plutonium.

Purified plutonium product solutions were divided into several batches, each of which was treated by HAN to convert the plutonium to the trivalent state before oxalate precipitation. Precipitates were filtered, dried, and calcined at 500°C to produce PuO_2 . The only measurable gamma-emitting fission products remaining in the oxide product (^{95}Zr and ^{125}Sb) were present in low concentrations (13 and 17 MBq/kg of plutonium respectively) and did not affect the radiation properties

of the product. Cross contamination of the oxide product with ^{244}Cm from the oxalate precipitation equipment was not a problem, since the ^{244}Cm concentration was only $2\text{ }\mu\text{g/g}$ of plutonium. The oxide product, which contained a total of 392 g of plutonium, was packaged for shipment to HEDL. Oxide product samples containing another 15 g of plutonium were transferred to the Analytical Chemistry Division and the Metals and Ceramics Division for further tests and analyses.

2.2 PRODUCT CONVERSION

R. E. Norman

This task focuses on the development of engineering technologies for converting U-Pu in nitrate process streams to solid oxides suitable for fuel pellet fabrication. Two product conversion methods, gel-sphere and thermal denitration, have been selected for development at ORNL. These methods, together with other candidate processes, will be evaluated as a preliminary step toward selecting a reference process for the HEF.

2.2.1 Thermal Denitration Conversion Development

In the thermal denitration process, the mixed nitrate feed is converted directly to a mixed oxide by heating. The principal criteria for the evaluation of products are the density and microstructure of pellets when the product oxides are fabricated into pellets by standard procedures.

Lab-Scale Studies

J. C. Mailen, D. J. Pruett, D. R. McTaggart, and W. B. Stines

Two batches of U-Pu oxide powder have been produced from two different feed solutions by thermal denitration. The results of an analysis of the first batch were reported previously.¹ Pellets fabricated from the second batch had a density of 95% of theoretical with good microstructure. A 60-g sample of the second batch was sent to Battelle-Pacific Northwest Laboratories (PNL) for evaluation. A batch of PuO_2 powder (with no UO_2) was also prepared by thermal denitration. Two PuO_2 pellets fabricated from this powder had densities of 89 and 90% of theoretical.

Process and Equipment Development

P. A. Haas, R. D. Arthur, and W. B. Stines

Comparisons of thermal analyses behavior patterns have shown cerous nitrate to be the best stand-in for

the thermal denitration behavior of plutonium. Small-scale denitrations of plutonium nitrate solutions have been reported by Rockwell Hanford Operations to show caking or mastic-stage problems similar to those for the denitration of $\text{UO}_2(\text{NO}_3)_2$ solutions. Technical grade cerous nitrate was tested using the ORNL modified denitration flowsheet. Mechanical operation was very good with easy discharge of a fine product powder with a microscopic shape and size appearance very similar to UO_3 that had been prepared using the modified flowsheet. Four UO_3 denitration products were prepared and shipped to PNL. Three of the products were made with a continuous rotary kiln and with feeds of different acidities; one was a batch denitration product.

2.2.2 Gel-Sphere Conversion Development

The gel-sphere conversion process utilizes internal gelation in the preparation of UO_2 - PuO_2 spheres. An ammonia donor and a complexing agent are mixed with the heavy-metal nitrate solution at a temperature near 0°C to form a broth. Gelation of broth droplets occurs rapidly in a heated immiscible liquid. Previous work on this process under the Fuel Refabrication and Development Program provides a good base for specific equipment concepts and technology required for application in the HEF. The Pu-U gel-sphere conversion studies are a joint effort with PNL and HEDL in the development of gel-derived fuel pellets.

Chemical Flowsheet Development

M. H. Lloyd, J. L. Collins, R. L. Fellows,
S. H. Shell, D. H. Newman, and W. B. Stines

Development work on mixed oxide gel-sphere conversion has been concentrated on the preparation of additional material for PNL fabrication studies. New furnace equipment that will accommodate a larger batch size was installed and tested. Excellent gel-sphere products were obtained with this equipment. Two batches have been prepared and shipped to PNL. One batch of 118 g was prepared in a single sphere-forming run. The second batch of 220 g was prepared in four sphere-forming runs. Based on previous experience, the spheres will be good feed material

for pellet fabrication. A third batch of spheres (~150 g) prepared last quarter was also shipped to PNL for evaluation.

An evaluation of microsphere preparation at the reference ammonia donor:metal ratio of 1.6 has been completed. This work was conducted to obtain a better understanding of regions where poor gel-sphere strength can be encountered. These parallel series of experiments were performed with pure uranium feeds, U-5% Pu feed, and U-10% Pu feed. As the plutonium content decreased from the reference value of 25%, the gelation time decreased because plutonium significantly retards uranium gelation. However, in each series, the urania crystallite size increased with the increasing active $\text{NO}_3^-:\text{U}$ mole ratio; two distinct urania phases formed. For the two plutonium-containing feeds (5 and 10% plutonium), a nitrate ratio was found in which the phase with small crystallites was eliminated. Satisfactory products could not be produced with the pure uranium feed using the same conditions.

Process and Equipment Development

A. D. Ryon, P. A. Haas, R. D. Spence, A. L. Rivera,
V. L. Fowler, R. C. Lovelace, W. B. Stines,
and T. L. Yount

The development of equipment and glove boxes for the gel-sphere line in Building 3019 was continued. Testing of glass gelation columns has led to the selection of a design for use in this system; shakedown tests are under way. Tests have also been performed with the dryers fabricated for use in this line using UO_3 gel spheres made in Building 3541. The test results provided useful information on the drying operation and also suggested control schemes.

The development of equipment for the cold (uranium-only) engineering facility was continued. A test of the continuous wash column operation was made with $700\text{-}\mu$ UO_3 gel spheres. The dried gel has a good surface appearance and a very uniform, light yellow color; this is evidence of uniform washing and no mechanical damage. Gel movement was maintained throughout startup, steady-state operation, and shut-down, with a countercurrent flow of washed gel and no pockets of stationary solids. There were some

cyclic fluctuations in solid and liquid levels, but these appear to be self-limiting and do not require changes in liquid flow rates.

Tests on rotary drop formation (Couette) were continued. Test results indicated that the uniformity of drop diameters is not as good as the best results for static mixers.

Work continued on the production of dry gel spheres for use by HEDL. The inventory of prepared spheres was increased to ~150 kg; 70 kg was shipped earlier.

2.2.3 Building 3019 Facility Modifications

F. L. Daley, B. C. Finney, F. E. Harrington, C. C. Haws, J. R. Parrott, W. D. Simpson, and D. F. Williams

Facilities are being developed in Building 3019 to prepare kilogram quantities of mixed heavy-metal (U-Pu) oxides suitable for pellet preparation by the gel-microsphere and thermal-denitration routes.

Laboratory 209 modifications are complete. The Analytical Chemistry Division has begun to prepare space for sample archives so that Laboratory 210 may also be reconditioned.

Five glove boxes that had been assembled and tested in Building 3503 were transferred to Building 3019. A crafts strike lasting seven weeks, followed by the accumulated priority maintenance jobs, combined to delay the installation of these glove boxes and the associated services and auxiliaries for nine weeks.

The environmental assessment for the product conversion facility has been submitted for approval, the safety analysis report has been through two internal reviews, and the nuclear safety review has been issued for comment.

2.3 MATERIALS COMPATIBILITY

J. C. Griess

The purpose of this task is to identify the best materials for fabricating components for advanced fuel reprocessing plants. Experimental work has been conducted at Battelle-Columbus Laboratories under subcontract, which expired September 30. The last quarter of the contract was used to complete various phases of the test program and to prepare final reports.

2.3.1 Battelle-Columbus Corrosion Studies

J. A. Beavers, R. R. White, and W. E. Berry
(Battelle-Columbus Laboratories)

Off-Gas Corrosion

Corrosion produced by moist iodine vapors at 25 and 40°C was essentially the same on austenitic stainless steels (Fe-Cr-Ni alloys), ferritic stainless steels (Fe-Cr alloys), iron, and nickel. On the other hand, pure chromium was not attacked. Attack was the same in the presence or absence of oxygen, but in all cases the removal of water from the system prevented corrosion. A report describing the results of all experiments in simulated reprocessing off-gas environments was completed and is being prepared for distribution.²

Corrosion in HNO₃-HF Solutions

The effect of heat treatment on the corrosion of Inconel 671 in boiling 8 M HNO₃-0.05 M HF was determined. After annealing at 1200°C, the corrosion of air-quenched specimens was about a factor of 2 less than that for furnace-cooled specimens. Metallography showed that the air-cooled specimens were single-phase, whereas the more slowly cooled material contained two phases, one of which was rich in nickel.

Connector Corrosion

Tests with two type 304L stainless steel Gamah connectors were conducted. One of the connectors had a type 304 stainless steel ring and the other had a Ti-6Al-4V alloy ring. The connector with the stainless steel ring began to leak between 400 and 500 h after the start of the test, whereas the one with the titanium alloy ring remained leaktight for the total test period of 1750 h. Metallographic examination of the failed connector showed severe stringer and intergranular attack on the seal ring and significant end-grain attack on the ends of the type 304L connector. Examination of an unused stainless steel seal ring from the same material as the one that corroded showed that the material contained numerous nonmetallic stringers. Attack apparently began at these locations and extended through the thickness of the ring.

A final report that includes all of the results from the experimental program not previously presented in topical reports was prepared and submitted to the CFRP for review and subsequent publication.

2.4 EVALUATION OF ALTERNATE EXTRACTANTS

F. J. Hurst

The objective of this task is to identify extractants that are superior to tributyl phosphate (TBP) for fuel reprocessing.

Tests were initiated to determine the distribution of the sodium salt of di(2-ethylhexyl) phosphoric acid (Na-DEHP) between 0.6 M TEHP and sodium carbonate solution. These data are needed to specify the most effective conditions for washing this principal degradation product from the solvent.

It can be expected that the distribution coefficient (aqueous/organic) for this relatively long-chain compound will be less favorable than for its TBP analog (Na-DBP). The distribution coefficient increases rapidly as the aqueous sodium ion concentration is decreased; therefore, more efficient washing is obtained with low sodium carbonate concentrations.

Measurement of the distribution coefficients (aqueous/organic) as a function of the aqueous sodium carbonate concentration showed that the coefficients decreased as the amount of Na-DEHP in the system was increased; the change results from Na-DEHP reaching its solubility limit in the aqueous phase. The indicated solubility limits in 0.3, 0.2, and 0.1 M

Na_2CO_3 solutions at 25°C are about 0.001, 0.003, and 0.017 M respectively. It is evident that effective removal of the Na-DEHP cannot be obtained in a single washing stage and that a two-stage crosscurrent contacting system will be needed in which the solvent is first contacted with about 0.3 M Na_2CO_3 solution and then with water or very dilute sodium carbonate solution. In a test of this procedure, 40 volumes of solvent were contacted with one volume of 0.3 M Na_2CO_3 and then one volume of 0.02 M Na_2CO_3 at room temperature. About 5% of the Na-DEHP was removed in the first contact and 75% in the second. Use of a larger volume of the more dilute solution would be needed to provide complete removal. The washing efficiency can also be improved by increasing the temperature to 40°C, which increases the distribution coefficients and improves the phase separation.

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3. Laboratory R&D

W. S. Groenier and J. H. Shaffer

Laboratory research and development activities provide basic studies on proposed processing steps and explore new process options. An emphasis is placed on solution properties in dissolution and solvent extraction steps, and attention is being given to several aspects of plutonium chemistry and solvent extraction technology. Hot-cell and glove box activities examine the processing properties of a variety of fuel materials and include solvent extraction process testing in mixer-settler and pulsed column equipment. New analytical methods for process control are being developed and adapted where possible for high-level gamma operations.

3.1 LABORATORY DEVELOPMENT

J. T. Bell and W. D. Bond

Laboratory development activities during FY 1981 have been concerned with basic studies of plutonium chemistry and fission product behavior in aqueous process streams. The development and evaluation of cleanup processes for organic solvent and aqueous recycle streams was also pursued.

3.1.1 Plutonium Polymerization

L. M. Toth and M. M. Osborne

Polymer reflux experiments have been successfully completed for samples of 0.05 *M* Pu in 1.0, 2.0, and 3.0 *M* HNO₃ using electrochemical methods to control the oxidation of Pu(IV) to Pu(VI). The applied potential was effective in maintaining 20 to 25% Pu(IV) in the solution. In 1 and 2 *M* HNO₃, polymer was visually observed within 2 h as a cloudy suspension. In refluxing 3 *M* HNO₃, polymer was observed by Tyndell light scattering after 4 h. In all cases the polymer precipitated, leaving essentially none in solution. Similar studies at 105°C with pressurized sealed systems to prevent refluxing showed longer times of 4 and 10 h for

polymer formation in 1 and 2 *M* HNO₃ respectively. These results show that plutonium solutions which normally would not be expected to polymerize can form and precipitate polymer under refluxing conditions.

3.1.2 Solvent Cleanup

J. C. Mailen, O. K. Tallent, and K. D. Pannell

Solvent cleanup tests used 2.54-cm (1-in.)-diam packed beds containing 80 g (12-in. height) of sodium-treated 12- to 42-mesh silica gel and 7.62-cm (3-in.)-diam fluidized beds containing ~500 g of lithium-treated 60- to 200-mesh silica gel. In general, the fluidized bed columns operated better than the packed bed columns. With a 7.62-cm fluidized bed, there was a minimum of disturbance by air bubbles and no channeling; however, there were some problems in stripping and regenerating the fluidized bed column.

An investigation of the degradation of normal paraffin hydrocarbon (NPH) diluent by nitration with HNO₃ solutions and NO₂ gas has continued. The principal degradation products (nitro, nitrite, and alcohol compounds) are formed primarily from the alkane constituents and not from impurities. The rate

of NPH degradation has been measured as the percentage conversions of NPH to the mononitrated compounds at 23°C; this rate is slow, being <0.2% in 24 h under most acid conditions. The rate increases significantly at higher temperatures, reaching 28% with the solvent in contact with 8 M HNO₃ at 106°C for 24 h and ~10% with 4.0 M HNO₃ at 104°C for 24 h. Secondary degradation products such as butyllauryl phosphate are believed to form when NPH is degraded in the presence of tributyl phosphate (TBP), because plutonium retention after Na₂CO₃ washes in such degraded solvents is significantly increased. The NPH degradation products (and secondary products) are not removed effectively by traditional wash and solid adsorbent methods; the use of 0.25 M KMnO₄–0.25 M Na₂CO₃ washes, while partially effective, produces MnO₂ precipitates resulting in severe phase separation problems.

3.1.3 Fission Product Studies

D. J. Pruett, J. C. Mailen, and W. B. Howerton

Studies of the coextraction of pertechnetate anions by uranyl cations into TBP-HNO₃ systems are continuing. In dilute solutions (<0.01 M in each reagent), the coextraction reaction can be quantitatively described as a simple exchange between one nitrate anion in the uranyl denitrated di-TBP complex in the organic phase and a pertechnetate anion in the aqueous phase. However, as the concentrations of the various species are increased and the temperature is varied between 25 and 60°C, the description of technetium distribution is more complex and requires further study.

The reaction between nitric acid and sucrose is being studied to determine if fission product precipitates will form during the denitration of waste solutions. A palladium precipitate forms within the first few minutes of the reaction. Characterization of the precipitate is incomplete, but it is not an oxalate and appears to contain cyanide groups in one or more of the ligands. Rare earth oxalates form only under relatively extreme conditions of high concentrations and low acidity. It was found that both Mn(II) and Fe(III), which are used to catalyze the nitric acid-sucrose reaction, catalyze the nitric acid-oxalate reaction even more effectively, preventing the accumulation of oxalate in the reaction mixture.

3.1.4 Solvent Recycle

D. W. Holladay, W. D. Bond, and W. B. Bolden

In concluding this phase of solvent cleanup studies, a continuous run with the aqueous phase in recycle was conducted using ⁹⁵Zr as an analytical label to confirm loading capacities in the aqueous phase and removal efficiencies for the major extractable components. The number of load cycles (volume of organic treated per unit volume of aqueous inventory) which can be achieved is controlled by the solubility of Zr (~0.02 M) and DBP (~0.1 M) and the resultant zirconium dibutyl phosphate compounds. Concentrations of all the extractable components in the organic raffinate were <0.001 M (limit of detection) for up to 100 aqueous load cycles. Based on minimum values determined for the scrubbing coefficients, these effluent concentrations correspond to decontamination factors (DFs) after 100 cycles of at least 10 for DBP, UO₂²⁺, NO₃⁻, and Zr. Based on the distribution of 0.1 mCi of ⁹⁵Zr as the radioactive label, the scrubbing coefficient for Zr(IV) was determined to be in excess of 10 000. In previous experiments using x-ray fluorescence for the analytical procedure, it had been possible to obtain a value of only 500. A coefficient value of 10 000 would provide a DF of ≥100 for zirconium after 100 load cycles (assuming no precipitates and good phase separation).

3.2 HOT-CELL DEVELOPMENT

D. O. Campbell and W. D. Bond

The hot-cell development task includes tests of solvent extraction flowsheet options in alpha-contained mixer-settler (ORNL) and pulsed column (SRL) equipment, specific tests in hot cells using irradiated fuel samples to study various reprocessing operations (with an emphasis on fuel solubility and insoluble residue characterization), and the procurement of irradiated fuels for hot-cell and solvent extraction flowsheet studies.

3.2.1 Plutonium Flowsheet Tests

B. A. Hannaford, W. D. Bond, and G. D. Davis

Two coextraction-costripping experiments were carried out with 0.6 M tri(2-ethylhexyl) phosphate (TEHP). These experiments conclude a series designed

to (1) establish flowsheet conditions for fast breeder reactor (FBR) fuel compositions, using dual scrub streams and dual strip streams; (2) demonstrate automatic controller response to a step increase in the aqueous feed (HAF) flow rate; and (3) measure plutonium losses to the stripped solvent (HCW) as a result of a step decrease in the strip solution (HCX) flow rate.

One experiment demonstrated that a 20% reduction in the HCX flow rate resulted in a twentyfold increase in uranium loss to the spent solvent (to $\sim 0.07\%$), but produced virtually no change in the plutonium loss (0.0001%). In the extraction-scrub bank, losses to the raffinate [high activity waste (HAW)] were 0.0001% and 0.02% for uranium and plutonium respectively. These were the lowest HAW losses for the series of experiments and are roughly comparable to losses which were measured in earlier coextraction-costripping experiments with 30% TBP.

The second experiment measured the response of an automatic density controller to a 20% step increase in the HAF flow rate. About 250 min were required for the controller to restore the heavy metal loading in the organic phase of stage 28 to ~ 15 g/L, after an excursion to ~ 25 g/L. Loss of plutonium to the raffinate increased briefly (from 0.2% to $\sim 1\%$) early in the transient before returning to normal.

Approximately 50 sets of equilibrium data for uranium, plutonium, and nitric acid were collected from the series of mixer-settler experiments with 0.6 M TEHP at temperatures of 40 and 50°C . Analysis of the data by multiple regression was begun to establish improved equilibrium correlations, particularly for plutonium, for use in solvent extraction computer codes.

3.2.2 Solution Stability and Solids Characterization

D. O. Campbell, J. H. Goode, J. W. Roddy,
and P. C. Arwood

Characterization studies were continued in the hot cell using the insoluble residues left after dissolving irradiated (U,Pu) O_2 in nitric acid. The solids tested were from the dissolution of ~ 50 -g batches of fuel from the PNL-59 series of irradiations in the General Electric Test Reactor (GETR) and from the dissolution of kilogram quantities of fuel from the PNL-3, PNL-59,

and P-15 experiments at the ORNL Solvent Extraction Test Facility (SETF).

Sedimentation studies, based on Stokes' law, indicated that the individual residue particles agglomerated into larger aggregates and that the aggregate size increased with increasing acidity. In the dissolver solution, $\sim 94\%$ of the aggregate particles were larger than $10\text{ }\mu\text{m}$ and 40% were larger than $20\text{ }\mu\text{m}$. In water, the amounts larger than these sizes were ~ 7 and $<1\%$ respectively. These results indicate that the dissolver or cladding rinse solutions should contain nitric acid to prevent peptizing the residue into very small particles that would neither filter nor centrifuge.

Replicate determinations of density were made using the residue remaining after the dissolution (in 8 M HNO_3) of mixed oxide that had been irradiated to $\sim 37\,000$ MWd/t. The measurements indicated an apparent density of $7.75 \pm 0.12\text{ g/cm}^3$. The densities were determined by the displacement of water from a pycnometer by samples of residue. A UO_2 pellet, used as a control sample in the same equipment, gave a density of $10.57 \pm 0.01\text{ g/cm}^3$.

The Stoke's law sedimentation technique for determining particle size distribution was also applied to the solids remaining after dissolving 460 g of irradiated LWR fuel in HNO_3 and washing the residue with HNO_3 . The sedimentation data indicated that $>95\%$ of the agglomerated residue particles were larger than $4\text{ }\mu\text{m}$ and $\sim 10\%$ were larger than $20\text{ }\mu\text{m}$.

After the residue was washed and ultrasonically dispersed six times in water for particle size measurements, the particle aggregates were smaller (as in earlier work with LMFBR residues); the aggregates did not, however, peptize as was expected. The data from these tests indicated that 95% of the aggregates were larger than $4\text{ }\mu\text{m}$ and 90% were larger than $\sim 10\text{ }\mu\text{m}$; however, the largest particle agglomerates were only $\sim 25\text{ }\mu\text{m}$ in diameter as compared to $\sim 100\text{ }\mu\text{m}$ in diameter in 3 M HNO_3 .

3.2.3 Pulsed Column Facility

C. E. Coffey, O. M. Ebra-Lima, and D. J. Reif
(Savannah River Laboratory)

Studies with plutonium present in the system were started in the SRL Pulsed Column Facility

(PCF). Four runs were made with simulated LWR fuel dissolver solution containing 2 g/L of Pu, 153 g/L of U, and 3 M HNO₃.

Although the PCF has provisions for monitoring the aqueous exit stream density in real time, these values were not useful in detecting steady-state operation because of solvent entrainment.

Three columns were used in the runs to provide the functions of extraction, scrubbing, and costripping. The feed point to the extraction column was varied to change the length of the extraction section and to determine the effect of this change on metal recoveries.

Measurement of the density of the aqueous solution leaving the scrub column indicated that steady state in this column was achieved 50 to 60 min after system startup.

3.3 ANALYTICAL DEVELOPMENT

D. A. Costanzo

This task is concerned with the development of analytical methods, equipment, and support facilities for the overall program. Included are the development of chemical, nuclear, radiochemical, x-ray fluorescence, and mass spectrometry methods suitable for high-burnup, highly radioactive fuel; the remotization and automation of analytical equipment; and the development of sampling requirements, mechanisms, and laboratory investigations to support HEF design studies.

3.3.1 X-Ray Fluorescence (XRF) Systems Development

J. H. Stewart, Jr. and C. J. Sparks, Jr.

The design and fabrication of optical systems for a proof-of-principle XRF spectrometer for the analysis of highly gamma-active samples has been completed. The linear position-sensitive x-ray detector was tested and installed on the spectrometer, and the electronic timer-scalers and control modules were connected for the initial testing of the spectrometer. A sample holder was also designed and fabricated. The total optical system design proposal report was issued. Cold testing of the integrated spectrometer has been initiated.

A joint effort among the Chemical Technology, Metals and Ceramics, and Analytical Chemistry Divisions is under way to develop an x-ray fluorescence capability for quantitative analysis of highly radioactive residues from head-end dissolver solutions using the shielded

scanning electron microscope (SEM) in Building 3525. Computer software has been developed using a similar instrument at the Oak Ridge Gaseous Diffusion Plant (ORGDP) and nonradioactive materials. These developments will be transferred directly to the ORNL instrument.

3.3.2 Spark-Source Mass Spectrometry (SSMS) Development

R. J. Warmack and J. A. Carter

Dissolver solutions and residues are being analyzed by the new high-gamma-level SSMS facility. Striking differences in isotopic abundances for certain elements (e.g., molybdenum and polonium) were observed when comparisons between solution and residue were made. The isotopes for most elements, however, agreed well within ORIGEN code calculations.

Preliminary work has been initiated to improve elemental sensitivity factors for the samples by using prepared (cold) standards. An on-site computer facility is being installed and will be used for data analysis and future control applications.

3.3.3 Hot-Cell Analytical Instrumentation

L. N. Klatt and D. E. Goeringer

The design and fabrication of the titrant delivery unit for the prototype microcomputer-controlled remote titrator has been completed. The unit incorporates several mechanical concepts used in the previously developed remote pipetter system. The electronic interface providing for computer control of the titrant delivery unit has been completed, the digital plotter that will provide hardcopy of the titrator output has been received, and the interface to the microcomputer controller has been developed. Integration of the components is in progress. The initial software design for control of the prototype titration system is complete; modifications in the control program will be made as system evaluation begins. A computer simulation study of the effect of electronic noise on the ability of a smoothing-differentiation algorithm to locate the end point of a titration was completed. Results suggest that the technique should give accuracies and precisions that are <0.2%.

The hot-cell version of the remote pipetter system installed in Building 2026 is performing as designed. A few minor control program modifications have been made to further facilitate use of the system.

4. Engineering Research

W. S. Groenier and R. M. Counce

The engineering research studies represent the first phase of the equipment development program and provide a basis for further design and development of specific engineering test components. Emphasis is placed on the identification, study, selection, development, and characterization of unit processes through the application of basic or fundamental experimental engineering techniques.

Studies of the voloxidation, dissolution, feed preparation, solvent extraction, and off-gas treatment processes are in progress, and development efforts are described. Work directed toward the development of voloxidizer and solvent extraction equipment is also reported.

4.1 VOLOXIDATION

M. E. Whatley

The voloxidation process is being developed as a dry head-end method for removing tritium from spent uranium reactor fuel prior to aqueous processing. The process is based on the reaction of the oxide fuel with oxygen or air at a temperature range of 720 to 920 K, where the release of tritium and, to a lesser extent, some of the other fission products occurs when UO_2 is restructured to U_3O_8 . The rationale for process development assumes that it is more desirable to remove tritium from the fuel and concentrate it into a relatively small volume than to dilute the tritium with water in subsequent aqueous processing steps. Additional benefits of voloxidation include oxidation of residual sodium in the fuel prior to dissolution, an increase in the rate of dissolution, and provision of a temperature-controlled surge capacity between shearing

and dissolution. The overall basis for this process step is currently being reviewed by a task force that will make recommendations, as appropriate, for future research and development requirements.

An investigation of the applicability of rotary kilns to the voloxidation process has been continued by performing heat and mass transport tests using a small rotary kiln and continuing a kiln-seal evaluation study. Tests of a 0.5-t/d voloxidizer system are also described; this is a major effort that features the test and evaluation of commercial components.

4.1.1 Full-Scale Experimental Voloxidizer (FSEV)

B. B. Spencer

Tests of a full-scale voloxidizer system are continuing for simulated operating conditions. A new kiln drum with prototypical internals has been installed. Activities addressing the operation and the instrumentation and controls of this unit are described.

Operations

B. B. Spencer, M. E. Whatley, S. A. Richardson, and D. R. Trotter

Data on the FSEV off-gas filter show that there is an ~ 5.4 -cm H_2O pressure drop through the clean filter for each liter per second of flow. During run 17, the filter began to plug as evidenced by the increasing pressure drop readings. The filters have since been cleaned and reinstalled.

Run 18, a 108-h campaign, has been completed. The primary reasons for this FSEV run were to (1) demonstrate drum temperature profile control, (2) obtain data on the cooling stack performance, and (3) test the drive system at a high rotational speed (4.0 rpm in a working range of 0.4–5.6 rpm). Preliminary results indicate that when the temperature of zone 4 (which is close to the middle of the furnace) is maintained at $500^\circ C$ and all of the other zones are maintained at $600^\circ C$, the cooling stack for zone 4 is removing ~ 6 kW of heat. This value will be compared with the heat removal value predicted by the existing mathematical model. No problems were encountered with the drum drive system. Prior to this run, an air tank was added to the solids recirculating subsystem. A solenoid valve was installed to activate the recycle air pulse. These modifications were implemented to regulate the volume of air added to the voloxidation system from this source. During the run, the solids recirculating subsystem was observed to perform satisfactorily. Following the run, the voloxidizer was operated in the surge volume mode until the drum ceased to accumulate material (i.e., until material began to flow out of the drum); measurements of the solids inventory profile were then obtained.

Instrumentation

W. F. Johnson and J. K. Yarbrough

A new furnace temperature control scheme was installed prior to run 18 and was used through the run. It involves a cascade control arrangement between software and hardware controllers associated with the furnace heaters. The new configuration was subjected to a number of furnace setpoint adjustments during the run, and preliminary results indicate that it not only responds well to setpoint changes, but also prevents

simultaneous furnace heating and cooling as experienced with the old control scheme.

Problems have been encountered with the telemetry system used with thermocouples attached to the voloxidizer drum to measure drum temperatures. An effort to determine the cause of sporadic readings is under way.

Data Analysis

B. B. Spencer, M. E. Whatley, and D. R. Trotter

Data logged on 21 cassette tapes during run 18 have been edited and placed on magnetic tape. Extraction of the data in the form of plots of zone temperatures, differential pressure across the filter, off-gas flow rate, feed rate, and cooling stack thermal load vs time is complete.

4.1.2 Rotary-Kiln Heat and Mass Transport Studies

B. B. Spencer, M. E. Whatley, and D. R. Trotter

Analyses of the gas-phase residence-time-distribution (RTD) data collected from the small voloxidizer have continued. Under isothermal conditions and with high solids holdup ($\sim 50\%$ inventory of quartz sand), the dispersion numbers calculated, using the new model, are considerably smaller than those calculated from the ordinary dispersion model. This was expected since the increased deadwater fraction contributed more significantly to the spread of the distribution.

A planned group of experiments to study the effects of a temperature field on gas-phase dispersion was completed. Again, a 50 vol % holdup of sand was used. Air flow rates and drum rotational speeds were varied, and drum temperatures ranged up to $200^\circ C$.

Additional experiments are under way using sheared prototype fuel to determine the effects of the presence of large solid particles on gas dispersion and on the exchange rate of the bulk gas with gas trapped in the solids bed.

4.1.3 Rotary-Seal Tests

J. G. Morgan and D. J. Kington

The development of seals to isolate the voloxidizer interior from the surrounding atmosphere is being continued. As the operating temperature is reached, thermal expansion causes the discharge end of the

prototypical voloxidizer kiln to extend 1.5 in. horizontally. A corrugated metal diaphragm is currently proposed for use as part of the end seal subassembly to accommodate this movement. A base design has been established for this diaphragm, using the stress analysis computer program NEPSAP. A 3/4-scale model of this diaphragm design has been ordered for testing in the seal test stand.

4.1.4 Rotary-Kiln Corrosion Test

J. G. Morgan, W. D. Holland, and D. J. Kington

Tests to evaluate potential voloxidizer materials of construction are continuing. Two rotating drums constructed of type 316 stainless steel and Incoloy 800 have been exposed to a moist iodine atmosphere at a temperature of 650°C. Each drum has mixing flights and contains sand and stainless steel slugs as simulated spent fuel.

The corrosion test with type 316 stainless steel was terminated after 4000 h of operation. Ultrasonic wall thickness measurements were made, and the results are being analyzed. The other corrosion experiment with an Incoloy 800 drum is continuing.

4.1.5 Temperature Measurement

J. G. Morgan and D. J. Kington

A new method for measuring the temperature of a rotating surface (such as a voloxidizer drum) is needed. Previous methods employ optics or electronics; these methods are generally not reliable for service in a radioactive environment. Experimental work has started on a concept which uses a parabolic reflector to concentrate the heat flux from the drum surface on to a thermocouple junction at the focal point.

Although a gold surface is most efficient for reflecting infrared radiant energy, a gold plating on stainless steel will diffuse into the substrate at voloxidation temperatures. Recently, a successful test was made with a reflector that contained an intermediate buffer layer of SiO₂ which prevented diffusion of the gold. The test, which was made at 650°C, was run for 1000 h.

4.1.6 Tritium Retention System Development

R. M. Counce and W. D. Holland

The purpose of this activity is to design a pilot-plant-scale drying system for voloxidizer off-gases.

Present efforts have been directed toward (1) establishing the thermal diffusivity of the packed drying bed and (2) studying procedures for condenser design of a system consisting of a vapor and a noncondensable gas.

4.2 DISSOLUTION

The objective of this task is to develop the technology for the dissolution of liquid-metal fast breeder reactor fuels in nitric acid that will provide high metal recoveries and maximize the volatilization of iodine from dissolver solutions. Nuclear criticality and off-gas control considerations are two of several important factors that require dissolver design and operation for breeder fuels to be performed within rather narrow limits. Continuous dissolving methods are being emphasized; these appear to offer superior solutions to these problems. An evaluation of an advanced dissolver design concept and the development of a method for providing solids agitation in a digester tank are included in the current effort. A material balance model for compartmentalized rotary dissolvers is also being developed.

4.2.1 Advanced Rotary Dissolver Development

W. E. Eldridge

Dissolver development to provide future dissolver alternatives is being continued. The concept being investigated features a spiral-screw baffle for solids movement; liquid flow is countercurrent to the flow of solids.

A series of tests designed to study the behavior of liquid transfer in the advanced rotary dissolver has been completed. Data analysis indicates that some deadwater regions exist in the current model and some degree of liquid channeling is evident. These are problems that can be corrected by internal modification of the drum.

The experimental test program has been concluded. Sufficient information is now available to design and construct a larger-scale version of the present advanced model.

4.2.2 Solids Suspension Tests

B. E. Lewis and J. F. Lockmiller

The digester tank test stand has been fabricated to study various means of suspending solids in simulated digester operation.

A study of the effect of sheared fuel spacer wires on solids suspension in the digester tank has continued. The annular air supply pulsed lift tube was tested with 25 g of 2.54-cm-long by ~ 0.16 -cm-diam stainless steel wires, using various air injection elevations in a solution of $\text{Ca}(\text{NO}_3)_2$ with a density of $\sim 1.3 \text{ g/cm}^3$. The annular air supply was not successful in keeping the wires off the bottom of the tank. The use of a satellite 1-cm-diam air supply line in place of the annular air supply provided much better results. All wires were kept in suspension except for occasional entrapment between the inside wall of the tank and the satellite air supply line. This problem can be easily corrected by centering the air supply line between the tank wall and pulsed lift tube. Also, with a single satellite air supply line operating at low air flow velocities ($\sim 58.5 \text{ cm/s}$, based on lift tube area), there was a tendency for wires to accumulate on the bottom of the tank in dead spaces opposite the supply line. At higher air velocities ($\sim 87.7 \text{ cm/s}$) as much as 100 g of wires was maintained in suspension.

In other tests with a multiple air supply, the presence of a central eductor line within a modified pulsed lift tube did not appear to interfere with the suspension of 100 g of wires in the lift tube at air velocities of $\sim 26.3 \text{ cm/s}$ up the lift tube. However, about half the wires were held up inside the tube and not ejected.

Additional runs were made with the modified multiple air supply pulsed lift tube using tungsten powder and 100 g of spacer wires. Various air flows and solids concentrations were studied. Analysis of the data is not yet complete; however, the initial results indicate a fairly high degree of suspension for the lower solids concentrations.

The effects of large amounts of foam on the operation of the pulsed lift tube were studied by adding various quantities of soap to the tank during operation. Large amounts of foam caused the overflow of both liquids and solids from the test tank. Smaller amounts of foam (~ 3 - to 4-cm-thick layer above the liquid level) did not appear to interfere with the tank operation; however, a complete analysis of the data has yet to be made. Prototypic amounts of foaming are difficult to estimate; however, large amounts of foam would be

objectionable in the Hot Experimental Facility digester tanks.

Tests were also run using 149 g of dissolver residue from an iron dissolution run in the prototype dissolver. The residue consisted primarily of agglomerates of small spheres of iron, iron dust, hull fragments, and about three spacer wires. All but the larger spherical agglomerates (~ 0.64 -cm-diam) were suspended. The gap between the bottom of the lift tube and tank bottom appeared to be too small to pass the large agglomerates.

4.2.3 Concentration Profile Studies

B. E. Lewis

A computer code has been developed to model the liquid and solid phase inventories in the continuous rotary dissolver. The code provides stagewise values of dissolved heavy metal and particulate concentrations. This code is useful for predicting dissolver performance under upset conditions and in providing information for criticality calculations.

Recent efforts have been directed at improving the particle balance portion of the code, upgrading the operational efficiency, and initiating documentation in the form of a user's manual.

4.3 FEED PREPARATION

R. M. Counce

The aqueous feed discharged from the primary dissolver system will contain solids (undissolved fission products, corrosion products, etc.) and will require clarification prior to solvent extraction. Preparation of the feed for solvent extraction will also include adjustment of the plutonium valence and the nitric acid concentration, as well as treatment to remove iodine. This task also encompasses the development of a variety of other processes associated with the liquid portion of fuel reprocessing, such as secondary dissolution methods (with more corrosive reagents and for fuel constituents not soluble in the primary dissolver), accountability systems, and fluid transfer and metering systems.

Present emphasis is on the testing of an extractive distillation system for nitric acid having application to the Iodex off-gas treatment process, development of a fluidic pump, and development of feed clarification methods.

4.3.1 Extractive Distillation of Nitric Acid

R. M. Counce and L. E. Thompson

The system being investigated represents a simplified concept for the concentration of nitric acid by extractive distillation. This step is necessary to convert low-strength nitric acid to the hyperazeotropic concentrations necessary for the Iodex process. The current two-pot experimental system uses $\text{Mg}(\text{NO}_3)_2$ as a dehydrating agent.

Available vapor-liquid equilibria information for the $\text{Mg}(\text{NO}_3)_2\text{-HNO}_3\text{-H}_2\text{O}$ system have been empirically correlated; these correlations are being incorporated into a topical report being prepared on the experimental investigation of the two-pot concept for nitric acid concentration.

The experimental apparatus has been modified to study vapor-lift pumping for the recycle of the $\text{Mg}(\text{NO}_3)_2$ liquid phase within the system. If successful, this pumping concept will further simplify the system by the elimination of mechanical pumping.

4.3.2 Fluidic Pump Development

R. M. Counce, G. V. Smith, and L. E. Thompson

The use of fluidic pumps with no moving parts offers an attractive alternative for the movement of radioactive and hazardous fluids. Data have been collected and analyzed for a fluidic pump using an axisymmetric reverse-flow diverter (RFD). Experiments were conducted to determine the effects of various geometrical parameters, operating conditions, and fluid properties on the performance of the RFD and pumping system. Acceptable agreement was found to exist between experimental data and theoretical predictions from the mathematical model of the RFD and pumping system. Future efforts will be directed toward the development of a fluidic pump to produce a steady continuous flow.

4.3.3 Feed Clarification Equipment Development

C. E. Coffey, O. M. Ebra-Lima, and
G. C. Rodrigues (Savannah River Laboratory)

Studies are under way at Savannah River Laboratory (SRL) to evaluate alternatives for clarification of dissolver solutions for subsequent solvent extraction operations. This activity used both nonradioactive

solutions and dissolved light-water reactor (LWR) fuel solutions to test alternative equipment.

Bench-scale clarification tests with dissolved irradiated LWR fuel in the continuous De Laval Gyrotester* centrifuge have begun in the SRL shielded cell facility. Scouting tests compared the settling rate of Dresden-1 fuel residue with that for Oconee-1.

Dresden-1 fuel (7.3 kg) was obtained from ORNL to provide a consistent and appropriate feed material for bench-scale tests. This is an LWR fuel irradiated to 23 780 MWd/t and cooled for ~6 years. The gamma activity is sufficient for adequate sensitivity in clarification measurements. Because its original enrichment was only about 1.5%, the remaining fissile content amounts to only 83 g of ^{235}U equivalent. This will greatly simplify disposal after completion of the program.

After complete cold assembly and operation of the apparatus in mock-up cells, dissolving and voloxidation equipment was set up in the shielded cell along with the De Laval Gyrotester centrifuge. The centrifuge bowl was modified to perform more like a basket centrifuge by removing the disk pack and center feed guide.

Approximately 1 kg of Dresden-1 fuel was voloxidized and dissolved to a final uranium concentration of ~200 g/L in 3 M nitric acid. This solution is being fed to the centrifuge at feed rates and centrifugal field conditions which correspond to those run in scouting tests.

A scouting test to compare the settling behavior of the Dresden-1 fuel residue with that of Oconee-1 showed that the Dresden residue settles approximately six times as fast. The average particle diameter of Dresden-1 residue is ~0.15 μm .

4.4 SOLVENT EXTRACTION

M. E. Whatley

The development of solvent extraction techniques for the Purex process recovery of advanced fuels is continuing at several sites. The primary objective of an activity at the Argonne National Laboratory (ANL)

*Trade name.

is to design, fabricate, and test annular centrifugal contactors of critically safe dimension that will provide the basis for a subsequent effort at SRL to provide a multistaged system. At ORNL, efforts are under way to improve and apply the SEPHIS and MATEX computer codes for flowsheet engineering studies. Additional work is in progress at the General Atomic Company (GA) to perform contactor improvement studies, test flowsheets, and test instrument application concepts.

4.4.1 Centrifugal Contactor Development

R. A. Leonard, R. H. Pelto, and A. A. Zeigler (ANL)

Work is continuing in the development of annular centrifugal contactors. Two different contactors are being studied; one has a 12-cm-diam rotor, the other a 4-cm-diam rotor. Based on the development of the 12-cm contactor at ANL, a prototype multistaged 12-cm contactor has been designed at SRL and is now being fabricated. This unit will eventually become a part of the 0.5-t/d Integrated Equipment Test (IET) facility at ORNL. Work is continuing at ANL on specific single-stage parameters pertinent to the design and operation of the 12-cm contactor in a multistaged array. Assistance is being provided to SRL as needed.

The 4-cm contactor is being used to evaluate multistaged operation of large-scale contactors, as it does not have the high resistance to phase inversion previously encountered with miniature (2-cm-diam rotor) contactors. Tests of the four-stage 4-cm contactor reported below include tests where three liquid phases were present and single-stage tests of changes of the contactor mixing zone to improve extraction efficiency.

Multistaged Contactor Operation

Multistaged uranium extraction efficiency tests were completed using the four-stage 4-cm contactor. The results show that individual stage efficiencies average 90.5% and are equivalent to single-stage operation, where the stage efficiency is 91.8%. The unit reaches steady state quickly, within three solution residence times.

In the 12-cm contactor, the air-controlled weirs can be adjusted to flush the organic phase out of the unit when the contactor is shut down. Since the 4-cm contactor has no air-controlled weir, a new method was

devised for flushing the organic phase out of the contactor. This new method will also be useful for flushing out particles that accumulate at the interface between the two liquid phases in the separating zone of the rotor.

Improved Contactor Design

A study of the mixing zone has continued with the objective being to maintain a high extraction efficiency over a wide range of throughputs without sacrificing liquid height in the mixing zone or seriously altering the organic-to-aqueous (O:A) ratio at which phase inversion occurs.

Three changes to the mixing zone (a notch at the bottom edge of the rotor, a taper on the bottom surface of the rotor, and a narrower annular gap in the mixing zone) seemed to improve the operation of the mixing zone. These changes were further evaluated by measuring the effect on extraction efficiency, annular liquid height, and the O:A flow ratio at which phase inversion occurs.

Preliminary extraction efficiency results using the single-stage 4-cm contactor indicate that the efficiency increases as the annular gap decreases. In addition, at low flow rates, a tapered rotor bottom gives a further increase in efficiency. In general, mixing zone changes which increased extraction efficiency also increased the annular liquid height.

The narrower annular gap had the largest effect on mixing zone operation; benefits are both a higher extraction efficiency and a more favorable O:A flow ratio at phase inversion. In fact, the extraction efficiency of the 4-cm contactor was increased from 92 to 98–99%, values comparable to those obtained with the 12-cm contactor. To obtain the benefits of the narrower annular gap, the contactor must operate at a higher annular liquid level. For the 4-cm contactor operating at high throughputs, the annular liquid height almost fills the mixing zone. When the annular liquid height exceeds the height of the mixing zone, the dispersion will partially bypass the rotor making contactor performance unsatisfactory. Thus, the annular gap is decreased until the benefits of increased extraction efficiency are balanced by the loss of contactor performance that results as the annular liquid height exceeds that of the mixing zone.

To relate visual observations of the mixing zone in the 4-cm contactor to that in the 12-cm contactor, a transparent housing was built for the 12-cm contactor. Initial results show that 12-cm contactor operation should be good from 3 to 20 L/min at 1760 rpm. At lower flows (e.g., 2 L/min), some loss in extraction efficiency would be expected. Based on the 4-cm contactor work, it is expected that a 12-cm contactor with a tapered rotor bottom would operate with high extraction efficiency at flow rates well below 2 L/min.

Operation with Three Liquid Phases

Using the four-stage 4-cm contactor with three liquid phases present (a modified Thorex flowsheet), it has been shown that single-stage and multistaged contactor operation is possible if the aqueous weir diameter (or the aqueous weir pressure for contactors with air-controlled weirs) is changed. By reducing the flow to one-third the normal rate, a stage could be designed to handle either two or three phases. To operate a multistaged contactor at the normal flow rate, the number of liquid phases in each stage must be determined and the design of each stage adjusted accordingly. When this is done, the presence of three liquid phases in several stages of a multistaged annular centrifugal contactor does not interfere with operation.

4.4.2 Flowsheet Engineering Studies

R. T. Jubin and M. E. Whatley

The MATEX computer code, which models the steady-state behavior of solvent extraction systems, has been modified to reduce computer costs by removing unnecessary statements and storage locations. Testing of the code has continued with successful solution of trial problems.

The *Users Guide to MATEX* was also written and should be published early in FY 1982.

4.4.3 Alternate Contactor Studies

G. E. Benedict and

L. J. Olguin (General Atomic Company)

Tri(2-ethylhexyl) phosphate (TEHP) has been selected as a possible alternate solvent to tributyl phosphate (TBP) for solvent extraction activities connected with nuclear fuel reprocessing. An activity

plan has been prepared for preliminary tests with 51% TEHP solvent.

Pulsed Column Tests

L. J. Olguin and D. A. Setzer

Tests have been conducted with 51% TEHP in the A-column extraction section. Results show that the extraction column flooding frequency is greater with 51% TEHP than with 30% TBP at low volume velocities. Attempts to operate the extraction column at volume velocities greater than 1.7 cm/s (1500 gal h⁻¹ ft⁻²) gave flooding frequencies much lower than with 30% TBP. The height equivalent to a theoretical stage (HETS) value based on the organic phase was 2.1 m (6.8 ft) for the top section of the column in this test. However, buildup of the degradation product [di(2-ethylhexyl) phosphate (DEHP)] may have influenced the column efficiency and capacity.

Six stripping column tests were also performed with 51% TEHP to determine the shape of the flooding curve. Previous studies at ORNL have shown that uranium has a higher O:A phase distribution ratio at low acidities in TEHP than in TBP. Therefore, the strip column was operated with an O:A flow ratio of 1:3, resulting in a lower uranium concentration in the 1CU stream. The strip column flooding curve for TEHP closely approximated the TBP flooding curve developed with an O:A ratio of 1.0:1.25.

Sodium Carbonate-TEHP Wash Study, Robatel Centrifugal Contactor

L. J. Olguin and D. A. Setzer

The Robatel centrifugal contactor has been tested for applicability as a liquid-liquid centrifuge for the Na₂CO₃-TEHP wash system. Laboratory experiments showed that the two liquid phases are difficult to separate. Initial Robatel washing tests with 30% TBP and 0.25 M Na₂CO₃ were successful. Complete phase separation was achieved in the range of 2000 to 3000 rpm and 1.0 to 1.25 L/min total flow rate. In a similar washing test with 51% TEHP and Na₂CO₃ solution, the organic phase did not separate completely for any of the operating conditions tested. The test results show that (1) increasing the Na₂CO₃ concentration decreased the volume of entrained aqueous phase in the organic phase, (2) reducing the number of Robatel

stages from eight to three had no effect on the separation of the phases, (3) foaming of the combined phases occurred at low (0.05 M) Na_2CO_3 concentrations, and (4) decreasing the Robatel rotational velocity reduced the aqueous holdup in the organic phase.

TEHP-Uranium Equilibrium Study

D. A. Setzer

A bench-scale study was conducted to determine the equilibrium distribution of uranium between 51% TEHP and 0.01 M HNO_3 at 50°C for use in evaluating the efficiency of the stripping column. Results from a previous stripping mode equilibrium study conducted at ambient temperature were compared to the study at 50°C. At the higher temperature, the uranium was loaded preferentially into the aqueous phase. At ambient temperature, however, more uranium distributed to the organic phase than to the aqueous phase.

4.5 OFF-GAS PROCESSING

This task consists of the development of various processes for the treatment of reprocessing plant off-gases to ensure that environmental goals are met. Work includes the continued development of the noble gas and ^{14}C selective absorption process and the formulation of plant engineering design criteria. The use of solid sorbents for various off-gas purification applications is also being evaluated. Tests of advanced sorbent materials are under way at ORNL, and work on an integrated high-temperature gas-cooled reactor (HTGR) off-gas treatment system is proceeding at GA. Savannah River Laboratory (SRL) is involved in a new effort which will lead to an off-gas processing demonstration using a sidestream of a radioactive off-gas stream at the Savannah River Plant (SRP).

4.5.1 Fluorocarbon Absorption Process Development

D. K. Little, R. S. Eby, and J. L. Patton (ORGDP)

Studies have continued at the Oak Ridge Gaseous Diffusion Plant (ORGDP) to develop a selective absorption process for ^{85}Kr and ^{14}C removal. These studies have focused on three areas: operation of the single-column, operation of the product purification equipment, and process application studies. Operation of the single-column pilot plant attempted to identify any

differences between operations using a feed gas containing air and those with a feed gas containing nitrogen. Nitrogen is the normal process gas used because of its convenience. These tests were delayed because of a loss of capacity of the reboiler heater. After heat diagnosis, it was decided to resume testing at a reduced power level until repairs could be made. The tests are currently in progress.

The test runs of the product purification equipment (cold trap studies) that have been completed focused on operating conditions and performance parameters for the cold trapping of krypton, xenon, and carbon dioxide. Gamma scanning equipment was installed to facilitate the study of trap plugging and component distributions during operation.

Process application studies have been initiated in response to an interest in using the selective absorption process to alleviate an expected market shortage of ^{85}Kr . Discussions with SRL personnel focused on the use of the proposed Integrated Hot Off-Gas Facility (IHOGF) as a good starting point for off-gas technology development and as a producer of krypton to meet the expected demand. Unfortunately, developmental goals and construction details of the IHOGF did not appear amenable to continuous production or efficient recovery of krypton. It was felt, however, that a new facility similar to the IHOGF, but dedicated to production, could produce ~50 000 Ci/year and therefore meet the expected demand. Located at the SRP, the plant would cost about \$8–10 million. Further investigation of this application of the selective absorption system is continuing.

4.5.2 Integrated Hot Off-Gas Facility

C. E. Coffey, O. M. Ebra-Lima, A. L. Blancett, A. J. Hill, Jr., S. R. Smith, M. C. Thompson, and R. C. Waggoner (Savannah River Laboratory)

Activities are under way at SRL to build and demonstrate the proposed IHOGF. This facility will treat an actual fuel reprocessing off-gas using a sidestream from SRP.

Design of the first phase of the IHOGF by Allied-General Nuclear Services (AGNS) is proceeding on schedule. The process and instrument drawings were completed and approved. Preliminary drawings of the

equipment and layout of the packed-bed columns have been received for review. A tentative schedule for fabrication of the facility modules by AGNS was also received. Long-term delivery items were identified. The scope of on-site work for the IHOGF was completed by the SRP Project Department.

The estimated total cost of IHOGF Phase I is \$3 million, including escalation, contingencies, and design.

The study of analyzers for NO/NO_x and NH₃ is complete. Specifications are being prepared for purchasing the instruments required for process control and operating data collection. Alternative process control systems and data acquisition/logging systems are now being evaluated.

Instrumentation required for safe operation of the IHOGF has been identified. Each of the monitors will incorporate high/low alarms and provision for automatic shutdown, as needed.

The SRP F-Area off-gas was sampled on May 28. Excessive moisture caused plugging of the NaOH trap after 1 h of operation, but samples were obtained for analysis. The moisture and CO₂ traps will be changed to avoid plugging.

An economic and engineering analysis is being carried out to evaluate and compare the various methods for ⁸⁵Kr collection and concentration. The study is using available data on cost, equipment specifications, and sizing. Factoring methods will be used to adjust the data to selected flows and to escalate costs to 1981 dollars.

4.5.3 Solid Sorbent Tests

R. T. Jubin and J. D. Randolph

There are several locations within the HEF reference flowsheet where solid sorbents are being proposed to treat off-gases. Sorbents, such as metal-substituted mordenites and silicic acid, are being evaluated for those applications.

Tests have been conducted to evaluate the iodine loading performance of low silver-exchanged mordenites (LAgZ). Silver utilization of this material is ~100% under many loading conditions. This compares to ~60% silver utilization of fully exchanged silver mordenite (AgZ). To determine the effects of NO, NO₂, H₂O, bed temperature, H₂ concentration, and silver exchange

on the CH₃I trapping capacity of the LAgZ, a scoping study was conducted using an eight-run fractional factorial experimental design.

Initial analysis of the data has indicated that (1) increased loadings occur with increased silver content (as would be expected), (2) higher loadings occur at 200°C than at 250°C, (3) the presence of 1% hydrogen in the gas stream improves loading, (4) moisture has a detrimental effect on the loading of CH₃I, (5) the presence of NO₂ has a detrimental effect on the loading, and (6) the observed effect of NO on loading was smaller than the response error and is not significant.

Several of the responses appear to be contradictory to those found for AgZ, for example, the effect of moisture.¹ Future work is planned to evaluate these differences and to select an optimum degree of silver exchange to minimize waste and cost.

4.5.4 Integrated HTGR Off-Gas Treatment System

G. E. Benedict and L. Abraham (GA)

Radon Adsorption/Decay Test

L. Abraham, K. Y. Higuchi, and P. M. Hirsch

Average decontamination factors (DFs) for radon (~10 and 100) were obtained with a nitrogen carrier gas flow rate of about 17 m³/h (10 scfm) in radon adsorption and decay tests using a 345-mm (13.6-in.)-diam column and hydrogen mordenite bed depths of 1.07 m (3.5 ft) and 1.52 m (5.0 ft) respectively. The DF is the ratio of integrated alpha counts in the feed to those in the column effluent.

External sources of electronic interference produce relatively high electronic noise-to-signal ratios in the radon detection system. Since this effect would be more pronounced at the higher DFs expected in subsequent tests to be conducted at adsorber bed depths of up to 3.05 m (10.0 ft), attempts have been made to reduce the electronic noise-to-signal ratio. For the same reason, the configuration of the detection chamber has been modified to improve the alpha detection efficiency.

CO₂/Krypton Separation Test

L. Abraham, K. Y. Higuchi, and P. M. Hirsch

A time-of-flight mass spectrometer is used to measure krypton concentrations in the CO₂/krypton

separation column feed and effluent streams. A procedure was developed for calibrating the mass spectrometer, and calibrations were completed for krypton concentrations from 10 to 10 000 ppm. The mass spectrometer readout computer was interfaced with a Hewlett-Packard 9825A data acquisition and processing system. The test system valving arrangement has also been changed to permit rapid changeover from the radon test to the krypton test configuration. A shake-

down run of the CO₂/krypton separation system was completed.

REFERENCE

1. R. T. Jubin, "Organic Iodine Removal from Simulated Dissolver Off-Gas Streams Using Silver-Exchanged Mordenite," *Proc. 16th DOE Air Cleaning Conference*, CONF-801038, p. 519 (October 1980).

5. Engineering Systems

M. J. Feldman

The scope of the work performed in the area of Engineering Systems includes the development of prototype equipment and the conceptual design of the Hot Experimental Facility (HEF). The design of prototype equipment is based on other research and development efforts, primarily those of the Process and Engineering Research and Development section of the CFRP. These efforts include the development of individual equipment items and process systems. The Integrated Equipment Test (IET) facility will be used for the synergistic testing of the equipment and the testing of the remote operation capabilities and characteristics of these components and systems.

The conceptual design of equipment and systems to be incorporated into the HEF will be derived from these development efforts. Work is also being conducted in the areas of safeguards, nuclear engineering, and environment and safety. These areas are in support of the equipment, systems, and HEF design efforts.

5.1 ENGINEERING STUDIES AND ANALYSIS

J. G. Stradley

The various studies reported in this section are not specific to any one functional research and development area but are important to the overall fuel reprocessing program. Specific generic areas being studied are safeguards, nuclear engineering, and environment and safety. Other studies perform analyses in broad areas that overlap individual tasks and provide background and recommendations for decisions pertaining to programmatic guidance and direction.

5.1.1 Engineering Analysis

Evaluation of Utilization of the Barnwell Nuclear Fuel Plant in Support of the U.S. Breeder Reactor Reprocessing Program

B. F. Bottenfield, D. C. Hampson, E. D. North, R. H. Rainey, J. N. Herndon, F. L. Peishel, D. P. Reid, J. G. Stradley, and L. L. Thomas (Allied-General Nuclear Services)

An earlier study was performed to evaluate the potential contributions that the Barnwell Nuclear Fuel Plant (BNFP) could make to the U.S. breeder reactor reprocessing program. The usefulness of the plant in

performing technical and programmatic demonstrations of breeder fuel reprocessing was evaluated. It was concluded that the existing BNFP facility could not be used to demonstrate the reprocessing of breeder fuel; however, the colocation of a demonstration breeder fuel reprocessing plant with the Barnwell plant did appear feasible. The concept underwent an engineering analysis to evaluate and expand on the preliminary conclusions reached earlier. The facilities, equipment, and processes have been defined in greater detail than was done in the earlier study; a cost estimate was also prepared.

No major engineering problems were identified that resulted from the colocation of a demonstration breeder fuel reprocessing plant with the BNFP. The general conclusions of the earlier study were verified.

US/UK Exchange Agreement Subtask: Mechanical Head-End, Heat Generation, and Heat Transfer
M. J. Haire, J. C. Ryman, and T. D. Welch

The experimental setup and the conduct of the first set of temperature measurements were observed in the Prototype Fast Reactor Post Irradiation Examination Caves at Dounreay, Scotland. Temperature measurements will be made on as many of the 12 fuel assemblies planned for the next reprocessing campaign as is compatible with reactor and reprocessing schedules. Centerline and wrapper temperatures will be measured before and after sodium cleaning operations.

A review of the benchmark problems indicates that the same answers are obtained with US and UK codes when the same assumptions and input data are used. Input data (i.e., nuclide libraries) used in the US (ORIGEN) and UK (FISPIN) computer codes have yet to be compared.

The United Kingdom is measuring concentrations of important nuclides in irradiated Prototype Fast Reactor fuel. This data will be used to validate the US ORIGEN code's decay heat calculations.

The United States and United Kingdom have complementary research programs to measure emissivity. The value of emissivity is an important input in fuel assembly temperature calculations. The UK program will

measure the emissivity of sodium and sodium-wetted steel surfaces. The United States is currently measuring emissivities after various types of sodium cleaning operations are performed on the steel surfaces.

Heat Transfer Experimental Program

**M. J. Haire, T. D. Welch, and
 C. A. Rhodes (University of South Carolina)**

The objective of this research is to obtain support information required for the design of cooling of deshrouded liquid-metal fast breeder reactor fuel assemblies during the fuel disassembly and shearing process. Experiments have been performed on an electrically heated, simulated fuel assembly bundle. The fuel assembly was cooled by gaseous jets impinging on the outer row of simulated fuel rods. An experimental sensitivity study of the parameters thought to influence heat transfer characteristics was conducted. The parameters being studied are heat generation rate, gas flow rate, gas supply temperature, and pressure. A recommended gas flow rate, gas supply pressure, and nozzle array design will be determined from the investigation.

The experimental data indicate that the required flow rate of coolant decreases when using a larger number of smaller diameter jets. The feasibility of the jet-cooling technique has been demonstrated, but the measured flow rates required for cooling are higher than calculated. The next step of the investigation will be to reduce the large number of variables and to decrease the flow rate of the gas required for jet cooling.

Gas-Flow Studies

T. D. Welch

The results of heat transfer calculations for Conceptual Design Study (CDS) fuel indicated that provisions must be made to cool these assemblies during handling, disassembly, and shearing for decay times $< 1\frac{1}{2}$ years. A recirculating cooling system has been proposed for the required cooling gas. A study has been made of the capital cost for equipment associated with the recirculating system vs a once-through system. The once-through gas cooling system

and associated facility equipment/systems has some greater costs than those associated with a gas recirculating system. These costs may be offset by other considerations, such as, no development costs and less maintenance.

Integration of the HTGR Reprocessing Demonstration with the Hot Experimental Facility
J. J. Shefcik (General Atomic Company)
and J. G. Stradley

The General Atomic Company is continuing a study to develop concepts and specifications for integrating the High-Temperature Gas-Cooled Reactor (HTGR) fuel reprocessing demonstration with the HEF. The design criteria flowsheets, layout drawings, cost estimate, and report associated with the study were completed.

5.1.2 Nuclear Engineering

J. F. Mincey

The task objective is to provide calculational support and technical guidance for the design of reprocessing equipment where criticality and radiation damage are significant problems.

PNL Critical U+Pu Solution Experiments
B. M. Durst, R. C. Lloyd (Battelle-Pacific Northwest Laboratory), R. T. Primm III, and J. T. Mihalcz

Preparations for the final two experiments with nitrate solutions containing ~380 g/L of (U+Pu), ~67 wt % Pu/(U+Pu), are in progress. During this reporting period, four experiments were completed on nitrate solutions containing ~468 g/L of U+Pu and ~67 wt % Pu/(U+Pu).

Proof-of-principle, subcritical, and reactivity measurements were completed. The measurements appear to be of high quality; the data will be analyzed in FY 1982.

Los Alamos National Laboratory (LANL) U+Pu Mixed Oxide Critical Experiments
R. T. Primm III, J. F. Mincey, and R. E. Malenfant (LANL)

Initial test results of a prototype canister design were obtained. The purpose of the canister is to facilitate U+Pu oxide handling during the actual critical experiments. The U+Pu oxide-filled canister was neutron- and gamma-radiographed to determine the

distribution of the moderator within the canister. In addition, a replacement measurement was performed in the PARKA critical assembly at LANL to determine the reactivity worth of the canister and to irradiate dosimeter wires to obtain an axial neutron flux profile. These data are currently being evaluated.

5.1.3 Environment and Safety

M. B. Sears and D. W. Williams (WARD)

This task provides guidance and assessments to (1) ensure that HEF activities meet statutory health, safety, and environmental requirements, and (2) minimize the potential on- and off-site impact of the facility.

The environmental assessment of the HEF was completed and forwarded to the DOE.

The magnetic tape containing the digitized house location and other population data for the Oak Ridge vicinity and a copy of the NUS computer code, POPDIG, for accessing the data have been received from the NUS Corporation. These data were compiled in support of siting the HEF facility. The population code has been run successfully on the PDP-10 computer at ORNL from on-line storage, and a procedure for operating the POPDIG code at ORNL has been prepared. Since these data might be useful in the event of an emergency in the Oak Ridge area, attention was focused toward facilitation of the use of the program with the PDP-10 user.

5.1.4 Safeguards Task

H. T. Kerr

The safeguards task is responsible for ensuring the development and implementation of effective safeguards technology for reprocessing plants as may be necessary to satisfy both domestic and international safeguards objectives. The current focus is on the development of advanced material control and accounting concepts.

Microscopic Process Monitoring
S. J. Hurrell, J. W. Wachter, T. L. Hebble, and R. M. Tate

The majority of the safeguards effort remains focused on the conceptual development and demonstration of the microscopic process monitoring (MPM) concept. Over the past several months, software

systems have been developed to permit testing of the MPM concept at the Barnwell Nuclear Fuel Plant (BNFP). The second and third plutonium cycles of the BNFP have been operated for several one-week periods, called miniruns, with natural uranium feed (no plutonium).

During this reporting period, the safeguards efforts were focused on analysis of data from the sixth minirun (May 13–19) and on participation in the seventh minirun (August 18–24) at the BNFP. These miniruns included demonstrations of the MPM concept with the following major objectives:

1. Conduct a comprehensive process monitoring demonstration using an on-line remote terminal.
2. Evaluate binary status signals for selected pumps and valves into the MPM logic for detecting abrupt diversions of nuclear material.
3. Determine lower limits of the MPM diversion capability in dynamic tanks.

One of the major technical objectives of the miniruns was to establish the capability to remotely direct the MPM analysis program from an off-site terminal. A terminal was established at ORNL, and the MPM analyses at the BNFP were controlled from this remote terminal. At times this telephone link was lost for various reasons; but, presumably, these data-link problems can be resolved by using a more sophisticated telecommunication system.

Equipment and software problems occurred which resulted in the loss of some data. Early in the seventh minirun, a defective serial link between the data acquisition computer (RDAS) and the program development system (PDS) computer and other equipment problems caused a loss of ~12 h of data that probably cannot be recovered. A software problem also occurred during the seventh minirun. The program had a tendency to get into an infinite loop after a few hours of normal operation. The program had to be terminated and restarted each time it (the program) went into the loop. This prevented continuous monitoring of the minirun except when manpower was available to watch the output terminal.

Another major technical objective of these miniruns was to determine the value of binary sensors that provide an on/off status indication for selected pumps and valves as a means of identifying batch transfers between tanks. The MPM software was modified to use this type of information, and some of the desired sensors were installed for the miniruns. As originally planned, the binary sensor outputs were not continuously monitored; their outputs were to be accessed at 2-min intervals. Unfortunately, the equipment being monitored was operated in an intermittent fashion with some status changes occurring during time intervals of <2 min; consequently, several status changes could not be clearly identified.

During the seventh minirun, a microprocessor was used to indicate both the current pump status and whether a change in the status had taken place in the last 4-min period. The MPM software only evaluated the binary data when a change in status was indicated. Unfortunately, the interrupt-driven system designed to signal the change in status was not working properly; therefore, the MPM binary software was never called into action. A computer program capable of simulating the signal from the microprocessor should be created so that the MPM binary logic could be tested on taped data.

The previously compiled minirun process data taken at 4-min intervals were used to perform a mass balance for selected process tanks. Uranium mass transfers were determined, and statistical techniques were used to indicate significant trends in the mass data. The technique works very well with suitable instrumentation, but it is not effective in areas with excessive instrument noise. With improved instrumentation, the diversion detection capability would be greatly enhanced.

In spite of the numerous software and hardware problems, the minirun did have some positive results. The remote on-line monitoring capability was generally successful despite frequent breakdowns, and all of the process data was taped and will be analyzed to determine the lower limit of detection capability, especially during continuous filling or draining of tanks.

Dynamic Accounting

H. T. Kerr, E. D. Blakeman, and T. L. Hebble

The first iteration of a dynamic accounting assessment for the head-end and separations area of the HEF has been completed and summarized in a draft report by the Safeguards Division of Los Alamos National Laboratory. Basically, the report describes an optimum set of measurement requirements for four dynamic accounting performance levels in selected areas of the HEF. The draft report contains an enormous amount of detailed information, therefore, some additional effort will be required to produce an executive summary for those readers who are not accountability specialists. Work will continue on the assessment to include a sensitivity study for the chemical separation area of the HEF.

The results of the sensitivity study, which will be expanded to include the product conversion area, should be a major influence on the future development of safeguards instrumentation. A computer code has been developed to optimize the combinations of measurements that would give a desired performance level at the lowest level of measurement difficulty. Measurement difficulty, as used in this context, is related to the amount of research and development required to obtain a given capability.

Operations Monitoring

S. J. Hurrell

A cooperative effort with Sandia Laboratories on the development of the operations monitoring concept is continuing. A paper describing operations monitoring concepts for a reprocessing plant has been redrafted, and some effort is being made to identify a practicable demonstration of some operations monitoring concepts.

A meeting was held with the staff of Sandia Laboratories to discuss the progress and future direction of the operations monitoring interaction. It was agreed that for the short term, the future direction of operations monitoring should take a more localized approach to diversion detection. This localized approach involves the analysis of only a few instruments focused on one operation in the facility. This analysis would be performed by a microcomputer located near the instruments.

Integrated Equipment Test (IET) Safeguards Studies

J. W. Wachter

The safeguards staff is continuing active participation in the IET planning meetings and is trying to identify specific safeguards demonstrations that would be feasible in the IET facility. A report has been drafted which summarizes the following major topics:

1. safeguards concepts that may be tested,
2. IET equipment involved,
3. instrumentation needed,
4. computer capacities needed, and
5. possible impacts on the IET schedule and operation.

A detailed study of the IET process flow and instrument drawings has been initiated for the purpose of identifying the facility components and instruments that may be used in material accounting demonstrations. The initial output from this effort will be an informal report that shows what demonstrations can be conducted with currently planned instrumentation and identifies upgraded or additional instrumentation that would be needed for expanded demonstrations.

5.2 COMPONENT DEVELOPMENT

E. D. North

Component Development work is a follow-on to the Engineering Research effort and is responsible for designing, fabricating, and demonstrating the performance of pilot-plant process equipment capable of remote operation and maintenance.

The dissolution and solvent extraction tasks are currently active.

5.2.1 Dissolution

F. E. Weber

The Component Development effort in the dissolution area is directed toward designing, constructing, and testing a 0.5-t/d pilot-plant-scale dissolver system. The system consists of a remotely maintainable rotary dissolver integrated with the support equipment necessary to provide a functional continuous dissolution system.

Prototypical Rotary Dissolver

Dissolution experiments with the sheared, iron-magnetite-filled fuel bundles began this quarter. A

42-h test with blanket assemblies had no problems with transport of the stainless scrap. The final stage-by-stage inventory of stainless scrap was uniform and close to the expected value. A 12-h test with a dummy driver fuel assembly failed to operate as expected. No stainless scrap discharged from the dissolver during the experiment. This failure was traced to an improper reverse cycle. Misalignment of a sensor caused the computer software for controlling drum rotation to operate improperly. The problem was corrected, and the experiment was repeated with no iron-magnetite. The solids transport behavior was acceptable and similar to previous tests. The stage-by-stage inventory increased near the feed end of the dissolver, with a total inventory slightly above the expected value.

Dissolver Support Systems and Process Equipment

The design of a new drive and support system for the dissolver began this quarter. The system consists of a close-coupled, ac, frequency controlled motor and self-aligning support rollers.

Fabrication of new blocking devices for the liquid transfer slots was also started this quarter. The devices incorporate suggested improvements discovered during the testing of the original blocking devices. These new devices should lower the solids transport problem to acceptable levels.

Plans for moving the dissolver system to the Remote Operations and Maintenance Demonstration (ROMD) area were begun this quarter. The move is scheduled to begin in January 1982.

Dissolver Instrumentation

Improvements to the dissolver control console continued this quarter. When complete, the console will display information <1 s after the process controller receives the data.

5.2.2 Advanced Centrifugal Contactor Program

**A. L. Olson (Allied-General Nuclear Services),
O. M. Ebra-Lima, and M. E. Hodges
(Savannah River Laboratory)**

The advanced contactor test facility has been completed and is being checked out. A few, rather typical, start-up problems were encountered and are

being corrected. Poor welds in the large storage vessels have caused considerable delay. Failure of the flow measurement and control system was unexpected. Replacement of the turbine meters will be completed next quarter.

Limited hydraulic runs on the single-stage, 12-cm, centrifugal contactor have been conducted using manual control and rotameters for flow measurement. The unit operates satisfactorily at a rate equivalent to 0.25 t/d (2 L/min aqueous and organic). Operation at 0.5 t/d had been previously demonstrated.

Fabrication of the 8-stage contactor is nearly complete. Shipment of the components to Savannah River Laboratory (SRL) will be made late this quarter. Assembly of the rotors, motors, and the support frame will be performed at SRL. Operation and testing of the 8-stage unit will be performed next quarter.

5.2.3 Solvent Extraction (IET)

**G. L. Borsheim (Exxon Nuclear Co.)
and M. V. Keigan**

The process flowsheets and equipment layout drawings for the IET solvent extraction system were revised to reflect the deletion of one pulse column. (Scrubbing will be accomplished by one of the centrifugal contactor 8-packs.) Advanced conceptual engineering work in the areas of piping and structural concepts and equipment data sheets was initiated. The solvent extraction system costs were reestimated.

5.3 SYSTEMS DEVELOPMENT

N. R. Grant

The Systems Development work is directed toward the development of certain major mechanical components and systems required for fuel reprocessing. The goal of Systems Development is to design, fabricate, and operate pilot-scale, prototype, remotely operable equipment.

The tasks assigned to this area are the disassembly and cutting systems, voloxidation equipment, receiving and storage systems, special remote systems, remote control engineering systems, waste handling systems, and procurement of depleted uranium fuel for use in equipment and process development.

5.3.1 Disassembly and Cutting

S. A. Meacham

The disassembly and fuel cutting efforts are responsible for the mechanical preparation of the fuel for the succeeding steps of voloxidation and dissolution. The task will develop equipment that will remove the undesired components (such as inlet and outlet nozzles) from the fuel assembly and cut the remaining portion of the assembly into short pieces. The sheared product will expose the contained fuel for subsequent oxidation (in the voloxidizer) and leaching (in the dissolver). The goal is to produce the necessary design, equipment, and data required for the successful operation of prototype units in the IET and similar equipment in the HEF.

Disassembly Systems

Remote disassembly system (E. C. Bradley, R. E. Eversole, and T. W. Semple). The final report on the evaluation of the conceptual design for the remote disassembly equipment and the revised system design requirements have been compiled by Westinghouse Advanced Reactors Division (WARD). Authorization was given to WARD to start the detail design phase.

Laser cutting development (B. S. Weil). The final report for the United Technologies Research Center laser cutting development subcontract was received. The installation of new servo drive equipment on the laser cutting rig and the return of all government-owned equipment to ORNL has been scheduled.

The laser cutting system design requirements document has been completed. The necessary drawings are being prepared.

Site preparations were started to convert the former prototype fuel room to the laser generator acceptance test area. Delivery of the laser is scheduled for December 1981.

Shear Systems

Remote shear project (B. S. Weil, D. C. Watkin, J. H. Evans, and T. W. Semple). Planning Research Corporation is continuing the detail design phase of the remote shear system. This phase is approximately 60% complete.

Birdsboro shear activity (W. F. Schaffer, Jr.). The FY 1981 experimental shearing program to develop base-line data for prototypic tooling is complete.

Testing of hydraulic fluids for the remote shear (E. C. Bradley). A final report on the irradiation testing of high-water-base hydraulic fluids was issued this quarter.

5.3.2 Prototypic Voloxidizer

J. E. Van Cleve, Jr. and T. W. Semple

The contract with Planning Research Corporation to perform the detail design of the prototypic voloxidizer is now 70% complete. Three design review meetings have been held, and problem areas in both the mechanical and electrical designs were identified, discussed, and resolved. The work is proceeding smoothly and should be finished on schedule.

5.3.3 Receiving and Storage

S. A. Meacham and E. C. Bradley

Sodium Removal

The experimental program to determine process parameters for rapid sodium removal from spent fuel assemblies was completed this quarter by WARD. The draft of the final report was prepared.

Storage

Analysis of the irradiated, sodium-exposed cladding specimens by scanning electron microscopy was completed this quarter at Los Alamos National Laboratory.

5.3.4 Special Remote Systems

S. L. Schrock (Westinghouse Advanced Reactors Division), F. L. Peishel, and W. F. Schaffer, Jr.

Special remotely operable components and systems that are common to the major needs established by the requirements of the HEF conceptual design or by other tasks in Component and Systems Development are being developed by this task. These include remote pipe and electrical connectors and jumpers, a remote piping repair system, and a remote automatic liquid sampling system.

Remote Connectors and Jumpers

Several improvements to line connectors and jumpers, including tools to handle and operate the connectors, are being developed under this subtask. The primary improvements desired in the line connectors are the development of radiation-resistant seals to replace presently utilized Teflon seals, a decrease in the size and cost of the connectors, and improvements in the reliability and ease of remote handling.

A test stand was designed, constructed, and installed to test various types of connectors. This facility is capable of testing connectors for mechanical integrity and sealing reliability under realistic or severe conditions of stress, temperature, and vibration. Installation, shakedown testing, and personnel training for the test stand were completed during the previous quarterly period, and a test plan was prepared. Testing has been initiated and will continue into the next fiscal year.

Remote Piping Repair System

The objective of this task is to develop remote pipe cutting, welding, and inspection techniques that could be used on the interconnecting piping in lieu of remote line connectors. Based on visits with vendors of automatic cutting and welding equipment, it has been determined that the state of the technology for these activities is acceptable. However, modifications are required to make the equipment usable in the HEF by adapting the handling fixtures for use with manipulators. The equipment and its supporting structure must also be designed to interface properly with the piping and pipe support system in the HEF.

A subcontract was placed during the last period with Battelle-Columbus Laboratories to perform a preliminary design of a piping repair system. This effort will address the adaptation of commercial tools to the cutting, cleaning, welding, and inspection of wall-mounted piping in the HEF and will recommend any additional development efforts that may be required. During this period, the preliminary design

effort continued, and monthly progress reviews with Battelle were conducted. Battelle proposed the use of an adaptive welding concept for the repair welding. With this concept, the weld head is instrumented to provide information to the control system which adjusts the welding parameters. This approach can accommodate reasonable variations in wall thicknesses as, for example, would be encountered in a corroded pipe. It can also either eliminate the need for post-weld inspection or make that inspection more efficient by indicating areas where defects are most likely to be found. This approach has the potential for simplifying the pipe repair system and giving superior quality welds.

The design of wall brackets on which the pipe repair system will be mounted is also being performed. This design work is proceeding on schedule and will be completed at the same time the Battelle effort is finished.

Remote Sampling System

An advanced automatic liquid sampling system for the HEF has been designed. The system consists of three subsystems: an automatic self-propelled vehicle capable of extracting liquid samples from each of the process modules; a track system, which guides and provides support for the vehicle; and a sampling station, which is the interface between the vehicle and the process module. The detailed designs of a prototype track system and sample station were previously completed. The detailed design of the vehicle was completed during this period.

The track system and sampling station are being fabricated with a scheduled delivery of January 1982. These items will be installed on the process module mockup in ROMD. Fabrication of the vehicle will be initiated early in the next fiscal year. This fabrication will be divided into two parts - the mechanical subsystems and the electronic control package. Both fabrications will be performed in the ORNL shops and will require approximately six months to complete. Long-lead material items for the vehicle were also ordered during this period.

5.3.5 Remote Control Engineering

J. Garin (Westinghouse Advanced Reactors Division),
B. J. Bolfig, J. N. Herndon, and G. E. Smith

Servomanipulator

H. L. Martin, W. H. Leavell, P. E. Satterlee, and
M. A. Dinkins

Servomanipulator development involves two major activities: (1) the procurement of an industry-standard system and (2) the development of an advanced servomanipulator system. Each system consists of a set of two manipulator slave arms and an interface package that has positioning equipment for TV cameras and an auxiliary hoist.

The industry-standard servomanipulator (Fig. 5.1) is being procured from Central Research Laboratory (CRL). This system features brushless dc servomotor actuators that drive each joint and a distributed digital multiplexing system to reduce cable handling problems. Design and fabrication of the CRL industry-standard servomanipulator system is continuing. Fabrication of the mechanical portion of the system is essentially complete.

Fabrication and assembly of the four manipulator arms (two master and two slave arms) are almost complete except for the installation of the joint actuators. The camera positioning system fabrication to be mounted above the arms is also complete. The CRL control system is 75% complete. The remaining design items include the operator control console and system installation drawings. All other parts are being fabricated.

The objective of the advanced servomanipulator development is to provide improvements over the industry-standard servomanipulator system. Features being developed include modularity, remote maintenance provisions, radiation hardening, and improved reliability. The conceptual design of the system has been completed. There are, however, several areas that are not clearly defined; therefore, a single-arm prototype system will be designed and fabricated in-house during the next two years. The results of this design effort and the reliability testing program to follow will provide guidance for procurement of a complete two-arm system beginning in FY 1984. Assembly

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Fig. 5.1. Industry in-cell (slave) manipulator system.

design for the shoulder, elbow, and wrist for the prototype system was initiated.

The advanced system will feature anthropomorphic kinematics, increased modularity for improved remote maintainability, and an electronics control package designed for future radiation hardenability. To develop these concepts, an actuator test stand is being designed that will measure mechanical, electrical, and thermal performance of proposed joint actuators.

The conceptual design of the wrist, which is a key feature of this system, has been completed. In addition, a full-scale plastic model of the advanced anthropomorphic wrist mechanism was completed, which demonstrates fabricability of this complex mechanism. It is planned to procure two pairs of the advanced anthropomorphic wrist mechanism for installation on the industry-standard servomanipulator system for initial trial and demonstration.

Man/Machine Interface, Hand/Eye Coordination
M. M. Clarke (Oak Ridge Associated Universities),
J. Hawk, and R. Crutcher

The development of an optimal man/machine interface for the HEF is critical in terms of increasing system efficiency and minimizing system downtime. Work has been completed in five areas: (1) time studies, (2) small-volume camera specifications, (3) identification of operator skills, (4) test plans for small- and large-volume camera coverage, and (5) selection of man/machine interface variables.

Small-volume camera specifications have been completed for prototypic remote tasks using the remote handling simulator. Camera type and position have been defined with respect to the completion of these tasks using Model-8 mechanical manipulators. The required operator skills have been identified, and recommendations for future HEF operator selection and training programs have been made.

A preliminary test plan has been prepared for the evaluation of small- and large-volume camera coverage and for man/machine interface requirements. The plan addresses the following specific issues: (1) task specifications, (2) operator-computer and interoperator task allocations, (3) design of displays and controls to meet or exceed human factors, and (4) optimization of

or exceed human factors, and (4) optimization of overall control station architecture. In addition, the Human Engineering Division of the U.S. Air Force, is providing informal technical assistance regarding anthropometric standards for integrated hand controls and for computerized modeling of man/machine interface.

In the area of remote viewing, a prototype camera positioning system has been fabricated and is being prepared for installation. This system is to be used to determine remote viewing and camera positioning requirements for small-volume viewing coverage. A pair of master/slave servomanipulator arms, on loan to ORNL from the Princeton Plasma Physics Laboratory, will be mounted with the camera positioning system and will perform simulated HEF work tasks so that various viewing configurations can be evaluated.

No sources were located for radiation hardened color cameras. Human factors experiments are in progress to evaluate color viewing for performing remote maintenance tasks.

Power Arm
H. L. Martin and D. P. Kuban

A power manipulator arm (180-kg capacity) is being developed to perform heavy work tasks at slow speeds. A PaR-3000 model arm is presently used to develop control methods.

The development work on actuator selection of this type of system is continuing. A test stand has been fabricated that will be used to evaluate actuators that combine the features of pancake harmonic gear reducers and dc rare-earth permanent magnetic motors. This stand will be installed within the next reporting period.

Wireless Data Transmission

The objective of this activity is to develop a method of transmitting video and control data with maximum reliability in a hot-cell environment. Several candidate techniques for free space (wireless) signal transmission were identified in a study, and an experimental test plan to identify a workable system was proposed. The recommended method of signal transmission by inductive loop is being implemented for test and evaluation.

A transmission line coupler system (inductive loop) for wireless control and video signal transmissions between the in-cell transporter/manipulator system and the out-of-cell operator console has been fabricated. The system is undergoing bench testing and is being prepared for testing.

Reliability Engineering

The purpose of this activity is to provide reliability engineering for the other remote control engineering activities. Efforts in the tribological design review of the advanced servomanipulator are essentially complete with submission of the draft final report. Improved power transmission component (gears, bearings, etc.) requirements for the advanced servomanipulator to enhance system reliability and recommendations for methods of lubrication for long-term use in a radiation environment similar to the HEF are included in this report.

5.3.6 Uranium for Tests

N. R. Grant

This task is divided into three areas: (1) the procurement of dummy assemblies as feed material for the disassembly/shear tests, (2) the procurement of depleted uranium dioxide as feed material for the voloxidizer and/or the dissolver, and (3) the disposal and/or recycle of uranyl nitrate.

The fabrication of 28 assemblies — six CFRP core type, six CRBR blanket type, and sixteen FFTF type, all containing aluminum oxide pellets — has been completed by the Exxon Nuclear Company. Delivery was

made at the end of August and the assemblies were placed in the new storage area completed earlier this year.

There were no activities in areas 2 and 3 above.

5.4 HEF CONCEPTUAL DESIGN

**B. F. Bottenfield, F. C. Davis, J. N. Herndon,
G. F. Kalb, M. V. Keigan, D. Macdonald, E. D. North,
F. L. Peishel, D. P. Reid, and O. A. Rogers**

The conceptual design of the HEF, an engineering-scale fuel reprocessing plant, is being performed by UCC-ND with the assistance of an architect-engineer and other subcontractors. The HEF is conceived as a versatile reprocessing test facility capable of handling the complete spectrum of breeder reactor fuels for the various fuel cycles now being considered. This facility will allow testing of breeder fuel cycles consistent with the overall breeder development program. The HEF will demonstrate advanced fuel reprocessing technology under plant operating conditions with irradiated fuel and will establish a basis for reprocessing capability to the breeder industry.

The final conceptual design report (CDR) was published in July and distributed in August.

The issuance of the CDR completed the work assigned under the project directive. The conceptual design was prepared under the technical supervision of the Fuel Recycle Division and the Engineering Division of UCC-ND by Bechtel National, Inc., the architect-engineer, and the following subcontractors: General Atomic, Los Alamos Technical Associates, Planning Research Corporation, and Sundstrand.

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6. Integrated Equipment Test Facility Operations

O. O. Yarbrow

The IET Facility Operations will provide for the synergistic testing of process equipment and flowsheets prototypical of the Hot Experimental Facility (HEF) and also for testing the prototypical remotization aspects of specific complex components in the system. Preparations for these efforts will include (1) IET line-item construction, (2) component development equipment installation, and (3) operation of the plant at the desired test conditions.

Construction of the General Plant Facility (GPF) portion of the IET was completed during FY 1980. This GPF construction area and utility modifications in existing buildings provided the location for two operational parts of the IET — the Remote Operation and Maintenance Demonstration (ROMD) and the Integrated Process Demonstration (IPD). The IPD will address the issues of integrated operation in the predominantly chemical processing portion of the system. The ROMD will focus on testing the remote aspects of the complex mechanical head-end processing equipment. The ROMD will demonstrate and verify critical remote maintenance techniques and procedures. The process equipment located in the ROMD and the IPD will be integrated to permit overall simulated reprocessing system operations.

Laboratory experiments were performed to acquire spectral data necessary to design an in-line photometric plutonium monitor. The multiwavelength photometer used for uranium measurements will be modified for this application.

Tests of the free acid monitor have been performed to determine the effects of entrained 30% tributyl phosphate (TBP) and particulate material. The effect of TBP was unobservable, and no persistent blockages occurred in 24 h of particulate testing. The conductivity cell has been tested for radiation effect.

Experiments have been performed to evaluate the filter and resonance self-indication methods of determining the isotopic fractions in spent fuel and blanket subassemblies. A report describing the methods and potential advantages of each is being written.

A study to evaluate the use of current nondestructive assay instruments for measuring the fissile masses in various waste and product canisters has been completed.

6.1 IET LINE-ITEM TECHNICAL COORDINATION

O. O. Yarbro

This task provides the support necessary to review the line-item design work performed by UCC-ND Engineering and the fabrication and construction work performed by outside contractors. It will also interface the line-item activity with other Fuel Recycle Division tasks.

6.1.1 IET Contract

E. L. Nicholson

The IET equipment installation contractor, Hicks and Ingle Company, has installed all the major process equipment items. Process and service piping installation is progressing satisfactorily, and electrical work has been started. The ROMD cranes and simulated shield walls have been installed, and service piping is complete.

6.1.2 Remote Operation and Maintenance Demonstration

E. L. Nicholson

The trolleys and telescoping tube assemblies for the manipulators and camera mounts were installed on the bridge and gantry cranes. The vertical bridge manipulator was installed. Power wiring and control cables were installed and connected to the ROMD operating console. New gears were installed in the bridge and gantry crane drive trains by the vendor, and satisfactory operation was obtained. Electrical installation is ~90% complete. The simulated shield walls and windows have been installed. Delivery of the television system (the last major item that must be installed in the ROMD) has been delayed until October.

6.1.3 Integrated Process Demonstration (IPD)

E. L. Nicholson

Major process equipment items have been installed by Hicks and Ingle. All holes have been cut in the floors and walls for equipment, piping, and electrical installation; major ports, such as the emergency exit hatchway, are complete. Most of the structural steel platforms, equipment supports, ladders, and stairways have been installed, and sampler boxes have been installed on the high-bay platforms. Off-gas headers

have been installed in the chemical-makeup, high-bay, and basement areas; floor drain piping changes in the basement have also been completed. Substantial progress has been made on process and service piping in all areas. Electrical installation work was delayed slightly because of interferences with planned cable tray holes in floors and walls.

The delivery schedule for the distributed data acquisition and control system has been delayed because of malfunctions with the tape drive units. Shipment is now scheduled for mid-February.

6.2 IET OPERATIONAL ENGINEERING SUPPORT

W. W. Evans

This task is dedicated to coordinating the installation of component development equipment in the IET facility.

Work on resolving problems in connection with planning the equipment installation in the ROMD area has continued.

A brief summary of the agreements and commitments made concerning the major problem areas discussed this period in the ROMD equipment installation plan follows:

Laser location. Management approved the recommendation to place the laser equipment initially in the fuel fabrication room and then move it into the basement area when space is available. This decision will allow UCC-ND Engineering to "fix" the location and continue planning on this basis.

Dissolver relocation. The schedule for moving the dissolver has been developed and issued for comment. Line-item funding has been approved for the dissolver support platform. The specification package for fabricating the platform has been prepared and submitted to outside contractors for bid. The design for the tank liner required for the trench in which the digester tanks will be set is complete and is being reviewed.

ROMD equipment transmitters. Considerable progress has been made in locating the transmitters and process control modules for the ROMD equipment. The plan now is to locate the major portion of the transmitters and process control modules in an area

within the component development basement. According to an environmental study of the area, the electronic equipment should not be affected by the basement environment.

A special task force has been organized to identify the interface requirements of the IET equipment and to prepare an overall report projecting the resource allocations required by IET operations between FY 1982 and FY 1985. This work will be completed next quarter. Thus far, the main emphasis of the task force has been to (1) prepare a draft IET criteria document, (2) develop interface control documents, and (3) prepare an updated equipment installation schedule.

6.3 IET OPERATIONS COORDINATION

J. Q. Kirkman and J. M. McNair

The major long-term functions of this task are planning and execution of IET operational campaigns.

Present activities have generally involved preparing the groundwork for developing detailed operating procedures for the IET. These procedures are key elements in executing safe, controlled operations; their preparation depends heavily on having detailed documentation on all facility equipment and on having accurate flowsheets for all process systems. Operating procedures for the facility remote maintenance equipment in ROMD and process flowsheets for component-development-supplied systems have received major emphasis during this reporting period.

Planning activities to prepare for delivery, installation, and initial operation of the acid fractionator process module mock-up were completed. The process module mock-up is the first component assembly that will be installed in ROMD for remote maintenance demonstrations. A draft of the test plan for the process module mock-up, describing the various remote maintenance tasks to be demonstrated was completed. The objectives and operations identified in the test plan will be utilized to develop detailed remote operating procedures for the demonstrations scheduled to begin during December 1981.

In a parallel effort with process module mock-up work, a list of operating procedures needed to operate the IET facility remote maintenance equipment was

produced, and work was initiated on drafting the detailed procedures. The basic course of action adopted involves writing a detailed set of operating instructions for each piece of remote maintenance equipment in the facility and structuring the procedures so that they can be easily identified with the main transport device (primary, secondary, or vertical bridge) with which they will be normally associated. The procedures needed to support the remote demonstrations on the process module mock-up have highest priority and will be completed by November 1981.

Three system design description and philosophy draft reports were completed by UCC-ND Engineering and submitted for Program approval. These documents are to be developed for all aspects of the IET process system and will contain a basic system description, a discussion of the design philosophy, and the design bases for all equipment in a system. In addition to providing a record of the design bases for an IET system, these documents will be useful as initial training material for IET operators.

Flowsheets for the IET dissolver system and the chilled water system were reviewed, and additional instrumentation details were incorporated to bring these drawings to full process and instrumentation drawing standards. The dissolver system drawings have been signed by Program personnel and transmitted as approved.

A document defining the functional requirements for an IET solids feeding system has been prepared and is presently being translated into design criteria for this equipment. The basic objective of the feed system is to provide equipment capable of simulating the batch-type feed stream expected from the prototypical shear.

6.4 IET PROCESS PLANNING AND ANALYSES

E. L. Nicholson and J. M. McNair

The efforts in this task will provide an important link between the IET demonstration facility and the design of the HEF. In the future, various flowsheet options will be studied and evaluated in the IET, and instrument functional requirements applicable to a reprocessing plant will also be identified and evaluated.

There was no significant activity in this task area during the last quarter, since major emphasis is being placed on line-item construction.

6.5 INSTRUMENTATION AND CONTROLS

S. M. Babcock

This task is responsible for the development of process instruments and data management systems for fuel reprocessing in the areas of process control, accountability, and safeguards.

6.5.1 Systems Development

R. W. Hayes and J. A. McEvers

Draft reports have been completed describing development in the areas of resource management to prevent deadlocks and network communication studies.

Work is continuing on adapting the RSX-11M operating system to support dual-ported disks. Software modifications have been completed and are now being tested.

6.5.2 Analytical Sensor Development

**H. H. Ross, J. E. Strain, D. A. Bostick, D. M. Taylor,
R. E. Harper, D. D. McCue, and D. McNeilly**

In support of a rotary dissolver evaluation campaign, an in-line electrochemical iron monitor was designed, built, and calibrated in the range of 0 to 100 g/L of Fe(III) in 1 to 7 M HNO₃. The iron-related signal produced by the cyclic voltammeter was measured by a "peak-grabber Bristol-interface" and displayed in the data handling system. Unfortunately, the sample line and electrochemical cell plugged very early in the run by undissolved iron, and the electrodes were broken in an effort to clear the stoppage. Coincidental to the in-line development, a titration method for the rapid off-line determination of Fe(III) and free acid was developed.

A mini-loop to simulate a flowing process stream is now under construction to aid in the testing of in-line sensors. The multiwavelength photometer will be installed in the loop.

The possibility of detecting trace levels of uranium in nitric acid streams by fluorescent techniques is

essentially zero. All attempts to circumvent the quenching of the uranyl fluorescence by nitrate ion have been uniformly unsuccessful.

The data necessary to design an in-line spectral plutonium monitor is now in hand and is being assembled into a report. It appears practical to measure both the total plutonium and the Pu(III):Pu(IV) ratio over the desired range (2–100 g/L) with good (<5%) accuracy.

The new in-cell component for the photometric uranium monitor is complete and has been optically tested. Complete system tests will begin when testing of the mini-loop is completed. Installation sketches have been prepared and submitted for use in the IET piping design.

The effect of entrained 30% TBP on the operation of the free acid monitor was found to be unobservable. Measurements indicate that the vaporization of acid samples in the free acid monitor generated no measurable nitrite in the evaporator effluent. Particulate material was added to the simulated process stream to determine whether flow stoppage would occur inside the acid monitor. A slight adjustment of the flow-metering tube remedied an initial stoppage, and thus far no stoppages have occurred for a total testing time of 24 h. Further testing is planned. A stainless steel and quartz conductivity cell for use in the free acid monitor was fabricated, tested, irradiated, and tested again, with good results.

6.5.3 Physical Sensor Development

**R. L. Anderson, G. N. Miller,
S. C. Rogers, and W. L. Zabriskie**

A specially designed circuit was constructed to interface the Panametrics torsion wave ultrasonic level sensor electronics (Panatherm) to a Hewlett-Packard (HP) 53165 100-MHz Universal Counter. Laboratory tests comparing the high resolution counter data to the Panatherm data (sampled and averaged by the HP-85 microcomputer) showed that the 50-ns time base resolution of the Panatherm can be further resolved to 10 ns through time averaging.

To achieve temperature and density profiling, notches were machined at 254-mm intervals on the

1270-mm sensor (five zones). Echoes are generated at each of these notches, and information relative to temperature and density thus avails itself for each corresponding zone.

The transducer section of the torsion wave ultrasonic level sensor will be subjected to condensed vapors of the nitric acid surrounding the waveguide, if there is no seal employed between these two sections. Remendur, a nickel-based alloy used for the magnetostrictive element of the transducer, was immersed in a 4 *M* solution of nitric acid (the expected concentration of the condensed vapors) to test its corrosion resistance. Remendur was found to be highly reactive to nitric acid; therefore, a seal must be used or the remendur must be otherwise protected from these corrosive vapors. A labyrinth seal (a pressure differential across a series of orifices) can provide a seal without interfering with the acoustic signals, but will add some complexity to the sensor. Diaphragm-type seals tend to severely distort the acoustic signals; however, a new design, which minimizes this effect, has been fabricated, and the preliminary results are promising.

6.5.4 Nondestructive Assay

C. W. Ricker, E. D. Blakeman,
G. G. Slaughter, and F. C. Difilippo

During this reporting period, separate experiments were performed to evaluate the filter and resonance self-indication methods for the nondestructive determination of the isotopic fractions in spent fuel and blanket subassemblies. Analyses of both experiments have shown favorable results and indicate that either method may be used to measure the fissile isotope fractions in fuel subassemblies. However, conclusive proof of the efficacy of either method cannot be obtained until experiments using fuel containing plutonium and/or fission detectors containing plutonium isotopes are performed.

A draft report has been prepared which summarizes the results of these two experiments and makes recommendations toward the selection of one method of isotopic evaluation for further investigation. Included in this selection process is a passive method by which

the isotopic fractions are inferred indirectly from the neutron background emitted by the fuel. The passive method offers advantages over the other two methods because of its simplicity and its high probability for successful application. The passive method, however, indirectly measures the isotopic fractions and therefore may not be acceptable from a safeguards viewpoint. Recommendations made in the report are based on comparisons such as this and other trade-offs. The ultimate selection of a method to be examined during the next fiscal year will depend on the relative importance of these trade-off factors.

A draft report has been completed on the results of a study to evaluate the use of current nondestructive assay instruments for measuring the fissile masses in various waste and product containers in the HEF.

6.5.5 Mechanical Surveillance

D. N. Fry, W. T. King, and C. M. Smith

A preliminary report on the feasibility of monitoring material movement in the prototype dissolver was submitted. It was concluded in this report that material transfer can be detected if one knows where in each revolution it is expected. Since backmixing and material accumulation were thought to occur in some of the dissolver stages, the absence of material transfer indications in some of the vibration signals should not be interpreted as a failure of the monitoring method, but perhaps as a demonstration of its usefulness.

Additional vibration data were obtained for one and three revolutions per minute operation with prototypic fuel and dissolution. These vibration signal recordings are being analyzed.

6.6 IET INSTRUMENTATION AND CONTROLS

B. G. Eads, S. M. Babcock, W. F. Johnson, D. D. McCue,
J. A. McEvers, H. H. Ross, and G. R. Wetherington, Jr.

This task is responsible for Instrumentation and Controls (I&C) support for IET Planning and Operations and for coordinating line-item I&C with the total facility I&C systems.

The development of control systems criteria and concepts is continuing in conjunction with the IET

task force and with the design for the control system for disassembly and cutting. The capabilities of currently available control systems are being evaluated in light of the line-item procured control system with the view of defining a concept for the overall IET control system.

An internal report outlining the capabilities and limitations of the current Component Development Data Acquisition and Control System (CODEDACS) has been prepared. This report is to be used as a part of the process to determine the future utilization of CODEDACS.

7. HTGR Fuel Reprocessing

W. S. Groenier and J. H. Shaffer

Process and engineering research and development activities in high-temperature gas-cooled reactor (HTGR) fuel reprocessing technology are being conducted at General Atomic Company (GA). These activities are limited to HTGR-specific head-end process and laboratory studies. Additional activities at GA on solvent extraction and off-gas treatment processes have generic applications in fuel reprocessing and are described in previous sections of this report.

7.1 HEAD-END STUDIES

W. S. Rickman, General Electric Company

This task is concerned with the implementation of the US/FRG cooperative program to verify the GA dry head-end pilot plant design concepts using fuel produced by the FRG. The task also involves the design of a Joint Prototype Head-End Processing Facility (JPHF). The FRG approved the total funding for CY 1981 FRG efforts on the JPHF conceptual design.

7.1.1 United States – Federal Republic of Germany Cooperation on Gas-Cooled Reactor Spent Fuel Treatment

W. S. Rickman, J. B. Strand, and N. D. Holder

Check-out runs were completed on the 20-cm-diam primary burner using an inert particle (ThO_2) start-up bed. The inert particle bed contributed to much smoother steady-state operations and required fewer operator adjustments than when burning with a crushed fuel element bed. Startup was difficult, however, and after tailburning, the product contained 11% carbon. A 20% carbon bed used for startup in another run did not significantly improve startup with the inert particle bed. The induction-heater holding-bed

temperatures were above 900°C for tailburning. Product carbon content was <10%.

The high-temperature rotary valve from the 40-cm-diam burner was used on the 20-cm-diam burner. A surge hopper and two GEMCO disk valves were also installed above the rotary valve in the below-bed fines recycle system. The two GEMCO valves were checked for leaks and tested for closure at temperatures up to 204°C (400°F). Cold shakedown tests of the 20-cm-diam burner below-bed fines recycle system used Tygon tubing so that recycle could be visually verified. A valve-operating sequence was established by which the system successfully recycled fines for 4 h.

During four fines burning tests, GEMCO valve gaps were adjusted on-line until the valves operated consistently. The pocket capacity of the rotary valve was reduced to minimize the effect of cold fines on bed temperatures. A new fines rotary valve was bench-tested and installed for use in the last two burning tests. The operation was varied until the fines could be successfully burned. Results indicate that bed and vessel temperatures should be 50°C higher than specified in the experimental plan. Reducing the plenum gas flow by 50 L/min also helps increase the residence time of

the fines bed and reduces the temperature differential across the bed. More deep-bed tests are planned to confirm planned low-enriched uranium test conditions.

7.2 LABORATORY STUDIES

G. E. Benedict, General Atomic Company

This task includes the specific development and evaluation of technology for the dissolution of high-density FRG fuel and the separation of solids from the dissolver product using a vertical centrifuge. A supporting effort for solvent extraction development is also included.

7.2.1 Inertial Filter/Centrifuge Tests

C. J. Hostbjoer

Additional tests were made of the inertial filter/centrifuge system to check the low-filtration efficiencies

measured during previous tests. The new tests gave better results, and the low efficiencies determined by the earlier tests are blamed on a combination of inconsistent analytical procedures and equipment problems.

7.2.2 Dissolution Tests

G. J. Hostbjoer

Five airlift acid recirculator tests were conducted in the 0.5-m-diam dissolver. Recirculation rates for the tests ranged from 0 (no recirculation) to 14.2 L/min. Any recirculation rate >5 L/min (2.5% reactor vol/min) decreased the dissolution time to ~25% of that required for the reference run. All work done with the airlift recirculator has been documented.

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