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LMFBR Fuel Reprocessing Program Progress Report for Period July 1 to September 30, 1976

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LMFBR FUEL REPROCESSING PROGRAM PROGRESS REPORT
FOR PERIOD JULY 1 TO SEPTEMBER 30, 1976

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Date Published: November 1976

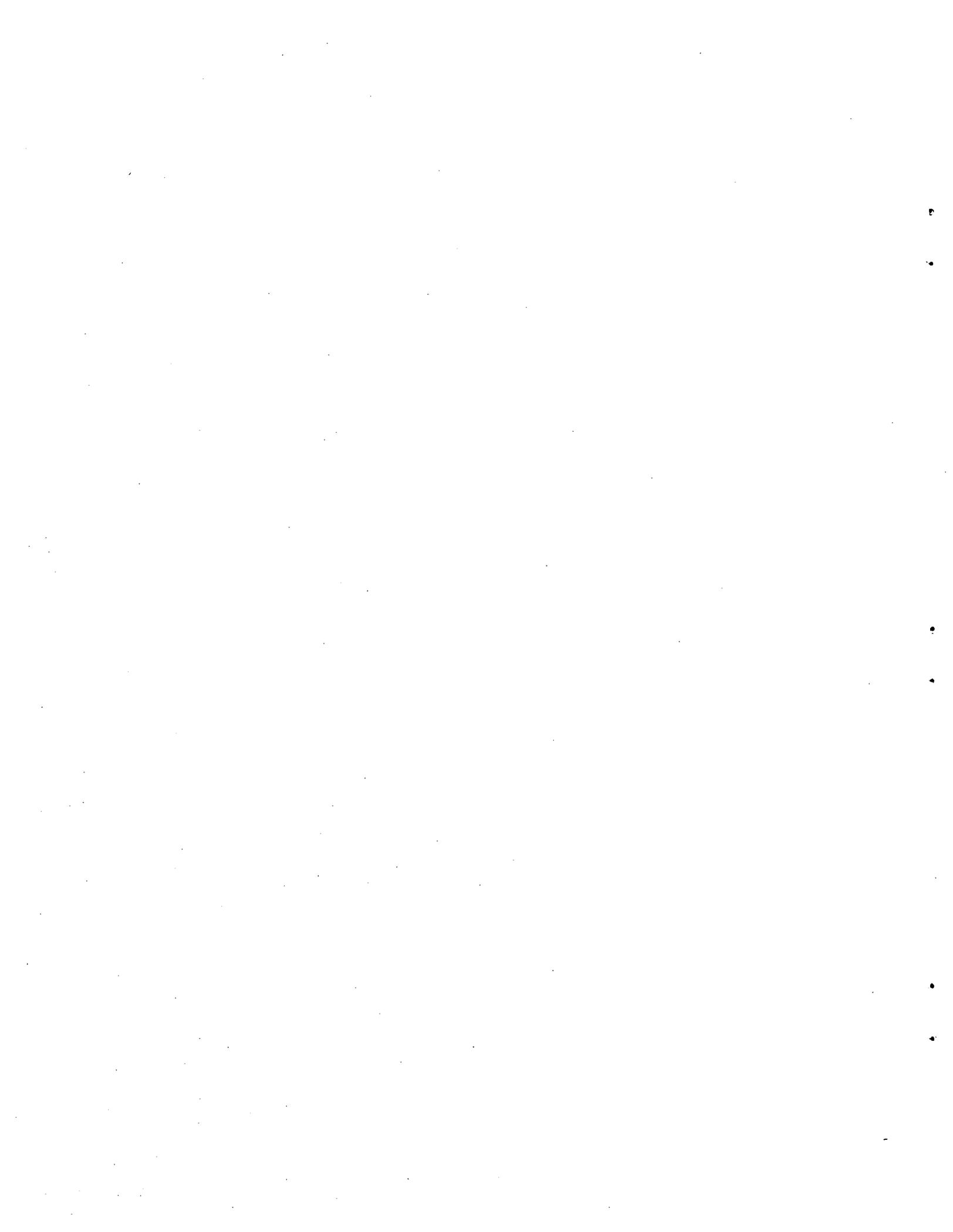
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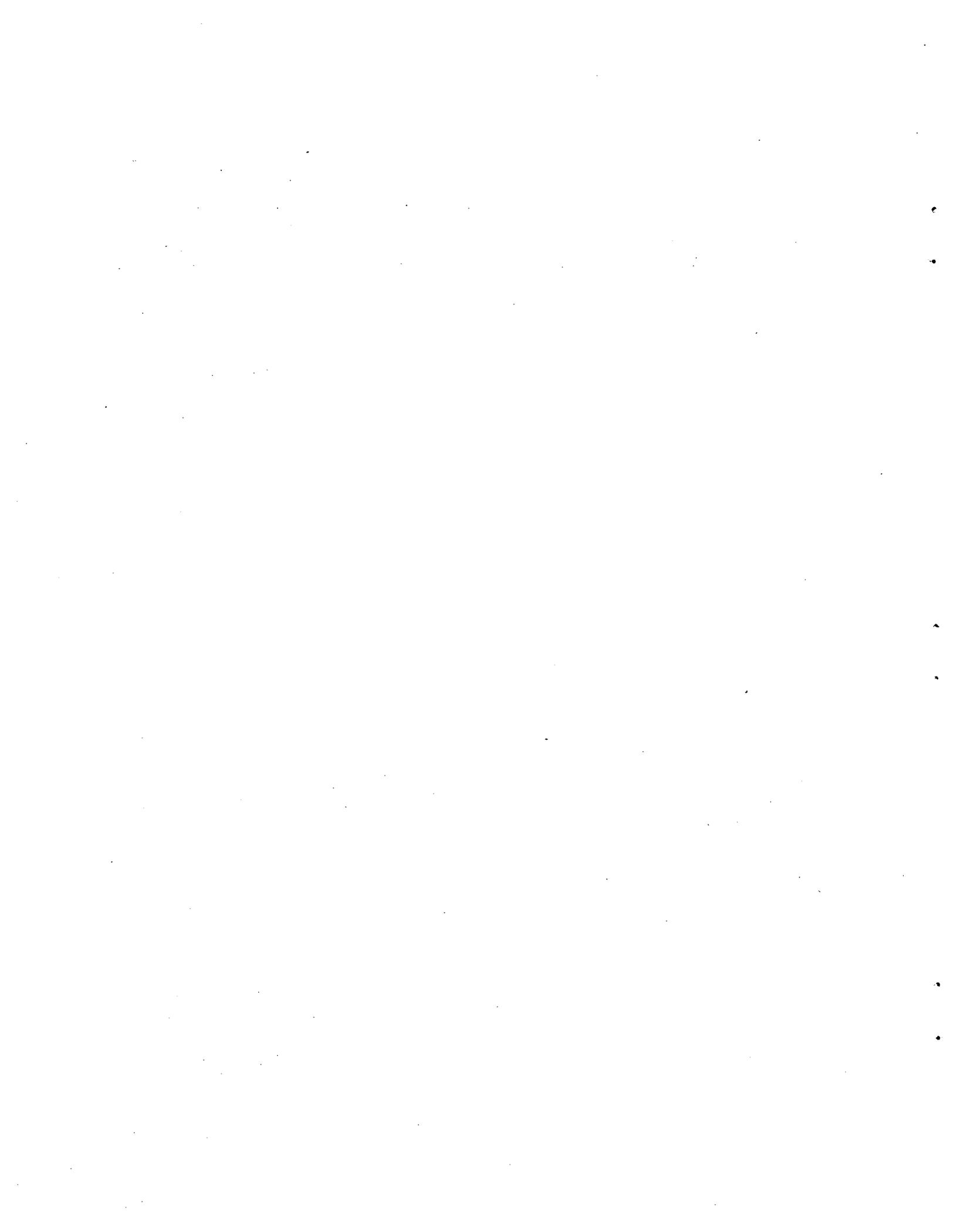
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1. HIGHLIGHTS

The LMFBR Fuel Reprocessing Program is aimed at providing technology on which to base the design of commercial reprocessing facilities that will be required by the late 1990s. The process and equipment development phase of the program should culminate in a hot pilot-plant process demonstration tentatively scheduled for 1988. This will allow the requisite lead time of about 10 years for the design, construction, and startup of a commercial facility. Feed for the hot pilot plant will be available from the Fast-Flux Test Facility (FFTF) and the Clinch River Breeder Reactor (CRBR), which are scheduled to begin discharging core elements in 1980 and 1984 respectively.

1.1 Development of Criteria for the Hot Pilot Plant (HPP)

The basic functional criteria for the HPP have been drafted and reviewed. They are now being incorporated into a document entitled Preliminary Design Criteria for the Conceptual Design of an LMFBR Fuel Reprocessing Hot Pilot Plant and are scheduled to be transmitted to ERDA on October 1, 1976. The report on the Commercial-Scale Reprocessing Plant is being assembled and summarized; it is scheduled to be completed by November 1, 1976, and to be published by January 1, 1977. These dates represent some delay in the schedule, which reflects management decisions preparatory to authorizing the publication of the criteria (a key event in an ongoing responsibility) and the effect of key personnel called to testify at the CRBR and AGNS hearings.

1.2 Laboratory and Hot-Cell Studies

Major efforts in the decommissioning of the hot cells in Building 4507 have been completed.

Earlier values of the distribution of NO_2 between air and nitric acid are corrected to take into account the formation of dimer in HNO_3 .

Studies of the dissolution of PuO_2 microspheres show that the surface area increases during much of the dissolution period.

The use of HI solutions to recover plutonium from primary dissolver residue continues to show promise. The plutonium remaining after evaporation of an HI solution to dryness was dissolved in 8 M HNO_3 to yield a plutonium solution with a low iodine content.

1.3 Engineering Research Studies

The development of the continuous rotary dissolver is continuing. This quarter the emphasis was centered on tests of pilot-plant-type dissolver internals in the 1-ft-diam unit and operational tests of the 4-ft-diam unit, including solids handling and liquid backmixing. A rotary seal test is described.

Tests of a sieve-plate column for scrubbing NO_x from air with water or dilute HNO_3 have shown that more than 80% of the NO_x in the feed gas can be removed in three plates.

The remaining fluorocarbon absorption process pilot-plant tests scheduled under campaign 3 have been completed with the removal of greater than 99.9% of carbon dioxide and xenon impurities. Process modeling studies are still in progress.

Results of chemical studies of stainless steel corrosion by R-12 solutions in the presence of iodine and water show that methyl iodide is miscible in R-12.

The final report on process reliability prepared by the Kaman Sciences Corporation has been issued.

Continuing development of the Iodox process has identified the extent to which methyl iodide decontamination factors (DFs) are lowered in the presence of NO_2 and water vapor.

Vibration analysis instrumentation installed on the prototype rotary dissolver indicated that a roller bearing support was defective. In other developments, we are pursuing studies to identify a system

for assaying the fissile content of spent fuel and to supply process sensors and controls for the various process tasks.

1.4 Component Development

Seventeen areas of component development have been identified as worthy of investigation. Three of these (disassembly and fuel cutting, voloxidation, and dissolution) have been under way for some time. This quarter has seen the addition of a new area, receiving and storage investigations. Although the subtask involves a large study, component development is only involved in a few specific tasks such as subassembly handling, subassembly cleaning, and subassembly storage. The status of this task is reported in Sect. 5.5.

The preliminary design of the first disassembly machine is almost completed, and detailed design is scheduled to begin in the near future. Two cutting tests were conducted on an alligator shear with "pinking" blades, but the results were not considered successful. The detailed design of the 0.5-ton/day dissolver is essentially complete; materials for fabrication are on order. A series of tests to determine the extent of corrosion of stainless steel during storage in water has been initiated on a subcontract. The matrix investigation will test the effect of sodium removal techniques as well as variable water conditions.

The layout showing the potential position of all component development equipment in Building 7603 is being finalized. It will be completed just in time to provide input for the start of installation drawings for the dissolver system.

1.5 Integrated Prototype Equipment Test Facility (IPET)

The conceptual design report of the IPET facility was completed in July and is scheduled for publication in early October. The cost is estimated to be \$16 million, an increase of \$6 million over the previous estimate.

The process steam boiler is essentially complete; distribution headers are currently being installed. Progress was initiated on the facility process off-gas system, the demineralized water system, and crane modifications. A study of the potential waste streams from component development and IPET activities was completed and a direction for solution of the waste disposal problem was selected; cost estimates are now being made.

2. DEVELOPMENT OF CRITERIA FOR THE HOT PILOT PLANT (HPP)

A. R. Irvine, B. F. Bottenfield, J. E. Kahn, E. L. Nicholson,
F. L. Peishel, T. Shapiro, D. C. Watkin, and O. O. Yarbrow

During this report period, the total effort of the Engineering Studies Group has been devoted to the development of criteria for an IMFBR fuel reprocessing HPP. The development of these criteria is being accomplished by, first, studying several of the major problems in the design of a large-scale reprocessing plant as a guide in determining the character and some of the development objectives for the pilot plant. The items being studied are: (1) remote maintenance methods; (2) feasibility of recycle of various liquids and gases; (3) effects of using a low-oxygen-content atmosphere in the head-end, solvent extraction, and related support cells; (4) effect on facility and operation of varying preprocessing decay time from the 120-day base case to 60 and 360 days; and (5) arrangement of shielded facilities, with special attention given to equipment and facilities for fuel receiving, cleaning, and storage, head-end mechanical and dissolution equipment, and conversion of products to oxide.

The proposed criteria are complete and are being submitted to ERDA for approval. Studies of the major problems are also complete; however, assembly of the report will require about five weeks. The scheduled publication date for the CRF study is January 1, 1977.

It is concluded that the IMFBR fuel reprocessing pilot plant should, where feasible, allow for the test of alternative solutions to significant fuel reprocessing problems which require testing under radioactive plant conditions. The most significant of these problems, or problem areas, are

as follows: (1) fuel receiving, cleaning, and storage; (2) preparation of the heavy metal contained in a spent fuel assembly for separation from fission products via solvent extraction; (3) control of fission product emissions; (4) control of exposure of operating personnel to ionizing radiation; (5) reliability of plant operations; and (6) economy of reprocessing insofar as is consistent with the other objectives.

Because of the need for early demonstration of the capability for satisfactorily reprocessing fast breeder reactor fuel, the equipment and systems used in the initial pilot-plant campaigns should provide the maximum reasonable assurance of acceptable performance. Subsequent campaigns should be used to test alternative equipment items and processes in an effort to demonstrate improved process efficacy and to reduce the processing cost.

The major functional criteria for the pilot plant are described in the subsections that follow.

2.1 Input Material

2.1.1 Fuel assemblies

The HPP shall be capable of storing and reprocessing oxide fuel assemblies from the FFTF, CRBR, PLBR, GCFR, and early commercial reactors. However, only the special tooling needed to accommodate the FFTF and CRBR core assemblies, blanket assemblies, and control rods shall be available at startup.

2.1.2 Decay time

The fuel assemblies received at the HPP shall have decayed for a minimum of 60 days. The minimum decay time for assemblies prior to reprocessing shall be 90 days.

2.1.3 Shipping cask

The fuel assemblies shall be shipped to the HPP in a cask similar to the LMFBR fuel shipping container described in AMCO-02-R-107. The

coolant presently planned for use in this cask is Dowtherm A; however, the design of the HPP shall assume that all shipments are made using sodium or an inert gas in the cask. The HPP will not have capability to receive assemblies from casks that have Dowtherm A coolant. It shall be assumed that the CRBR, PLBR, and GCFR assemblies will be shipped in a similar cask with the same cask-to-cell interface characteristics and will use either sodium or inert gas as a coolant. However, provision shall be made to accommodate variations in cask design and cask-to-receiving-cell interface systems.

2.1.4 Cask transporter

Provision shall be made in the HPP to accept casks shipped on either railroad cars or trucks.

2.2 Functional Requirements

2.2.1 Reprocessing capacity

The HPP shall be designed for a nominal instantaneous throughput of 0.5 metric ton of heavy metal per day. Specific consideration shall be given during design to ensure effective operation of all process steps at a throughput of 0.25 metric ton/day. Operation at 0.25 ton/day is necessary to provide for extended periods of operation during the early years of the program when the supply of typical fuel is limited.

2.2.2 Process description

The core and blanket assemblies shall be processed to recover and purify uranium and plutonium using a series of steps, including shearing, voloxidation, dissolution and feed preparation, and Purex-based solvent extraction. The uranium and plutonium products shall be converted to an oxide form. Equipment for off-gas treatment, liquid and acid purification and recycle, and waste handling shall be included.

2.2.3 Accountability

The HPP shall be designed and operated in accordance with applicable codes and Federal regulations regarding accountability of source and fissile materials.

2.2.4 Storage of feed material

The fuel storage facility shall have the capacity for storing 500 fueled assemblies and 70 control assemblies. This capacity will provide a fuel storage surge capacity equivalent to 100 days' operation of the HPP at the nominal 0.5-ton/day capacity with elements containing 100 kg of heavy metal. This storage capacity is consistent with the requirement that the receiving and storage facility be capable of accepting and storing assemblies that must be shipped from the FFTF and the CRBR in 1986 and 1987, prior to startup of the HPP. An estimated 400 fueled assemblies and 50 control assemblies will be discharged from the FFTF and the CRBR during 1986 and 1987. The storage system shall have the provisions for detecting, handling, and separating, if necessary, fuel assemblies suspected or known to be defective.

As a design objective, the heat dissipation capacity for the storage pool shall be 1.5×10^6 W. This dissipation capacity is consistent with a storage capacity of 570 total assemblies with a representative CRBR mix of core, blanket, and control assemblies with an effective cooling time of 150 days.

2.2.5 Process cell ventilation system

The process cell ventilation system shall be capable of both "closed" cycle, low-air-volume throughput operation with local cooling and once-through, high-volume operation.

As a design objective, the total uncontrolled air leakage to the process cells shall be 100 scfm.

2.2.6 Process off-gas systems

The process off-gas systems, including the dissolver off-gas, shall be based on a once-through flow with influx of cell atmosphere to the process equipment limited to a minimal volume.

2.2.7 Storage and shipping of plutonium product

Storage capacity for 5000 kg of PuO₂ product shall be provided. This is approximately 100 days' output of the HPP when operating at the nominal 0.5-ton/day throughput. Facilities for packaging and shipping plutonium product in an approved manner shall be included in the HPP.

2.2.8 External contamination of casks

On the arrival of a cask, its external surfaces are to be rinsed free of road dust and surface contamination. This operation shall be conducted at a receiving station designated for that purpose. Facilities to collect and monitor the rinse solution prior to disposal or treatment (as required) shall be provided.

The external surfaces of casks shall be free of radioactive contamination when shipped from the HPP. Cask decontamination and monitoring facilities shall be provided.

2.2.9 Cask maintenance

The areas of responsibility for cask maintenance have never been defined; nor have agreements been reached. The criteria package, in the absence of clear direction, states a design position that responds to the problem. Alternative design directions can be followed when clear direction by ERDA is provided. It is essential that an adequate understanding and a solution to this problem be developed through technical discussions between the cask designers, reactor sites, and HPP design team.

Equipment shall be provided at the HPP to pump contaminated sodium out of the casks into storage tanks. Facilities for decontaminating the internal surfaces of casks shall also be provided. No facilities shall be provided for sodium reprocessing. Space shall be available for limited maintenance operations.

2.3 Operating and Maintenance Requirements

2.3.1 Support facilities

The HPP shall be an essentially independent site, providing all the facilities and services necessary for routine operation and maintenance of the fuel storage and processing activities. The services include water supply, sanitary waste disposal, electrical service, steam and compressed gas supplies, access roads, and rail spurs. Support facilities to be provided include on-site maintenance shops, mockup areas, operating galleries, piping and sampling galleries, instrumentation and control rooms, laboratory and analytical services, cooling systems, warehousing, and offices. Existing ORNL facilities shall be used, wherever possible, to support nonroutine activities at the HPP.

2.3.2 Plant availability factor

As a design objective, the target on-stream availability for the HPP shall be 60% of the time-rated capacity. However, the HPP facility and associated equipment should demonstrate the feasibility of commercial reprocessing with an 80% on-stream capability, assuming additional redundancy may be required in the commercial plant.

2.3.3 Design life

The design life of the facility shall be 30 years. The design life of specific equipment items may be less, depending on considerations such as technology, economy, etc.; however, spare part inventories and design features for replacement of defective items shall be consistent with the availability factor requirements. Surge capacity shall be provided at appropriate points in the process to permit short-term shutdown of certain components without causing interruption of the entire operation.

2.3.4 Development equipment

All development equipment for the HPP shall be designed to demonstrate concepts that are applicable in a commercial-scale plant. Nondevelopment equipment need not be prototypical.

2.3.5 Maintenance of contaminated equipment

Equipment that will become gamma radioactive shall be located behind shielding walls which are capable of maintaining radiation levels in routinely occupied areas ≤ 0.1 mR/hr. Maintenance of radioactive equipment shall, in general, be performed by remote means to minimize the exposure of maintenance personnel. Contact maintenance of an equipment item shall be permitted in cases where the combination of radiation level, time required for the maintenance operations, and frequency of maintenance would limit personnel exposure to the design objective of ≤ 500 mR/year.

The transfer of equipment, fuel, waste, etc., into and out of contaminated areas shall be performed in a controlled manner in order to minimize the release of contaminants to routinely occupied areas. Facilities shall be provided for handling contaminated equipment, as required, to achieve remote decontamination, remote maintenance, contact maintenance (following decontamination), interim storage, and preparation for disposal.

2.3.6 Equipment interfaces

The design of specific in-cell equipment shall be compatible with the environmental conditions and the remote viewing, manipulation, and maintenance provisions of the cell in which it is located.

2.4 Waste and Effluent Management

2.4.1 Fission product emissions

A major objective of the HPP is to demonstrate practical means to limit, to very low values, the release of fission products to the environment. The effluent control systems included in the HPP shall be capable of demonstrating retention factors which, if applied to a 5-ton/day commercial LMFBF fuel reprocessing plant on a typical site, would yield a whole-body dose of ≤ 1 mR/year at the site boundary.

As a design objective consistent with the above goal, the effluent control systems of the HPP shall be capable of providing overall plant retention factors as follows: ^{14}C , 5; ^3H , 5; ^{85}Kr , 100; ^{131}I , 1×10^7 ; ^{129}I , 4×10^4 ; Ru, 5×10^6 ; particulates, 2×10^{11} ; transuranics, 2×10^{11} .

2.4.2 Disposition of high-level liquid waste

High-level liquid waste shall be concentrated and solidified on a current basis in a manner acceptable for subsequent transfer to a Federal repository. The shipping casks and cask transporters will not be provided by the HPP.

Facilities for the storage of solidified high-level waste shall be provided with a capacity equivalent to that produced from one year's operation of the HPP at the nominal 0.5-ton/day rate. Storage capacity equivalent to one year's operation of the HPP at the nominal 0.5-ton/day rate shall be provided for concentrated high-level waste prior to solidification.

The conversion of high-level liquid waste to a suitable solid form is a part of the HPP design and construction effort. While the equipment and facility provisions are included as part of the HPP task, the development of the technology is a separate ERDA responsibility. Transfer of that technology to the HPP design effort must occur at a fixed milestone.

2.4.3 Disposition of metal scrap

Metal scrap originating from fuel assemblies, the various process steps, and nonrepairable process equipment shall be consolidated and packaged in a manner acceptable for subsequent transfer to a Federal repository. The waste shipping casks and transporters will not be provided by the HPP.

Facilities for the storage of consolidated metal scrap shall be provided with a capacity equivalent to that produced from one year's operation of HPP at the nominal 0.5-ton/day rate. As a design objective, the heavy (uranium and plutonium) metal content of metal scrap, prior to consolidation, shall be reduced to a value $\leq 0.1\%$ of plant input.

2.4.4 Process liquid recycle and disposition

The HPP shall contain equipment for the recovery, purification, and recycle of water and acid for use in all process functions. The amount of water added to the process shall be minimized, and excess water shall be extensively decontaminated prior to release. The level of radioactivity

in the discharged water shall be consistent with the overall fission product emission criteria. Capability shall be provided for future addition of systems for long-term storage treatment or solidification of excess water, as a potential tritium control demonstration.

2.4.5 Suspect liquid waste

Nonprocess, potentially contaminated wastes such as changeroom showers, sink effluents, and fire protection water discharges from potentially contaminated zones shall be routed to a collection system for monitoring and processing, if required, before release.

2.4.6 Miscellaneous noncombustible solid waste

Facilities shall be provided for the handling and packaging of noncombustible solid waste in a manner suitable for permanent disposal.

2.4.7 Contaminated combustible waste

Combustible waste, including any waste process organics, shall be reduced to a noncombustible material suitable for disposal via a suitable combustion process.

2.5 Plant and Public Safety

Specific regulations, codes, and standards that apply to the facility shall be established during the early phases of the HPP concept design and shall include the basic areas described below.

2.5.1 Nuclear safeguards

Federal regulations for the safeguards of nuclear materials shall be followed.

2.5.2 Personnel radiation exposure

The radiation exposure of plant operating and maintenance personnel shall be in accordance with Federal regulations. A design objective for

the HPP facility and equipment is to limit this exposure to 500 mrem/year, which is significantly below existing regulations.

2.5.3 Nuclear criticality

Nuclear criticality control shall be in accordance with Federal regulations; the techniques that are used shall be applicable to commercial reprocessing plants.

2.5.4 Personnel safety

Safety standards for plant personnel shall be in accordance with Federal regulations.

2.5.5 Facility structural requirements

Structural requirements for the buildings shall be in accordance with established codes and standards, including those for natural phenomena such as earthquakes, tornadoes, and flooding.

2.5.6 Recovery from abnormal conditions

A safety analysis of the facility systems shall be made and appropriate features incorporated to ensure safe recovery from abnormal conditions or potential accidents with an acceptable risk to plant personnel.

3. LABORATORY AND HOT-CELL STUDIES

B. L. Vondra

Process research and development directed toward the definition of process parameters and conditions for the complete flowsheet were continued. Studies presently in progress are examining:

- (1) Base metal zeolites for iodine trapping.
- (2) Solubility of PuO₂ in HI.
- (3) Fixation of iodine wastes in concrete.
- (4) Removal of organic contaminants for aqueous recycle streams.

- (5) Head-end process steps with irradiated fuel.
- (6) Recycle of nitrogen oxides for gaseous reagent usage.

The major efforts in the decommissioning of the hot cells in Building 4507 have been completed. Current plans call for installation of equipment in the midquarter and initiation of hot-cell development in November.

3.1 Hot-Cell Operations

J. H. Goode, R. G. Stacy, V. C. A. Vaughen, E. C. Hendren, G. K. Ford

3.1.1 Development of in-cell equipment

We have reported the success of the continuous tritium monitor to detect and follow $^3\text{H}_2\text{O}$ concentrations in air, and have discussed the problem with interference by ^{85}Kr when present. The lack of success of sparging to remove the krypton interference led us to turn our attention to another possible culprit. Since our source of ^{85}Kr is the burner off-gas from HTGR fuel burnings, we suspected $^{14}\text{CO}_2$. This has been confirmed by finding ^{14}C in the scintillator liquid in a batch shakeout test.

It may be possible to modify our system to separate and report both $^{14}\text{CO}_2$ and $^3\text{H}_2\text{O}$ concentrations, certainly a benefit to the analysis of LWR voloxidizer off-gases.

Testing of other in-cell equipment was delayed in favor of using all personnel for decommissioning the Curium Recovery Facility (CRF). All of the equipment, however, was put in operating condition by the ORNL shops, and is ready for further cold-testing as manpower becomes available.

3.1.2 Decommissioning the Curium Recovery Facility

Ninety-five individual entries in air-line suits were made into the curium-contaminated CRF during the quarter. The 2-ton, 13 x 3 x 9-ft-tall Equipment Maintenance Glove Box, located above Cells 3 and 4 in Building 4507, was separated from the floor, sealed, crated, and sent to storage in the ORNL Solid Waste Storage Facility.

Sandblasting was used to remove curium-contaminated paint from cell access holes and the concrete walls of the hot cell in the CRF. On completion of the sandblasting, the sand was bagged-out of the cells, and the surfaces were successively cleaned with a vacuum cleaner and washed with detergent-water mixtures. Subsequent Health Physics surveys indicated a few hot spots that required additional cleaning, but the cells were generally at sufficiently low contamination levels to permit preparations for resurfacing and repainting. These included removal and patching of unnecessary glove ports and windows in the sheet-metal alpha barrier in the rear of each cell, cutting and beveling piping for later welding, masking pipe fittings for protection from paint, etc. Anchor-bolt holds were filled and the sandblasted concrete walls of Cell 3 plastered with stucco. Two coats of white epoxy paint were sprayed onto each cell and their access ports. After removal of the "vestibule" and "tent" used as contamination-control zones for personnel working in the cells, installation of fixed equipment in the cells will begin. These include plenums for cell ventilation exhaust filters, floor pans, work surfaces, etc. (Sect. 3.1.3).

3.1.3 Miscellaneous

The Garden Carrier No. 2 (AEC-OR-USA 6088/BLF) cask was returned from Battelle-Columbus Laboratory with the second shipment of irradiated UO_2 from the Carolina Power and Light H. B. Robinson-2 reactor. A portion of this second shipment was repackaged for another group at ORNL, and a portion of the first shipment was packaged and sent to the Savannah River Laboratory along with an empty cask (D-38, AEC-OR-USA 5787/BLF) to pick up recycle SRP solvent for laboratory tests. The Garden Carrier was then sent to the Aerojet Nuclear Company, at Idaho National Engineering Laboratory, to pick up three complete rods from the H. B. Robinson reactor assembly B05.

Figure 3.1 shows the assembled working surface and fuel storage array ready for installation in Cell 3, Building 4507, where shearing and voloxidation head-end studies will be performed. The ORNL Criticality Committee has approved the storage of 1.6 kg of $^{235}\text{U} + ^{239-241}\text{Pu}$

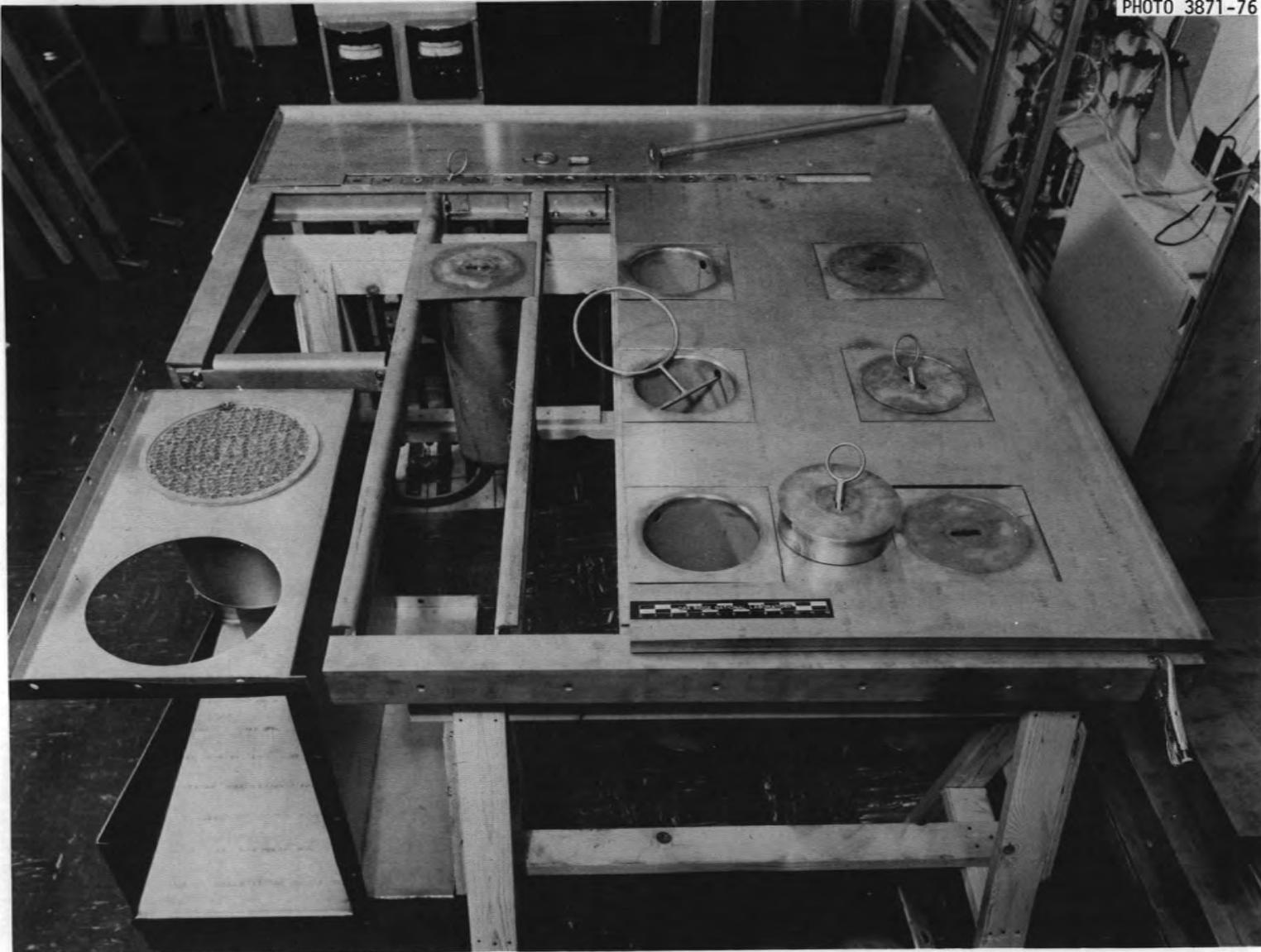


Fig. 3.1. Work tray and fuel storage wells for voloxidation cell.

in the seven 7-in.-diam wells and sixteen 1-in.-diam wells. Lead-filled covers provide shadow-shielding overhead. Figure 3.2 shows the all-bolted construction of the support structure, the removable storage wells, and the cell exhaust plenum and a drop-in HEPA filter.

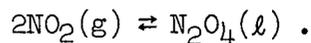
Figure 3.3 shows the intercell (Cell 2-3) transfer port with the doors closed and the sliding tray centered. Figure 3.4 shows one of the folding doors open and the trays projecting into a "cell."

Fabrication is in progress on floor pans for Cell 4 and a new sample chute for Cell 3. Design of a replacement Equipment Maintenance Glove Box and access hole shielding plugs is under way. Modification of a cask pedestal to place above the Cell 3 sample chute is in progress.

3.2 Distribution of NO₂ Between Air and Hyperazeotropic Nitric Acid

J. C. Mailen

The distribution of NO₂ between air and hyperazeotropic HNO₃ is an important aspect of the operation of an Iodox iodine trapping system since NO₂ can reverse the trapping reaction. An earlier reported analysis¹ of the measured values of the NO₂ distribution has been found to contain an erroneous factor of 2 due to failure to properly account for the formation of dimer in solution:



The proper values for this equilibrium constant,

$$K = \frac{c_{\text{N}_2\text{O}_4}^{1/2}}{c_{\text{NO}_2}},$$

are given below for a temperature of 25°C:

c_{HNO_3}	K
15.7	108
18	152
20	256

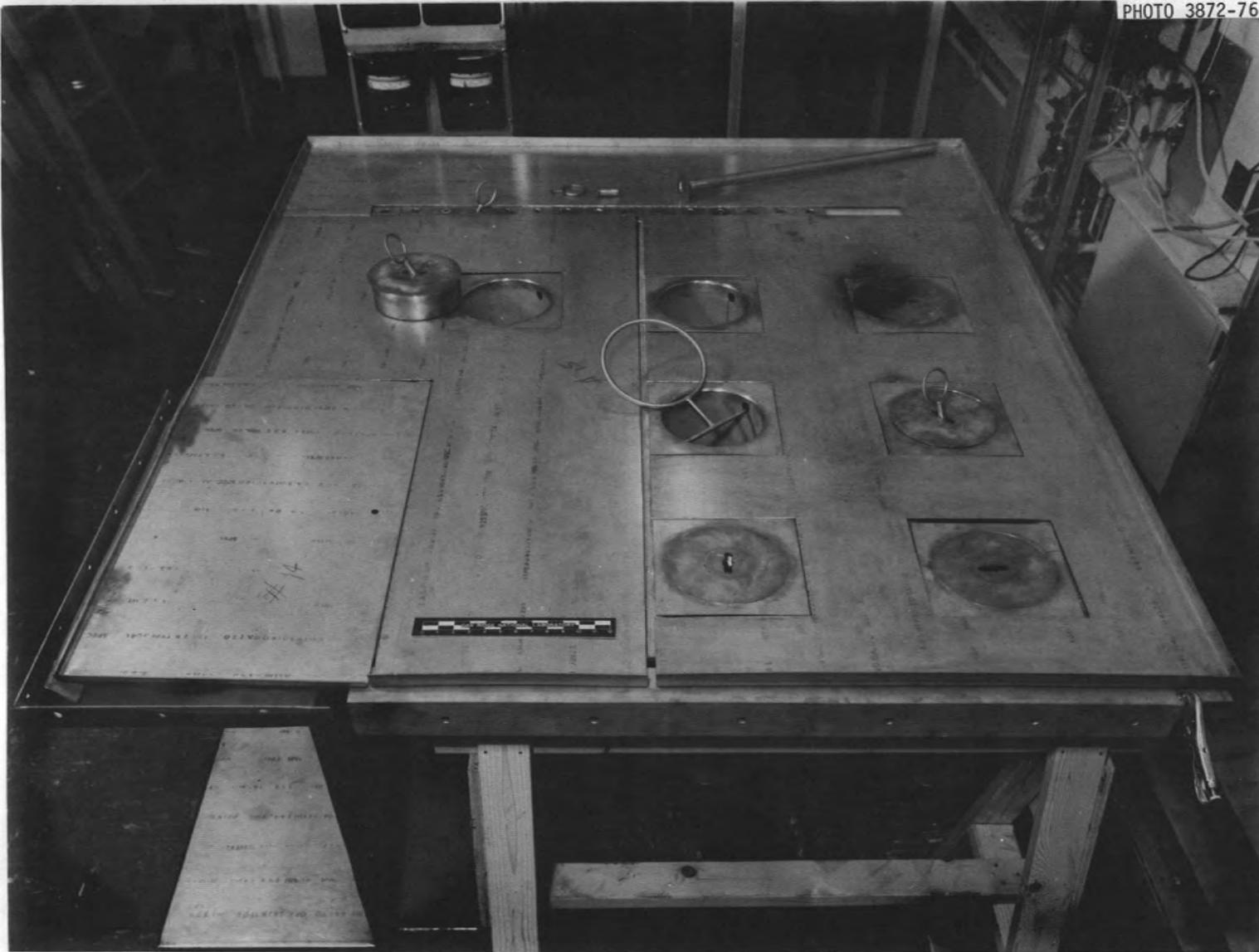


Fig. 3.2. Partially assembled work tray and fuel storage wells, showing bolted construction and cell exhaust plenum and HEPA filter.

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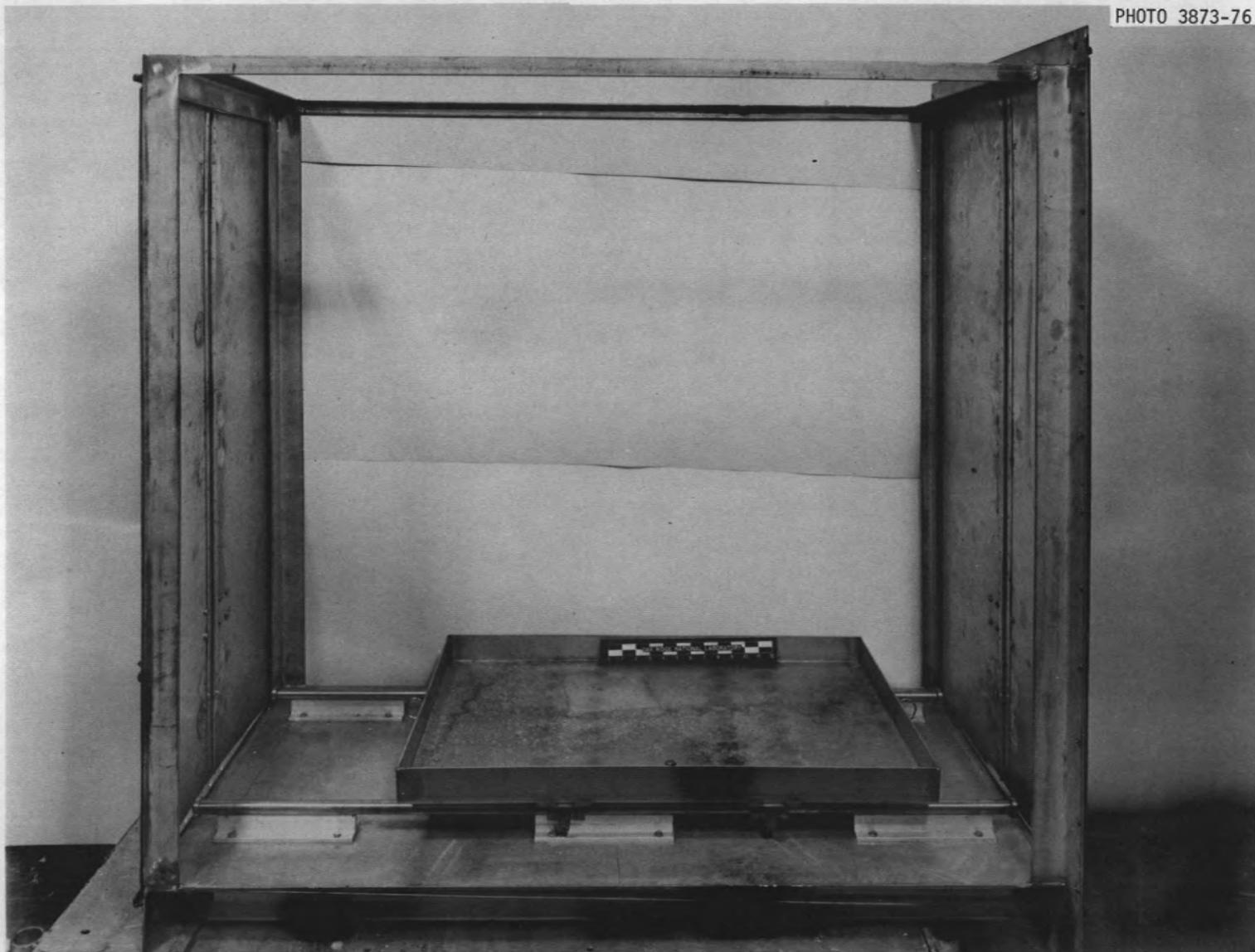


Fig. 3.3. Intercell transfer port.

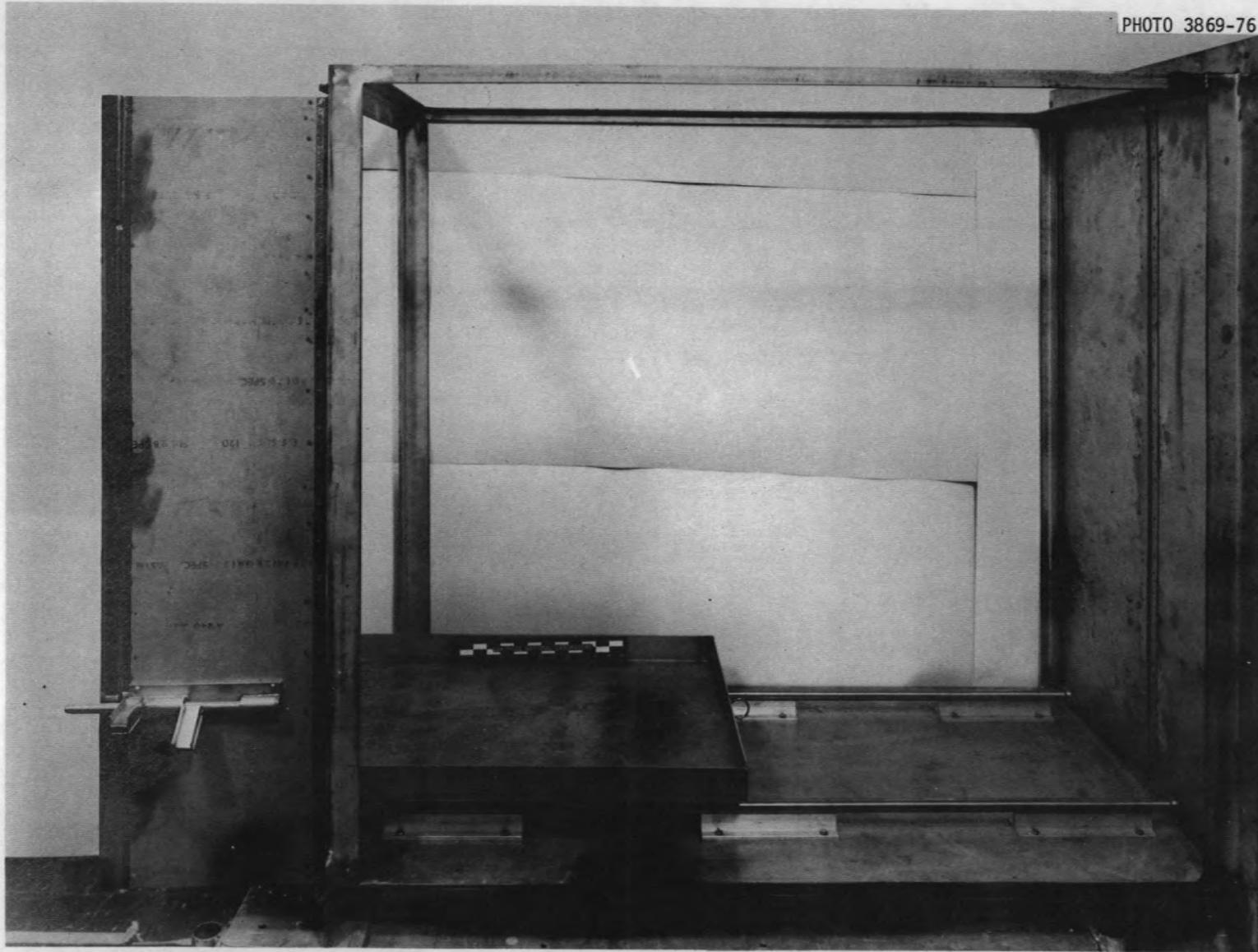


Fig. 3.4. Intercell transfer port with manipulator-operated door in open position.

A satisfactory fit of these data is given by:

$$\ln K = 0.2 C_{\text{HNO}_3} + 1.56,$$

where C_{HNO_3} is molar concentration.

3.3 Plutonium Dioxide Dissolution Studies

O. K. Tallent and K. D. Williams

We are continuing to examine methods for dissolving the plutonium residues² remaining after dissolution of IMFBR-type fuel rods in HNO_3 . Criteria for acceptable dissolution methods for these residues include that the dissolvent volume be kept low, that the addition of foreign ions be held to a minimum, and that as few as possible new process steps be involved. Recently obtained experimental data relative to PuO_2 dissolution in HI solutions are reported here. Information on solids characterization during PuO_2 and UO_2 dissolution is also presented.

3.3.1 Dissolution in aqueous HI solutions

Kinetics data for PuO_2 dissolution in aqueous HI solutions have been previously reported.² The primary emphasis of the PuO_2 dissolution work at this time is directed toward developing process methods for the recovery of plutonium from HI dissolver solutions and subsequent recycle of the solutions.

One HI recycle process method under consideration at present involves dissolution of PuO_2 -bearing residues in HI solution, distillation and evaporation of dissolver solutions to dryness, recycle of distillates to dissolve more solids, and dissolution of dried PuOI cakes in $8.0 M \text{HNO}_3$.

In a preliminary test, a 1-g quantity of highly refractory PuO_2 microspheres was dissolved in 25 ml of $6.35 M \text{HI}$ at $\sim 128^\circ\text{C}$ in $< 8.0 \text{ hr}$. The microspheres in the original state had a density of 11.0 g/cc and a surface area of $0.012 \text{ m}^2/\text{g}$. The resulting dissolver solution, which had a dark blue color characteristic of Pu(III) was distilled to dryness.

The dried cake was dark blue, and the distillate had a plutonium concentration of $< 1 \times 10^{-5} M$. The dried cake was divided into three approximately equal portions. One portion was dissolved in 10 ml of 8.0 M HNO_3 to yield a 0.123 M Pu(IV) solution with an iodide concentration of $< 0.0008 M$. The other two portions are being reserved for analyses and additional experiments.

3.3.2 Characterization of PuO_2 and UO_2 solids during dissolution

Records and reports from previous PuO_2 and UO_2 dissolution work are currently being reviewed. The two particular aspects of earlier work being reviewed at present involve PuO_2 and UO_2 surface changes during dissolution and the small fraction of PuO_2 and UO_2 that is slow in dissolving.

Occasionally, papers are found in the literature which report that the surface area of a given batch of PuO_2 , UO_2 , or ThO_2 decreases as the batch is dissolved. Such decreases in surface area are usually calculated values based on assumed particle size and geometry changes and do not reflect actual experimental data. In the past, a number of PuO_2 dissolution problems have been ascribed to the presumed surface area decreases. Such decreases are frequently used to explain observed reductions in PuO_2 or ThO_2 dissolution rate with time. We have shown in previous reports that decreases in PuO_2 microsphere dissolution rates in HNO_3 -HF- H_2O solutions are, in large part, due to the formation of Pu(IV) fluoride complexes.

Microphotographs taken during one of our PuO_2 microsphere dissolution experiments indicate that there is an increase--not a decrease--in PuO_2 surface area throughout a significant fraction of the dissolution time. The microspheres used in this experiment initially had a density of 11.0 g/cc, a surface area of 0.012 m^2/g , and a diameter of 149 to 117 μ . They had been calcined at 1150°C in argon--4% hydrogen. One-half-gram batches of the microspheres were digested in 12.0 M HNO_3 --0.04 M HF at 100°C in all-Teflon equipment for varying periods of time.

Microphotographs of samples of the PuO_2 left undissolved are shown in Fig. 3.5. The first microphotograph shows the microspheres in the

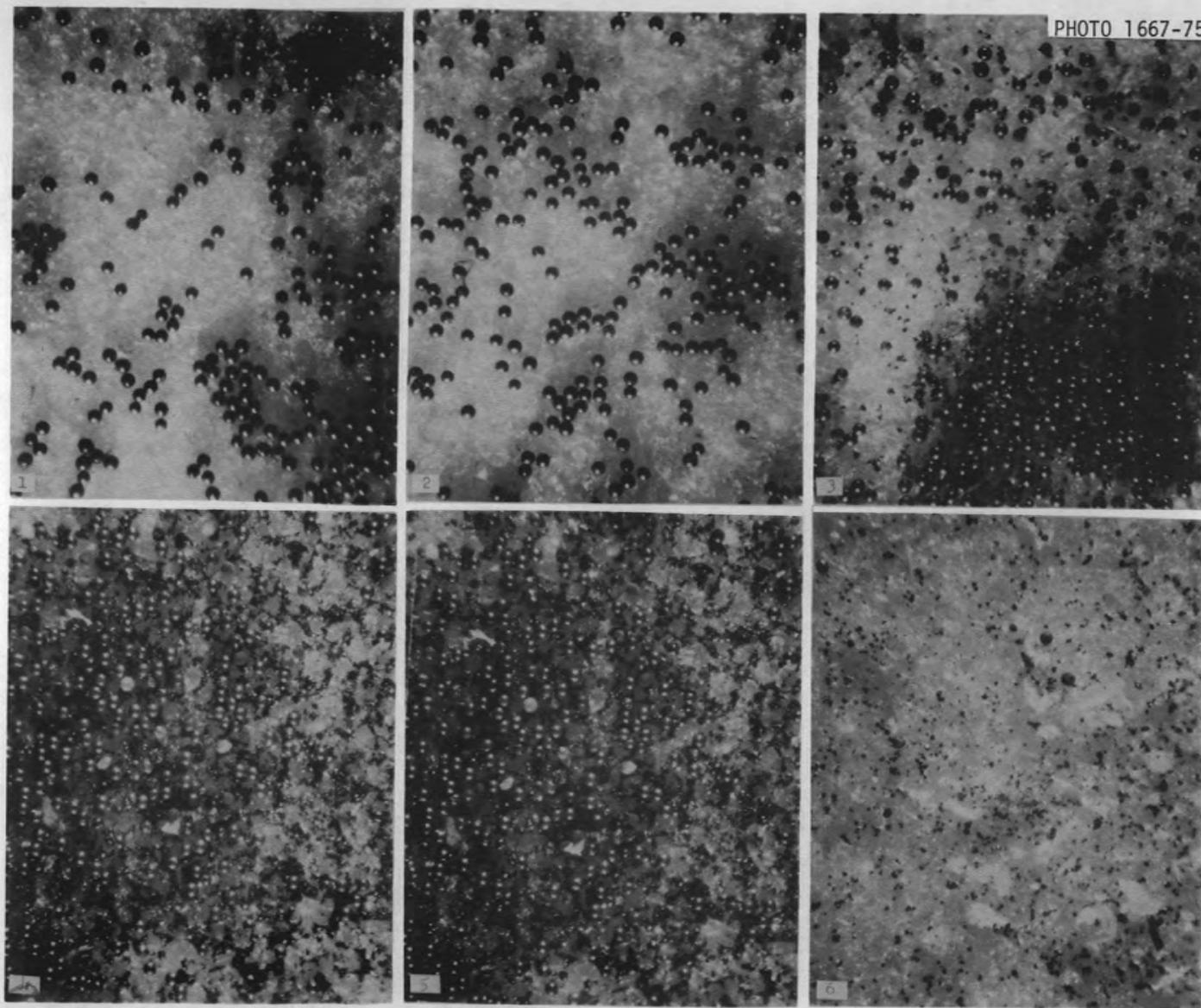


Fig. 3.5. Plutonia microspheres at various stages of dissolution
in 12 M HNO₃--0.04 M HF.

original state, while the other five show microspheres and fragments at times during dissolution when the PuO_2 was 10.1, 22.4, 33.2, 51.4, and 85.3%, respectively, dissolved. Inspection of the microphotographs reveals that the net surface area of the undissolved PuO_2 increases throughout a significant fraction of the dissolution period due to roughening of the surface of the microspheres. It is also of interest that a few of the microspheres appear to be dissolving at a much slower rate relative to the others.

Tests with UO_2 microspheres have given results similar to those obtained for the PuO_2 microspheres. These tests were conducted by digesting 1.0 g of UO_2 microspheres with a density of 10.44 g/cc, a surface area of $\sim 0.01 \text{ m}^2/\text{g}$, and a diameter of 350 to 420 μ in 25-ml volumes of 6 M HNO_3 at boiling temperature in glass equipment. The dissolution times were varied. At the end of each dissolution, dissolvent samples were taken for uranium analyses. The microspheres were washed, dried, and photographed.

Microphotographs taken of the UO_2 microspheres at various stages of dissolution are shown in Fig. 3.6. The first microphotograph shows the microspheres in the original state, while microphotographs 2, 3, 4, 5, 6, 7, and 8 were taken after 0.25, 0.50, 1.08, 1.50, 2.00, 6.13, and 7.71 hr, respectively, of dissolution time. It can be seen that, as the microspheres dissolve, their surfaces roughen and that the dissolution time is much longer in some cases than in others. As microphotograph 8 shows, there are large differences in the sizes of the microspheres. Even though 90% of the uranium has dissolved, some of the microspheres are almost the original size. The reason for the differing dissolution rates for the microspheres is unknown at this time since all the microspheres were prepared and calcined as one batch under the same conditions.

It should be noted that our results indicating increased surface areas of microspheres during dissolution is in agreement with information presented by A. L. Uriarte³ in 1965. This study showed that measured surface areas of mixed UO_2 - PuO_2 particles in fuel pellets increased significantly during much of the period of their dissolution in HNO_3 solutions.

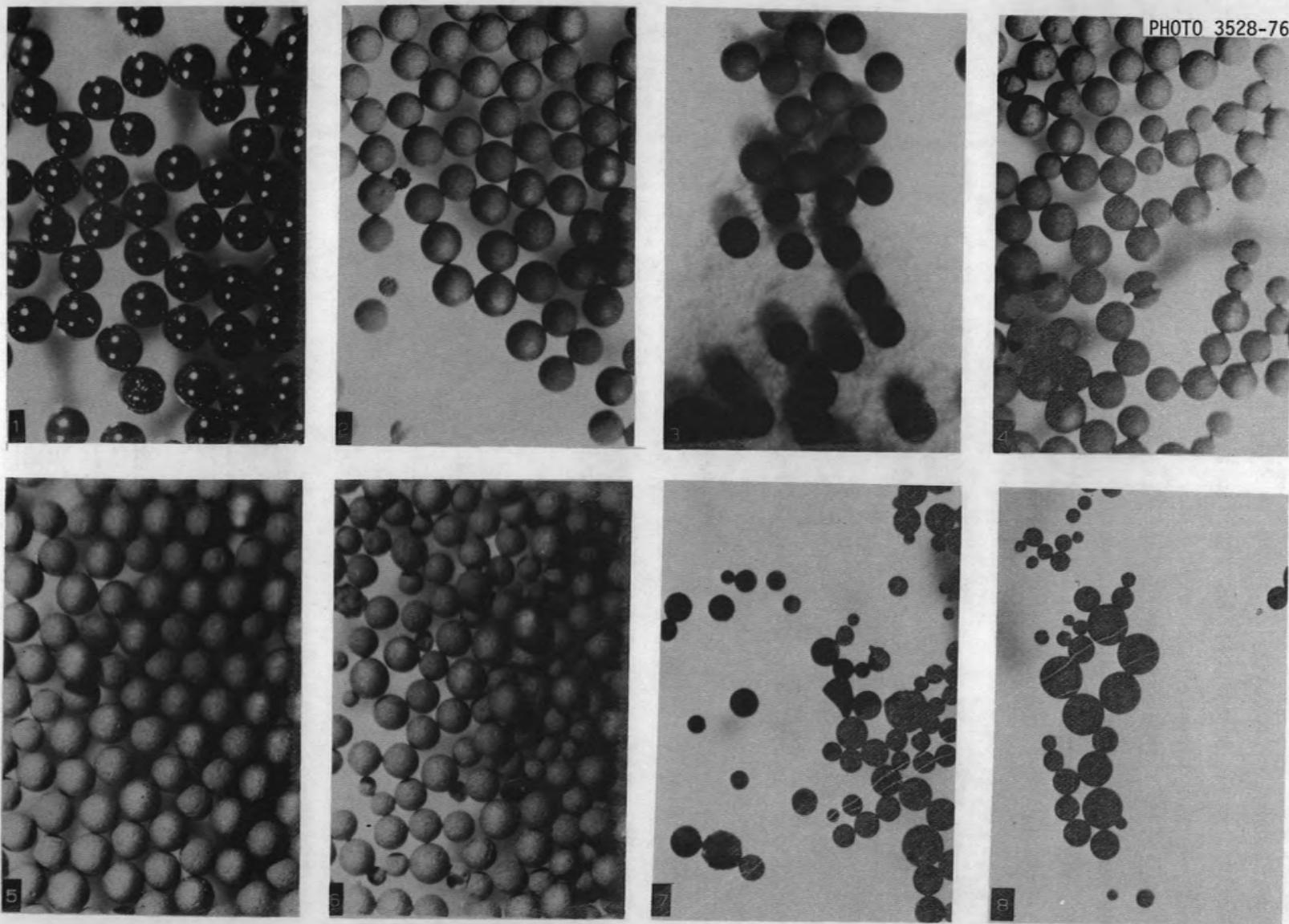


Fig. 3.6. Urania microspheres at various stages of dissolution in 6.0 M HNO_3 .

At some time in the future, we expect to isolate samples of the slow-dissolving microspheres and to obtain surface area, chemical impurity, density, grain boundary, crystallite size, and scanning electron microscope data for comparison with data from microspheres that dissolve at a normal rate. The information obtained is expected to be useful in determining why a small fraction of the microspheres dissolve at a much slower rate. In addition, the information is expected to be useful in defining conditions for the preparation of fuels that will dissolve faster and more completely.

3.4 Stability of Iodate in Iodox Waste Storage Tanks

W. E. Clark and W. B. Howerton

Small-scale irradiation experiments indicate that little, if any, volatilization of I_2 will occur from irradiated solutions of iodic acid (HIO_3) in azeotropic HNO_3 as long as the solutions are vigorously boiling. This conclusion is confirmed by experiments in which the radiolytic generation of nitrite was simulated by introducing N_2O_3 gas into a boiling solution of HIO_3 . Free I_2 formed rapidly in cooler solutions. It appears that only a minimum sparge will be required to prevent iodine volatilization from the Iodox storage tank, but that rapid formation of free iodine may occur if boiling is allowed to cease.

The effective G-value for generation of HNO_2 in the boiling storage solution was found to be approximately 9.7.

REFERENCES FOR SECTION 3

1. J. C. Mailen and W. B. Howerton, p. 56 in Aqueous Fuel Reprocessing Quarterly Report for Period Ending March 31, 1974, ORNL-TM-4587 (September 1974).
2. W. D. Burch et al., IMFBR Fuel Recycle Program Progress Report for Period January 1 to March 31, 1976, ORNL/TM-5463 (June 1976).
3. A. L. Uriarte and R. H. Rainey, Dissolution of High-Density UO_2 , PuO_2 , and UO_2 - PuO_2 Pellets in Inorganic Acids, ORNL-3695 (April 1965).

4. ENGINEERING RESEARCH STUDIES

W. S. Groenier

Initial equipment concepts are being developed and evaluated, and engineering data on which to base equipment designs are being obtained, in the various engineering research studies reported here. Studies of the dissolution, voloxidation, NO_x scrubbing, and Iodox processes are presently in progress. Development efforts will be defined, and investigations will be initiated in the future in several other areas.

All work directed toward the retention of krypton in the fluorocarbon absorption process, as well as the development of instrumentation and control methods, is managed under this part of the program and is also reported here.

4.1 Voloxidation

M. E. Whatley and D. C. Hampson

The voloxidation process has been proposed as a head-end method for removing tritium from the fuel prior to aqueous processing. Based on previous experimental work, it appears that this objective can be met by heating the oxide fuel to 450 to 600°C (723 to 873 K) in flowing oxygen or air. Volatilization of tritium and other fission products results as the UO_2 in the mixed-oxide fuel becomes restructured during oxidation to U_3O_8 . Early removal of tritium from the fuel into a relatively small volume is desirable in order to avoid extensive dilution of the tritium with water in the subsequent fuel dissolution step. A secondary benefit of voloxidation is the oxidation of residual sodium in the fuel prior to dissolution.

The rotary-kiln studies are described and reported as part of the LWR Fuel Reprocessing and Recycle Program,¹ which now provides funding for voloxidation work. The studies are directly applicable to either LWR or LMFBR fuels.

4.2 Dissolution

The objective of the dissolution task is to ensure that IMFBR fuels can be dissolved in HNO_3 to give high metal recoveries. Dissolution characteristics of the fuels can vary widely, depending on plutonium content, method of preparation, and irradiation history. Consequently, extensive leaching data will be obtained in the overall program to determine the effects of such variables. Other efforts are directed toward understanding the behavior of iodine in the dissolver system. Criticality control and off-gas considerations call for dissolver design and operation to be performed within rather narrow limits. Continuous dissolving methods, which appear to offer superior solutions to these problems, are being emphasized.

During this report period, the major emphasis was focused on the ongoing development of a continuous, rotary dissolver.

4.2.1 Studies of the one-fourth-scale rotary dissolver

J. Q. Kirkman

The glass-enclosed section of the 1-ft-diam (0.3-m-diam), three-stage rotary dissolver is currently being modified to accept a set of scaled stage internals identical in design to those planned for the pilot-plant-scale dissolver (see Sect. 5.4). Performance testing under simulated operating conditions is planned subsequent to completion of the modification work.

The purpose of this modification and testing is to investigate the behavior of both the single skewed baffle and the gravity-induced liquid transfer concepts that are being incorporated in the stage design of the pilot-plant-scale dissolver. These two features are new and do not exist in our present operating models.

Both the 1-ft- and the 4-ft-diam (see Sect. 4.2.2) designs have two internal baffles per stage. One baffle agitates the solids within a stage during forward drum rotation, while the other transfers solids to the next dissolver stage on reversal of rotation. The new, single skewed-baffle

arrangement represents a simplification compared with present designs since both the solids agitation and transfer functions are performed with only one baffle.

The transfer of liquid from stage to stage is presently accomplished by two different methods. In the 1-ft-diam model, the stage bulkheads are perforated with four circumferential rows of holes near the outer edge; in the 4-ft model, radially placed buckets in each stage dip liquid from one stage and physically transfer it to the next in a manner similar to that observed with a waterwheel. The perforated bulkhead concept suffers certain disadvantages such as the tendency toward liquid back-mixing and potential solids plugging. In addition, the waterwheel concept is mechanically complex. To avoid these problems, the new stage design utilizes a solid bulkhead with one circumferential row of slots placed on a diameter which will maintain a specified liquid depth. The entire dissolver is inclined 5° from horizontal so that liquid flows by gravity from stage to stage, spilling through the bulkhead slots.

Placing one stage of suitably scaled pilot-plant dissolver internals into the glass-lined section of the 1-ft-diam dissolver provides a means of testing the new baffle and liquid transfer concepts described above. The geometrical scaling between the 1-ft-diam model and the larger pilot-plant design has been established by holding the ratio of liquid volume to total stage volume constant. Hydraulic similarity with regard to passage of the agitation baffle through the liquid will be attained by adjusting the rotational speed of the 1-ft-diam model.

The objective of the planned testing is to visually observe the performance of the internal stage design being developed for the pilot-plant-scale dissolver. Specifically, we will note and record the following functions on high-speed film:

- (1) Solids agitation with a single skewed baffle.
- (2) Solids transfer with a single skewed baffle.
- (3) Stage-to-stage liquid transfer in the presence of liquid disturbances caused by the rotating baffle.

Equipment modifications are essentially complete. Testing is scheduled for October 1976.

4.2.2 Four-foot-diameter dissolver prototype

B. E. Lewis

The 4-ft-diam rotary dissolver (Fig. 4.1) is composed of a single dissolution stage coupled with inlet and outlet sections. This dissolver is enclosed within a boxlike structure for off-gas control and does not require mechanical shaft seals. Provisions have been made to recycle the solids from the discharge section to the feed section via a conveyor belt, bucket elevator, and feed chute containing an atmosphere isolation valve. The equipment has been assembled and in operation for a total time of ~ 97 hr, during which solids handling characteristics, leak rates, solution backmixing, and liquid carryover have been studied. The liquid carryover and solution backmixing tests are currently in progress while preparations are being made to study the fines washing capability of the dissolver.

Solids handling characteristics of the 4-ft-diam dissolver were studied in a deliberate attempt to plug the dissolver using prototypical porcelain-filled IMFBR sheared fuel elements along with C- and S-shaped hooks. These hooks represent simulated shroud pieces and were comprised of 1-in.-wide stainless steel strips 6 to 14 in. long. Each hook was given an inventory control number and recycled through the dissolver system along with ~ 50 kg of a typical feed mixture (70 wt % hulls, 30 wt % shroud) of sheared fuel. During the first period of about one week, 17 C-type hooks of assorted size were continuously recycled through the dissolver. Holdup of the hooks in the dissolver increased until nine appeared to have become permanently lodged. Inspection of the exit section of the dissolver showed the nine hooks, along with pieces of shroud and hulls, hanging on the lip of the solids exit chute (see in Fig. 4.2). The hooks were subsequently removed from the dissolver, and the run was continued using an increased inventory of 26 C-type hooks along with the 50-kg feed charge. After about one week, the inspection ports were again removed to exhibit six hooks hung on the solids exit chute along with pieces of shroud and hulls. At this point, 12 S-shaped hooks of assorted sizes (6, 8, 10, and 14 in. long) were prepared. These new hooks increased the total hook supply to 38. The entire inventory of hooks was added to the dissolver in a deliberate attempt to

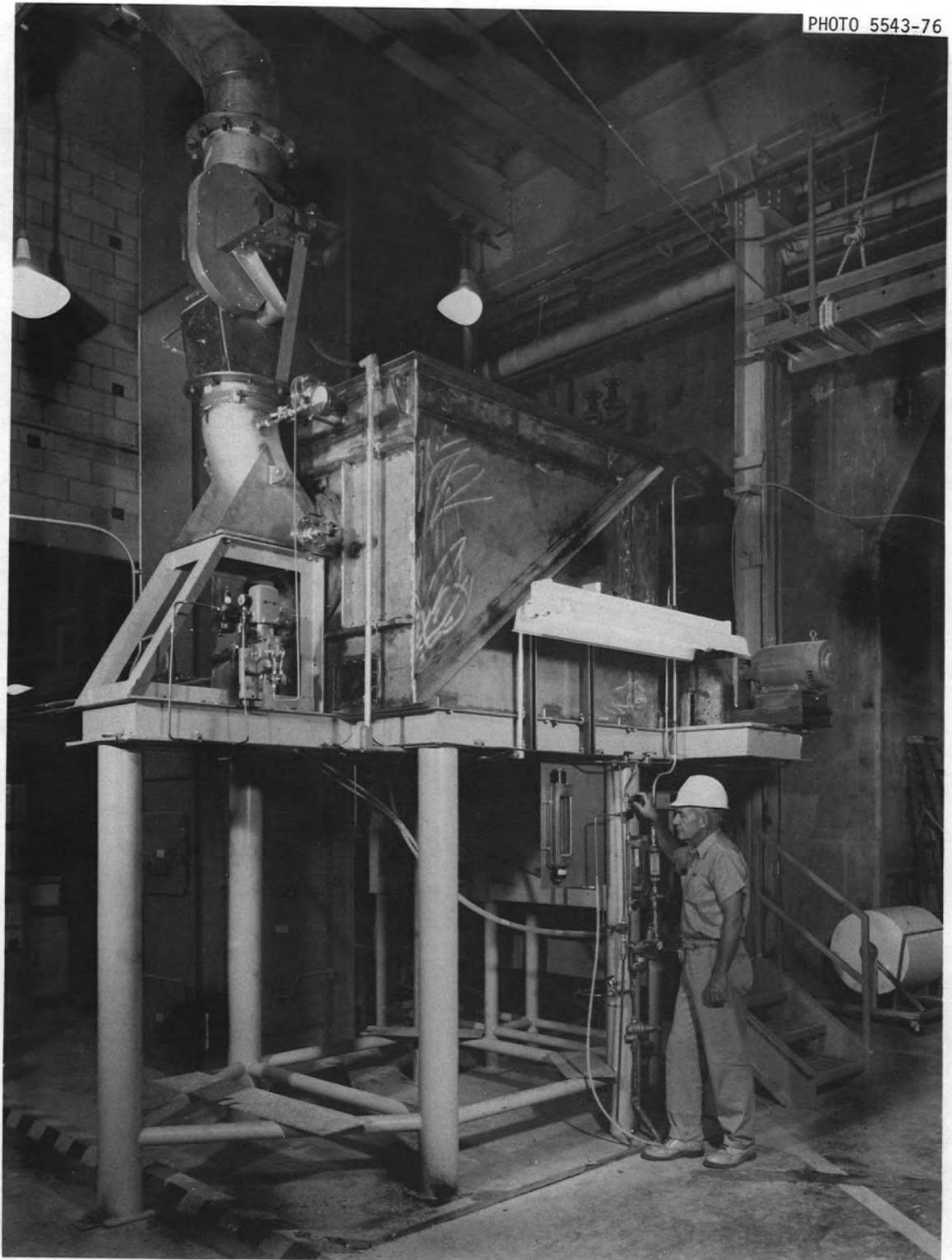


Fig. 4.1. Four-foot-diameter dissolver system.

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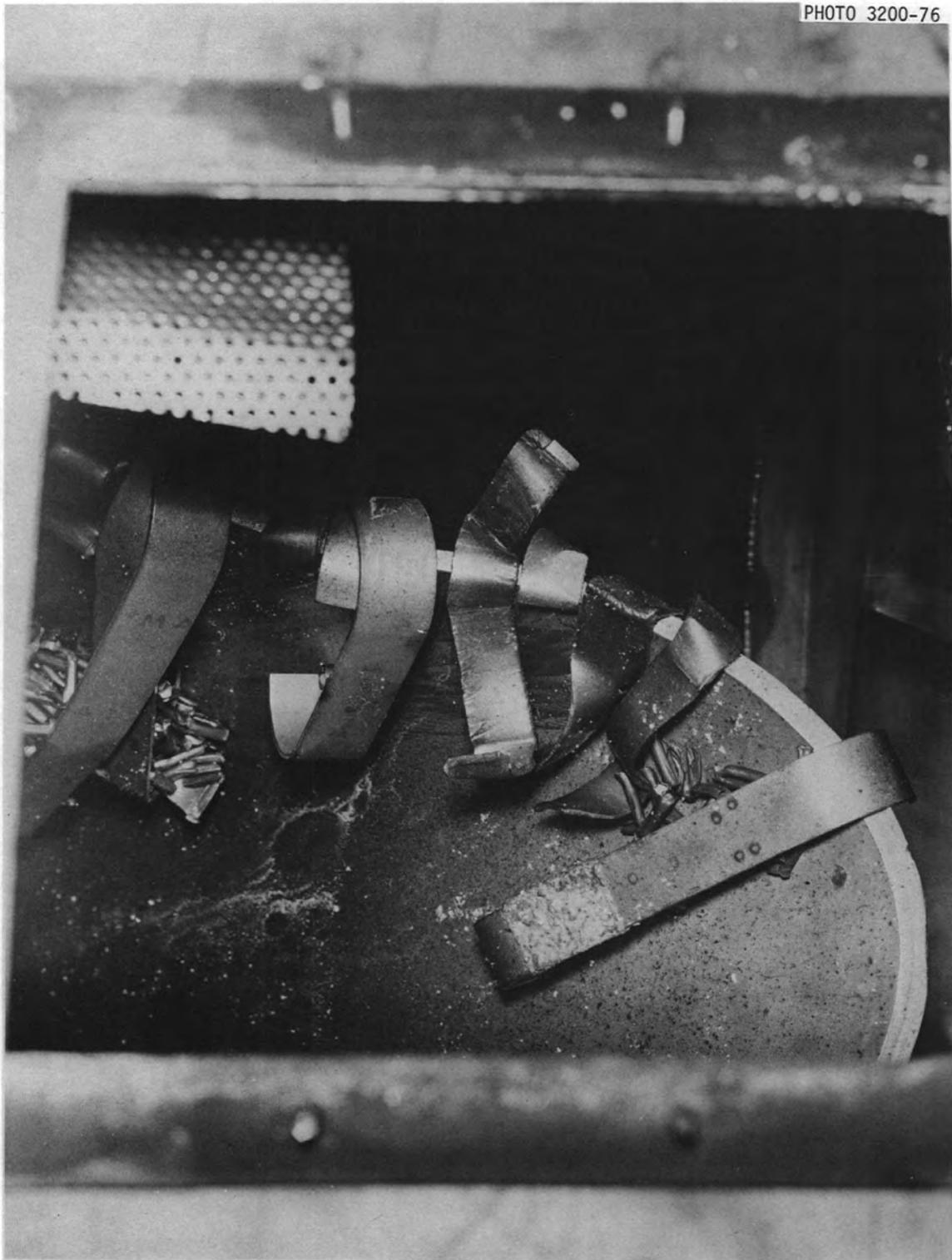


Fig. 4.2. Solids exit chute showing holdup of hulls, shrouds, and hooks.

block the transfer of solids. After ~ 1 day of operation, the solids exit chute became blocked by the accumulation of hulls, shroud pieces, and hooks on the lip of the chute. This test was repeated to determine the minimum number and size of the available S-shaped hooks required to block the dissolver. No blockage occurred until all but three of the hooks had been added to the dissolver. The solids exit chute was the only part of the 4-ft-diam dissolver where holdup or blockage occurred.

Because of the difficulty in sealing large-diameter rotating objects, there are no seals between the stationary and rotating parts of the 4-ft-diam rotary dissolver. Instead, the dissolver drum is enclosed in a box-like stainless steel housing with the stationary inlet and exit chutes coming through the housing into the rotating drum. This leaves approximately a 1/8-in. gap between the stationary and rotating parts. The gap is above the liquid level. The rate at which the fluid in the dissolver leaks out through the gap by splashing was measured for a variety of liquid feed rates and drum rotational speeds. It was found that the leak rate increased with both drum speed and feed rate. At a drum speed of ~ 5 rpm and a feed rate of ~ 2.6 gpm, the leak was approximately one-third of the total feed stream. In the pilot-plant dissolver, we intend to purge the housing with steam and force an inleakage of steam through the gap. This should minimize leakage.

Preliminary backmixing tests have been completed by placing a known volume and concentration of HNO_3 in the middle section of the dissolver, and known volumes of water in the exit and inlet sections. The dissolver was then rotated in the reverse or material transfer direction. Samples of the liquid in each section of the dissolver were titrated to determine the acid content and hence the degree of backmixing. Thus far, results indicate negligible backmixing. The liquid carryover tests are being performed in a similar manner, with dilute HNO_3 being placed in the center section of the dissolver along with a charge of hulls and shrouds. The hulls currently being used are only partially filled with ceramic material. The feed charge is then transferred out of the dissolver, and liquid samples are obtained. Titration of the samples gives an indication of the amount of liquid transferring with the hulls and shrouds.

From the first two runs that have been made, it appears that ~ 40 cc of liquid transfers with each kilogram of hulls and shrouds. Similar tests will be conducted using hulls both completely filled with ceramic material and completely empty.

Accelerometers and hydrophones have been installed on four of the eight pillow-block supports of the 4-ft-diam dissolver. These will be used to measure and record the vibration signature of the operational dissolver. The signature will be used for identification of future problems, including plugging. Data obtained in initial runs have already aided in the location of a defect in the roller support mechanism.

The water rotameter originally installed for the dissolver was not of sufficient capacity to supply the liquid flow rate that is currently required; therefore, a larger (4-gpm) rotameter and a pressure regulator have been added. The pressure regulator helps to maintain a constant flow. Modification of the bucket-elevator feed hopper has been made to aid in the collection of large quantities of feed materials. Also, the original bolt-down inspection ports on all three sections of the dissolver have been replaced by easily removable latch-on plates.

4.2.3 Operational testing of ferrofluidic seals

B. E. Lewis

The remote and chemically severe environment of a nuclear fuel reprocessing plant requires reliable and easily maintained sealing systems. We are beginning an investigation of ferrofluidic seals for this application. In a ferrofluidic seal, the gap between the stationary and rotating parts is filled with a ferrofluid, which consists of colloidal iron particles suspended in a liquid medium. The fluid is held in place by the use of permanent magnets built into the seal housing. Irradiation tests of the ferrofluid recommended for use by the manufacturer indicated no serious problems regarding fluid degradation. Therefore, for the purpose of continued evaluation of ferrofluidic seals, design and fabrication of a one-fourth-scale dissolver seal test stand were completed. We view this type of seal as a possible backup to the main concept of a purged housing for the rotary dissolver. The seal test stand, which has

been assembled on the ground floor of Building 7603, will be used to simulate the operating conditions in the rotary dissolver. The tests to be performed over the next 10 to 12 months will examine the sealing capability of the ferrofluidic seal when exposed to various pressures, temperatures, degrees of misalignment, and materials similar to those expected in the operational rotary dissolver. Support equipment for the experiments is currently being ordered and installed. Testing should begin in October 1976.

4.3 Feed Preparation

The aqueous feed discharged from the dissolver will contain solids (undissolved fission products, corrosion products, etc.) and therefore will require clarification prior to solvent extraction. Preparation of the feed for solvent extraction will also include adjustment of the plutonium valence and HNO_3 concentration, as well as treatment to remove iodine.

Iodine removal methods proposed to date include the use of $\text{NO}_2\text{-H}_2\text{O}_2$ or $\text{NO}_2\text{-O}_3$ gas mixtures as sparge streams in the feed preparation tanks. The presence of a large amount of NO_2 in vessel off-gas streams may have a deleterious effect on the performance of the Iodox process for iodine removal from gas streams. Efforts during this report period have been aimed at the development of a method for removing nitrogen oxides from the off-gas streams associated with feed preparation vessels.

4.3.1 Nitrogen oxide scrubbing

R. M. Counce

Engineering studies of a nitrogen oxide removal step using an absorption process have been initiated to provide the necessary technology for the design of larger-scale absorption equipment. During this report period, some modifications were made to the gas analysis system previously described² and experimental studies of NO_x absorption were begun. The purpose of this report is to briefly describe these modifications and present results of the initial experimental work.

The gas analysis system has been modified for improved accuracy and to allow for gas sampling and temperature measurements on a stagewise basis. A revised schematic flowsheet of the experiment is given in Fig. 4.3. The results of NO_x gas analyses provided by the "in-line" analyzer have compared closely with gas analyses provided by W. R. Laing of the Analytical Chemistry Division. The results of runs ER-9, -10, -11, -12, -15, -17, and -18 will be reported. The amount of time to reach steady state was explored in run ER-9; approximately 5.5 hr was required. The NO_x concentration profile in the sieve-plate column as steady state was being attained is shown in Fig. 4.4. The gas and liquid flows were steady after approximately 300 sec of run time, and liquid was recirculated (under ideal flow conditions) to provide a complete inventory change at intervals of approximately 600 sec. During the time required to reach steady state, a slight increase in the gas temperatures in the column was recorded.

The effects of the HNO_3 concentration of the scrubber liquid on the profiles of NO_x concentration and temperature in the sieve-plate column are shown in Figs. 4.5 and 4.6. The temperature profile for the column during a run with a feed gas composed of air and steam alone is also shown in Fig. 4.6. In general, varying the acid molarity in the indicated range had little effect on the NO_x removal efficiency of the system and the temperature behavior was not as extreme as expected. A sparge of the liquid holdup tank, which was described previously, appeared to improve the NO_x removal efficiency (see Fig. 4.7).

In general, the performance of the sieve-plate column as an NO_x removal device appears very satisfactory at this point in the experimental program.

Other variables to be studied are NO_x concentration in the gas feed stream, steam concentration in the feed gas stream, gas flow rates, and liquid flow rates.

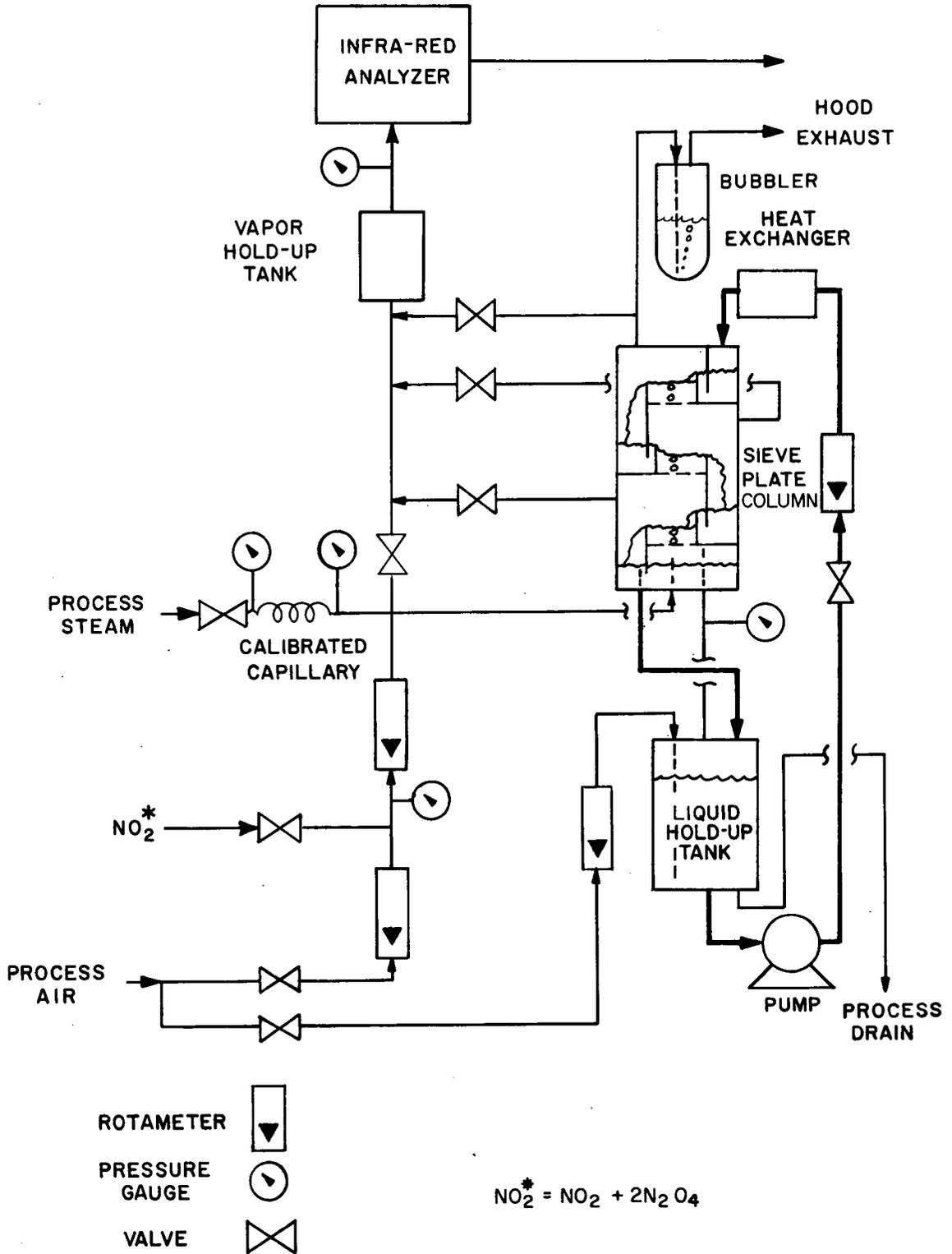


Fig. 4.3. Equipment used in the NO_x removal study.

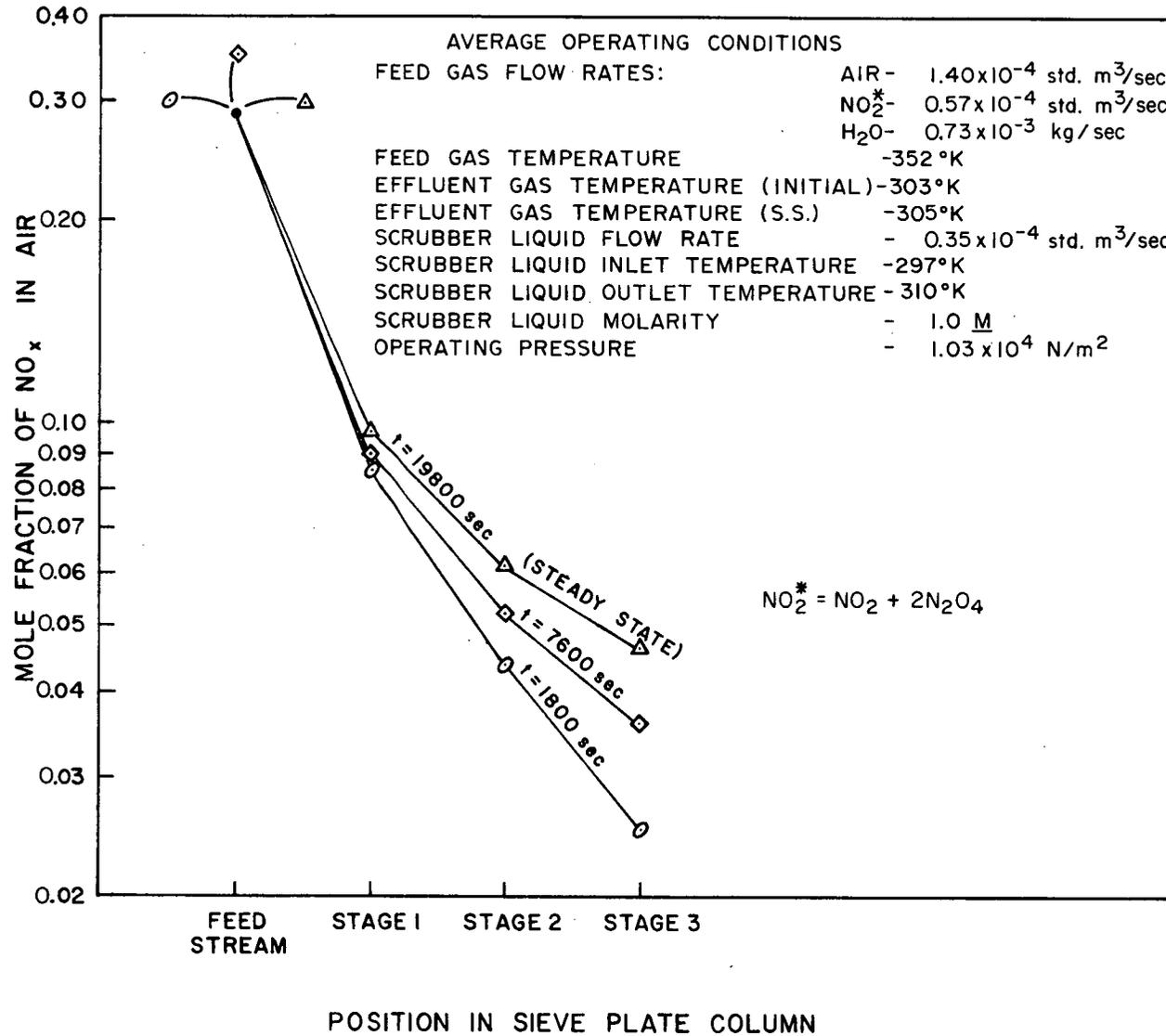


Fig. 4.4. Concentration profile obtained for ER-9 in sieve-plate column during approach to steady state in run ER-9.

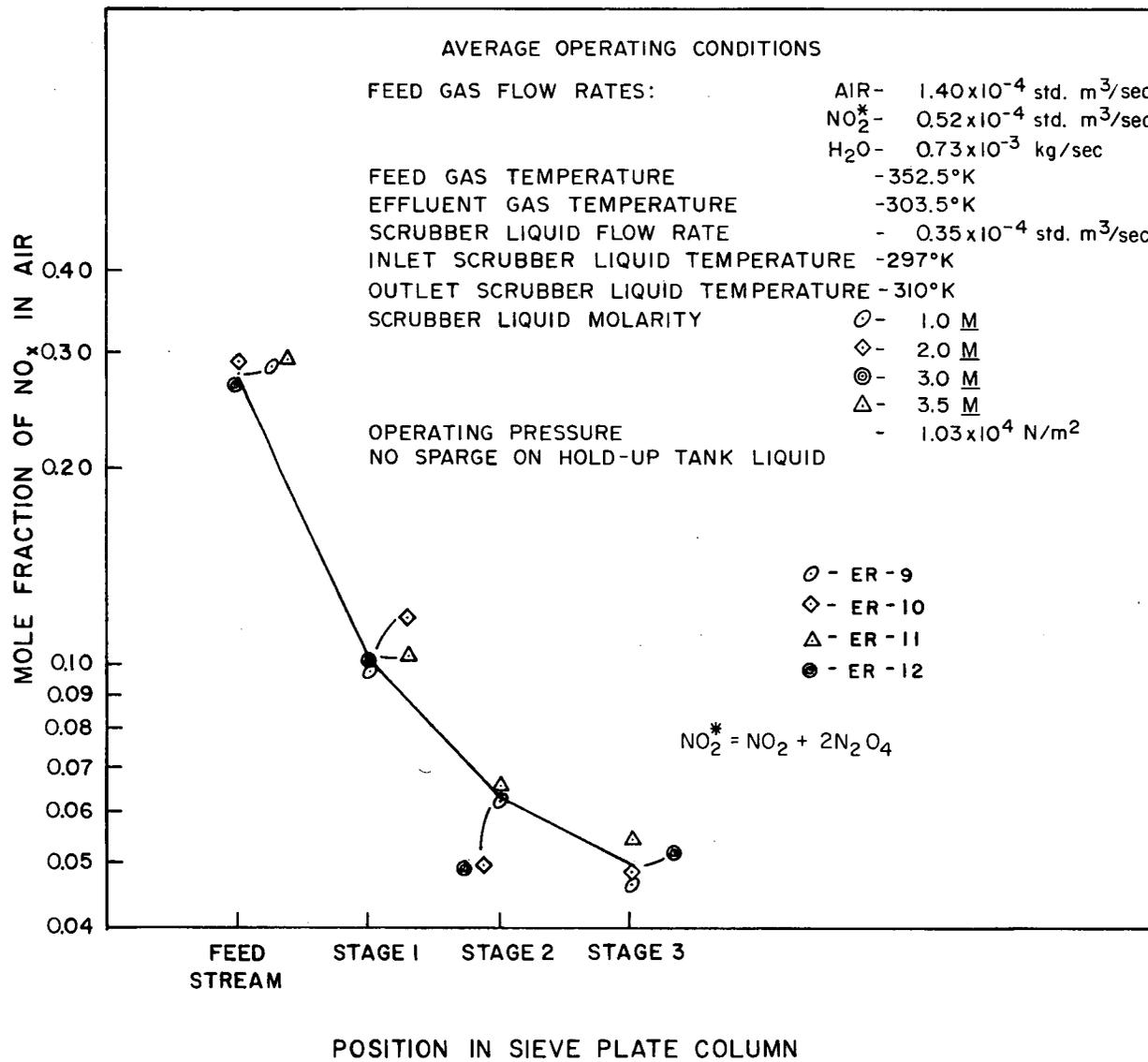


Fig. 4.5. Steady-state NO_x concentration profiles in sieve-plate column for scrubber liquid having HNO₃ concentrations of 1, 2, 3, and 3.5 M.

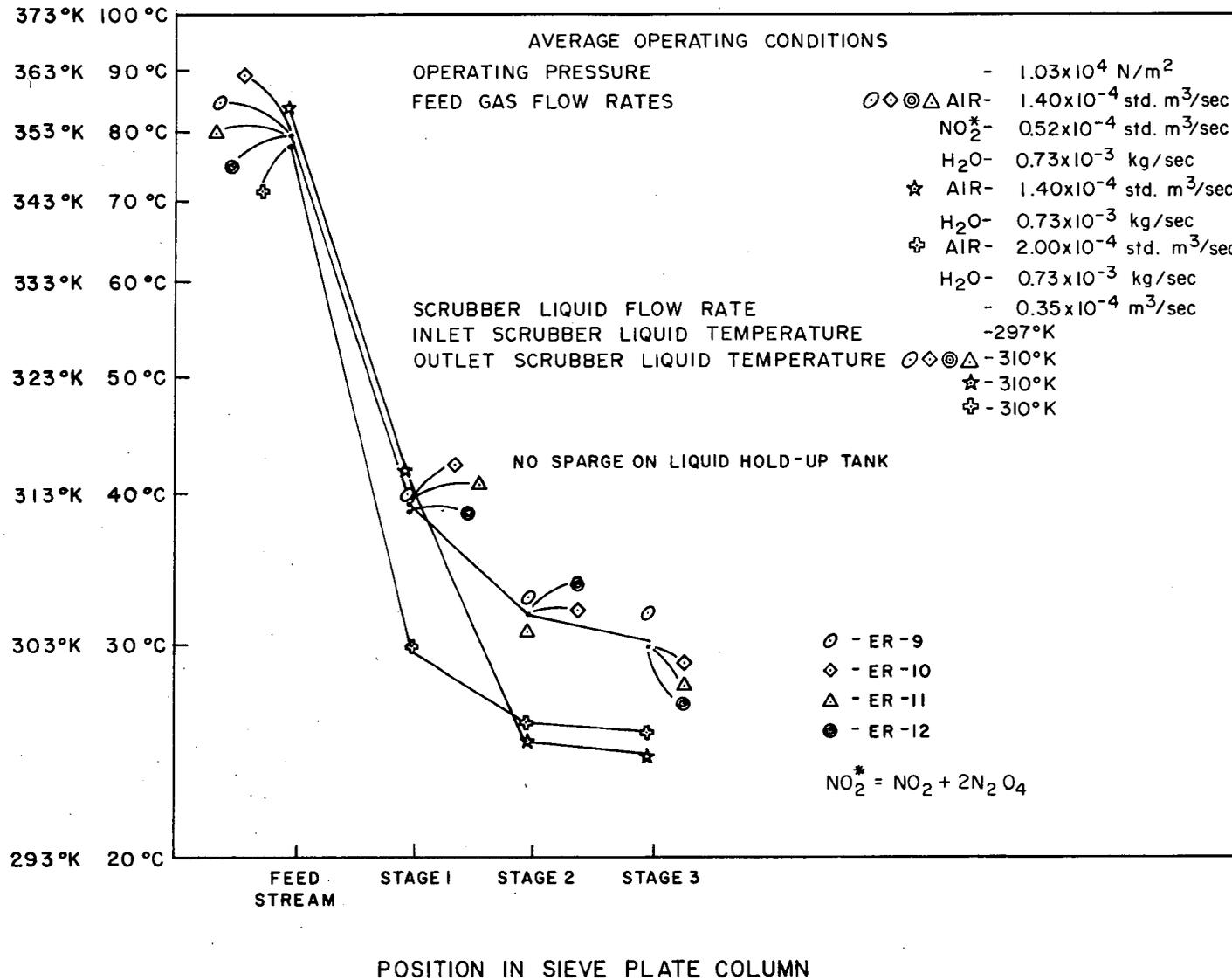


Fig. 4.6. Steady-state temperature profiles in sieve-plate column for scrubber liquid having HNO_3 concentrations of 1, 2, 3, and 3.5 M.

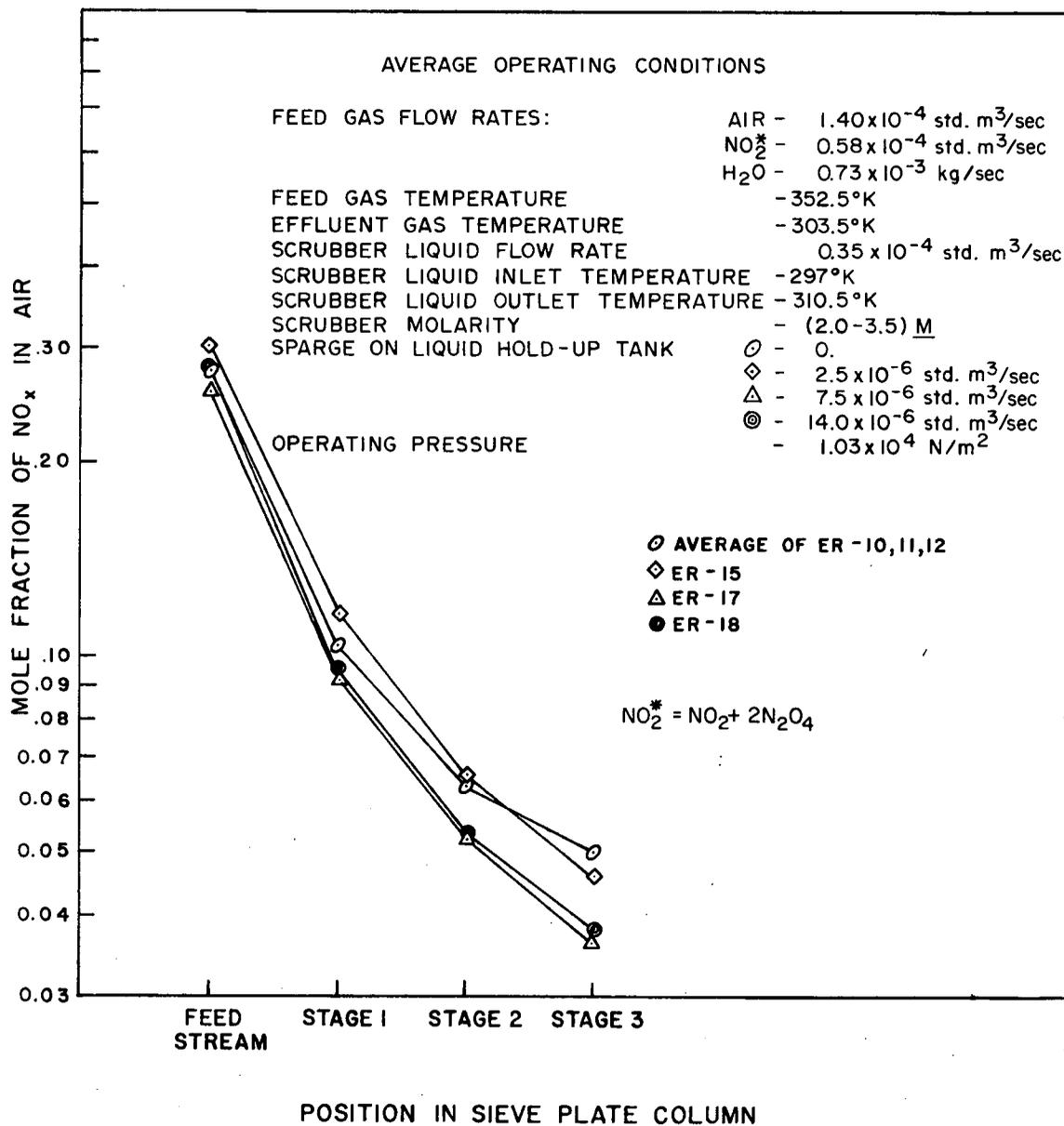


Fig. 4.7. Steady-state NO_x concentration profiles in sieve-plate column for different air sparge flow rates of the liquid holdup tank.

4.4 Fluorocarbon Absorption Studies*

M. J. Stephenson

This program is concerned with the development of the Krypton Absorption process on an accelerated basis. The program now includes expansion of the pilot-plant development, performance of a proposed system reliability analysis, and a study of the chemical effects of impurities in the fluorocarbon solvent on the process and equipment.

4.4.1 Pilot-plant operation

M. J. Stephenson, R. S. Eby, V. C. Huffstetler, and B. E. Kanak (ORGDP)

The remaining pilot-plant tests scheduled under campaign 3 have been completed. This effort was directed toward further defining the general process disposition of the various feed gas components, including carbon dioxide, nitrogen dioxide, iodine, methyl iodide, water, and xenon, and the effects of these components on the operability and overall performance of the process. Test results with nitrogen dioxide, iodine, and methyl iodide were reported previously.² Work with carbon dioxide and xenon is reported here. Pilot-plant tests with water have been rescheduled for the next experimental campaign because the necessary analytical equipment was not available during this operating period.

The results of the carbon dioxide and xenon removal tests are summarized in Fig. 4.8; krypton data are included for comparison. In these runs, the absorption step was conducted at a pressure of 300 psig and a temperature between -25 and 0°F. An infrared gas analyzer was used to establish carbon dioxide removals, while gamma scintillation equipment gave the corresponding xenon concentrations. Only tracer levels of activity (e.g., from 1 to 3 Ci of ^{133}Xe) were used during the xenon tests. Xenon and carbon dioxide absorber removals in excess of 99.99% were measured in pilot-plant tests conducted at the same absorber conditions of pressure, L/G ratio, and temperature that yielded about 99% krypton removal. Carbon dioxide removals were slightly higher than those measured for xenon, consistent with the component solubilities. Based on pilot-plant data, H_{OG} values for Xe and

*Jointly funded by IMFBR Reprocessing and LWR Fuel Reprocessing and Recycle programs.

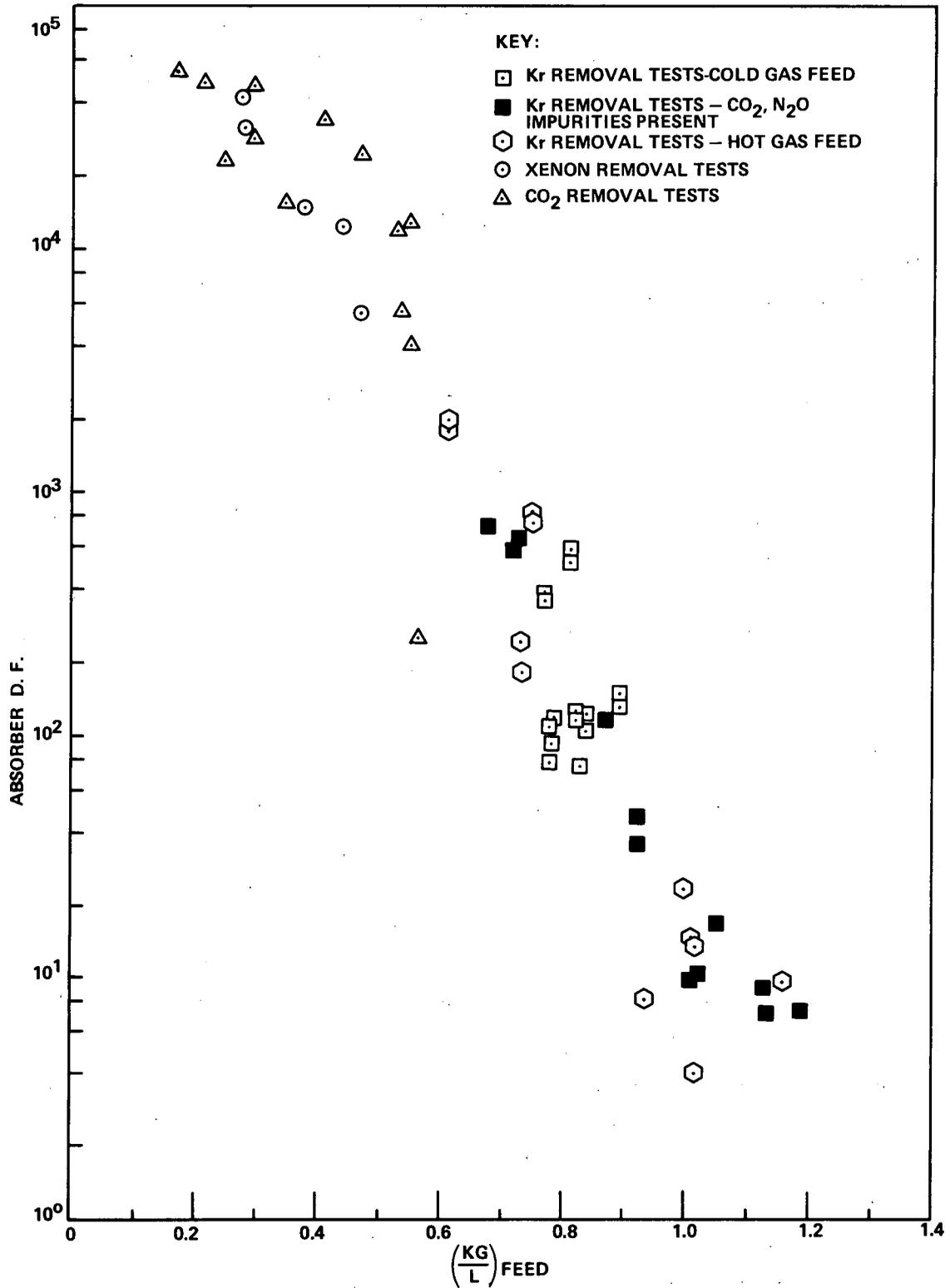


Fig. 4.8. Summary of recent pilot-plant tests with krypton, xenon, and carbon dioxide (9.0-ft absorber column, 300 psig, and $0^\circ F$).

CO₂ were 6 to 10 in. The final stripper column was operated at 15 psig with varying L/G ratios. Measurable amounts of carbon dioxide and xenon were found in the recycle solvent for those tests in which stripper L/G ratios were greater than 2.0; however, the absorber performance was only slightly affected by the recycle in those cases where the process removals exceeded 99.99%. At lower stripper boilup rates (e.g., even when the resulting L/G ratio was as high as 3.0), krypton was not detected in the recycle solvent. This observation is also consistent with component solubilities.

Based on the results of the third pilot-plant operating campaign and other considerations, several pilot-plant modifications were identified as being desirable to improve the efficiency and effectiveness of the process. Most of these flowsheet changes were made this quarter. Specifically, the packed height of the absorber column was increased from 9 ft to 15 ft, an absorber feed gas cooler/desublimer was built and installed in the absorber feed gas line, and solid-state reboiler heater controllers were installed on the fractionator, stripper, and solvent still reboilers. More contacting stages in the absorber will allow the use of lower solvent-to-gas flow ratios, which will improve the overall efficiency of the process.

The feed gas cooler/desublimer will permit evaluation of the effectiveness of cold trapping feed gas components such as nitrogen dioxide, water, and iodine. This heat exchanger is fitted with an external gamma scanner for determining iodine loading profiles and has provisions for washout and regeneration. The solid-state reboiler controllers give automatic heater control capability; manual controls were previously used. The boilup rate of the fractionator is controlled by a selected temperature point on the packed column, the stripper by the column ΔP , and the solvent still by the reboiler liquid level. The automatic heater controls will allow for better compensation of process transients. Digital output wattmeters capable of $\pm 1/4\%$ power measurements have been installed on all three reboilers to give a better account of system energy requirements. Repiping of the solvent still to allow for better reflux control is the only pilot-plant modification identified through the experimental program that remains to be accomplished. This work is in progress.

4.4.2 Process application

M. J. Stephenson, R. S. Eby, V. C. Huffstetler, and B. E. Kanak (ORGDP)

Process modeling studies are continuing. At present, individual process components are being examined in the light of present theory and available data. Heat exchanger calculations, summarized in Fig. 4.9, show that the feed gas cooler/desublimer will remove high concentrations of water and iodine. In this case, the cold trap pressure was taken as 200 psig and the effect of various operating temperatures investigated. At the nominal operating temperature of the absorber (i.e., -25 to 0°F), water and iodine removals are calculated to be between 99 and 99.9%. The inlet water and iodine concentrations were assumed to be 1.4 mole % and 69 ppm respectively. With a nitrogen dioxide inlet concentration of 0.69 mole %, only a small quantity of this component would be condensed. Higher operating pressures will decrease the equilibrium partial pressures of the individual feed gas components and hence increase their removal.

In view of the relatively low water solubility limit of the solvent at the temperature of the process and the possible corrosion problems that could develop if large amounts of free water are present, the function of the feed gas cooler/desublimer might prove necessary to ensure the overall plant operation and integrity by significantly reducing the amount of water that might otherwise contact the solvent. Also, the bulk amount of the iodine fed to the process would end up in the heat exchanger and thereby minimize the amount of iodine in the liquid circuit.

In moving toward the objective of identifying preferred process equipment for the demonstration plant, available gear- and turbine-type solvent pumps are being reviewed. The selection of a gear or turbine pump would eliminate inherent hydraulic problems associated with the popular liquid-end diaphragm metering chemical pump now used in the pilot plant; it would also significantly reduce process maintenance requirements. Because of head limitations, two or more of the gear or turbine pumps will be needed in series to achieve the absorption pressures required by the process. After the alternate pump has been selected, it will be installed in the pilot plant for evaluation. Also, since this type of pump has no

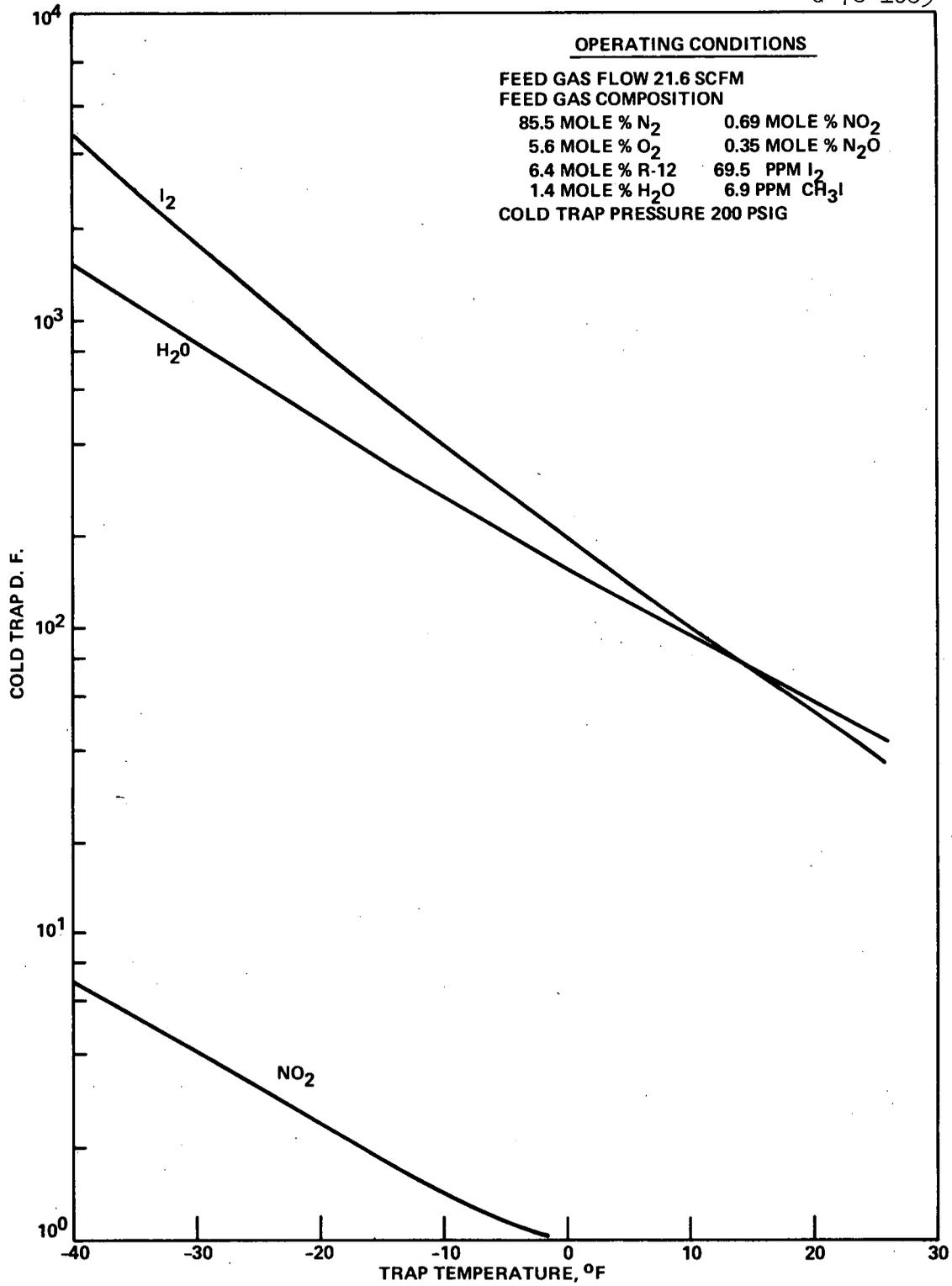


Fig. 4.9. Calculated cold trap removals of various feed gas components as a function of trap temperature.

built-in metering capability similar to that of the diaphragm unit, development of a suitable means of flow control will be required.

4.4.3 Chemical studies of contaminants in off-gas processing

L. M. Toth, D. W. Fuller, and J. T. Bell

The chemical research on off-gas components in R-12 refrigerant has been concerned with two major topics during this quarter: (1) the corrosion of the type 304 stainless steel vessel by R-12 solutions containing iodine and water, and (2) the general physical and chemical behavior of methyl iodide in the R-12 system.

The corrosion experiments were initiated during the previous reporting period. A first-order rate of I_2 loss at $0^\circ C$ was observed when 50 to 100 ppm of water was added to the R-12 solution, and the rate expression²

$$\frac{d(I_2)}{dt} = -k't \quad (1)$$

was proposed because of the limited solubility of water in R-12. At that time we suggested that it might therefore be possible to obtain useful fundamental data on type 304 stainless steel corrosion in the small-scale spectrophotometric system. We attempted to demonstrate that the rate of I_2 loss was independent of the amount of added water as long as the R-12 solution remained saturated with water, but found that inadequate mixing in our system produced a reaction mechanism which was apparently diffusion controlled. The work was temporarily postponed until a more sophisticated stirring mechanism was constructed. This system has recently been completed, and corrosion measurements will be resumed during the forthcoming months.

Attention was directed at the general physical and chemical behavior of methyl iodide in R-12. Methyl iodide was found to be miscible in R-12 up to 2.3 moles/liter. It forms stable solutions in R-12 and does not attack the type 304 stainless steel container. By monitoring the electronic absorption band of CH_3I at 256 nm and 257 nm (molar extinction coefficient = 230 ± 50 liter mole⁻¹ cm⁻¹) in the liquid and vapor phases, respectively, a mole fraction distribution coefficient, $D = x_l/x_v$, for the -40 to $+300^\circ C$

range was measured and was fit by least-squares to the expression

$$\log_{10}(D) = -1.1 + 476/T, \quad (2)$$

where T is temperature in K.

These data are preliminary and are thus subject to revision after our final measurements are complete. Because methyl iodide shows no solubility limits, no saturation data (as was given for I₂) are available.

Design and construction have begun on a simple exhaust system which will satisfy safety requirements for handling nitrogen dioxide in our spectrophotometric system. Experiments incorporating nitrogen dioxide in the R-12 system are expected to begin during the fall quarter upon completion of the installation of the safety equipment.

4.4.4 Reliability studies

W. E. Unger and D. E. Wood*

Kaman Sciences Corporation has completed the work covered under their subcontract (P. O. ORNL 11X 81721, Req. UCN-1127) with the issuance of their final report, Availability Analysis of the Selective Absorption Pilot Plant, KSC-6212-1. A portion of the summary of this report is provided below.

....The current study is an analysis of the reliability of the pilot plant, including modifications proposed for use in a future demonstration plant.

Availability was chosen as the measure of success, since the plant can remove most of the krypton even with occasional outages, as long as the outage time is short. This led to the extensive use of standby equipment and bypasses instead of full on-line redundancy....

Several conclusions can be drawn from the overall results.

1. The expected outage time of 15 hours will exceed the krypton removal requirement (90%) by a large enough factor so that more accurate failure data and/or additional redundancy should not be necessary.

*Senior Research Scientist, Nuclear Services Program, Kaman Sciences Corporation.

2. Occasional failures can be tolerated since short repair time is just as important as low failure rate in minimizing total outage time. Thus standby redundancy (with a short outage for standby startup) is adequate instead of on-line redundancy or automatic switching to eliminate the system failure.
3. Availability of standby units nearby for quick replacement or in-place (ready to start) is important to reduce outage time.
4. Standby units need not be as reliable as the primary unit since the reliability of the standby system depends on correct operator action.
5. The availability of a component and its bypass will be one or two orders of magnitude better than the component alone for the normal range of operator reliability of 0.9 to 0.99. Higher availability could be achieved with automatic switching or on-line redundancy, but this was not necessary in the current study.
6. Long outages, even though rare, should be avoided since the krypton retention limit could be exceeded during the particular year when the outage occurs. Thus complex equipment like the columns should have enough parts available for fabrication and installation in a few days, although a complete spare would not be necessary.
7. Access for repair is important to minimize outage time.
8. Items beyond the still output may need provision for purging and recycling the gas, isolation of sections to reduce the amount to be recycled, and access for contact maintenance, if possible.
9. Product storage should be in units small enough so that a failure would release substantially less than 10% of the year's production.

4.4.5 Engineering design criteria studies

(No report this quarter.)

4.5 Iodex Process

The removal of volatile fission products from process and effluent streams is an important part of IMFBR fuel reprocessing. Iodine retention factors approaching 10^7 are desired when processing 90-day-decayed fuel. The Iodex demonstration system incorporates iodine scrubbing, collection

of the solid pentavalent iodine, and acid recovery. Studies of the Iodox process, which utilizes hyperazeotropic HNO_3 to scrub volatile iodine compounds from air and to convert the iodine to the nonvolatile pentavalent form, were continued.

4.5.1 Demonstration of the Iodox process

B. A. Hannaford

Iodox demonstration experiments IXD-5 and IXD-6 were carried out to investigate the effects of nitrogen dioxide alone and nitrogen dioxide--water vapor, respectively, on the DF for methyl iodide. Measured DFs in the two experiments were similar; the water vapor in IXD-6 added to the deleterious effect of the nitrogen dioxide as a result of the decrease in acid concentration.

Iodox Demonstration Experiment, IXD-5. The previously reported experiment (IXD-4) to measure the deleterious effect of NO_2 was inconclusive because steady-state conditions were not attained. In IXD-5, the introduction of nitrogen dioxide to the feed gas was begun early in the experiment in order to maximize the steady-state period. Sampling and ^{131}I counting were continued over a 460-min period, including a near steady-state interval of about 90 min in the concluding portion of the run (Fig. 4.10). The increase in the ^{131}I gradient in the bubble-cap column coincided with a decrease in flow rate of scrub water from 11.1 ml/min to 7.9 ml/min, which occurred at about 230 min. This caused a significant increase in the acid concentration throughout the column.

The iodine DFs calculated for the final run interval are presented in Table 4.1. The deleterious effect of nitrogen dioxide on plates 1 and 2 was very similar to the results observed in run IXD-4. The overall column DF calculated from plate-to-plate values was in very good agreement with the DF calculated from gas samples alone.

The retention factor for ^{131}I in the iodine concentrator was ~ 230 , compared with a value of 1900 in an earlier buildown experiment in which no external source of nitrogen dioxide was present. Iodine present in the air purge flow from the concentrator condenser was collected on silver

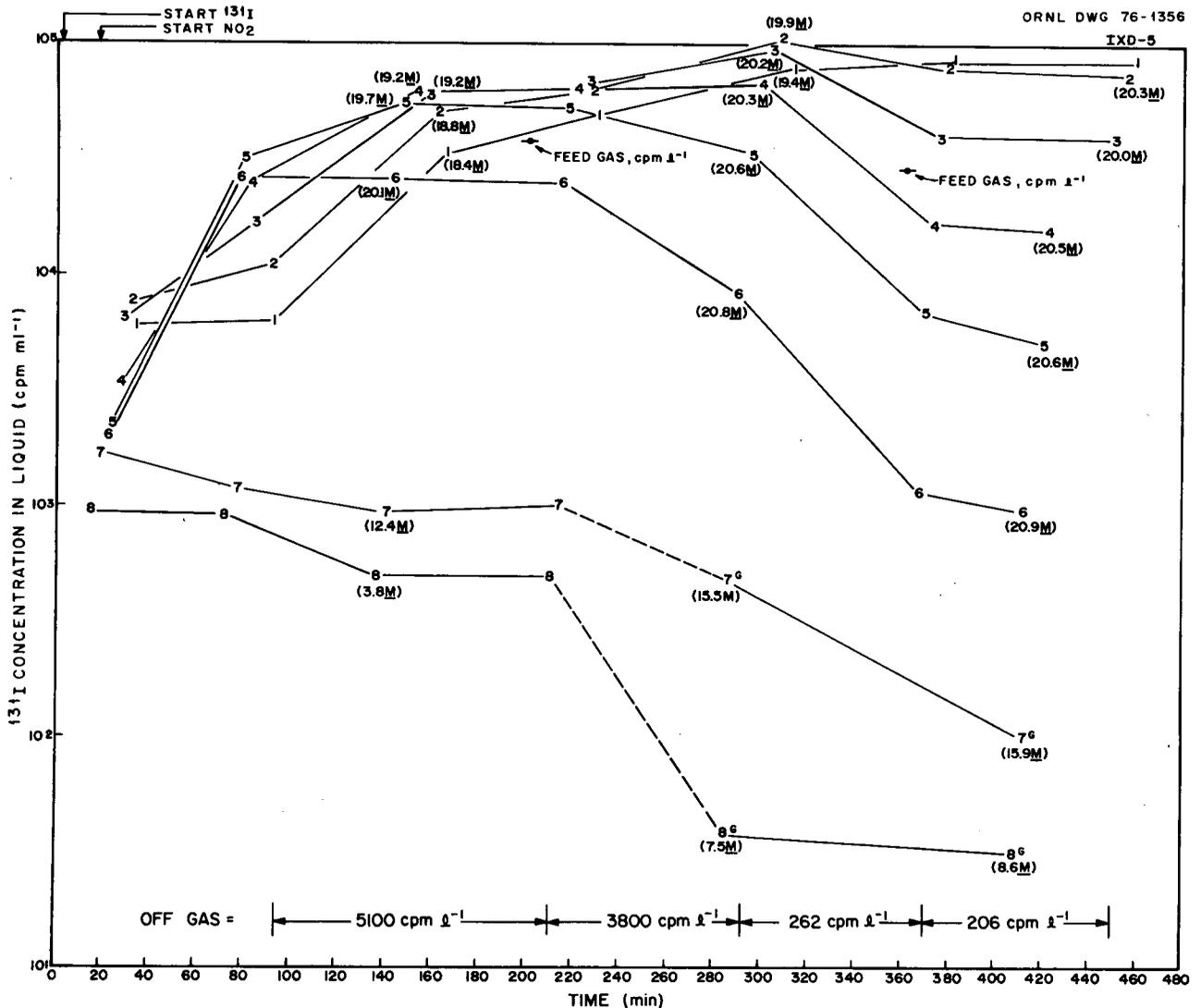


Fig. 4.10. Run IXD-5. Concentration of iodine in liquid and gas streams vs time for scrubbing ^{131}I -traced CH_3I from air--2% NO_2 with fuming nitric acid. [Feed rate of air--2% NO_2 = 95.6 std liters/min (1.59×10^{-3} m^3/sec); feed rate of 21.6 M HNO_3 to plate 6 = 35 ml/min (5.8×10^{-7} m^3/sec); feed rate of water scrub to plate 8 = 7.9 ml/min (1.34×10^{-7} m^3/sec). Methyl iodide concentration in dry feed gas = 0.000205 g I/liter. Nitric acid concentrations are given in parentheses. Superscript G's designate samples that were counted in glass for maximum accuracy at low ^{131}I levels.]

Table 4.1. Run IXD-5: DFs^a for ¹³¹I-traced
CH₃I in air--2% NO₂

Plate number	Iodine DF
1	1.04
2	1.91
3	2.43
4	2.30
5	4.51
6	2.99
Average ^b	2.30
Column, overall	150.
Column, overall ^c	164.

^a At a run time of 400 min.

^b Average = $[(DF)_1 \times (DF)_2 \times \dots \times (DF)_6]^{1/6}$.

^c Based on ¹³¹I concentrations and flow rates of gas streams only, taking the average of two feed gas samples (33,740 counts min⁻¹ liter⁻¹) and the final off-gas sample (206 counts min⁻¹ liter⁻¹).

zeolite traps to show that about 1.7×10^{-4} fraction of the iodine feed was escaping by this route during the final 150 min of the experiment. This compares with a value of 10^{-7} fraction during experiment IXD-2, in which no NO_2 was fed to the system.

Iodox Demonstration Experiment, IXD-6. The purpose of this experiment was to measure the combined effect of nitrogen dioxide and water vapor in the feed gas containing methyl iodide. Preparations for the experiment included calibration of the humidifier to supply up to 2.8 g of water vapor per minute and revision of the acid sampling and counting system for more efficient flushing.

Counting results for run IXD-6 show reasonably constant ^{131}I concentrations throughout most of the run for the upper plates (Fig. 4.11). The unusually low ^{131}I concentration on plate 1 resulted from the low acid concentration caused by the incoming water vapor. The improving performance of the column in the latter part of the run was probably a result of the observed increase in HNO_3 concentration of the recycle acid from the extractive distillation system.

Table 4.2 summarizes the results of the experiment after 520 min of run time. The very low DFs observed are probably the consequence of dilution of the acid on all plates by an excessively high water scrub rate, and of the acid on the bottom plate by the water vapor in the feed gas. The significance of the DF of < 1 for plate 1 is that iodine was refluxing between plates 1 and 2 as the solution from plate 2 continuously entered plate 1, where the equilibrium was less favorable due to the lower acid concentration. The DF values for these plates as calculated by Mailen³ were in good agreement with the observed values.

The retention factor for iodine in the iodine concentrator was 200, substantially the same as that obtained for run IXD-5. Iodine leaving the concentrator condenser with the air purge amounted to only about 3.5×10^{-6} fraction of the iodine fed during the final 60 min of the run.

Acid recycle by extractive distillation. Operation of the extractive distillation system following the column revision that was reported earlier was generally satisfactory in both runs. Iodine in the contaminated acid

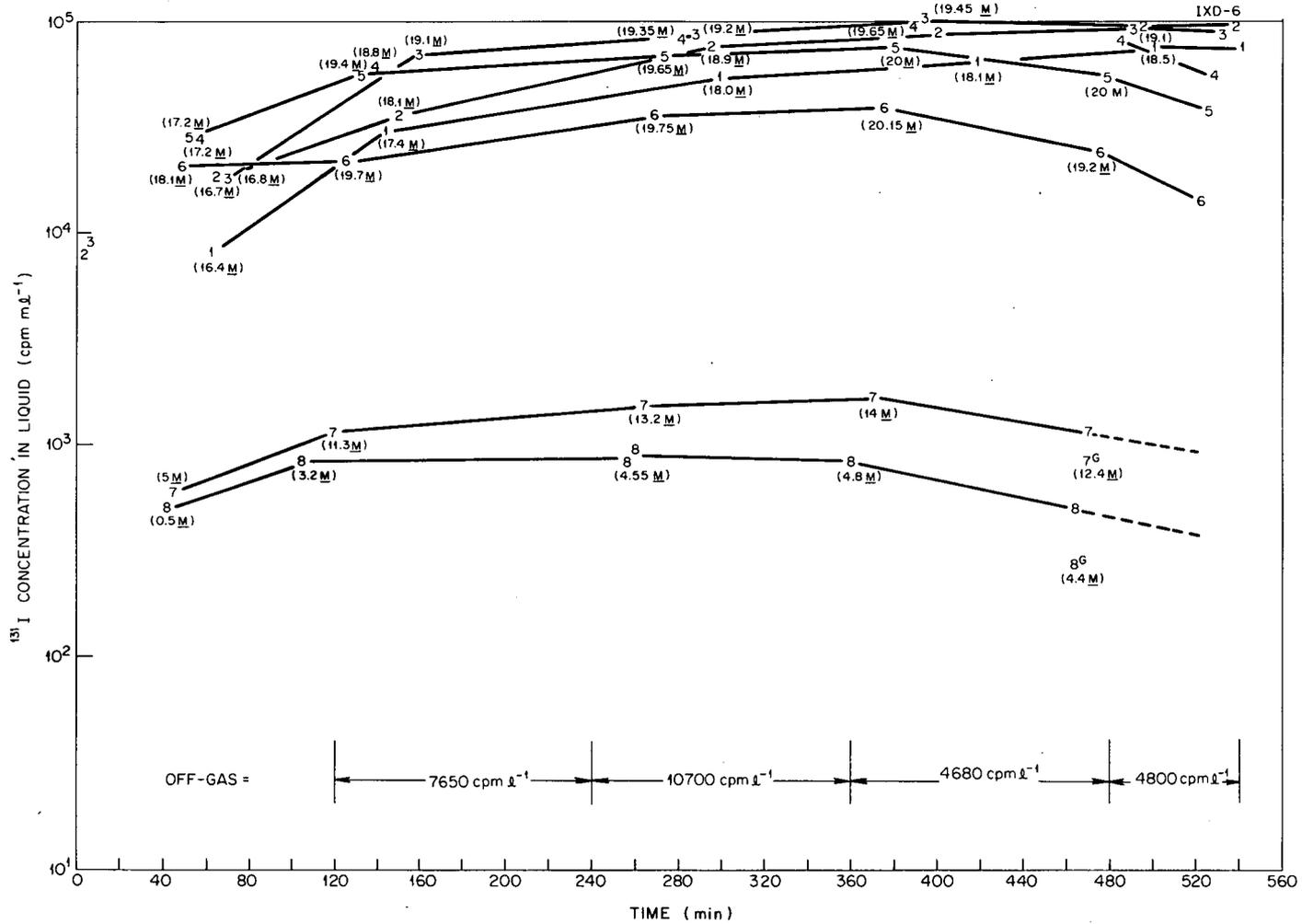


Fig. 4.11. Run IXD-6. Concentration of iodine in liquid and gas streams vs time for scrubbing ¹³¹I-traced methyl iodide from air--2% NO₂--2% H₂O with fuming nitric acid [Feed rate of gas mixture = 100 std liters/min (1.66 x 10⁻³ m³/sec), feed rate of 21.9 M HNO₃ = 40 ml/min (6.63 x 10⁻⁷ m³/sec); feed rate of water = 11.3 ml/min (1.97 x 10⁻⁷ m³/sec). Methyl iodide concentration in feed gas = 0.000237 g I/liter. Nitric acid concentrations are given in parentheses.]

Table 4.2. Run IXD-6: DFs^a for ¹³¹I-traced
 CH₃I in air--2% NO₂--2% H₂O

Plate number	DF
1	0.8
2	1.05
3	1.46
4	1.39
5	2.00
6	2.42
Average ^b	1.42
Column, overall	8.3
Column, overall ^c	11.

^aAt a run time of 520 min.

^bAverage = $[(DF)_1 \times (DF)_2 \times \dots \times (DF)_6]^{1/6}$.

^cBased on gas flow rate, ¹³¹I concentration in the off-gas (measured), and ¹³¹I concentration in the feed gas (calculated).

feed to the extractive distillation system was largely retained within the salt phase; the observed accumulation rate of about $0.36 \text{ cpm g}^{-1} \text{ min}^{-1}$ was in very good agreement with the material balance estimate. Roughly 2 to 3% of the iodine in the feed reported to the acid product, and a similar fraction to the raffinate. Thus greater than 90% of the iodine accumulated in the salt phase in these short runs. Continued accumulation in longer runs will be measured as part of planned future long-term operations. Presumably, a small bleed of salt solution may be necessary to limit iodine buildup if breakthrough to acid product or raffinate streams does not occur. The effect of iodine on salt solution stability is an area identified for future laboratory investigation.

Work planned for the immediate future would have involved a change from methyl iodide to I_2 as the contaminant. However, an additional experiment with methyl iodide will first be carried out in which the HNO_3 concentration will be increased throughout the column by reducing the flow of water to the acid vapor scrubbing section and by increasing the feed rate of HNO_3 to the column.

4.6 Instrumentation and Control Systems

N. C. Bradley

The overall objectives of this task are to develop instrument systems and engineering techniques for the fuel reprocessing program and to provide a basic instrumentation and control technology for application to commercial plants. In addition, instrumentation and control systems are required in support of laboratory experiments and highly specialized tests associated with the components and process development tasks in the LMFBR Fuel Reprocessing Program.

4.6.1 Nondestructive assay

G. L. Ragan, R. S. Booth, T. J. Burns, J. D. Jenkins, F. R. Mynatt,
J. E. Rushton, C. R. Weisbin, and L. R. Williams

Work in this subtask continues to be concentrated on the development of a system for the measurement of the fissile content of incoming spent

fuel elements by nondestructive assay techniques. Activity during this quarter has been primarily in the areas of detector evaluation, system design and optimization, and planning for the forthcoming proof-of-principle experiments.

A small neutron source is now available for use in the evaluation of detectors, especially to establish a neutron energy scale. It consists of an assembly that uses a gamma-ray source consisting of 2 mCi of ^{232}U to irradiate a D_2O converter to produce 0.20-MeV neutrons, or a beryllium converter to produce 0.85-MeV neutrons. This source will be used to evaluate two types of detector (methane-filled and ^3He -filled) at several filling pressures.

Calculations have continued in support of system design and optimization, involving variations of the basic design reported earlier.⁴ We have tried using a 5-cm-thick tantalum filter (between fuel and the D_2O or beryllium converter) in place of the original composite filter composed of 4 cm of lead plus 1 cm of $^{10}\text{B}_4\text{C}$. The tantalum filter is almost as good as the composite in absorbing low-energy (poorly penetrating) interrogating neutrons, and is more efficient in transmitting fission-signal neutrons from fuel to detectors and in attenuating gamma rays from the spent fuel.

Initial estimates of neutron backgrounds resulting from fuel gamma emissions irradiating a D_2O or beryllium converter indicated that D_2O was acceptable, but that the lower photon energy threshold of the beryllium (γ, n) reaction led to an unacceptably high ratio of fuel-gamma-induced neutron background to the unavoidable neutron background directly emitted by the fuel. The revised ORIGEN calculations reported last quarter² reduced this ratio by a factor of 9 (2 for lower fuel gammas and 4.5 for higher fuel neutrons). These factors, combined with the additional tantalum absorption (another factor of about 2) discussed above, may make the use of a beryllium converter acceptable. Calculations to evaluate the tantalum-plus-beryllium combination are being made, and preliminary results are encouraging.

If a beryllium converter can be used, a lower gamma photon energy will be required; therefore, additional isotopic gamma sources

(especially ^{124}Sb) are being considered. Antimony-124 has frequently been used along with beryllium in other NDA systems since it is easily produced and relatively inexpensive. On the other hand, we have been finding that the preferred isotopes (^{228}Ra , ^{228}Th , and ^{232}U) for use with D_2O are not readily available and are quite expensive.

A commercial x-ray machine is being considered as an alternative photon source for use with either a D_2O or a beryllium converter. Preliminary calculations with a D_2O converter indicate that a pulsed 4-MeV unit, which is commercially available and is being widely used today by industries and hospitals, can produce a fission neutron signal during the pulse that is about 25 times the neutron background from the fuel. This ratio is far more desirable than that (about unity) which we can hope to attain with an isotopic source of reasonable size. The rapid alternating measurement of signal plus background (during the pulse) and background only (between pulses) should further improve the accuracy and speed of the measurements. We are making further comparisons of the relative merits of x-ray and isotopic sources, including their reliability, economy, and convenience of operation.

Plans for the forthcoming proof-of-principle experiments are becoming better defined. The TURF facility seems to be well suited for the experiments; apparently, sufficient space for them will be available there. A test sample for the demonstration experiments which simulates an IMFBR blanket subassembly may be made of pins loaded with low-enrichment UO_2 pellets, such as those used in operating light water reactors. Provision will be made for including a few pins at selected positions that have a different enrichment or contain plutonium; these will be moved around to check spatial sensitivity factors. Other experimental procedures will be as outlined in the last quarterly report.² Provision will be made, if feasible, to make tests of performance under conditions using (or simulating the use of) an x-ray machine as the photon source.

4.6.2 Process sensors and controls

N. C. Bradley, J. M. Jansen, Jr., W. F. Johnson, R. M. Tate, D. N. Fry, G. N. Miller, M. V. Mathis, and C. M. Smith

Programmable logic controller control development. Development activities for the use of a programmable logic controller (PLC) has resulted in successful applications at the Iodex demonstration facility and the 4-ft-diam dissolver test facility. A third application is planned for control of the dissolver seal test demonstration, which is similar to control of the 4-ft dissolver. A fourth application of a PLC is for control of the fuel assembly shear, compactor, and fuel feed mechanism. The latter application, which is the most involved to date, contains the requirements for the system to be operated in automatic, semiautomatic, and manual modes. The PLC selected for the task will be of a maximum configuration for inputs, outputs, and memory capacity. Instrument flowsheets and operational state diagrams have been designed. The next step will be to produce the logic ladder diagrams for programming of the controller's memory.

Large control programs require significant portions of time to develop; therefore, efforts are currently under way to prepare a computer program that can easily and quickly produce a logic program for the PLC using statement language from the operational state diagrams. Hardware that has recently been purchased will allow communications from the programming computer directly to the PLC's memory for simple, rapid loading. The development of this capability will further reduce the costs of implementing this type of logic control.

Computer for shear development. The PDP-11T40 computer system, which has been temporarily installed in the Instrumentation and Controls Building, is continuing to be checked out. Some adjustments have been required for the tape recorders. Specifications have been completed for purchase of the display terminal, line printer, and 32 K additional core memory. Specifications for the digital input and output systems and the analog input system are nearing completion now that data acquisition requirements for the fuel assembly shear have been finalized.

Fluidic instrumentation. The application of a fluidic gaging system for determining the condition of the shear blade appears very promising. Approval was granted to procure a system proposed by General Electric's Fluidic Specialty Section; however, contact with this group has revealed that they are no longer in business. Consequently, efforts have been started to locate another company knowledgeable in fluidic systems.

Data acquisition systems. The computer-based data acquisition and analysis system being developed for the fuel assembly shear will enable large quantities of data to be taken and analyzed in real time; in addition, they will be readily retrievable for later analysis. This concept of data acquisition is also required for the IPET facility so that large quantities of data can be produced quickly and become readily available for use in the design of the HPP.

A conceptual design and cost estimate has been produced for an IPET data acquisition system.

Vibration analysis instrumentation. Vibration measurement and analysis has several possible applications to mechanical operating equipment. One possible application presently under study is a plugging detection system for the rotary dissolver. The first method of frequency measurement is one using four sensitive accelerometers attached to the mounting bolts at each of the four rotating drum bearing supports (Fig. 4.12). Hydrophones were also placed at two locations to provide sensors with measurement capabilities for frequencies up to 300 kHz.

The acoustic emissions from the dissolver were first measured and tape recorded to determine the background levels for the drum rotating empty, with and without the transport elevator and other equipment in operation. During this set of measurements, pairs of unusual noise bursts were recorded, one leading the other by 0.25 to 0.5 sec and equivalent to 0.9 to 3.6 g's. Figure 4.12 also shows the time traces from the four accelerometers during a single burst of noise. The recurrence frequency of the pulse pairs agreed with the rotational speed of the four drum support rollers. This type of vibration pulse indicated the possibility of support bearing problems; thus a recommendation was made that this be

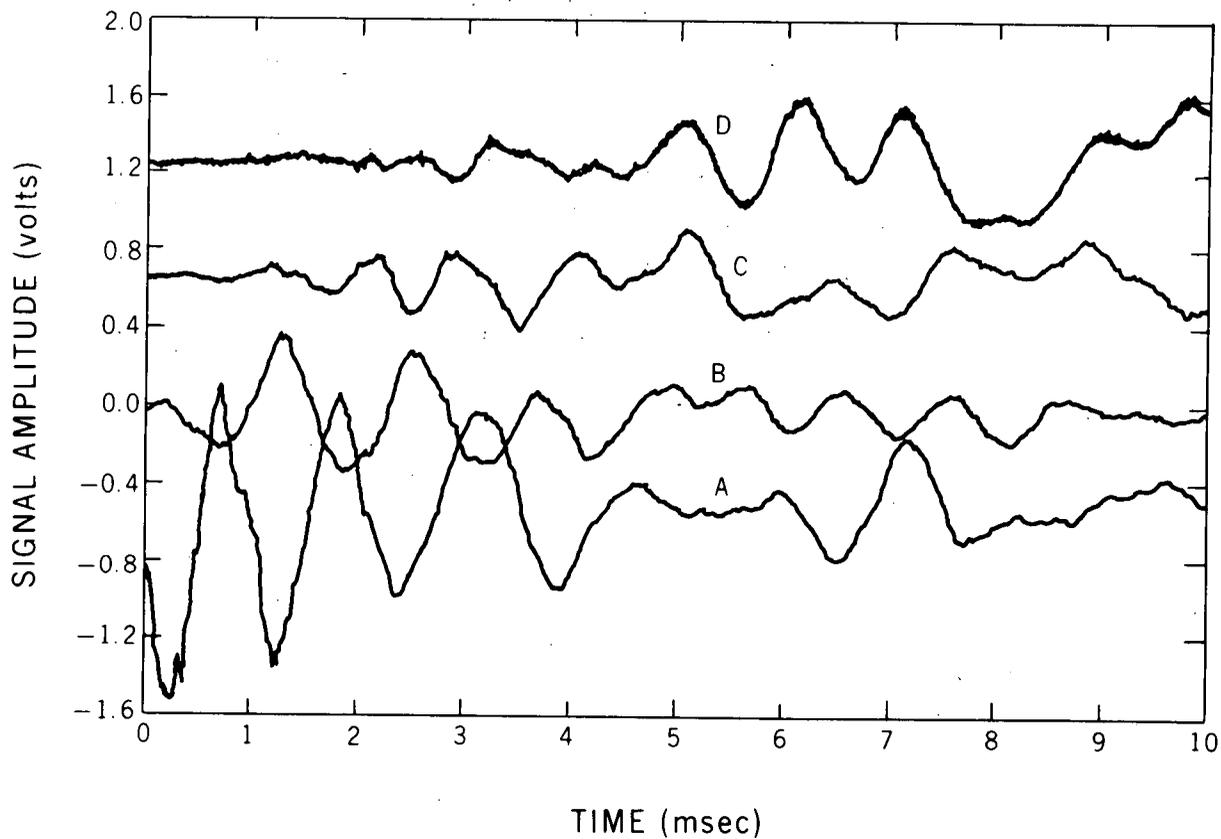
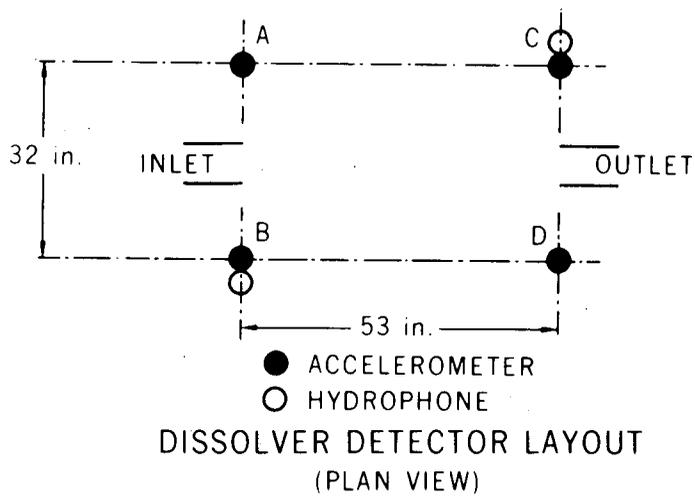


Fig. 4.12. Detector location and accelerometer time traces obtained in preliminary test of instrumentation for the LMFBFR-FRP dissolver.

investigated. The dissolver was shut down and dismantled. On inspection, the drum support rollers were found to be deformed; in addition, there were indications of difficulties with the bearings. The investigation of the problem is continuing.

The next vibration measurements will be made after completion of repairs to the dissolver to permit determination of background levels. Following this, a series of measurements will be made with various loadings of simulated chopped fuel, beginning with small amounts and gradually increasing to an amount that will cause plugging.

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3. J. C. Mailen, ORNL, personal communication, Sept. 12, 1976.
4. W. D. Burch et al., IMFBR Fuel Recycle Program Progress Report for Period July 1 to December 31, 1975, ORNL/TM-5477 (August 1976).

5. COMPONENT TESTING

N. R. Grant

5.1 Disassembly and Fuel Cutting - Contracted Work

N. R. Grant, C. D. Watson, and B. S. Weil

This portion of component development is being performed by the Gulf + Western Advanced Development and Engineering Center. The work is divided into four tasks: machining (rotary shearing), disassembly, shearing, and subassembly cutting.

5.1.1 Machining (rotary shearing)

Gulf + Western (G+W) has designed a test machine that will cut fuel with an eight-cutter mandrel at slow speed (10 rpm). Since the action produced is more typically a shearing than a machining action, it has been designated as a rotary shearing subtask. All of the necessary equipment and components have been ordered and/or received. These items will be assembled and tests conducted during the next quarter.

5.1.2 Disassembly

ORNL has submitted a design specification to G+W for the first (push-type) disassembly machine. G+W has been preparing the preliminary design for this prototype unit. Several design review meetings have been held, and at the end of this quarter the preliminary design is nearly complete. The detailed design is scheduled to be complete by the end of the next quarter. The major part of the FY 1977 contract with G+W will be directed to completion of this machine. The plan is to have the machine operating at ORNL by the end of the next fiscal year.

5.1.3 Shearing

G+W completed fabrication of "pinking" blades and components to allow testing on a leased 45-hp alligator shear. Two sets of tests were conducted during this quarter. The equipment added to the shear is shown

in Fig. 5.1. Note that the feed is introduced at an angle of 45° in order to cut the shroud into rectangular pieces. The subassembly used as feed is preflattened to simulate the operation of the gag. After several cuts, the subassembly has the appearance shown in Fig. 5.2. Product from the first test (Fig. 5.3) was mostly unsatisfactory; most cuts were not successful in breaking the shroud into distinct pieces. In addition, some long pieces of "fuel" were produced, indicating that the gag was not holding the upper rows of elements in place and thereby allowing them to pull out during shearing. Also, "fuel" pieces were flattened on the ends.

For the second test, a clevis block and swivel were added to the gag to allow the mechanism to hold the subassembly more tightly (Fig. 5.4). Although the product still did not look as good as it should, improvement was evident (Fig. 5.5). A hydraulic cylinder was added to the gag to provide additional holddown, and the final few cuts were made (Fig. 5.6). This improved cutting of the shroud but caused "fuel" pieces to be trapped in the corner of the shroud.

On an overall basis, ORNL has reevaluated alligator shears and has determined that more tests are needed before proceeding with the design and fabrication of a \$500,000 prototype unit. Since the present plan of leasing time on existing commercial shears does not allow the freedom for a testing program, ORNL will purchase a commercial unit. This shear will be modified as necessary to provide the test program information. In addition, new blade designs will be investigated.

5.2 Disassembly and Fuel Cutting - ORNL Program

C. D. Watson and B. S. Weil

Experiments during this report period have been limited principally to the G+W disassembly test rig. The installation and testing of new shear instrumentation on the ORNL straight-sided shear and the verification of shear dimensions for new equipment being fabricated have prevented significant efforts relative to shearing. Several test cuts have been completed to check out the machine and to provide sheared product for other experiments.

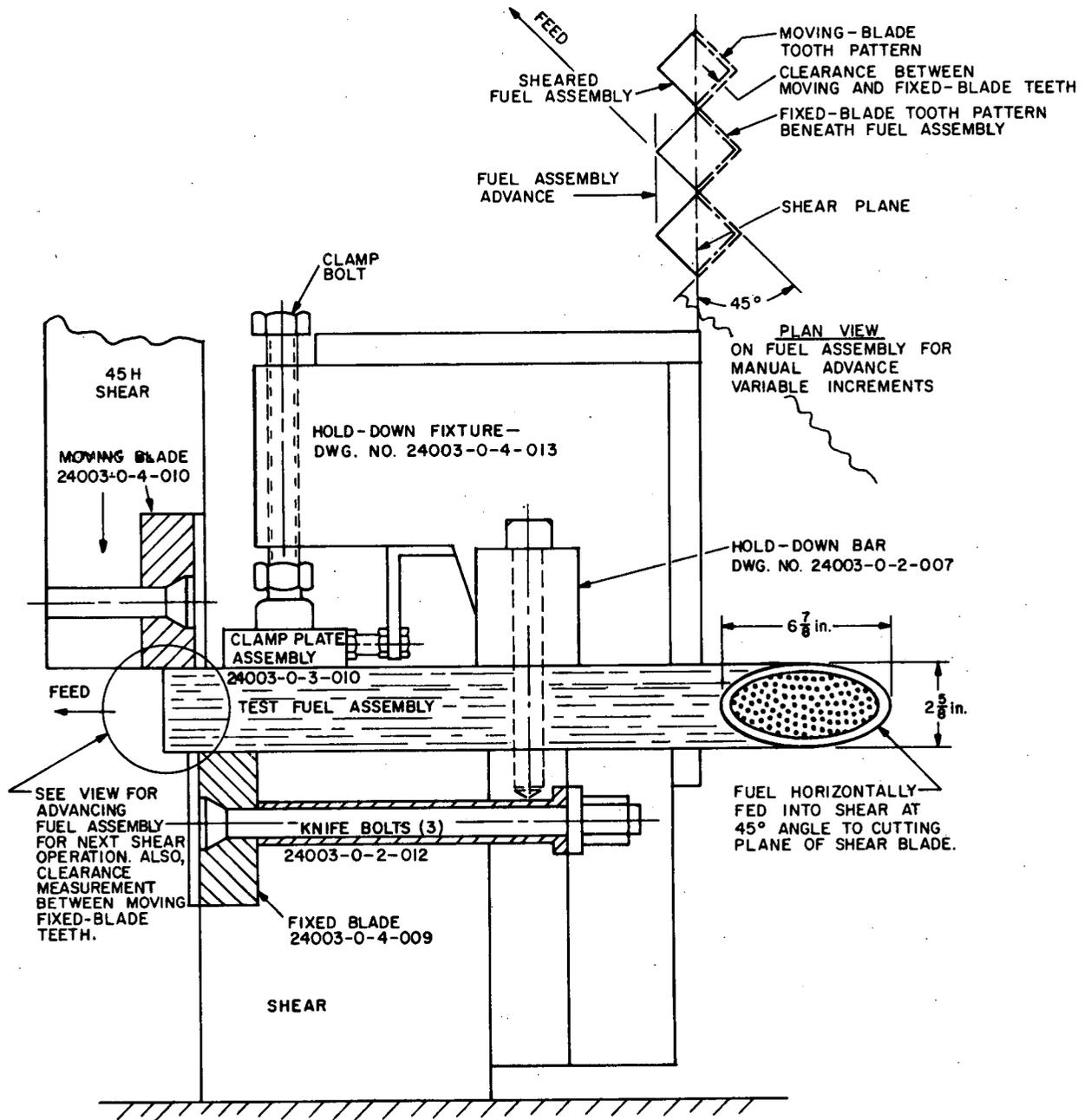


Fig. 5.1. Test assembly - side elevation.

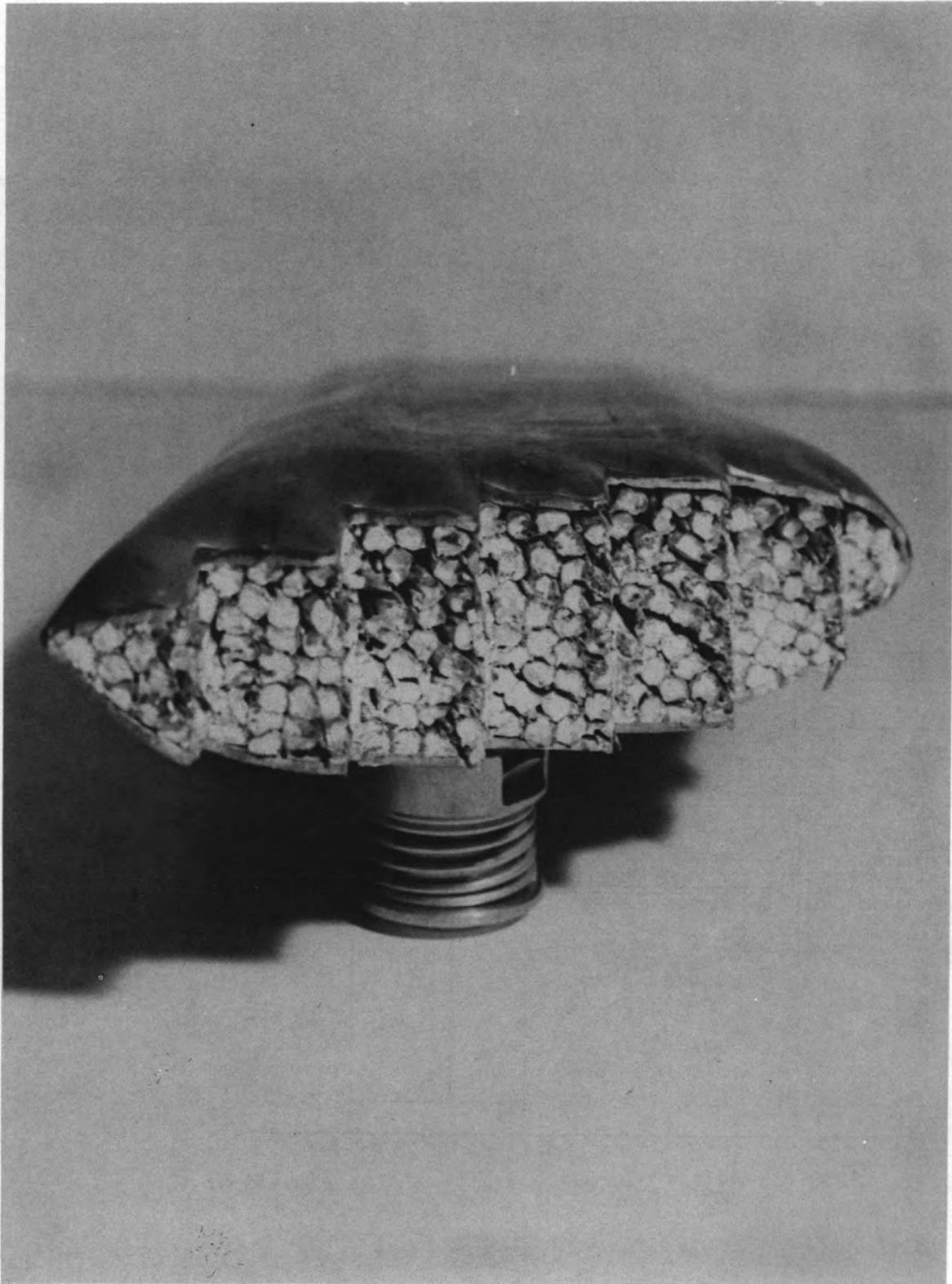


Fig. 5.2. End view of sheared fuel assembly.

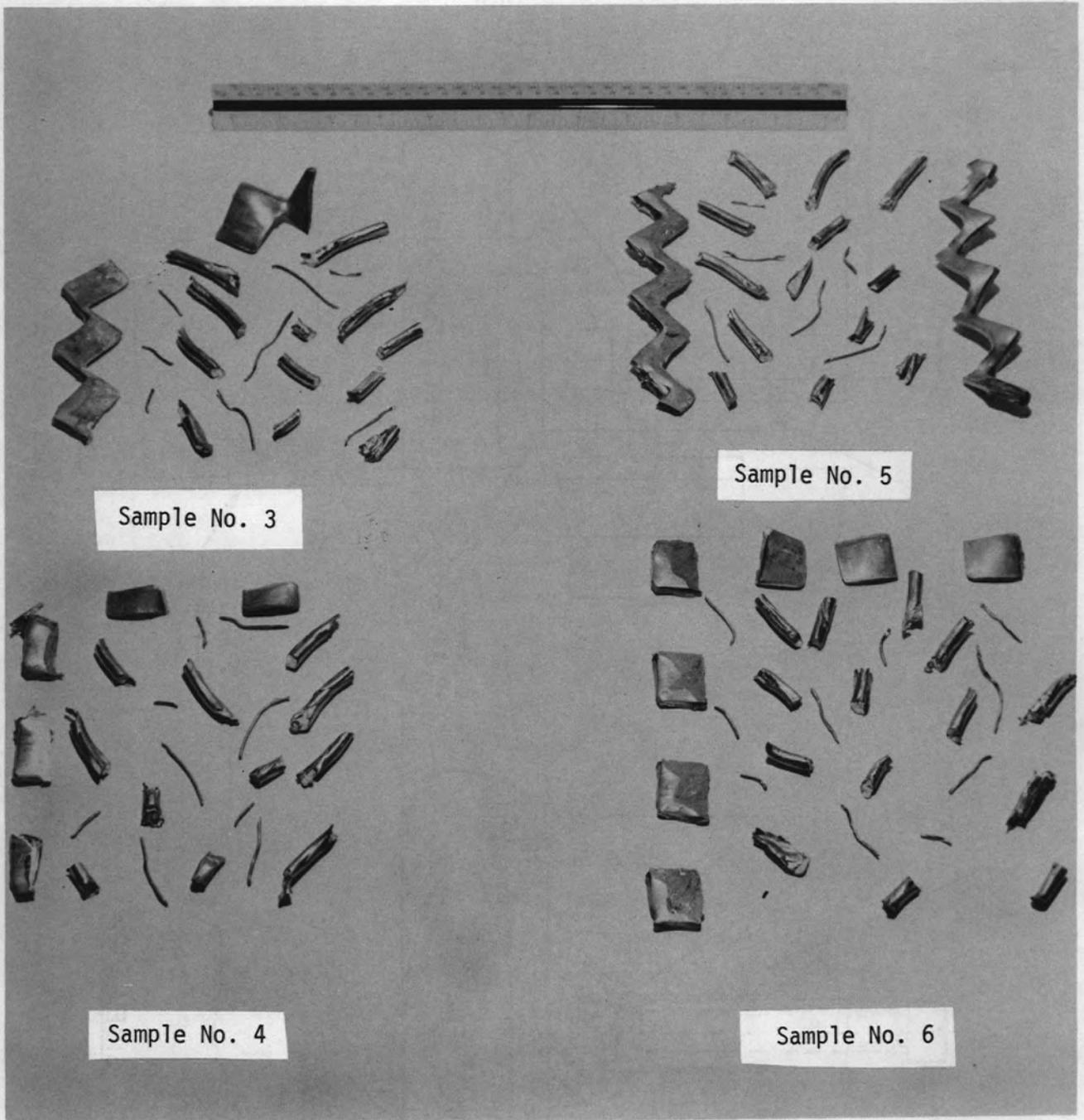
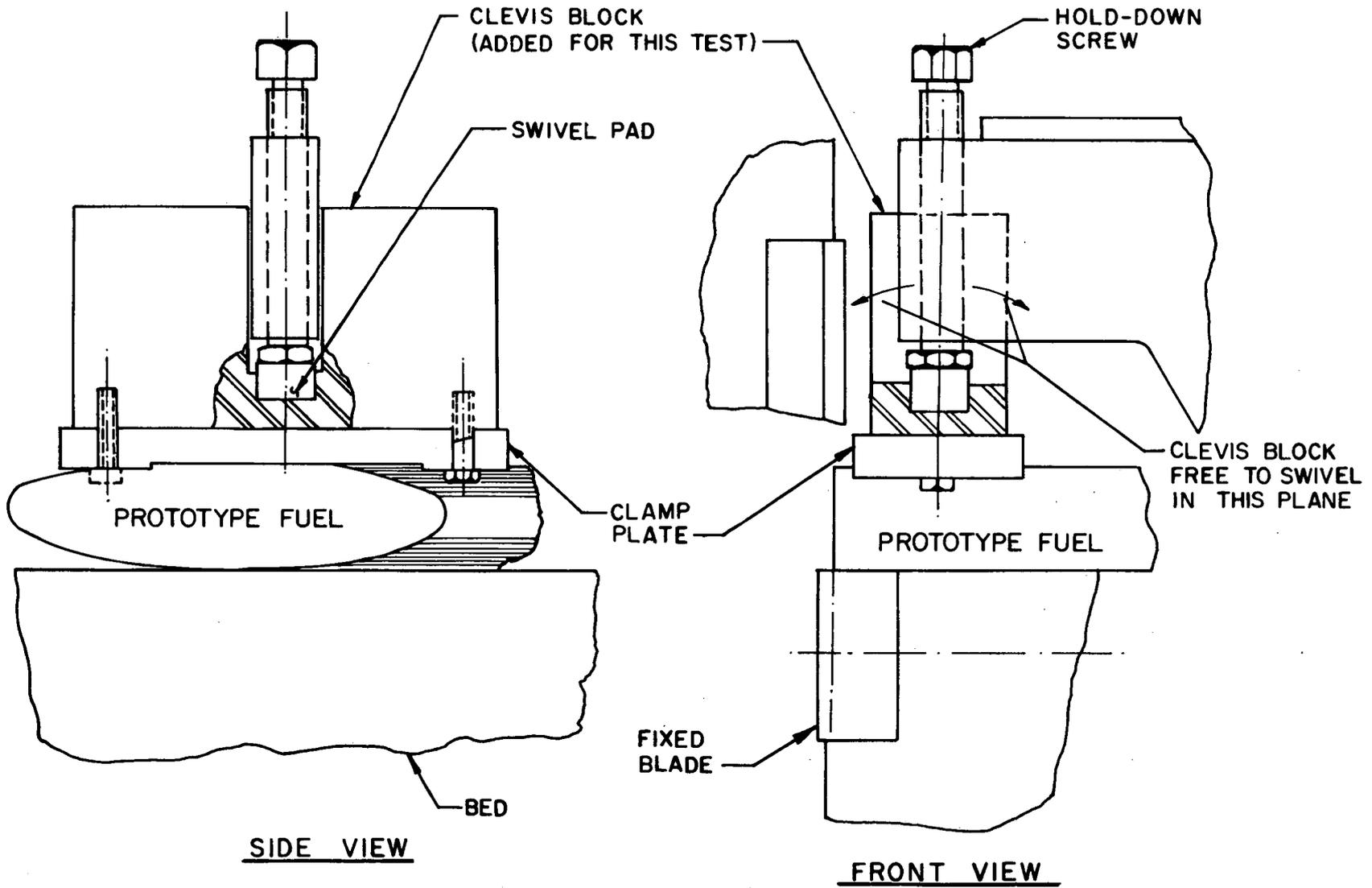


Fig. 5.3. Product characteristics for sample Nos. 3-6.



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Fig. 5.4. Test apparatus as modified since previous test.

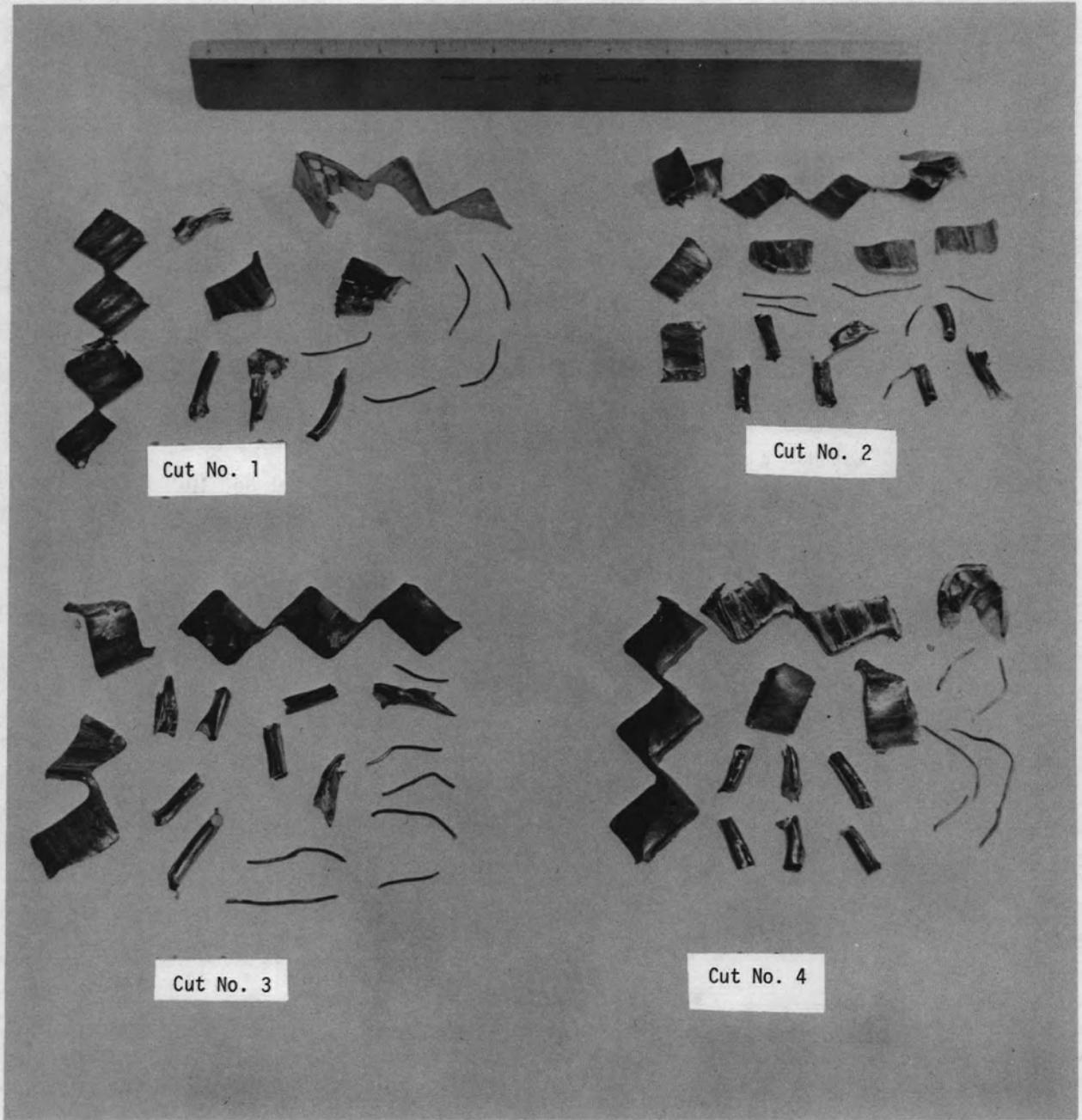


Fig. 5.5. Product characteristics for cut Nos. 1-4.

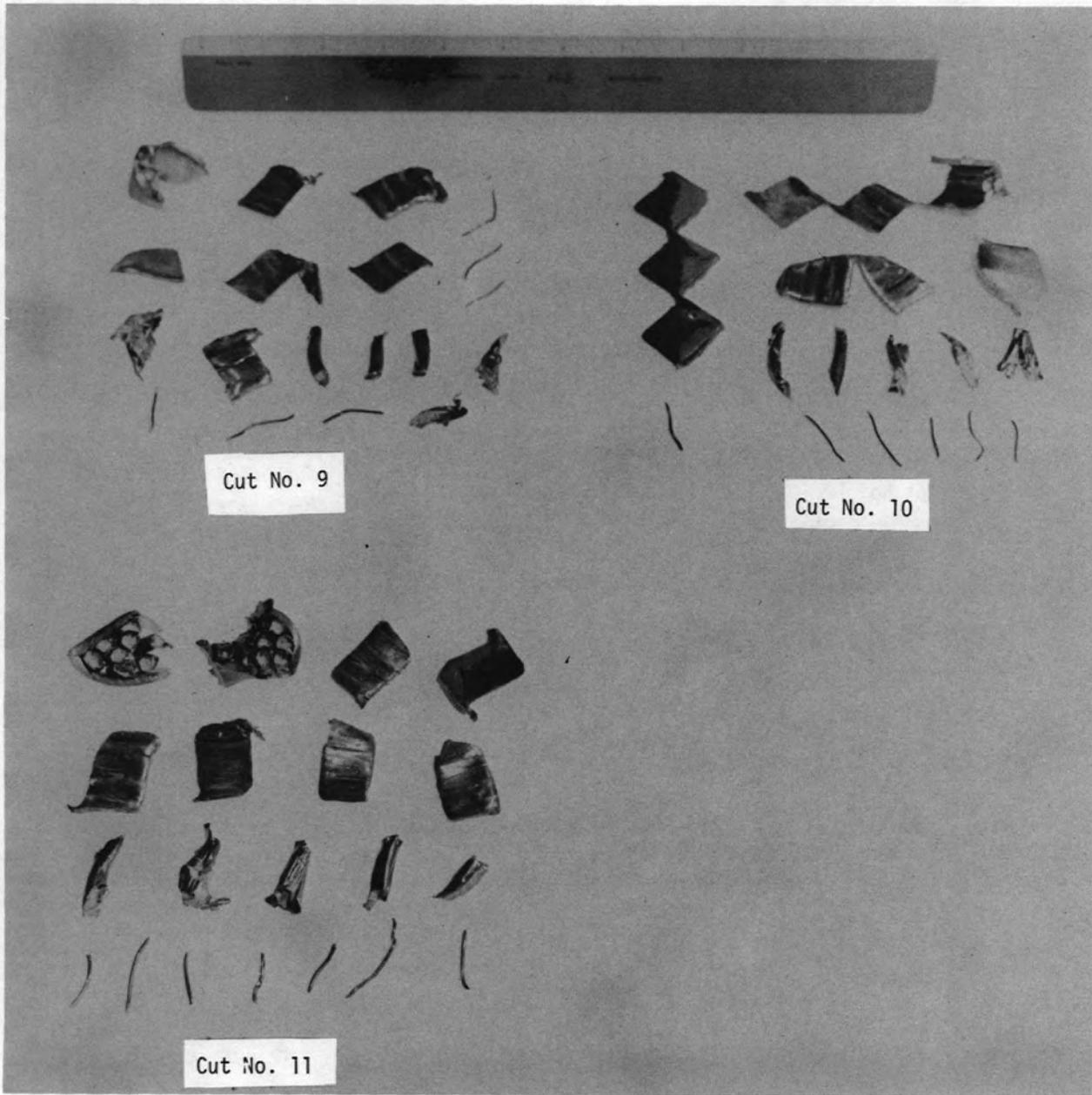


Fig. 5.6. Product characteristics for cut Nos. 9-11.

Work related to the G+W disassembly test rig has continued to yield data concerning the magnitude of frictional resistance to pushing simulated fuel bundles from within ductile prototype shrouds. Phase II of the testing was completed this quarter. Phase II testing was done using 1.1-m (45-in.) bundles and 2.9-m (115-in.) shrouds instead of the 35-m (14-in.) bundles of Phase I. Table 5.1 summarizes the results of Phases I and II. Phase III of the disassembly test was interrupted until a more reliable method of applying wire wrap to 2.6-m (102-in.)-long prototype rods can be found. Currently, efforts are under way to design and fabricate equipment that can produce the necessary, uniform prototype fuel.

Within the past five years, significant advances in laser technology have been made to warrant investigation of the laser's in-cell application to disassembly. As of this writing, Sciaky Brothers, Incorporated, of Chicago, Illinois, and Union Carbide Corporation, Linde Division, of Indianapolis, Indiana, have expressed a willingness to provide future assistance. Both companies have received samples of prototype fuel for some preliminary feasibility testing to be carried out during the next report period.

New instrumentation was installed on the shear to be compatible with the data acquisition DEAC PDP 11/40 computer. Since it is desirable to determine the condition of the shear blade without process interruption, future tests will be carried out to ascertain whether a relationship exists between blade condition and some quantitative parameter such as expended energy. Therefore, in addition to power, pressure, and position, the current instrumentation records the total energy expended during a typical shearing cut. Along these same lines, equipment vibrations might be used to establish shear blade operation by monitoring frequency patterns and peak vibration amplitudes to generate a "machine-print" of good operation. Therefore, a triaxis accelerometer and an FM recorder have been installed on the shear to collect vibration data, and later computer analysis of the data will establish the operation's base line.

Fabrication of a new 200-ton swing-out compactor, push-pull shear ram, and feeding device has begun; delivery is scheduled for sometime during the next report period. In addition, purchase orders have been issued to acquire material to begin fabrication of prototype radial blanket subassemblies.

Table 5.1. Results of disassembly tests for Phase I and Phase II

Bundle bladder pressure [bars (psig)]	Maximum force (net) [N ^a (lb _f)]	Maximum average power (W)
<u>Phase I (35-cm bundle, 91-cm shroud)</u>		
2.0 (29)	11,398	230
3.5 (51)	16,464	230
5.0 (73)	31,661	230
<u>Phase II (1.1-m bundle, 2.9-m shroud)</u>		
2.0	16,002	74.5
3.5	46,101	-
5.0	98,679	131

^a N = newtons in kg-m/sec² = 0.2248 lb-force.

5.3 Voloxidation

D. C. Hampson and M. E. Whatley

The criteria and design specification package for a voloxidizer was issued to four companies during this quarter. This package requests bids for the design and fabrication of a prototype unit to be operated at ORNL. Responses are due early in October. In addition, a design criteria and cost estimate has been completed for the voloxidizer auxiliary system. These items will be designed by UCC-ND Engineering.

5.4 Dissolution

J. Q. Kirkman

Design of the pilot-plant-scale rotary dissolver is essentially complete. A check of the detailed drawing and a formal design review are currently in progress. Final reviews are scheduled to be completed, and the dissolver to be released for fabrication, by approximately October 15, 1976.

UCC-ND Engineering started design of the auxiliary components and systems necessary to support dissolver component development and testing about August 1, 1976. Initial efforts have involved identifying long-lead-time components, initiating development of procurement specifications, and producing a set of preliminary system arrangement drawings. The total engineering design task is defined by criteria, flowsheets, and material balances supplied by the project staff, and encompasses auxiliary systems such as solid materials feeding, liquid makeup, dissolver product handling, and acid recovery. Components contained in these auxiliary systems include tanks, coolers, condensers, scrubber, conveyors, and steam generator.

Specifications for the first component, the NO_x scrubber, are scheduled to be released for bids on October 1, 1976. Procurement actions for the remaining system components will be initiated during the period October 1, 1976, to February 1, 1977.

5.5 Receiving and Storage

S. A. Meacham* and N. R. Grant

During this report period, an outline of areas to be investigated relative to receiving and storage of spent LMFBR fuel was initiated. Typical areas and questions pertaining to these areas include:

- (1) Cask handling: pre-unload preparations, seal to unload systems, post-unload cleaning.
- (2) Subassembly handling: method, cooling, subassembly cutting.
- (3) Subassembly cleaning: method, cooling, hydrogen disposal, disposal of radioactive liquid and solid (particulate) waste.
- (4) Storage: storage of leaking vs nonleaking subassemblies, advantages of water vs sodium, requirements for water vs sodium treatment.

This quarter, emphasis was given to areas (3) and (4), spent LMFBR fuel cleaning (sodium removal) and subsequent storage. One immediate concern of the overall LMFBR fuel reprocessing technology is the problem of selecting a safe and efficient method for removing the sodium from the spent fuel assemblies as well as choosing a proper means of storage before the fuels are recycled. Although a sodium pool may be desirable for storing the LMFBR spent fuels, economic considerations dictate the need of evaluating the feasibility of a water storage pit for the LMFBR spent fuels. The objective of the WARD/ORNL program¹ is to determine the corrosion behavior of sodium-exposed stainless steels, after various sodium removal procedures, under water pit storage conditions. These preliminary results will be used to determine whether a potential problem exists in the storage of sodium-exposed fuel assemblies under water.

The WARD/ORNL program, which was initiated this quarter, will consist of five phases to be conducted concurrently:

- (1) The literature will be reviewed to determine the effect of irradiation on the corrosion behavior of stainless steels (sodium-exposed if available) in water.
- (2) The prototypical FFTF and CRBR fuel cladding material, 20% cold-worked 316 stainless steel which has been exposed to sodium at temperatures from 1200 to 1360°F for periods of approximately 5000 hr,

*Westinghouse Advanced Reactor Division (WARD), Madison, Pa.; on loan to ORNL.

will be gathered and identified before it is exposed to sodium again in order to recondition the surface under the same conditions as experienced previously.

- (3) Sodium-removal procedures, including the alcohol process, the water vapor/argon process, and the steam/argon process, will be reviewed and employed on the test samples. (Argon and nitrogen are considered interchangeable for this test.)
- (4) Preliminary water testing of sodium-exposed specimens that are clad with 20% cold-worked type 316 stainless steel and have been reconditioned and cleaned in accordance with (2) and (3), respectively, will be initiated. In addition, specimens of annealed type 316 stainless steel with surfaces typical of material deposited in the low-temperature region of a dynamic sodium system will be considered. Furthermore, an evaluation of the effects of released fission products and sodium-logged fuel on the water storage pit relative to purification, detection, and maintenance of low activity levels within the storage media will be reviewed.

The specimens that are selected will cover basically two types of mass transfer changes resulting from sodium exposure: One group of high-temperature specimens [see item (2) above] represents corrosive and selective alloy depletion conditions. Another group, prepared from type 316 stainless steel previously exposed to sodium from 700 to 900°F, will provide surface conditions that are prototypical of the mass transfer deposition region in a sodium--stainless steel system. Therefore, the specimens selected will exhibit various degrees of mass transfer changes.

The specimens will be exposed to water for times up to three months. Two types of water conditions, such as temperature and chemistry, will be studied: (1) those prototypical of the LWR water storage pit (high purity, 180°F), and (2) those expected to be encountered during "upsets" (high pH and/or high Cl⁻ content).

- (5) The preliminary data on type 316 stainless steel will be evaluated, and recommendations on water storage of spent LMFBR fuel will be

made. Additional work required to substantiate the preliminary results or for solving a problem will be outlined at this stage. In addition, testing of the complete fuel assembly and/or the advanced cladding alloys will be defined.

During this quarter, the following work has been accomplished or initiated:

- (1) The scope of the work involved in the WARD/ORNL Fuel Reprocessing Program Corrosion Studies has been divided into five phases designed to generate specific information regarding the feasibility of using water pit storage for LMFBR spent fuel assemblies.
- (2) A test matrix for sodium removal of sodium-corroded, type 316 stainless steel cladding (hot leg) and the deposit containing stainless steel tubing (cold leg) was prepared (see Table 5.2). In addition, the water chemistry for the corrosion studies was also delineated (see Table 5.3).
- (3) All design work was completed; the machining of previously corroded specimens was also completed. In addition, the facilities required for the sodium reconditioning have been defined.
- (4) A welding procedure was developed to permit the welding of the test specimens in an inert atmosphere. Using this technique, 30 specimens were fabricated from material that had been exposed to conditions which led to corrosion by sodium.
- (5) After the test assembly fabricated from 1325°F exposed material had been installed into a dynamic sodium loop, the first exposure run was begun. To date, approximately 200 hr of exposure time has accumulated on these specimens.

In addition, a review of sodium-removal procedures was conducted, and the EBR-II and CPP facilities were visited to discuss and review current sodium removal procedures and the storage of spent nuclear fuel.

Table 5.2. Test matrix for sodium removal and water exposure of sodium-exposed cladding material^a.

Sodium removal method	Test period (months)	Water bath			
		No. 1	No. 2	No. 3	No. 4
Water vapor/argon	1	0	X	X	X
	2	0	X	X	X
	3	XX	XX	XX	X
Steam/argon	1	0	X	X	X
	2	0	X	X	X
	3	XX	XX	XX	X
Alcohol	1	0	0	0	0
	2	0	0	0	0
	3	X	X	X	X

^a 0 = not tested; X = one specimen; XX = two specimens.

Table 5.3. Chemistry of water baths for cladding material exposure

Property	Conditions for water bath			
	No. 1	No. 2	No. 3	No. 4
Chloride, ^a ppm	≤ 0.15	≤ 0.15	≥ 500	≥ 500
pH	7.0 ± 0.5	10 ± 1 ^b	7.0 ± 0.5	10 ± 1 ^b
Oxygen content, μg/cc ^c	5.3	5.3	5.3	5.3
Sodium ion content	Not controlled ^d	Increased by NaOH added to increase pH	Not controlled ^d	Increased by NaOH added to increase pH
Conductivity, micromhos	≤ 5	Not controlled ^d	Not controlled ^d	Not controlled ^d

^a Adjusted with MgCl₂.

^b Increased via additions of NaOH.

^c Saturated at 180°F.

^d Will be measured.

REFERENCES FOR SECTION 5

1. "The WARD/ORNL Fuel Reprocessing Work Program on Sodium Cleaning and Subsequent Corrosion of Sodium Exposed Stainless Steel Under Water Pit Storage Conditions," ORNL Contract No. 7168, Westinghouse Electric Corporation.

6. INTEGRATED PROTOTYPE EQUIPMENT TEST FACILITY (IPET)

J. M. Chandler and W. F. Schaffer, Jr.

The conceptual design of the IPET facility was completed early in July 1976 in accordance with the conceptual design criteria package presented to UCC-ND Engineering. Draft copies were distributed internally and to ERDA Oak Ridge Operations for review. Comments received from ERDA in mid-September were minor; the schedule of the operating phases of IPD and ROMD was clarified, and additional justification for the data acquisition system and emergency power was included in the final version of the report. The report is scheduled for issue in early October. In brief, the data acquisition system is vital in storing process information in an orderly form to allow rapid analysis of process events as well as equipment performance for use by the engineering disciplines in the design of the HPP; the emergency power is necessary to minimize offenses to the operating environment by supplying the power necessary to keep the off-gas system operational during power outages. The conceptual design cost estimate for the project was \$16 million.

6.1 IMFBR Facility Engineering

The installation of the process steam boiler was completed in July by a lump-sum contractor (Hobson Construction Company). Initial startup tests under the supervision of the boiler fabricator's (Johnston Brothers, Incorporated) field representative (Industrial Water Chemicals Equipment Division) were conducted early in August. Two auxiliary systems required changes before the installation could be considered operational. A surplus tank supplied to the contractor by ORNL as part of the boiler blowdown system was not rated for full boiler pressure and had to be replaced. A modification of the fuel oil supply piping was also made to correct the operation of the feed pumps. The documentation on the steam boiler was reviewed to ensure compliance with ORNL quality assurance requirements. The files, which are now complete, have been transferred from the Project Engineer to the IMFBR Fuel Reprocessing Project.

Significant achievements were also made in several projects necessary to the progress of the component development activities. These are listed below.

- (1) Process off-gas system (Phase I). Capital equipment funding to the extent of \$110,000 was authorized for the engineering and advance procurement of essential equipment (i.e., the fan and filter assembly).
- (2) Demineralized water system. UCC-ND Engineering is engaged in a study for the refurbishing of this system for supplying the process water requirements for the component development work and for the process steam boiler. A cost estimate is also being prepared. Field work has started on the cleaning of the ion exchange beds, and procurement of new resin has been initiated.
- (3) Process steam system. The system for distributing process steam for Building 7602 is now being installed; completion is expected by the end of September.
- (4) Crane modification. The 23-ton crane system is being modified for pendant control in order to improve its utility for component development activities.

6.2 IMFBR Facility Waste

The research and development activities in the 7600 area associated with the IMFBR fuel reprocessing project will create from 1000 to 2000 gal of waste per day, which must be given special treatment. A waste management proposal for these wastes was prepared for management review. UCC-ND Engineering has been funded for a study, to culminate in a document containing an equipment list, piping flowsheets, equipment layouts, and a cost estimate for each of the four options presented in the proposal.

The plan was developed after a series of meetings held with ORNL, the Y-12 Plant, and ERDA waste management personnel. The IMFBR fuel reprocessing wastes contain HNO_3 as the principal contaminant, along with lesser amounts of phosphates, sulfates, and caustics. Small amounts of natural or depleted uranium may also be present in some waste streams.

In addition to the direct process waste streams, other sources of liquid waste included in the study were cooling water and storm-drain streams. Once-through cooling water and foundation water drains will be discharged to the environment. The existing sanitary waste disposal system is considered acceptable.

The proposed waste management plan shown in Fig. 6.1 is favored over the other options. This option utilizes the existing waste treatment operations at the Y-12 Plant, which will receive all the process waste streams from the 7600 area.

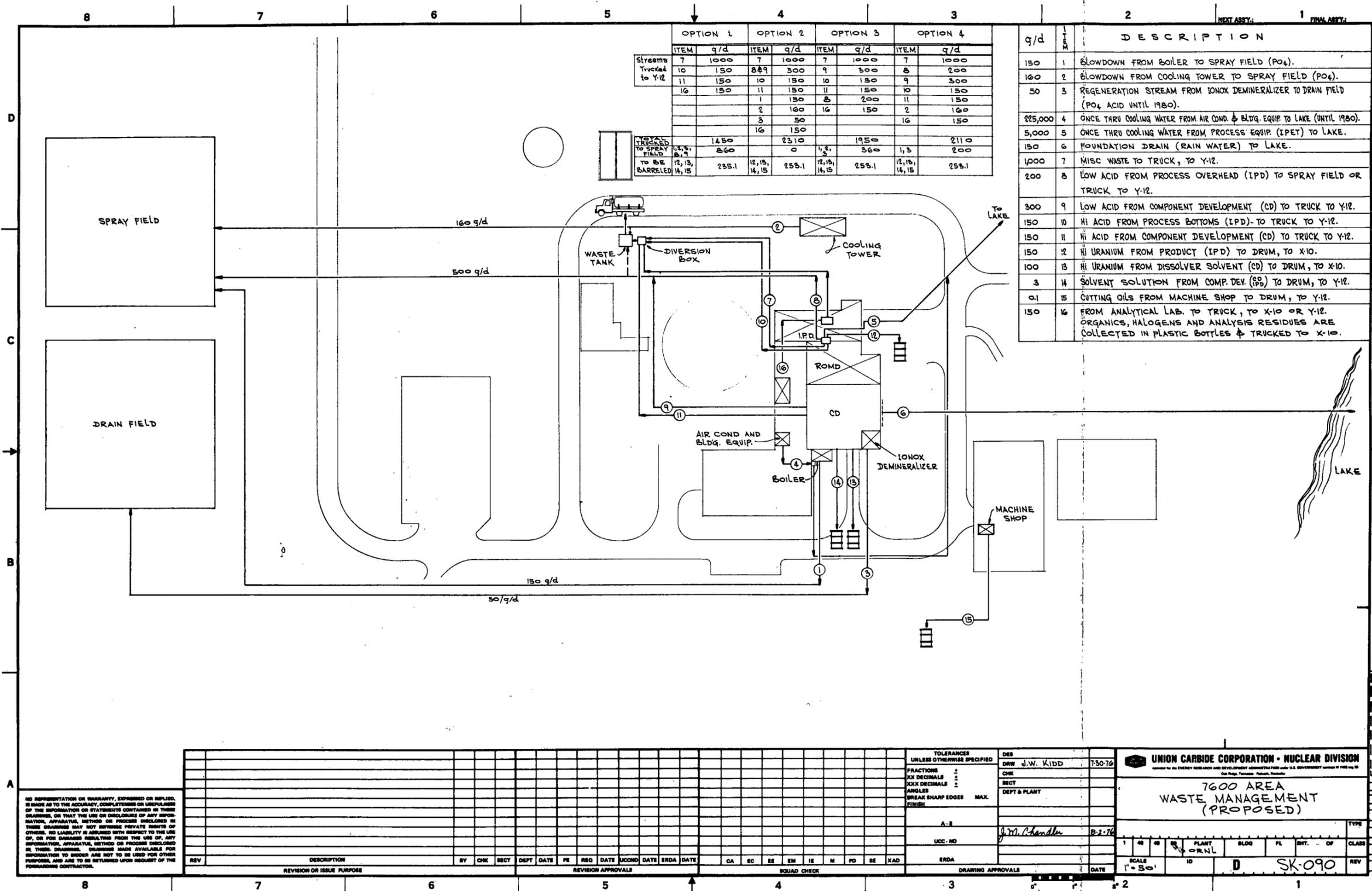
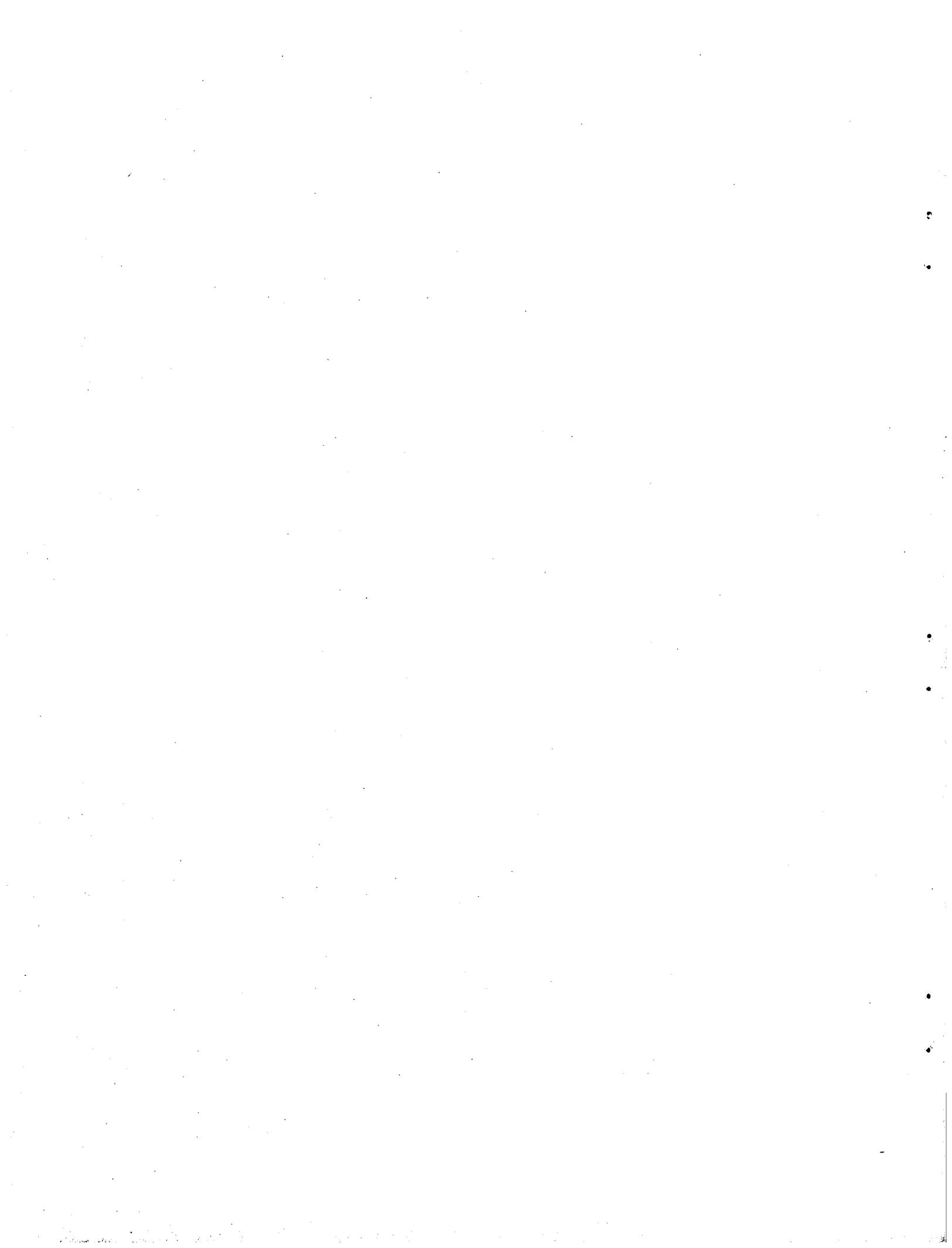


Fig. 6.1. Proposed waste management plan for the 7600 area.



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