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HTGR FUEL RECYCLE PROGRAM

**QUARTERLY PROGRESS REPORT
FOR THE PERIOD ENDING AUGUST 31, 1977**

**Prepared under
Contract EY-76-C-03-0167
Project Agreement No. 53
for the San Francisco Operations Office
U.S. Energy Research and Development Administration**

DATE PUBLISHED: SEPTEMBER 1977

GENERAL ATOMIC COMPANY

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ABSTRACT

This publication continues the quarterly series presenting results of work performed under the National HTGR Fuel Recycle Program (also known as the Thorium Utilization Program) at General Atomic Company. Results of work on this program prior to June 1974 were included in a quarterly series on the HTGR Base Program.

The work reported includes the development of unit processes and equipment for reprocessing of High-Temperature Gas-Cooled Reactor (HTGR) fuel, the design and development of an integrated pilot line to demonstrate the head end of HTGR reprocessing using unirradiated fuel materials, and design work in support of Hot Engineering Tests (HET). Work is also described on trade-off studies concerning the required design of facilities and equipment for the large-scale recycle of HTGR fuels in order to guide the development activities for HTGR fuel recycle.

INTRODUCTION

This report covers the work performed by General Atomic Company under U.S. Energy Research and Development Administration Contract EY-76-C-03-0167, Project Agreement No. 53. The work done under this project agreement is part of the program for development of recycle technology for High-Temperature Gas-Cooled Reactor (HTGR) fuels described in the "National Program Plan for HTGR Fuel Recycle Development" (GCR-76/19).

The objective of the program is to provide a demonstration plant for the recycle of HTGR fuels. This plant will demonstrate facility and equipment design and operating procedures which are licensable and commercially feasible for the reprocessing and refabrication of spent fuel from HTGRs. Work at General Atomic Company is concentrating on the following National Program tasks: Program Management and Analysis (Task 100); Reprocessing Technology Development (Task 200); Refabrication Technology Development (Task 300); HTGR Recycle Demonstration Facility (HRDF) design support (Task 600).

Task 100, Program Management and Analysis, includes the functions of overall planning, scheduling, budgeting, reporting, management control of the program, and coordination of activities.

Task 200, Reprocessing Technology Development, includes the definition of flowsheets, the development of components, and the definition of operating techniques, remote maintenance and or disassembly techniques, and coordination of fuel shipping and storage activities. Operations which must be developed include crushing of the fuel elements; burning the graphite in a fluidized bed-burner; separation of the fertile and fissile

particles; crushing the SiC coating on fissile particles; burning the crushed particles; dissolution of thorium and uranium in the burned, crushed particles; separation of the undissolved solids (SiC hulls, etc.) from the leachate; separation of the thorium and uranium from the fission products by solvent extraction; separation and purification of the thorium and uranium by solvent extraction; process and facility off-gas treatments to ensure releases are environmentally acceptable and in compliance with regulations; and the primary treatment of solid, liquid, and gaseous wastes from the process.

Task 300, Refabrication Technology Development, includes the definition of flowsheets, the development of components, and the definition of operating techniques, remote maintenance and/or disassembly techniques, and coordination of fuel shipping and storage activities. The refabrication begins with aqueous uranyl nitrate solution from the reprocessing facility and ends with fuel elements prepared for shipment to the reactor. The principal operations to be developed are loading the ion-exchange resin with uranium, resin carbonization, resin conversion, coating the converted resin with pyrolytic carbon and SiC, fuel rod fabrication, fuel element assembly, fuel and fuel element inspection, scrap recovery, and waste handling.

Task 600, HTGR Recycle Demonstration Facility, includes the design, construction, proof-testing, and operation of a demonstration facility for the recycle of HTGR fuel. The plant is to include all fuel cycle operations from the receiving of spent fuel elements from the reactors to shipping the refabricated fuel elements back to the reactors. The preconceptual design studies and the early conceptual design are to be used to guide the development work for reprocessing and refabrication processes and equipment. The results of the research and development tasks will in turn be used to guide the detailed design of HRDF.

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1. SUMMARY

General Atomic HTGR Fuel Recycle Program activities progressed on schedule during the quarter except for the 48-hour run on the 0.20-m primary burner, which is being repaired.

Crushing of fueled HTGR fuel elements has commenced. No major operating or process difficulties were discovered in the four tests completed to date. Material balances have demonstrated material unaccounted for (MUF) quantities consistently no greater than $\sim 0.3\%$ of the charge, which is well within the $\leq 0.5\%$ target. The quantity of oversize crusher product from the fueled elements has been consistently reduced compared with that obtained from the unfueled elements tested in Phase II of the Activity Plan. Dust containment within the ventilation enclosure has been completed, and no release of radioactive materials has been detected.

Two runs of extended duration, high bed carbon operation were made on the 0.40-m burner. Results indicated that either in-bed or above-bed fines recycle with the parallel, pressurized hopper pneumatic transport system was acceptable for fines burning. The above-bed recycle resulted in less fuel particle breakage and buildup of noncombustible oxides/hulls in the fines system and was thus selected as the preferred injection location.

Air classification tests were performed to complete qualification testing. This included preoperational component testing, FSV type TRISO/TRISO fuel particle classification, and LHTGR type TRISO/BISO fuel particle classification.

The detail design of the engineering-scale dissolver-centrifuge has been completed, and equipment detail design and system arrangement drawings have been released for fabrication or procurement.

Three heel dissolution runs were made in the 0.13-m dissolver. Based on bench-scale tests, 40% heel was selected as the optimal thoria-to-Thorex ratio. Results of these 0.13-m dissolver runs will be reported in the next quarterly report after data analyses are complete.

Three solvent extraction feed adjustment runs were completed during the quarter. The purpose of these runs was to test equipment modifications and flowsheet requirements for intercycle concentration.

Seven solvent extraction runs were completed during the quarter. Runs 62 and 63 were used to determine the extraction efficiency of the Robatel centrifugal contactor using an aqueous feed stream containing thorium. The SEPHIS computer code for the Thorex flowsheet was used to calculate theoretical values which were compared with the analytical results from the pilot plant operation.

Qualification testing of components and sub-systems in the solids handling system was completed using simulated feed materials. Additional system verification will take place during the forthcoming sequential operations of the head-end pilot plant. Installation of new support structures was completed for the crusher product bunker and the primary burner product bunker. Both bunker systems are fully operational; each bunker's weigh system is being monitored for performance improvements due to the structural changes.

Information previously generated in the Maintainability and Reliability Study is being integrated with the Reprocessing Plant Operating Mode Simulation Study. A failure mode analysis for dry head-end systems has been initiated. The final draft of the HRDF Head-End Remote Maintenance Report has been completed and is in review.

Flowsheets for the HRDF Thorex and Purex solvent extraction systems were reviewed and updated to reflect the current process development status. A GASP computer simulation language has been chosen as being most

suitable for modeling operating modes in an HRDF reprocessing plant. Development of a simple model of the fuel element crushing system is in progress.

As part of the nonproliferation study, Level I functional flow diagrams were prepared for specified processing areas in HTGR, LWR, HWR, GCFR, and LMFBR thorium fuel cycle options. Fuel material descriptions for discrete processing steps in HTGR fuel cycle options were included.

2. FUEL ELEMENT CRUSHING

2.1. SUMMARY

During the present reporting period, preparations for crushing fueled HTGR fuel elements began. A slot valve was designed, constructed, installed, and then tested while anode butt scrap graphite was being crushed for additional feed for primary burner tests. The valve was successful in preventing preferential discharge of crushed product from the equipment item nearest the product collection bunker.

Crushing of fueled HTGR fuel elements has commenced. No major operating or process difficulties were discovered in the four tests completed to date. Material balances have demonstrated material unaccounted for (MUF) quantities consistently no greater than $\sim 0.3\%$ of the charge, which is well within the $\leq 0.5\%$ target. The quantity of oversize crusher product from the fueled elements has been consistently reduced compared with that obtained from the unfueled elements tested in Phase II of the Activity Plan (AP521001). Dust containment within the ventilation enclosure has been complete, and no release of radioactive materials has been detected. The product size distribution has thus far been within the estimated limits of the primary burner feed requirements. Total particle breakage is $\sim 2.7\%$ of the fissile and $\sim 0.3\%$ of the fertile particles.

2.2. UNIFRAME TESTING

2.2.1. Introduction

Prior to the current tests, several test programs had been completed, including Phases I and II of the Activity Plan and 21 separate primary burner feed preparation tests. Phase I encompassed separate testing of

each of the major equipment items in the fuel element size reduction system (UNIFRAME) to demonstrate their capabilities, operating characteristics, and interactions with the subsystems. Phase II encompassed tests of the UNIFRAME as an integrated operation with all components functioning as a system. A series of 21 tests using anode butt scrap graphite as feed to the UNIFRAME were also completed to provide the necessary quantities of feed for the primary burner system and to obtain further operating experience with the UNIFRAME. All of these prior tests utilized unfueled HTGR graphite fuel blocks or the anode butt scrap graphite as feed. Some of the crushed product from the anode butt scrap graphite tests has already been used successfully in current primary burner tests.

The current tests include (1) continuation of the anode butt scrap graphite crushing tests to prepare the UNIFRAME for crushing fueled HTGR fuel elements and (2) commencement of Phase III of the Activity Plan, which encompasses fueled element crushing tests.

2.2.2. UNIFRAME Preparation for Fueled Element Crushing

2.2.2.1. Introduction. Previous tests utilizing anode butt scrap graphite had resulted in preferential discharge of the crushed product from the equipment item closest to the product collection bunker (Ref. 2-1). This was attributed to insufficient motive gas flow for pneumatic transport of the product due to diversion of the gas through the path of least resistance (i.e., equipment nearest the product collection bunker). The situation was initially avoided by installing the oversize crusher nearest to the product collection bunker and operating it intermittently to allow product to build up in the crushing cavity, thereby increasing the pressure drop and diverting the motive gas through the screener, ensuring its discharge.

However, to provide a more positive control and reduce operating variables, a slot valve was designed, constructed, and installed in the oversize crusher discharge (see Fig. 2-1). This valve provides a variable

Fig. 2-1. UNIFRAME oversized crusher slot valve

restriction in the discharge from the oversize crusher to the pneumatic transport line allowing control of the pressure drop, which prevents preferential transport of the oversize crusher product. The current crushing tests on the anode butt scrap graphite were used to test the slot valve in preparation for crushing fuel-loaded HTGR elements as well as to provide additional feed for primary burner tests.

2.2.2.2. Slot Valve Tests. Four separate tests (SCRAP-22 through -25) were completed utilizing anode butt scrap graphite to test the feasibility of the slot valve and to provide additional feed for primary burner tests.

In the initial test (SCRAP-22), the slot valve was set at its 50% open position and the screener failed to discharge during the crushing operation. After crushing was completed and with the screener housing containing its entire inventory of product, the slot valve was closed completely. Discharge of the screener product commenced immediately and automatically without assistance and continued without interruption until the entire product had been pneumatically transported.

The slot valve was reset at its 25% open position for the second test (SCRAP-23). Material discharge and pneumatic transport from both the screener and oversize crusher were complete and problem-free. Two more tests were made with the slot valve at its 25% open position (SCRAP-24 and -25). Both tests resulted in trouble-free transport.

At the conclusion of this series of four tests, the system was cleaned out to obtain a complete material balance. Material balance results for these tests and summaries of the material balances for the previous tests are given in Table 2-1. Material unaccounted for (MUF) individually in each of the current tests as well as for the entire four-run series was, in general, greater than had been observed in the previous 21 tests. The reason for the increased MUF is not known for certain but is probably the premature shutdown of the secondary and tertiary crushers. It has been a practice to shut down each major equipment item after it has completed its

TABLE 2-1
MATERIAL BALANCE RESULTS FROM SCRAP GRAPHITE CRUSHING

Test No.	Charge (kg)	Recovery (kg)	Gain (+) or Loss (-)	MUF (% of Charge)
Prior Tests				
SCRAP-1 to -9	700.150	695.329	(-) 4.821	0.68
SCRAP-10 to -18	863.751	860.804	(-) 2.947	0.34
SCRAP-19 to -21 and element 1-2319	<u>388.054</u>	<u>384.363</u>	<u>(-) 3.691</u>	<u>0.95</u>
Total	1951.955	1940.496	(-) 11.459	0.59
Current Tests				
SCRAP-22	97.395	92.908	(-) 4.487	4.61
SCRAP-23	96.942	95.583	(-) 1.359	1.40
SCRAP-24	97.395	94.677	(-) 2.718	2.79
SCRAP-25	<u>97.395</u>	<u>113.740</u> ^(a)	<u>(+) 16.345</u>	<u>16.78</u> ^(a)
Total	389.127	396.908	(+) 7.781	2.00
Total for All Tests				
SCRAP-1 to -25	2341.082	2337.404	(-) 3.678	0.12

(a) Includes material recovered from system cleanout at end of tests.

stage of the crushing operation. With the ventilation shrouding completely in place (installation was completed prior to test SCRAP-19), visual observation of the completion of secondary crushing is limited, and both the secondary and tertiary crushers may have been shut down prior to complete material discharge. To achieve complete cleanout after the conclusion of test SCRAP-25, the equipment was restarted and operated for several minutes. As can be seen from Table 2-1, this resulted in the recovery of several kilograms of material and an excellent overall MUF of 0.12% of the total charge during all 25 anode butt scrap graphite crushing tests. In the remaining tests all equipment items will be operated past their normal completion times up to a 15-min maximum to ensure a more complete cleanout.

2.2.3. Fueled Element Crushing

The Phase III system tests of the UNIFRAME as outlined in the Activity Plan (AP521001, Issue D, January 4, 1977) are being undertaken to provide data and observations on crushing fueled HTGR elements. The previous tests (Phase II of the Activity Plan) have demonstrated the system's capabilities with unfueled HTGR elements.

The major objectives of the Phase III system tests are to observe and compare, with each available type of fueled HTGR element:

1. The operation of the system, crushing behavior of the material, throughput rates, and product characteristics.
2. The system's material flow and radioactive material containment concepts.
3. The extent of fuel particle breakage.

The following fueled HTGR elements were made available and scheduled for use in the Phase III tests:

1. Three standard FSV H-327 graphite elements fueled with TRISO/TRISO particles (scheduled for tests UL-58, UL-59, and UL-60).
2. Three control rod FSV H-327 graphite elements fueled with TRISO/TRISO particles (scheduled for test UL-61 and optional tests UL-62 and UL-64 based on results from other tests).
3. Three standard LHTGR H-451 graphite elements fueled with TRISO/BISO particles (scheduled for tests UL-64, UL-65, and UL-66).

In addition to these nine fuel elements for the Phase III tests, 17 standard LHTGR H-451 graphite elements fueled with TRISO/BISO particles are available for testing of pilot plant sequential operations. Because of these forthcoming pilot plant sequential operations, a standard LHTGR element was selected for the initial Phase III test to obtain data and make observations at the earliest date possible in the event changes are required to minimize the risks to the sequential operations.

To date, the three standard LHTGR and one of the standard FSV fueled elements have been crushed successfully (Phase III tests UL-58, UL-64, UL-65, and UL-66) in the UNIFRAME without major difficulties or operating problems. Analysis and data gathering from the tests are in progress and partially completed.

In each of the five tests completed thus far, crushed product from the oversize crusher and the screener was collected separately for analysis of the respective particle breakage and product size distributions.

To obtain maximum recovery of the crushed materials from each test for a material balance, the screener, oversize crusher, and tertiary crusher were operated for 15 min after initial charging of the fuel element because of the inability to directly observe the conclusion of their operations, and the secondary crusher was operated for several minutes after observation of its conclusion of crushing due to the limited visibility of its

operation. In addition, a port was removed from the screener after shutdown and the normal residual material on the screen surface was brushed into the discharge trough to the oversize crusher. These two equipment items were then operated for an additional 3 to 5 min to completely clean out any oversize crushed material.

These modified operating conditions thus far have resulted in excellent material balances for all runs for which data are available. The largest amount of MUF for any single run was 0.29% of the charge and the total MUF for all four runs was 0.05% of the total charge. Material balance data are given in Table 2-2.

The quantities of crushed product discharged separately from the screener and oversize crusher relative to the total crushed product (i.e., product distributions) are shown in Table 2-3. Further comparison of results for fueled FSV and LHTGR elements will be performed after the final two FSV elements have been crushed (tests UL-59 and UL-60). However, the trend observed in the Phase II tests for the percentage of oversize crusher products to be less for crushed unfueled LHTGR H-451 graphite elements than for crushed unfueled FSV H-327 graphite elements seems to be continuing for the fueled elements. In general, the percentage of oversize crusher product also seems to be less from fueled elements than from their unfueled counterparts (Ref. 2-1).

In the initial Phase III test (UL-64), little oversize product was collected at the conclusion of testing. Investigation of this abnormality revealed that two large, wafer-like fragments from the anode butt scrap graphite crushing tests had lodged in the oversize discharge chute. This prevented material from entering the oversize crusher, filled the chute, and allowed the excess to overflow into the screener discharge. The material in the chute was subsequently crushed in the oversize crusher, but this upset condition rendered the data invalid for comparison of quantity and size distribution of oversize crusher and screener products. This blockage

TABLE 2-2
MATERIAL BALANCES FROM FUELED ELEMENT CRUSHING

Test No.	Charge (kg)	Recovery (kg)	Gain (+) or Loss (-)	MUF (% of Charge)
UL-58	129.090	128.724	(-) 0.366	0.28
UL-64	122.243	122.241	(-) 0.003	0.00
UL-65	121.300	121.652	(+) 0.352	0.29
<u>UL-66</u>	<u>122.763</u>	<u>122.523</u>	<u>(-) 0.240</u>	<u>0.20</u>
Total	495.396	495.140	(-) 0.256	0.05

TABLE 2-3
PRODUCT DISTRIBUTIONS FROM FUELED ELEMENT CRUSHING

Test No.	Oversize Crusher Product		Screener Product	
	kg	% of Total Product	kg	% of Total Product
UL-58	15.253	11.85	113.471	88.15
UL-64 ^(a)	0.915	0.75	121.326	99.25
UL-65	11.152	9.17	110.500	90.83
UL-66	8.591	7.01	113.932	92.99

(a) Blockage in the oversize chute resulted in abnormally low throughput to the oversize crusher. Data are invalid for comparison with other tests.

is unlikely to reoccur without the presence of the abnormally shaped scrap graphite, and it has not reoccurred in any of the tests thus far.

Due to the limited visibility through the ventilation enclosure, the system throughput rates cannot be established with great accuracy. However, observations through the fuel charging port in the tests thus far indicate that crushing is essentially complete in less than 5 min. The remainder of the 15-min running time is necessary only to ensure a more complete discharge of potential small residues of material. In only one test (UL-58) was any material holdup observed after the 15-min running time. In this test a small fragment (estimated to be 10 x 3.8 x 7.6 cm) remained in the secondary crusher. This fragment was moved from its original location in the crushing cavity via a rod and was crushed immediately upon restarting of the secondary crusher. Current plans are to investigate means of avoiding this in tests of the upgraded design.

Losses of radioactive dusts from the ventilation enclosure are detected via two continuous air monitors, one located near the fuel element charging port and one located near the screener and the oversize crusher discharge point, and by wipes of the floor and machine frame surfaces outside the ventilation enclosure at the end of each test. Results thus far indicate that dust escape from the ventilation enclosure did not occur or was in such small quantities as to be indiscernible from the normal background counts.

The screener and oversize crusher products from each individual run were reduced in a sample splitter to a size suitable for size distribution determinations using a standard sieve series. Data on the screener and oversize crusher product size distributions from the fueled element tests to date are presented in Table 2-4.

General trends indicate that the screener product from crushing standard LHTGR H-451 graphite elements is somewhat coarser than that from crushing standard FSV H-327 graphite elements, as was also observed with

TABLE 2-4
 SCREENER AND OVERSIZE CRUSHER PRODUCT SIZE DISTRIBUTIONS FROM FUELED ELEMENT CRUSHING

A. Screener Results

Test No.	Weight Percent of Screened Fractions (Mesh Size in μm)									
	+4750	-4750 +4000	-4000 +2800	-2800 +2000	-2000 +1000	-1000 +850	-850 +425	-425 +355	-355 +250	-250
UL-58	0	2.5	21.0	11.5	15.2	4.9	34.5	1.8	2.1	6.5
UL-64 (a)	2.1	5.0	19.7	11.7	15.8	14.8	21.7	1.8	2.2	5.2
UL-65	0	4.6	25.6	13.5	15.7	11.7	21.8	1.5	1.8	3.9
UL-66	0.2	3.7	25.2	13.2	15.6	10.6	22.3	1.5	2.1	5.6
Test No.	Cumulative Weight Percent Less Than Stated Size (Mesh Size in μm)									
	+4750	-4750 +4000	-4000 +2800	-2800 +2000	-2000 +1000	-1000 +850	-850 +425	-425 +355	-355 +250	-250
UL-58	100	100	97.5	76.5	65.0	49.8	44.9	10.4	8.6	6.5
UL-64 (a)	100	97.9	93.0	73.2	61.5	45.7	30.9	9.2	7.4	5.2
UL-65	100	100	95.4	69.8	56.3	40.6	29.0	7.2	5.7	3.9
UL-66	100	99.8	96.1	70.9	57.7	42.1	31.5	9.2	7.7	5.6

B. Oversize Crusher Results

Test No.	Weight Percent of Screened Fractions (Mesh Size in μm)									
	+4750	-4750 +4000	-4000 +2800	-2800 +2000	-2000 +1000	-1000 +850	-850 +425	-425 +355	-355 +250	-250
UL-58	3.5	9.6	17.6	19.4	24.9	2.9	11.6	1.5	1.8	7.2
UL-64 (a)	14.5	15.0	18.7	15.4	15.9	2.7	6.5	1.0	1.3	9.1
UL-65	10.4	12.7	16.6	16.2	19.1	4.2	10.0	1.9	1.8	7.1
UL-66	12.2	12.3	12.9	12.1	18.5	4.9	13.4	1.4	2.0	10.2
Test No.	Cumulative Weight Percent Less Than Stated Size (Mesh Size in μm)									
	+4750	-4750 +4000	-4000 +2800	-2800 +2000	-2000 +1000	-1000 +850	-850 +425	-425 +355	-355 +250	-250
UL-58	100	96.5	86.9	69.2	49.9	24.9	22.0	10.4	9.0	7.2
UL-64 (a)	100	85.5	70.5	51.8	36.5	20.5	17.8	11.4	10.4	9.1
UL-65	100	89.6	76.9	60.3	44.1	25.0	20.8	10.8	8.9	7.1
UL-66	100	87.7	75.4	62.5	50.4	31.9	27.0	13.6	12.2	10.2

(a) Blockage in oversize chute resulted in oversize discharge through screener. Data are invalid for comparison with other tests.

unfueled elements (Ref. 2-1). Both of these types of products contained little or no material (i.e., 0% to 0.2%) greater than 4750 μm in size. (Because of the upset condition in run UL-64, data from this run were disregarded.) Estimated burner feed requirements call for less than 5% material greater than 4750 μm and an approximate minimum of 63% material less than 2800 μm in size. Both of these conditions are easily met by the current screener products (Table 2-4) and by a combination of the screener and oversize crusher products in the current ratios observed in the Phase III tests.

The oversize crusher products obtained thus far were essentially the same in percentages of material from each of the screened fractions below 2000 μm for both types of fueled elements. However, the product from the standard FSV H-327 graphite element tends to be finer (i.e., smaller percentages of the largest material) above 2000 μm , resulting in much less material greater than 4750 μm in size.

The product size distribution trends will be evaluated more thoroughly and represented graphically in a future quarterly report after the Phase III tests are completed.

Analysis to determine particle breakage during the crushing operations is currently in progress. Analysis of breakage prior to crushing (i.e., through rod loading) and particle exterior contamination is pending. However, results from the first test (UL-64) indicate that partial breakage will be within reasonable bounds. In this test the total breakage was $\sim 2.7\%$ of the fissile and $\sim 0.3\%$ of the fertile particles.

2.3. CONCLUSIONS

Two methods are available for avoiding preferential discharge from equipment nearest the product collection bunker and ensuring complete discharge and pneumatic transport of the crushed product: a slot valve restriction in the oversize discharge and, as a backup, intermittent operation of the oversize crusher.

Crushing of fueled HTGR elements thus far has presented no major problems. Dust containment is complete, and material accountability has been well within limits. Product size distributions fall within the estimated limits of primary burner feed requirements, and particle breakage is within reasonable bounds.

REFERENCE

- 2-1. "Thorium Utilization Program Quarterly Progress Report for the Period Ending May 31, 1977," ERDA Report GA-A14441, General Atomic Company, June 1977.

3. CRUSHED FUEL ELEMENT BURNING

3.1. SUMMARY

Two runs of extended duration, high bed carbon operation were made on the 0.40-m burner. Results indicated that either in-bed or above-bed fines recycle with the parallel, pressurized hopper pneumatic transport system was acceptable for fines burning. The above-bed recycle resulted in less fuel particle breakage and buildup of noncombustible oxides/hulls in the fines system and was thus selected as the preferred injection location.

The 0.20-m burner tube was repaired, and all other components for the system upgrading were either ordered or are being fabricated.

3.2. INTRODUCTION

The results of short-term 0.40-m burner tests completed in the previous quarter (Ref. 3-1) indicated that several major equipment revisions were necessary to make the system more reliable during long-term operation. However, two major unknowns relating to the fines system remained after completion of these tests. The first was equipment reliability, and the second was the capacity of the system. The system's ability to recycle and burn the large mass of fines generated during the extended, high bed carbon operation necessary when the startup bed and feed material are ~85 wt % carbon had never been tested. The fines system had been confirmed only in operation with beds containing <50 wt % carbon. Tests involving several hours of operation at >50 wt % carbon were needed before the fines system could be considered reliable for future extended cycle use. Both in-bed recycle and above-bed recycle were tested under these operating conditions to determine which, if either, alternative provided satisfactory overall performance.

The 0.20-m burner wall failure and its implications were discussed in the previous quarterly report in terms of limitations in heating short, defluidized beds (Ref. 3-1). The activity in this reporting period centered around repair of the burner tube and concurrent upgrading of the burner system equipment. The configuration of the induction heater, the heater control system, and the operating procedures were modified to improve the capability to heat short beds. Also, the ability to preheat the inlet gas was added to the system design to provide flexibility and safety for operation with varied bed heights.

3.3. 0.40-m PRIMARY BURNER

3.3.1. Extended Combustion Tests with Fines Recycle

3.3.1.1. Introduction. The quantitative results of two runs which tested in-bed (Test C) and above-bed (Test D) fines recycle during extended, high carbon operation are described in Section 3.3.1.2, and conclusions based on these results are discussed in Section 3.3.1.3. These runs were the first tests at the most demanding conditions for the fines recycle system: an extended period of high carbon operation, simulating the first cycle in a primary burner operating campaign (Ref. 3-2).

A total feed quantity equivalent to eight crushed LHTGR type BISO/TRISO loaded blocks was used in each run (1.5 for a startup bed and 6.5 as feed). Tests C and D completed the preparations necessary for the 20-block burning campaign of Test E, as part of sequential operation of the head-end pilot plant. The results, together with the quantitative results of Test B (low carbon operation with fertile TRISO particles and in-bed fines recycle), indicated the preferred injection location and operating technique for fines recycle in future 0.40-m burner operation.

The qualitative results of Test B and the first attempt at performing Test C (C.1) were discussed previously and led to recommendations to (1) install an above-bed burner wall penetration for O_2 injection, (2) replace

the fines system bellows, which were high risk failure areas, (3) alter the fresh feed bunker outlet piping to avert feed bridging, and (4) revise the scope of operations planned for completion of Test C to better assess the existing in-bed, pressurized fines recycle system capabilities in long-term operation (Ref. 3-1). The results of the completion of Test C (C.2), an extended high carbon burning operation with in-bed fines recycle and mid-reactor O_2 injection, specifically concern these recommendations and are discussed in Section 3.3.1.2.

The particle breakage and resulting buildup of noncombustible oxides/hulls in fines recycled by pressurized, in-bed (vertex) injection are discussed in Ref. 3-1. The buildup of such noncombustibles was thought to be detrimental to fines burning efficiency in Test B. This fact and the generally negative condition of high particle breakage with in-bed recycle led to a decision to test the 0.40-m burner mid-reactor penetration as a concurrent above-bed fines and O_2 injection location. In previous tests, pressurized, above-bed and in-bed fines recycle had been shown to be equally effective in burning fines. Above-bed recycle had the benefit of less bed carryover and particle breakage (Ref. 3-3). These tests were done in the 0.20-m primary burner and required confirmation in the 0.40-m system. Test D was therefore planned as an operation similar to Test C, except that fines were injected above the bed instead of to the vertex (through the bed).

Figures 3-1 through 3-21 depict critical process parameters during these runs. They illustrate that Test B was a short-duration test of ~ 4 hr at low burn rate interrupted by a cool-down and restart. Tests C and D were extended tests with ~ 17 hr burning at high burn rates. Test C was shut down and restarted in consecutive days, while Test D was operated continuously. The other major operational difference between the tests was that fines were tailburned in Tests B and D, whereas fines recycle was stopped prior to Test C tailburning. (In-bed fines recycle tailburning with BISO/TRISO beds had been documented, whereas above-bed fines recycle tailburning with BISO/TRISO beds had not been done prior to Test D.)

3.3.1.2. Experimental Results. The discussion of Tests B, C, and D is divided into sections in which the pertinent results from each test are compared. The areas covered are:

1. Fines recycle.
2. Product characteristics.
3. Temperature distribution.
4. Bed fluidization/cooling.
5. Off-gas composition.
6. Feeding.
7. General equipment performance.

3.3.1.2.1. Fines Recycle

System Operability

The parallel, pressurized hopper fines transport system operated acceptably in all three tests. One significant transport difference was noted in the tests. The fines hopper transport pressure ranged from 70 to 83 kPa during the in-bed recycle tests, while the above-bed recycle transport pressure ranged from ≥ 83 to ≤ 138 kPa. Tests B and C used in-bed recycle via an ~ 1.4 -m length of 14-mm-i.d. tube and flex hose. The backpressure on the recycling fines (the fines hopper pressure during transport) included the minimal backpressure of this short transport line and the more significant backpressure of the fluid bed itself. Test D used above-bed recycle via an ~ 5.3 -m length of 14-mm-i.d. tube, ~ 3.4 m of which was a vertical riser. Hence, the bed backpressure was avoided, but the long transport line caused more backpressure with above-bed transport. The highest backpressure in above-bed recycle occurred during the period of peak fines inventory, while the lower backpressure occurred at the beginning and at the end of the test when the fines mass was low.

Fines Burning Efficiency

Fines burning efficiency for in-bed recycle was similar to that for above-bed recycle during the last part of Tests C and D when bed carbon was <50 wt %. The in-bed recycle of Test C was apparently more efficient than the above-bed recycle of Test D during the early period with >50 wt % bed carbon, since level detectors in the fines hoppers indicated a larger fines inventory in the early period of Test D than during the same period in Test C (see Figs. 3-1, 3-2, and 3-3 for bed carbon trends in Tests B, C, and D). Both techniques of fines recycle burned fines at a rate such that the volume of the existing fines system was never exceeded.

The difference between high carbon fines burning in Tests C and D may be discussed in light of the difference in quantities of mid-reactor O_2 flow used. (As shown in Fig. 3-4, no mid-reactor gas flow was used in Test B.) Figures 3-5 and 3-6 show the levels of O_2 flow in Tests C and D and indicate when mid-reactor O_2 flows were altered. The peak mid-reactor O_2 flow in Test C was $0.065 \text{ std m}^3/\text{min}$, added during the plenum O_2 ramp increase as the burn rate, fines generation, and burner system temperatures equilibrated. A similarly small amount of mid-reactor O_2 was added during the plenum O_2 ramp of Test D, but the fines buildup noticed after ~ 1 hr of operation at these O_2 levels indicated that this mid-reactor O_2 level was insufficient for above-bed recycle. The increases in vertex O_2 to $\leq 0.25 \text{ std m}^3/\text{min}$ which occurred early in Test C were replaced by increases in mid-reactor O_2 (see notes 1 and 3, Figs. 3-5 and 3-6). The fines inventory in Test D was relatively large at this point and required ~ 4 hr of operation before being reduced below a mass detectable by level devices in the fines system. After the Test D bed carbon weight percent had been reduced to <<50 wt %, the mid-reactor and vertex O_2 levels were altered to levels similar to those used throughout Test C (see note 4, Fig. 3-6). In contrast, mid-reactor and vertex O_2 had to be reduced for an ~ 1.5 -hr period during the >50 wt % carbon period of Test C when fines inventory reached such a low level that burner process conditions were destabilized. The full O_2 levels were reestablished and were held for the remainder of Test C after the recycling fines mass built back up (see note 4, Fig. 3-5).

Fig. 3-1. Off-gas concentration and bed carbon content in 0.40-m primary burner Test B

Fig. 3-2. Off-gas concentration and bed carbon content in 0.40-m primary burner Test C

Fig. 3-3. Off-gas concentration and bed carbon content in 0.40-m primary burner Test D

Fig. 3-4. Inlet gas flow in 0.40-m primary burner Test B

Fig. 3-5. Inlet gas flow in 0.40-m primary burner Test C

Fig. 3-6. Inlet gas flow in 0.40-m primary burner Test D

Process Effects of Fines Recycle

Fundamental differences between non-recycle and recycle periods of the tests and between in-bed and above-bed fines recycle were noted when certain process parameters were compared. The differences involved the final products of the run, the burner system temperature distribution, the bed cooling character, and the off-gas composition. In summary, it was noted that (1) in-bed recycle resulted in >4 wt % particle breakage in recycle to a TRISO bed and >5 wt % particle breakage in recycle to a BISO/TRISO bed compared with <1 wt % breakage with above-bed recycle to a BISO/TRISO bed; (2) in-bed temperatures were cyclically cooled by both in-bed and above-bed fines transport cycles, with the former having the larger effect; (3) the bed cooling air requirements were reduced by high rates of fines recycle, especially recycle through the bed; and (4) fines recycle generally increased off-gas CO volume percent levels and caused intermittent spikes and fluctuations of ± 5 vol % CO. These areas are discussed further in the following sections.

One process parameter response to fines recycle which requires separate mention is the bed pressure drop measured from taps into the vertex gas inlet and the burner off-gas outlet. The bed ΔP trends are plotted in Figs. 3-7, 3-8, and 3-9. The effect on the bed ΔP instrument of the short time lag in establishing fines flow when switching between hoppers is significant. During fines flow the bed ΔP indicates a higher level than between fines flow. This was used as an accurate monitor of the fines cycle in Tests B and C. The bed ΔP during the above-bed fines recycle of Test D shows the normal trend (see Fig. 3-9).

3.2.1.2.2. Product Characteristics

The final product from Tests B, C, and D included the "product bed" which was pneumatically transported out of the burner, the "plenum" material which had been forced through the distributor plate during the slugging bed operation, and the "final fines hopper" material. Material

Fig. 3-7. Bed, filter, and cyclone pressure drops (ΔP) in 0.40-m primary burner Test B

Fig. 3-8. Bed, filter, and cyclone pressure drops (ΔP) in 0.40-m primary burner Test C

Fig. 3-9. Bed, filter, and cyclone pressure drops (ΔP) in 0.40-m primary burner Test D

withdrawn in "fines samples" during Test C and D operation gave indication of the fines composition trends leading up to the final fines hopper material.

The fines recycle particle breakage values of >4 , >5 , and <1 wt % mentioned previously were for Tests B, C, and D, respectively. The material balance summary for these three runs (Table 3-1) indicates the total particle breakage magnitude as particle loss (item 22). The recycle breakage estimate calculated as shown in Table 3-1 (item 23) is the fraction of that particle loss which is attributable to operation of the primary burner. The difference between item 22 and item 23 is an estimate of breakage due to pneumatic transport of the product bed. This analysis is based upon screening; the presence of TRISO hulls in the $+355\text{-}\mu\text{m}$ particle fraction biases the data toward less particle breakage than actually occurred. Microscopic examination of the $+355\text{-}\mu\text{m}$ fractions indicated this to be especially true in the case of Tests B and C. Another biasing factor is that the pneumatically transported product breakage estimate would include some material broken within the fluid bed operation but not yet elutriated into the fines system or pounded into the plenum. However, the overall magnitudes are believed to be sufficiently valid to indicate the general trends.

The smallest total particle loss was 1.8 wt %, which occurred in Test D with above-bed recycle to a BISO/TRISO particle bed. About 1 wt % of the loss occurred in ambient product pneumatic transport as proportionally small quantities of $-355\text{-}\mu\text{m}$ oxides/hulls were found in the plenum or in the fines relative to those found in the transported product. The BISO/TRISO particle loss in the in-bed recycle of Test C was, in contrast, predominantly noted in the plenum and final fines hopper and fines sample fractions (see items 16 and 18, Table 3-1). In-bed fines recycle to the pure TRISO coated particle bed of Test B yielded the highest particle loss of the three tests. The proportionally highest loss as judged by the $-355\text{-}\mu\text{m}$ oxides/hulls was in the transport of the lower 30 wt % of the fluid bed at high ($\sim 650^\circ\text{C}$) temperature. This "hot" product removal particle breakage

TABLE 3-1
MATERIAL BALANCE SUMMARY FOR 0.40-m PRIMARY BURNER TESTS B, C, AND D

Item (a)	Test B		Test C	Test D	Comments
	Hot	Cold			
Combustibles Balance					
1. Carbon in (kg)	82		735	750	1. --
2. Product bed; +850 (kg)	0.04	0.0	0.329	0.065	2. --
3. Product bed; -850, +355(kg)	4.10	1.50	0.665	0.366	3. --
4. Product bed; -355 (kg)	1.20	0.48	2.957	0.033	4. Carbon fines
5. Plenum; +355 (kg)	0.34		1.655	1.033	5. Unburned whole particles
6. Plenum; -355 (kg)	0.22		6.631	5.050	6. Carbon fines and unburned broken particles
7. Final fines hopper; +355 (kg)	0.02		0.012	0.002	7. Graphite, unburned particles
8. Final fines hopper; -355 (kg)	6.30		10.40	0.923	8. No fines tailburn in Test C
9. Total carbon unburned (kg)	14.2		22.65	7.472	9. Sum of 2 through 8
10. Carbon out in fines samples (kg)	None		10.47	11.061	10. --
Noncombustibles Balance					
11. Particles in (kg)	268		197	415	11. --
12. Product bed; +850 (kg)	0	0	0.033	0.260	12. ~0.1 kg of -2000-μm agglomerates; only in Test D
13. Product bed; -850, +355 (kg)	69.1	156	105.5	328.4	13. Particles (some hulls)
14. Product bed; -355 (kg)	6.29	6.02	2.279	2.947	14. Hulls and oxides (some particles)
15. Plenum;+355 (kg)	5.19		78.84	79.01	15. Particles (some hulls)
16. Plenum;-355 (kg)	0.40		10.63	1.290	16. Oxides and hulls
17. Final fines hopper;+355 (kg)	0.51		0.041	0.099	17. Particles and hulls
18. Final fines hopper;-355 (kg)	4.99		1.280	0.276	18. Oxides and hulls
19. Noncombustibles out in fines samples (kg)	None		0.558	0.071	19. Oxides and hulls
20. Total oxides/hulls out; -355 (kg)	17.7		14.7	4.6	20. 14 + 16 + 18 + 19
21. Total particles out; -850,+355 (kg)	231		184.4	407.5	21. 13 + 15 + 17
22. Particle loss $\left(\frac{\text{No. 11} - \text{No. 21}}{\text{No. 11}}\right) \times 100$ (wt %)	13.8		6.4	1.8	22. Tests B and C low when compared with No. 20 - oxides/hulls
23. Recycle breakage estimate $\left(\frac{\text{No. 16} + 18 + 19}{\text{No. 20}}\right) \times 100$ (wt %)	4.2		5.4	0.6	23. --
24. Final bed carbon $\left(\frac{\text{No. 2} + 3 + 4 = C}{C + \text{No. 12} + 13 + 14}\right) \times 100$ (wt %)	6.6	1.2	3.5	0.14	24. Particle carbon <1 wt % except Test B - Hot (~6%)

(a) 850 and 355 are micron screen sizes.

was twice the "cold" product breakage (removed at ambient temperature) on a weight percent basis (compare item 14 with item 13, Test B, Table 3-1). The large quantity of oxides/hulls in the final fines hopper of Test B is discussed in Ref. 3-1 as a possible inhibitor to fines tailburning efficiency. The short-duration Test B operation yielded the smallest plenum content of noncombustibles of the three tests.

Final bed burnout was best in Test D, in which only 0.14 wt % final bed carbon remained (item 24, Table 3-1). The bed was tailburned using induction heating assistance in Tests B and D, but Test C was tailburned without induction heating. With the exception of the hot product bed of Test B, it was noted that in those final beds in which carbon was >1 wt %, the particle fraction -850,+355 was actually <1 wt % carbon (item 3, Table 3-1). The higher carbon in the particle fraction of the hot product bed in Test B apparently resulted from carbon inside TRISO particles which were cracked or partially broken in transport.

Carbon in recycling fines was tailburned to the minimum levels possible only in Tests B and D. The 6.3 kg of carbon remaining in the fines of Test B after the tailburn (item 8, Table 3-1) was mentioned above in the context of burning being inhibited by the 4.99 kg of noncombustible fines also recycling. The tailburn in Test D resulted in only 0.923 kg of remaining carbon fines mixed with 0.276 kg of noncombustible fines. The 10.4 kg of carbon fines mixed with 1.28 kg of noncombustible fines in Test C indicates the in-bed recycling fines composition when entering the tailburning phase and the generally higher concentration of oxides/hulls (and particles) in circulating fines relative to that in the above-bed recycle of Test D (compare Figs. 3-10 and 3-11).

A final observation regarding the characteristics of the product was that small ($\leq 2000 \mu\text{m}$) agglomerates of particles (see Fig. 3-12) were present in small quantities (<0.100 kg, item 12 in Table 3-1) in Test D. Agglomerates such as these are formed when a local area of marginal mixing occurs coincidentally with the presence of molten metals. Such metals stem

Fig. 3-10. Recycling fines oxide/hull content and fines sample history for 0.40-m primary burner Test D

Fig. 3-11. Recycling fines oxide/hull content and fines sample history for 0.40-m primary burner Test C

Fig. 3-12. Primary burner BISO/TRISO fuel particle agglomerates (typical)

from low melting eutectic compounds which are formed from trace levels of impurities released from the broken particles. Similar eutectics may be formed when fission products, present in irradiated particles, are released from broken particles. The local area of marginal mixing is discussed in Section 3.2.1.2.4.

The material size distribution of the feed and products from Tests B, C, and D are given in Figs. 3-13, 3-14, and 3-15, respectively.

3.3.1.2.3. Temperature Distribution

The in-bed temperature and its response to the two modes of fines recycle are illustrated in Figs. 3-16, 3-17, and 3-18. The $<550^{\circ}\text{C}$ fines act as a heat sink as they enter the $\sim 900^{\circ}\text{C}$ bed and cause a brief cooling period during the peak fines flow period. As one fines hopper empties, a short period of time is required before flow from the other hopper is established. The bed heats until the high flow period of the fines cycle reoccurs. The small mass of in-bed recycling fines in the low carbon Test B caused only a $\pm 10^{\circ}\text{C}$ bed temperature cycle (Fig. 3-16). The much larger mass circulating through the bed in the high carbon operation of Test C caused bed temperature cycles as great as $\pm 30^{\circ}\text{C}$ (Fig. 3-17). The above-bed recycle of Test D, which had the largest inventory of fines of the three tests, yielded maximum cycles of $\pm 15^{\circ}\text{C}$ cycles during the peak flow period (between 1500 and 1800) and $< \pm 10^{\circ}\text{C}$ cycles in the remainder of the test (Fig. 3-18). The loss of fines flow from one of the fines hoppers occurred several times early in Test D when the 0.1-m-i.d. inlet ball valve would bind (apparently due to non-isothermal conditions between the valve ball and body during the approach to equilibrium temperatures). These losses caused the abrupt bed temperature rises shown in Fig. 3-18 (see note 4). The bed temperature would restabilize after the above-bed fines flow was reestablished.

The above-bed temperature recorded about 0.5 m above the mid-reactor O_2 /fines inlet and approximately 2 m above the upper region of the non-slugging bed is shown as thermocouple No. 11 in Figs. 3-16, 3-17, and 3-18.

Fig. 3-13. Material size distribution for 0.40-m primary burner Test B

Fig. 3-14. Material size distribution for 0.40-m primary burner Test C

Fig. 3-15. Material size distribution for 0.40-m primary burner Test D

Fig. 3-16. System temperatures in 0.40-m primary burner Test B

Fig. 3-17. System temperatures in 0.40-m primary burner Test C

Fig. 3-18. System temperatures in 0.40-m primary burner Test D

This temperature rapidly increased and decreased upon starting and stopping of fines recycle, respectively. In the case of in-bed fines recycle, the above-bed temperature was very sensitive to mid-reactor O_2/CO_2 flows (see Fig. 3-17, notes 2 and 4). The above-bed temperature generally was hotter during in-bed recycle than during recycling above the bed. The abrupt above-bed temperature rise during the fines tailburning period of Test D (note 2, Fig. 3-18) is typical of what has occurred in some of the past 0.20-m burner work when in-bed carbon was reduced to a low level. The in-bed O_2 was not utilized, and a large O_2 front abruptly "broke through" the bed and ignited recycling fines in a "flash" burn situation. This burning was returned to normal fines tailburning levels by reducing in-bed O_2 and thus the O_2 reaching the fines (mid-reactor O_2 had been reduced prior to the flash burn; see notes 5 and 6, Fig. 3-6).

The minimum recycling fines temperature as recorded in the fines hoppers is given as thermocouple No. 9 in Figs. 3-17 and 3-18. The low fines volume in Test B yielded a maximum fines hopper temperature of $300^\circ C$, and hence this temperature was not included in Fig. 3-16. The fines hopper temperature peaked at $\sim 570^\circ C$ for the highest fines mass flow period of Test C and at $\sim 530^\circ C$ for Test D.

The in-bed wall temperature is presented as thermocouple No. 15 in Figs. 3-16, 3-17, and 3-18. The general trend, evident especially in the longer Tests C and D, was for the wall temperature to gradually reduce to a 650° to $700^\circ C$ equilibrium temperature. The lower equilibrium temperature applied for the above-bed recycle Test D. Two analogous periods are noted in the Test C and D wall temperature history. The Test C wall experienced an unusual "dip" (increased bed-to-wall ΔT) between about 1500 and 1700 hr. The Test D wall experienced a similar "dip" between 2146 and 2300 hr. The significance of this bed-to-wall ΔT perturbation is discussed in the following section.

3.3.1.2.4. Bed Fluidization/Cooling

The overall bed fluidization quality during heatup and combustion in the three tests was excellent. The 85 wt % TRISO/15 wt % -3/16-in. graphite bed of Test B began heating at 0.33 m/s superficial inlet gas velocity and had an overall bed ΔT of $<10^{\circ}\text{C}$ during the heatup. The 85 wt % -3/16-in. graphite/15 wt % BISO/TRISO particle bed required a slightly higher (~ 0.39 m/s) velocity for heating and had an overall bed ΔT of $<20^{\circ}\text{C}$ during heatup in Tests C and D.

The bed fluidization quality during combustion may be roughly assessed from the in-bed ΔT and the bed-to-wall ΔT and from the quantity of final bed agglomeration (which results partly from poor local mixing). As discussed above, the recycling fines cause a general cycling of the bed temperature. However, the overall bed ΔT can still be a valid monitor of good mixing. The average in-bed ΔT in the tests was 10° to 20°C . The peak in-bed ΔT observed was $\sim 40^{\circ}\text{C}$ and occurred in Tests C and D concurrent with the wall temperature "dips" mentioned in the previous section. The bed ΔT and wall ΔT increase in these runs, occurring at a period when fines inventory was known to be low, indicating that mixing quality degenerated with reduction of recycling fines. Inversely, fines recycle improves in-bed mixing, resulting in better in-bed and bed-to-wall heat transfer. The presence of a minor quantity of agglomerates in Test D may indicate that mixing quality (at least at some local area) is lower when fines recycling takes place above the bed.

The cooling requirements for each run are given in Figs. 3-19, 3-20, and 3-21. Test C required much less external bed cooling air than Test D or B. The temperature distributions discussed previously indicated that the heavy mass of in-bed recycling fines carried more heat axially into the above-bed and fines system zones of the burner. The result was less heat dissipation radially to the cooling air. The cooling air requirements were generally less than expected for the full burn rate in all the tests with fines recycle. The cooling air flow trends showed a general increase through a test.

Fig. 3-19. Bed cooling air flow for 0.40-m primary burner Test B

Fig. 3-20. Bed cooling air flow for 0.40-m primary burner Test C

Fig. 3-21. Bed cooling air flow for 0.40-m primary burner Test D

3.3.1.2.5 Off-Gas Composition

Figures 3-1, 3-2, and 3-3 show the volume percent CO off-gas monitored during Tests B, C, and D, respectively. The volume percent O₂ is also plotted. The remainder of the off-gas was CO₂ and may be obtained by subtraction.

The most significant off-gas result was the response of the CO volume percent to starting and stopping of fines recycle. Starting the cyclic mass flow of fines into the burner would start cyclic spikes and fluctuations in the volume percent off-gas CO (see note 1, Figs. 3-1, 3-2, 3-3). These fluctuations averaged ± 3 vol % with spikes as large as ± 5 vol %. Stopping fines recycle abruptly reduced the overall CO volume percent and resulted in decreased fluctuations (see note 5, Fig. 3-2).

The large CO increase shown in Fig. 3-3 (note 3) occurred during the high temperature "flash" burn of above-bed fines in Test D. In general, the off-gas trends showed increases of CO when temperature and/or fines flow increased.

3.2.1.2.6. Feeding

The fresh feed system experienced no flow inconsistencies during Tests C and D. The mechanism of the bridging which had occurred previously (Ref. 3-1) was apparently removed when the outlet piping of the feed bunker was revised. The rotary valve gave adequate feed metering performance.

The feed bunker maximum capacity during the runs was $\lesssim 1.5$ blocks. This was the high limit of the load cell device weight-measuring range. Burner feeding was interrupted for 4 to 10 min during refill of the feed bunker. Loss of the fresh feed flow into the burning bed caused 10° to 30°C elevations in the bed temperature. The bed temperature was restabilized when feeding resumed.

3.3.1.2.7. General Equipment Performance

The fully enclosed, guided bellows which replaced the ruptured 0.15-m-i.d. fines system bellows (Ref. 3-1) gave good performance through Tests C and D. The 0.10-m-i.d. fines system bellows that were found distorted after the initial Test C attempt (Ref. 3-1) were replaced with flange pipe sections, which also gave trouble-free performance in the operations.

The dual-bellows off-gas line from the burner to the cyclone was noted to be a high-risk failure area during the long-duration tests. The line was initially installed with about 13 mm offset. During the burner tests, this line was heated to about 700°C. The resulting thermal expansion increased the initial offset and caused the fines to impinge on the bellows corrugations. Plans for correcting this problem are discussed in Section 3.3.1.3.

The performance of the single bar linkage which replaced the Peaucellier burner alignment mechanism (Ref. 3-1) is discussed in Section 3.3.2.

The level sensors used in Tests C and D are discussed in Section 3.3.3.

3.3.1.3. Conclusions and Recommendations

3.3.1.3.1. Future Operation with Fines Recycle

The acceptable fines burning and the lower particle breakage obtained with above-bed fines injection indicate that this location for fines recycle is preferable to in-bed injection. The use of rapid increases in mid-reactor O_2 concurrent with the plenum O_2 ramp (in-bed burning increase) is advisable to help curtail the buildup of a large fines inventory early in a test. These increases should raise mid-reactor O_2 to ~ 0.250 std m^3/min as quickly as allowed by maintaining the off-gas O_2 volume percent

at ~2 wt %. Off-gas O_2 concentrations >5 wt % should be avoided, especially in the low bed carbon tailburning portions of the run when above-bed burning is likely.

If it is possible in 0.40-m primary burner equipment revisions, the burner tube length should be extended ~2 m and/or the off-gas line connecting the burner to the cyclone should be extended to give the recycling fines more residence time to react and burn.

If the above-bed fines injection is to continue to use the pressurized hopper system for transport, either the hopper system should be elevated to a position closer to the burner inlet or the 14-mm-i.d. riser line should be increased to ~17 mm i.d. (to decrease the transport hopper backpressure).

3.3.1.3.2. Process Effects of Fines Recycle

Fines apparently enter the bed even when injected above the bed. However, the bed temperature cycling and cooling requirements indicate that the effect of fines on the bed is much larger with in-bed recycle.

The levels of oxides/hulls forced into the gas distributor plenum and the quantity of particles and oxides/hulls in the fines of Test C relative to Test D indicate that the in-bed recycle causes slugging to be severely aggravated.

The cycling of off-gas CO volume percent during the cycling fines flow is apparently inherent to fines burning phenomena. The increase of CO generated by burning fine particles (<100 μm) has been discussed in terms of the faster gaseous diffusion away from the surface of small particles relative to large (>100 μm) particles (Ref. 3-4). The faster gaseous diffusion allows more CO to escape the region of the particle into the bulk gas without being reacted further to CO_2 . (It follows that higher temperatures would also generate faster diffusion and more CO as observed in

these operations.) These spikes and fluctuations may cause instability in the off-gas treatment train, and a surge tank of sufficient volume to contain ~5 min of burner off-gas flow is recommended to even out the volume percent CO concentration.

The degree of bed temperature cycling and the fines hopper temperatures are reliable process monitors of the quantity of fines recycling.

Large quantities of recycling fines apparently improve in-bed mixing and decrease in-bed wall cooling requirements. In-bed recycle appeared to have the greater effect in this respect. The general increase in cooling air required as a test progresses indicates the decrease of this fines recycle effect as the mass of recycling fines diminishes.

3.3.1.3.3. Product Effects of Fines Recycle

The higher breakage due to in-bed recycle causes a rapid oxide/hull inventory accumulation in the circulating fines. This may decrease fines burning efficiency and increase criticality problems in the fines system. These are contributing factors in the decision to recommend above-bed recycle.

The apparent excess of hulls found in the BISO/TRISO tests indicates that more TRISO particles break than BISO particles. Chemical analysis to separate uranium (from TRISO particles) and thorium (from BISO particles) in the product fractions will determine the amount of TRISO versus BISO particle breakage. Air classification of split samples of the product bed and plenum material will further aid in determining the BISO/TRISO particle ratio in the product material.

3.3.1.3.4. Equipment Effects of Fines Recycle

The offset in the dual-bellows off-gas line will be corrected by shortening the horizontal section of the line by 13 mm. A sleeve will be

designed and installed to fit inside the bellows in the vertical run. The sleeve will extend well beyond the convolutions of the bellows to eliminate any possibility of impingement.

The binding of the 0.10-m-i.d. fines hopper inlet valves during the rise of the circulating fines to their $>500^{\circ}\text{C}$ operating temperature was apparently due to the higher temperature of the valve internals relative to the valve body temperature. The valves have been externally insulated to decrease the heat loss and the non-isothermal temperature binding condition.

3.3.2. Peaucellier Mechanism Replacement

As discussed in the previous quarterly report, the Peaucellier alignment mechanism which guides the bottom end of the 0.40-m primary burner was replaced with a single bar linkage. The burner was operated during Tests C and D with the single bar linkage installed. No lateral motion was observed.

The single bar linkage is simpler and less expensive than the Peaucellier cell previously used and functions satisfactorily. It is recommended that this design be considered for all future burner alignment designs.

3.3.3. Level Sensors

3.3.3.1. Introduction. The following requirements were initiated for the 0.40-m primary burner system:

1. Bunker level sensors for the fines east and west bunkers (Fig. 3-22).
2. An overflow level sensor in the fines east and west feed line (Fig. 3-23).

77K1203

Fig. 3-22. Fines hopper level sensor probes for 0.40-m primary burner

FINES OVERFLOW
LEVEL SENSOR

BED LEVEL
SENSOR

77K1205c

MID-REACTOR
 CO_2/O_2 GAS
LINE

14-mm-I.D. ABOVE-BED
FINES RECYCLE LINE

Fig. 3-23. 0.40-m primary burner bed level sensor, mid-reactor gas/fines injection lines, and fines hopper overflow level sensor

3. A primary burner bed level sensor (Fig. 3-23).

4. A fresh feed bunker outlet flow indicator.

A single-element probe similar to those discussed in Ref. 3-1, Section 3.3.4, was used to meet these requirements.

3.3.3.2. Discussion. The level sensors listed as items 1, 2, and 3 were fabricated and installed according to Standard Level Sensor Installation Drawing No. 5190004 (Fig. 3-24). Two of these level sensors (items 1 and 2) and the fresh feed bunker flow indicator sensing elements are contained inside a 6.35-mm-o.d. stainless steel tube. The bed level sensing element (item 3) is contained inside a 12.7-mm-o.d. Inconel 600 tube. The fines bunker level sensors were installed in the inlet pipes approximately 0.3 m above the top of the bunkers and extended 50.8 mm down into the pipes. The overflow level sensor was installed in the fines inlet piping approximately 0.3 m below the "Y" connecting the off-gas cyclone and filter and extended 50.8 mm down into the pipe. The fresh feed bunker outlet flow indicator is located just below the feed star valve for the primary bunker. The sensing element is located in a horizontal plane to provide maximum sensitivity to the moving particulates. The burner bed level sensor was installed through a penetration adjacent to the mid-reactor gas penetration and extended 50.8 mm down into the vessel.

3.3.3.3. Conclusion and Recommendations. The sensors were used in primary burner Tests C and D and satisfactory results were obtained. An electrical problem was encountered with the burner bed level probe during the first test. High temperature destroyed the wiring insulation between the vessel wall and the outer clamshell and shorted out the sensor. This problem was corrected prior to the second burner test.

The sensors will be used in future operations as an aid in monitoring in-bed slugging conditions, fines hopper inventories, and fresh feed flow.

3.3.4. Load Cell Installation

In order to measure dynamic axial loading on the 0.40-m burner tube, it is planned to suspend the burner tube on load cells.

Two simple beams with load cells at each end pick up the lift points of the burner tube. Mechanical jacks on each end of the beam will lift the burner tube off the deck plate and provide the load path through the load cells.

The beams are now in fabrication, and the mechanical jacks have been ordered.

3.3.5. Burner Wall Penetrations

Penetrations for the mid-reactor, above-bed O_2 and the bed level sensors were created in the burner wall and through the exterior insulating shrouds. This work was accomplished without dismantling or disassembly of any burner components and with the burner remaining in place. A hole saw, mounted in a portable drill with a magnetic base, was used to cut through the shroud walls and the insulation. The pilot drill for the hole saw spotted the burner wall for drilling the 19-mm-diameter penetration of the Hastelloy X burner wall. Sawing and drilling for each penetration were done without changing the setup (see Figs. 3-25 and 3-26), and a new setup was made for the second penetration. Contoured Hastelloy X bosses were welded centrally on each penetration and tubular liners were installed and welded into the shroud penetration to complete the modification.

3.4. 0.20-m PRIMARY BURNER TUBE REPAIR AND SYSTEM UPGRADE

3.4.1. Introduction

The burnthrough of the 0.20-m primary burner is described in Ref. 3-1. The burner repair and the status of activities under way to improve the 0.20-m system are discussed below.

Fig. 3-25. Setup for cutting burner wall penetrations

Fig. 3-26. Elevation view of setup shown in Fig. 3-25

The current status of the repair of the 0.20-m primary burner is as follows:

1. Burner tube - The tube has been repaired by sawing out the damaged section and welding in a replacement (a detailed description is given in Section 3.4.2.1).
2. Susceptor/cooling shroud - in fabrication.
3. Off-gas cyclone - on order.
4. Off-gas piping - in fabrication.
5. Fines hopper - in fabrication.
6. Off-gas filter chamber - in fabrication.
7. Product can - in fabrication.
8. Gas distributor - undergoing modifications in-house.
9. Piping and instrumentation drawings - in review cycle.
10. Cooling air piping - in review cycle.
11. Electronic control procurement - all items on order.
12. Structural platform - completed.

3.4.2. 0.20-m Primary Burner Repair

The damaged portion of the burner tube has been removed and replaced with a new section (Fig. 3-27). The work was accomplished without removing or repositioning the burner from its operating position.

0.2-m-I.D.
NEW
SECTION

Fig. 3-27. 0.20-m primary burner repair

The repair plan involved the use of a rented guillotine power hack saw which mounted on the exterior of the burner tube. This saw was used to completely remove the damaged portion of the burner and the unpinned bottom transition section. The latter was salvaged for welding to the new 0.33-m pinned burner section. Figure 3-28 shows the guillotine saw being positioned for the final cuts to remove the ruptured portion of the burner. The transition section has been cut away, and the sawing equipment and the burner will be re-shrouded to eliminate radioactive contamination during cutting. One positioning "V" block and the cutting blade of the power saw are clearly visible in the photograph.

Since the burner tube is currently radioactive, all repair activities were performed by personnel wearing protective clothing and fresh air supply air hoods. All particulate-producing operations except for welding were prohibited or severely controlled. The saw operator wearing the air hood is seen in Fig. 3-29 during the removal of the damaged section. Similar protective equipment was worn by the welder and his helper during assembly welding.

The guillotine hack saw produced an excellent machined surface which was square and perpendicular to the vertical axis of the burner. No reworking of the weld joint surfaces was required during assembly of the burner components. The first assembly of the new Hastelloy X burner section is shown fitted for welding in Fig. 3-30 and finish welded in Fig. 3-31. Next the transition piece was positioned and welded. Figure 3-32 shows the transition section backing weld being made on the interior of the tube. Figure 3-33 shows the completely repaired burner tube ready for thermocouple installation.

Prior to welding the new pinned section to the existing burner tube, a weld test program was undertaken. This was done to determine the existence of any problems associated with welding the carburized interior surfaces of the old Hastelloy X burner tube and transition section to the new Hastelloy X repair section. The program also developed an acceptable repair welding

GUILLOTINE
SAW

DAMAGED
BURNER
TUBE

Fig. 3-28. 0.20-m primary burner repair: guillotine saw being positioned to cut away damaged burner section

GUILLOTINE
SAW
SHROUDED

SAW OPERATOR
IN AIR HOOD
AND PROTECTIVE
CLOTHING

Fig. 3-29. 0.20-m primary burner repair: saw operator and equipment during sawing operation

Fig. 3-30. 0.20-m primary burner repair: new burner section positioned for welding

Fig. 3-31. 0.20-m primary burner repair: upper weld completed between burner and new burner section

Fig. 3-32. 0.20-m primary burner repair: transition section being welded to new burner section; internal backing weld shown

Fig. 3-33. 0.20-m primary burner: completely repaired burner tube

procedure and aided in selection of Inconel 82 as a satisfactory weld filler material. In the test program three types of test welds were made on Hastelloy X material taken from each end of the damaged section of the burner: patch, bevel groove, and bead-on-plate welds. Test welding indicated that 100% weld penetration of the vessel wall from the outside would not be achieved without further experimentation. Since internal backing welds were possible on the burner tube, the welding plan was changed to specify backing welds for each weld joint prior to finish welding the 45° weld bevel.

Metallurgical examination was made of damaged and carburized Hastelloy X material. No evidence was found of possible problems in the micro-structure, and X-ray examination of test welds revealed no cracks or other undesirable defects in or near the welds.

3.4.3. Gas Pre-Heater for 0.20-m Primary Burner

3.4.3.1. Introduction. A gas pre-heater installation has been proposed for testing on the 0.20-m primary burner as a result of (1) mixing problems with small beds and (2) the recommendations of the design evaluation report for the primary burner. A low-cost catalog heater which can be readily installed to test the pre-heater concepts is described below.

3.4.3.2. Discussion. The recent burn-through of the 0.20-m primary burner was attributed primarily to poor mixing of the small BISO particle bed. The ability to pre-heat the fluidizing gas (CO_2/O_2 mixture) would have increased the velocity at the bed entry and might have permitted restart of the full bed mixing. A recommendation of the design evaluation report for the 0.40-m primary burner was to test gas pre-heating as an alternative to induction heating, based on cost and maintainability advantages. Consequently, two designs have been developed: one using a low-cost, readily available electric resistance heater which will pre-heat to 650°C and the other utilizing an induction heater design that will permit pre-heating to 800°C.

3.4.3.2.1. Electric Pre-Heater

A design has been developed which uses a catalog electric heater installation with sufficient flexibility to test the low bed restarting capability and permit a partial evaluation of gas pre-heating as an alternate or supplement to induction heating.

The proposed design is shown schematically in Fig. 3-34. A commercial electric heater is installed in the CO_2/O_2 gas line to the plenum as well as the CO_2/O_2 gas line to the vertex. The heaters are sized to maintain a temperature well in excess of the startup bed temperature for the initially high gas flows (up to $\sim 2.2 \text{ m}^3/\text{min}$) required for cold bed fluidization. As shown in Fig. 3-35, a 22.5-kW power supply will provide the required temperature difference. The estimated pressure drop is 69 kPa at $2.2 \text{ m}^3/\text{min}$ through the heater. The gas pre-heaters will permit a maximum 650°C pre-heat at flows below $1.1 \text{ m}^3/\text{min}$ and some reduction in exit temperature at flows between 1.1 and $2.2 \text{ m}^3/\text{min}$.

3.4.3.2.2. Induction Pre-Heater

A tubular heater has been designed with an outside diameter of 0.1 m and a concentric plug that results in a 1.27-mm annular space for gas flow. The gas velocity is relatively high, resulting in a high transfer coefficient, about $852 \text{ W/m}^2\text{-K}$ at the exit. The heater design is shown in Fig. 3-36 and the temperature profiles are shown in Fig. 3-37.

A heater of this design will heat $0.6 \text{ m}^3/\text{min}$ of CO_2 to 800°C in a heater length of 0.8 m with 17 kW input. The maximum heater wall temperature is 900°C . The heater will utilize the existing induction power supply from the 0.10-m secondary burner.

A thermal analysis of the heater has been completed.

Fig. 3-34. Gas pre-heater installation (bold lines designate new equipment)

Fig. 3-35. Gas pre-heater using existing power supplies

3-55

Fig. 3-36. Induction pre-heater design for 0.20-m primary burner

Fig. 3-37. Induction pre-heater temperature profiles

3.4.3.3. Conclusions and Recommendations. The proposed installation of a commercial electric gas pre-heater on the 0.20-m primary burner will allow evaluation of bed restarting, as well as an alternate system heating technique. If the electric gas pre-heating proves successful, the induction heater may be installed to test pre-heat capabilities to 800°C.

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- 3-3. "Thorium Utilization Program Quarterly Progress Report for the Period Ending November 30, 1976," ERDA Report GA-A14214, General Atomic Company, December 1976, pp. 3-1 to 3-14.
- 3-4. Stula, R. T., D. T. Young, and H. H. Yip, "Interim Development Report for Primary Burning," ERDA Report GA-A13546, General Atomic Company, January 5, 1976, p. 6-35.

4. PARTICLE CLASSIFICATION, CRUSHING, AND BURNING

4.1. SUMMARY

There were no particle crushing or secondary burning tests scheduled during this quarter. The particle crusher was disassembled for inspection.

A design evaluation of the 0.20-m secondary burner was completed. The evaluation included 13 features of the existing burner system.

Air classification tests were performed to complete the qualification testing. This included preoperational component testing, FSV type TRISO/TRISO fuel particle classification, and LHTGR type TRISO/BISO fuel particle classification.

4.2. 0.20-m SECONDARY BURNER SYSTEM DESIGN EVALUATION

4.2.1. Introduction

Possible modification of the existing dry head-end cold pilot plant engineering-scale equipment to reliable and maintainable equipment that is prototypical of the HRDF design has required an evaluation of the existing equipment design. This evaluation has taken into account performance, cost, ease of implementation of changes, impact of changes on the current HRDF design, and customer (ERDA) acceptance. Feasible alternative designs have been similarly evaluated. The entire evaluation has been completed.

4.2.2. Activity

The design evaluation encompassed the following 13 features:

1. Separability of upper and lower cooling shrouds.
2. Remote disconnects of burner assemblies.
3. The concept of a coolant pressure boundary (shroud) external to the vessel.
4. The method of heating the burner vessel and its contents.
5. The method of attachment of vessel thermocouples.
6. The method of fabrication of the susceptor.
7. The type of insulation used for the induction coil.
8. The design of the insulation bonnet assembly.
9. The design of the upper cap assembly.
10. The location of the solids feed entry port.
11. The product removal valve assembly.
12. The separability of the induction coil, susceptor, and insulation.
13. Control of the gap between the susceptor and burner tube.

For each of these features, information was compiled in the following sequence:

1. Technical evaluation of the existing system design.
2. Analysis of the existing system costs.

3. Selection and technical evaluation of alternative system designs.
4. Analysis of alternative system costs.
5. Comparison of the existing and alternative systems using value engineering techniques.

Based on this information, design options for each of the 13 features were selected to give the best "performance" (operability, maintainability, reliability, compliance with design criteria and technical specifications, licensability, etc.). Another set of options was selected to give the best "value" (ratio of performance and installed cost). These options are listed in Table 4-1.

The options given in Table 4-1 were evaluated and selected independently without consideration of interfacing with any of the other features. However, to consider the effect of the best designs in each case on the whole system, two integrated burner system design concepts that constitute the best "composites" of design options were developed. One concept represents the "best value" composite but may include inherent, relatively high development risks of failure to achieve design objectives (Table 4-2). The other concept represents the "minimum risk" composite but may result in only modest improvements in performance or cost (Table 4-3).

4.2.3. Conclusions and Recommendations

Based on the 0.20-m secondary burner system design evaluation, the following conclusions and recommendations are made:

1. This evaluation has led to recommendations for the parallel development of two integrated design concepts for a prototype secondary burner system. One concept utilizes the existing burner heating and cooling subsystems but simplifies a number of other features associated with remote maintenance and burner

TABLE 4-1
BEST OPTIONS FOR DESIGN FEATURES

Design Feature	"Best Value" Option	"Best Performance" Option
1. Cooling shroud separability	Provide an integral welded shroud with bellows.	Same as "Best Value."
2. Remote disconnects	Eliminate middle Grayloc flange. Relocate lower Grayloc flange to cooler zone below distributor. (HETF design)	Same as "Best Value."
3. Method of cooling burner/contents	Use internal gas-cooled tubes.	Existing pilot plant design.
4. Method of heating burner/contents	Blow hot gaseous CO ₂ through bed and external shroud.	Existing pilot plant design.
5. Method of vessel thermocouple attachment	Use spring-loaded thermocouples.	Existing pilot plant design.
6. Method of susceptor fabrication	No conclusion.	No conclusion.
7. Type of induction coil insulation	Use dry fiber blanket (e.g., FIBERFRAX).	Existing pilot plant design.
8. Insulation bonnet assembly design	Use a two-piece insulation assembly	Same as "Best Value."
9. Upper cap assembly design	No conclusion.	Eliminate filter plenum. Provide external blowback system. Use remote top flange. (HETF design)
10. Solids feed entry location	No conclusion.	Top entry.
11. Product removal assembly	Existing pilot plant design.	Existing pilot plant design.
12. Separability of coil, susceptor, and insulation	Use integral construction of coil susceptor and insulation.	Same as "Best Value."
13. Uncontrolled gap between susceptor and tube	Provide an integral welded shroud with bellows.	Same as "Best Value."

TABLE 4-2
"BEST VALUE" INTEGRATED BURNER SYSTEM DESIGN CONCEPT

Method of Cooling Burner - Internal gas-cooled tubes

- Eliminate external shroud.
- Eliminate insulation bonnet assembly.

Method of Heating Burner - Induction heating

- Eliminate susceptor (suscept directly on tube).
- Use dry fiber blanket (e.g., FIBERFRAX) insulation for induction coil.
- Provide integral construction of coil and insulation.

Remote Disconnects

- Eliminate middle remote flange. Relocate lower remote flange to cooler zone below distributor. (HETF design)

Miscellaneous Features

- Use spring-loaded thermocouples on vessel tube.
- Eliminate filter plenum. Provide external blowback system. Use remote top flange. (HETF design)
- Provide top solids feed entry.
- Retain existing product removal assembly valve.

TABLE 4-3
"MINIMUM RISK" INTEGRATED BURNER SYSTEM DESIGN CONCEPT

Method of Cooling Burner - External shroud

- Provide an integral welded shroud with bellows.
- Use a two-piece insulation assembly (cylinder + fixed top section).

Method of Heating Burner - Induction heating

- Investigate methods of fabrication of susceptor.
- Use dry fiber blanket (e.g., FIBERFRAX) insulation for induction coil.
- Provide integral construction of coil, susceptor, and insulation.

Remote Disconnects

- Eliminate middle remote flange. Relocate lower remote flange to cooler zone below distributor. (HETF design)

Miscellaneous Features

- Use spring-loaded thermocouples on vessel tube.
- Eliminate filter plenum. Provide external blowback system. Use remote top flange. (HETF design)
- Provide top solids feed entry.
- Retain existing product removal valve assembly.

operation; this concept involves minimum development risk to achieve design objectives. The other concept modifies the cooling subsystem and also eliminates or simplifies a number of other features associated with heating, cooling, remote maintenance, and burner operation. The modified cooling subsystem uses an internal gas-cooled heat exchanger. This second integrated design concept offers the potential for maximum cost reduction, simplicity of design, and ease of fabrication, but involves significantly greater risk. Therefore, it must be studied in more detail and tested in existing smaller-scale pilot plant equipment before implementation as part of the prototype secondary burner system. Methods of heating and cooling the prototype secondary burner and its contents should be determined before any other prototype design features are selected.

2. Cooling the burner with an internal heat exchanger (vertical array of parallel tubes) may be feasible from a process point of view but involves considerable risk at this time. Nevertheless, this concept warrants further evaluation to verify its feasibility. Several aspects of this concept must be evaluated in detail before a final decision is made:
 - a. Process-side differential pressure. It must be acceptably low.
 - b. Effects of the tubes on slugging, mixing, and other dynamic characteristics of the fluidized bed.
 - c. Means of supporting the tubes and the mechanical effects of the tubes, their supports, and inlet/outlet CO₂ piping on stresses, vibration, bending, erosion, etc.; and the effect of process gases on the corrosion of tubes and other internal components.

- d. Physical interference with and effect on performance of in-vessel filters by tubes and other internal components.

If a more detailed evaluation still indicates this concept is feasible, then it should be tested in both the 0.10-m and 0.20-m (engineering-scale) secondary burners. Based on three experiments with each burner, the cost of implementing this concept on the prototype 0.20-m secondary burner is estimated to be approximately \$40,600 [includes exempt and non-exempt direct labor charges for design engineering, installation, testing, and the cost of sub-prototype heat exchangers for the 0.10-m and 0.20-m (engineering-scale) burners].

3. The induction heating concept, which requires no further development on the secondary burner, should continue to be used. However, if cooling of the burner by an internal gas-cooled heat exchanger (item 2, above) proves to be infeasible and the external cooling shroud is used, then a combined bed and shroud hot gas (CO_2) heating concept should be evaluated further. In particular, the following aspects of this alternative design should be evaluated in more detail in order to reduce the risks inherent in this concept:

- a. Controllability.
- b. Heatup time. It must be compatible with the HRDF operating cycle and production (availability) requirements.
- c. Effects of the hot gas on the bed material, including attrition, elutriation, etc.
- d. Mechanical effects of the hot gas on the vessel and any internals, i.e., stress, vibration, erosion, etc.

If the results of such further evaluation appear promising, this alternative heating concept should then be tested on both the 0.10-m and 0.20-m (engineering-scale) secondary burners. The implementation cost of this concept on the prototype 0.20-m secondary burner is estimated to be approximately \$116,000 [including exempt and non-exempt direct labor charges for design, engineering, installation, testing, and the cost of a hot CO₂ delivery system for the 0.10-m and 0.20-m (engineering-scale) secondary burners].

4. If internal cooling proves to be feasible (coupled with induction heating), then design features No. 1 (cooling shroud), No. 6 (susceptor), and No. 8 (insulation bonnet assembly) should be eliminated from the prototype burner design. If internal cooling is not feasible, then the integrated burner system design concept presented in Table 4-3 should be developed.
5. The mid-vessel remote flange (0.20-m Grayloc remote clamp) should be eliminated from the prototype burner design. The lower remote flange (0.15-m Grayloc remote clamp) should be relocated to a cooler zone below the distributor.
6. A spring-loaded thermocouple and a tube-skin thermocouple should be installed and tested along with the existing vessel thermocouples on the 0.20-m engineering-scale burner. Particular attention should be given to their reliability and accuracy.
7. Methods of handling and installing dry fiber blanket insulation, such as FIBERFRAX, between the induction heating coils and the burner tube should be investigated and implemented once the method of heating the prototype secondary burner is established.
8. Those other features -- upper cap assembly, solids feed entry location, and the integral construction of the induction coil,

insulation, susceptor (if used), and burner tube -- which have already been incorporated into the HETF secondary burner system design should be incorporated into the HRDF design if HETF program testing verifies the feasibility of these designs for hot reprocessing operations.

9. The present design of the product removal valve assembly should be retained at this time for the prototype burner. However, if additional funds become available and schedules permit, two alternative designs should be tested on the 0.20-m secondary burner. These alternative designs include a concept similar to the HETF primary burner product removal valve assembly and a "push-pull" type elutriation system that completely eliminates the valve assembly. In particular, the accountability and operability of these concepts should be tested. Implementation costs for these alternative designs are \$10,000 to \$20,000, based on a requirement of two experiments to test each alternative design.
10. Remote maintenance fixtures should be evaluated in light of the results and recommendations of this study.
11. Future design evaluation studies of the 0.20-m secondary burner should include the following areas:
 - a. Optimization of the burner operating cycle, including heatup based on HRDF availability allocations and plant economics.
 - b. Determination of the optimum specification for unburned carbon in the burner product once leacher and solvent extraction impurity limits are defined.
 - c. In-vessel filter reinforcement.

4.3. CLASSIFIER COMPONENT TESTING

4.3.1. Introduction

The fuel particle classifier takes product from the primary burner and separates fissile from fertile particles. Prior to verifying the ability of the classifier to achieve such a separation, the various components had to be qualified in accordance with the Activity Plan (AP522101).

The following components were included in the qualification testing: filter-bunker, blower, feeder, weigh cells, bunkers, and recycle of fuel particles. A diagram of the classifier is shown in Fig. 4-1.

The results of the component qualification testing are discussed below.

4.3.2. Activity

4.3.2.1. Filter-Bunker

4.3.2.1.1. Test Procedure

The test procedure adopted for the classifier filter-bunker is intended to verify that the pressure drop is acceptable and to preload the filters so that material entering the bunker can be recovered. The results obtained from component testing in the solids handling system (Ref. 4-1) with in-bunker filters showed that no particles greater than 5 μm were passing through the sintered metal tubes. The taking of sample wipes from the filter exhaust line from the classifier filter bunker was felt to be unnecessary. The test procedure was as follows:

1. Using the variable speed blower, the pressure drop at 30% and 60% power was measured. (Note: 90% power was used with the solids handling bunkers, but the classifier filter-bunker is considerably smaller.)

Fig. 4-1. Schematic diagram of fuel particle classifier

2. With the gas flow at 60% power, approximately 2 kg of graphite fines was introduced into the conveying line. The weight added was noted.
3. With the blower off, one complete blowback cycle was actuated. The blower was restarted and pressure drop was measured. This was repeated for 10 complete blowback cycles. The burn-back fines were removed.
4. Steps 2 and 3 were repeated until 95% of the fines added were recovered.

4.3.2.1.2. Results

Step 1: 30% power, $\Delta P = 0.8$ kPa; 60% power, $\Delta P = 2$ kPa.

Step 2: Weight in = 2120 g, ΔP (60%) = 5.6 kPa.

Step 3: After 1 blowback cycle, ΔP (60%) = 2.9 kPa. After 10 blowback cycles, ΔP (60%) = 2.4 kPa. Weight out = 2095 g. Holdup = 26 g.

Repeat of Step 2: Weight in = 2095 g, ΔP (60%) = 4.6 kPa.

Repeat of Step 3: After 10 blowback cycles, ΔP (60%) = 2.7 kPa. Weight out = 2090 g. Holdup = 5 g.

4.3.2.1.3. Acceptance Criteria

Recovery: The Activity Plan calls for >95% after preloading; 99.8% was achieved (2090 out of 2095 g).

Pressure drop: The Activity Plan calls for 2.5 kPa when clean and <5 kPa when blown back. This was achieved.

Efficiency: The Activity Plan calls for 100% efficiency for particles >5 μm . This is the implication from the results obtained in the solids handling system (Ref. 4-2). Furthermore, 99.8% recovery indicates that the filters are efficient.

4.3.2.2. Blower

4.3.2.2.1. Test Procedure

A Brooks Indicating Rotometer is used to measure the flow of air into the classifier. A variable speed blower is used to modulate the air inflow rate. A Diogenes three-mode controller is used to regulate the blower in order to hold a preset air flow.

4.3.2.2.2. Results

Calibration of the rotometer indicated the visual readout was right on specification, but the electronic readout was skewed high 12%. No zeroing capabilities were provided, so the Diogenes controller input zero and full-scale were altered to compensate, thus giving correct visual readout in the control room Diogenes display.

The complete control loop was operated to determine suitable control parameters. These are:

Proportional band: 200%
Reset rate: 6 repeats/minute
No derivative mode

Response tests included blanking off the inlet air flow filter momentarily and watching system transient response. Return to set point was rapid, with stabilization within 10 to 15 sec. Throughout the ensuing TRISO/TRISO and TRISO/BISO classification tests, the air flow never varied more than 1% from the set point, even during initiation of feed addition.

4.3.2.2.3. Acceptance Criteria

The Activity Plan calls for the following:

Maximum temperature: <250°F

Maximum vacuum: 12 in. Hg

$\frac{\text{Field indication}}{\text{Transmitted signal}} = 0.99 \text{ to } 1.01$

Control system: <1% offset

<30 sec response to a 50% step change

All these criteria were met.

4.3.2.3. Feeder. The classifier feeder belongs to the solids handling system. A full description of the test procedure, results, and acceptability is given in Refs. 4-1 and 4-2.

4.3.2.4. Weigh Cells

4.3.2.4.1. Test Procedure

The maximum design basis load, 215 kg, was placed on each of the classifier particle bunkers and removed several times in order to adjust the electronic weigh systems for accuracy and repeatability. Reference 4-2 contains a description of the precautions required to obtain maximum accuracy.

4.3.2.4.2. Results

Initial results were disappointing. Investigation revealed that the outlet bellows on both the particle bunkers were misaligned. After the bellows had been reinstalled in a neutral position, the accuracy of the

weigh cells supporting both bunkers was within $\pm 0.5\%$ of the design load and the repeatability was within $\pm 0.1\%$ of the design load.

4.3.2.4.3. Acceptance Criteria

The Activity Plan calls for an accuracy of $\pm 0.5\%$ of design load and a repeatability of $\pm 0.1\%$. Therefore, the acceptance criteria were fully met.

4.3.2.5. Bunkers

4.3.2.5.1. Test Procedure

The flow rate out of each of the classifier product bunkers must not be too large for the conveying system. The calculated bunker volumes must be compatible with process requirements; i.e., each must be able to contain the maximum volume in the classifier feed bunker.

4.3.2.5.2. Results

The original design was based on the concept of controlling the flow rate out of the bunkers by using an orifice. Although this idea reduces the mechanical complexity of the system, it requires changing the size of the orifice in the following circumstances:

- When changing from BISO to TRISO fertile particles.
- When recycling from one or both of the product bunkers.

The application of small rotary valves was investigated (see Ref. 4-2). Such valves have since been installed and tested.

The component testing in the solids handling system included conveying material into a bunker until the high-level alarm was activated. Hence, the volume was determined experimentally. The design volumes of the bunkers filled from a pneumatic conveying system were based on a concave top

surface. In fact, the surface of the contents is nearly flat, thereby substantially increasing the volume. The volume of the classifier feed bunker was not determined experimentally because such an experiment would have put a severe strain on the fuel particle inventory. Based on experimental results with other bunkers, the classifier feed bunker volume was calculated as follows:

● Design volume	0.082 m ³
● Actual volume (calculated)	0.130 m ³

The classifier product bunkers are identical (see Fig. 4-2). The angle of repose, shown by a broken line, is assumed to be 45°. This is a conservative figure, since the actual value is likely to be less than 30°. The volume of the bunker is 0.150 m³.

4.3.2.5.3. Acceptance Criteria

The Activity Plan calls for complete discharge with less than 0.23 kg holdup. This was observed during recycle tests. The capacity required is the fuel particles in 9.1 fuel elements, which corresponds to 0.082 m³, which is also the design basis volume of the classifier feed bunker. Calculations have shown that the particle bunkers are large enough to contain more than the maximum volume of the feed bunker and that they are therefore acceptable.

4.3.2.6. Recycle of Fuel Particles

4.3.2.6.1. Test Procedure

The recycle system is used to return the classified (or misclassified) products from the fissile or fertile product bunkers to the feed bunker for reclassification (see Fig. 4-1). The recycle system was tested to verify its suitability for transporting fuel particles from the product bunkers to the feed bunker while determining approximate fuel particle breakage.

Fig. 4-2. Classifier product bunker

4.3.2.6.2. Results

Two types of recycle tests were conducted. In the first, approximately 50 kg of uncoated BISO fuel particles were recycled from the fertile bunker at 0.23 kg/s feed rate over a range of transport velocities. Pressure drop and temperature data were collected during the tests and compared with the predicted system pressure drop for the same feed rate. The results are shown in Fig. 4-3. Fuel particle breakage was observed to be approximately 2%. Particle breakage results are considered conservative since the transport conditions were varied over a wide range to attain maximum system performance data.

The other type of recycle test consisted of transporting 25 kg of uncoated TRISO fuel particles from the fissile product bunkers to the feed bunker over a range of transport velocities. Figure 4-4 compares the observed and predicted pressure drop results for a feed rate of 0.065 kg/s. The poor correlation of observed and predicted data was attributed to the low feed rate. A conservative particle breakage value of 7% was observed.

4.3.2.6.3. Acceptance Criteria

The acceptance criteria call for suitable transport from either product bunker to the feed bunker. This requirement was met.

In addition, a somewhat arbitrary value for acceptable particle breakage of $\leq 2\%$ is to be verified. The BISO test met this criterion and the TRISO test did not; however, as previously stated, the tests were not performed under optimum conditions for particle breakage.

4.4. TRISO/BISO FUEL PARTICLE CLASSIFICATION

4.4.1. Introduction

The fuel particle classifier Activity Plan (AP522101) includes a series of tests on TRISO/BISO fuel particle blends to verify scale-up from

Fig. 4-3. Observed versus predicted pressure drop results for classifier recycle system using uncoated BISO fuel particles fed at 0.23 kg/s

Fig. 4-4. Observed versus predicted pressure drop results for classifier recycle system using uncoated TRISO fuel particles fed at 0.065 kg/s

the unit operations equipment to the prototype level. These tests have been completed, and less than 1% crossover took place either way over the entire range of fuel blends tested. This is an improvement over earlier classification work on the modular classifier (Ref. 4-3) used in the unit operations tests.

4.4.2. Experimental

In initial shakedown tests, the classifier vacuum tended to draw air and particles through the feeder from the bunker with a subsequent loss of control of the feed rate. This was remedied by installing a vent line from the classifier to the feed bunker, which allowed air to be drawn through the 0.025-m vent line instead of the feeder.

The solid rate sensor had been eliminated in earlier testing due to problems caused by particle jamming and wide data scattering. It was necessary for these tests to obtain the feed rate from the time rate of feed bunker weight loss, using a calibrated weigh cell system.

Gas flow up through the column was obtained from an indicating rotometer that was checked against a calibrated rotometer before the test series began.

Feed material was burned-back, TRISO coated, WAR depleted uranium particles and ThO_2 BISO kernels blended into 63 wt % TRISO, 22 wt % TRISO, and 6.2 wt % TRISO batches to cover the high and low limits as well as the average core composition. Physical properties are given in Table 4-4 for both particle types. Table 4-5 contains complete information on each test run during these experiments.

Crossover of fuel particles was determined by both visual microscopic examination and chemical titrimetric techniques. Results obtained using these two methods correlated very well.

TABLE 4-4
MATERIAL CHARACTERISTICS

BISO kernels:	Mean diameter	523 μm ,
		standard deviation = 18 μm
	Particle density	= 9.60 g/cm^3
TRISO WAR particles:	Mean diameter	= 548 μm ,
		standard deviation = 40 μm
	Particle density	= 2.26 g/cm^3

TABLE 4-5
RUN PARAMETERS AND RESULTS FOR TRISO/BISO FUEL PARTICLE CLASSIFICATION TESTS

Run	Wt % TRISO	Batch Weight (kg)	Feed Rate (g/min)	Gas Velocity (m/s)	Wt % TRISO in BISO Stream	Wt % BISO in TRISO Stream
1	63	20	850	3.4	No classification	
2	63	20	850	5.1	1.9	0.4
3	63	20	900	6.9	0.3	2.1
4	63	20	940	7.6	0.03	3.4
5	22	28.5	1700/1000	7.6	0.01	7.1
6	6.2	20	1800	7.6	0.02	15.3
7	6.2	20	1900	6.0	0.11	0.01
8	22	24	1400	6.0	0.03	0.9
9	63	6.3	920	6.0	0.05	0.3

Twenty kilograms of the 63% TRISO feed was tested first at 3.42 m/s gas velocity (as specified in the Activity Plan). The maximum feed rate possible was 850 g/min or the equivalent of ~ 3 FEs/hr. This low feed rate yields a dilute gas-solid suspension, so no variation of feed rate was needed to improve classification. Full output feeder operation was used throughout the rest of the experiments. However, a higher gas velocity was evidently required, because all the material fell through the column to the bottom of the air classifier at this velocity.

Investigation to determine the reason for this poor performance led to checking the calibration of the rotometer on the unit operations modular classifier. The calibration was found to be low by $\sim 25\%$, which meant that more gas was flowing through the modular classifier than the rotometer indicated. Since this earlier work was used to set the gas flows in the air classifier Activity Plan, low gas flows resulted with subsequent poor classification.

The terminal velocity was calculated to be 4.4 m/s for the TRISO particles and 9.3 m/s for the BISO particles. The flow was raised 50% to 5.1 m/s velocity so it would be at least high enough to carry the TRISO particles over the top. The feed was remixed after sampling to yield an identical 20-kg batch of feed material for the next test. Particle feed rate was again 850 g/min at the maximum feeder setting. The increased gas flow was effective and resulted in a clean overhead stream of TRISO particles (2% of which went through the cyclone to the filter chamber), but the BISO stream was contaminated with $\sim 2\%$ TRISO particles.

Gas flow was increased in the next two runs while a constant feed composition was maintained. Subsequently, the feed composition was varied at two different gas flow rates. Details and results are given in Table 4-5.

It can be seen that keeping the bottom, or BISO, stream clean and relatively free of contamination from TRISO particles was no problem

throughout the tests, but the overhead stream of TRISO particles tended to be more susceptible to contamination with BISO particles. At the 7.6 m/s gas flow rate, significant amounts of whole BISO particles were carried over into the TRISO stream. During operation at 6.0 m/s, the only BISO material carried over was broken particles, chips, and dust. It may therefore be concluded that under proper gas flows and feed rates, the crossover in the TRISO stream will be strictly a function of BISO particle breakage in the classifier feed stream. Thus, any BISO particle breakage in the flowsheet prior to the classifier will result in crossover. It can also be concluded that under the proper operating conditions, the BISO stream will never contain over 1% TRISO particles, since the lighter, broken TRISO particles stay with the entrained TRISO stream.

It is of interest to note that with a 22 wt % TRISO particle mixture (the average contained in LHTGR blocks), the average rate of feed addition at full feeder output was 1400 g/min, or the equivalent of 5 fuel blocks/hr. This is sufficient to supply ten 0.40-m primary burners -- obviously a very large capacity.

4.5. TRISO/TRISO FUEL PARTICLE CLASSIFICATION

4.5.1. Introduction

Activity Plan AP522101 specifies a series of air classification tests using a FSV type TRISO/TRISO fuel particle mixture. The material contains enriched uranium and is from a primary burning-classification experiment performed in 1974 using the 0.10-m primary burner and the modular air classifier. A comparison was to be made between results from the earlier classification tests and current results obtained on the prototype air classifier.

4.5.2. Experimental

Feed material for the first four runs was split out of the same batch, while the fifth run used a different batch. Feed rates were adjusted using

the Syntron vibratory feeder controls. The actual feed rate was measured using a calibrated weigh hopper. Gas flow through the column was held constant using an automatic feedback control loop with a rotometer sensor and a variable speed blower for a final control element.

Overall material balances were as follows:

$$\text{Total material } \frac{\text{out}}{\text{in}} = 0.997$$

$$\text{Thorium } \frac{\text{out}}{\text{in}} = 1.052$$

$$\text{Uranium } \frac{\text{out}}{\text{in}} = 1.046$$

4.5.3. Results

The earlier modular classifier tests were completed in August 1974, and the material was kept in storage during the 3-year interim period prior to its use for the prototype classifier tests. Table 4-6 shows some representative results obtained with the modular classifier. Comparison of these results with those for the prototype classifier given in Table 4-7 shows considerably greater crossover was experienced with the prototype unit. Since comparable results on TRISO/BISO fuel classification showed improved classification with the prototype unit, the reasons for the greater crossover of TRISO/TRISO particles in the prototype unit are not clear at this time. Although the larger-than-expected crossover results for TRISO/TRISO particles were disappointing, crossover in both the fissile and fertile streams indicated that the gas velocities used were satisfactory and little improvement could be achieved with different flows. These tests indicated that secondary classification at a minimum would be required to achieve satisfactory particle separation.

The recommended test program for FY-78 on TRISO/TRISO fuel classification includes:

1. Studies with the full range of blends of TRISO ThO_2 /TRISO WAR fuel particles, which may become the reference fuel system.

TABLE 4-6
MODULAR CLASSIFIER TEST DATA

Feed Rate (g/min)	Air Velocity (m/s)	Fissile Stream Contamination (%)	Fertile Stream Contamination (%)
86	4.3	2.8	5.9
155	4.3	0.6	4.8
182	5.3	19.0	12.2

TABLE 4-7
PROTOTYPE CLASSIFIER TEST DATA AND RESULTS

Run	Batch Weight (g)	Feed Rate (g/min)	Air Velocity (m/s)	Fissile Stream Contamination (%)	Fertile Stream Contamination (%)
1	2782	90	4.3	17	17
2	2804	181	4.3	3.2	17
3	2819	222	4.3	45	15
4	2817	423	4.3	21	32
5	2467	175	5.3	14	14

2. Studies with the full range of blends of FSV type fuels using depleted uranium FSV TRISO fissile and reject FSV TRISO fertile fuel particles.

TRISO/BISO classification will be done only as a part of the sequential pilot plant operations or on a service basis for other operations.

4.6. PARTICLE CRUSHING

As discussed in the last quarterly report (Ref. 4-4), the fertile crusher experienced bearing and motor problems. As a result, the unit has been dismantled and made available for study.

The unit was carefully disassembled and assemblies and components were examined for evidence of failure, wear, or other contributory causes of the problems encountered with bearing overheating and motor overloads. The two bearings with reported difficulties have been forwarded to the bearing manufacturer's laboratories for analysis. Grease samples from each bearing were tray burned in a muffle oven to remove all hydrocarbons, graphite, and fuels and to expose metallic and silicon carbide residuals. This information, though inconclusive, was furnished to the bearing vendor along with other pertinent information on the problem bearings.

The boron carbide (B_4C) insert implanted in one side plate in the region of highly localized wear was closely examined after the recent crushing of 180 kg of particles. The surface of the insert appeared to be burnished in a characteristic pattern, but there was no measurable wear. The other insert of K703 material did not show evidence of any additional wear.

The fertile crusher has been reassembled with two new replacement bearings, with care being taken to avoid overlubrication, and with the preloads reduced from 90 in.-lb to 70 in.-lb per rod shaft. Sixty kilograms of fuel particles will be crushed while the electrical power

demand and crushing efficiency are observed. The unit will then be completely disassembled and all parts subjected to detailed dimensional inspection. Particular note will be made of wear and other abnormalities. Crushing roll surfaces will be surface analyzed for finish and contour change and roll diameters will be accurately measured. These data will be compared with previous measurements for indication of wear or wear trends.

REFERENCES

- 4-1. "Thorium Utilization Program Quarterly Progress Report for the Period Ending November 30, 1976," ERDA Report GA-A14214, General Atomic Company, December 1976.
- 4-2. "Thorium Utilization Program Quarterly Progress Report for the Period Ending February 28, 1977," ERDA Report GA-A14304, General Atomic Company, March 1977.
- 4-3. De Lesdernier, D. L., General Atomic Company, private communication.
- 4.4. "Thorium Utilization Program Quarterly Progress Report for the Period Ending May 31, 1977," ERDA Report GA-A14441, General Atomic Company, June 1977.

5. AQUEOUS SEPARATION

5.1. SUMMARY

By July 1, 1977, the detail design of the engineering-scale dissolver-centrifuge system had been completed. All equipment detail design and system arrangement drawings have been released for fabrication or procurement.

This system consists of a 0.5-m dissolver equipped with feed bunkers and a reflux condenser for dissolution of both fertile and fissile fuels, a continuous vertical centrifuge for separating insols from the mother liquor, a repulp vessel for rinsing residual salts from insols, and auxiliary equipment. When installed, it will complete the engineering-scale HTGR head-end reprocessing pilot plant.

Modification of the cold pilot plant for installation of this new system is under way in accordance with system arrangement drawings and defined utility requirements. Procurement and fabrication of process equipment are in progress, with delivery of most major components anticipated in late September. Installation of the engineering-scale system is expected to be complete by February 1978.

Three heel dissolution runs were made in the 0.13-m dissolver. Based on bench-scale tests, 40% heel was selected as the optimal thoria-to-Thorex ratio. Results of these 0.13-m dissolver runs will be reported in the next quarterly report after data analyses are complete.

Bench-scale studies were initiated during the quarter to assess the degree of ruthenium and iodine volatility during ThO_2 dissolution. It is

desired to control ruthenium volatility while volatilizing essentially all the iodine to keep it out of downstream vessel off-gas systems. Preliminary data indicate controllable ruthenium volatility which is directly related to dissolver acid concentration and sparge flow rate. Iodine volatility is poor from the high Acid-Thorex dissolver solution, perhaps due to oxidation to iodates.

5.2. ENGINEERING-SCALE DISSOLUTION

5.2.1. 0.5-m Dissolver-Centrifuge System

5.2.1.1. Introduction. The next step in the development of dissolution-centrifugation for HTGR reprocessing is the design and installation of the engineering-scale dissolver-centrifuge system. This system, together with the future insols dryer, will be incorporated into the engineering-scale cold pilot line. When installed, it will complete the engineering-scale HTGR head-end reprocessing pilot plant.

Included in the system are feed and product tanks, a dissolver and condenser, a centrifuge, and a repulp tank. Specifically, data will be provided on the dissolution of FSV and reference TRISO and BISO fuels, on the separation of the mother liquor from the undissolved solids, and on the repulping of the solids for better recovery of the heavy metal nitrates. Later in the program, solids drying will be included and equipment to demonstrate remote maintenance of selected components will be added.

5.2.1.2. System and Equipment Design. A perspective drawing of the 0.5-m dissolver-centrifuge system depicting the major components and the main piping is shown in Fig. 5-1. This drawing shows the redesign of the solids feed bunker support structure. Both bunkers have been placed at the same elevation, which permits personnel access to the bunkers for manual fill from the transfer can without ladders and associated safety equipment. The common-frame design of the bunker support structure is greatly simplified, as is the layout of the load cells.

Fig. 5-1. 0.5-m dissolver-centrifuge system

Each bunker is supported by three load cells and the tie rods. The tie rods are essential for the proper functioning of load cells because they prevent side load from being exerted on the cells. Further isolation of the weighing load cell from the dissolver is achieved with expansion bellows, so that the expansion of piping would cause minimal error in weighing accuracy. A rotary feeder is utilized to meter secondary burner product into the dissolver. The drive motor and gear box for the feeder are mounted on one of the support frame legs. A double universal joint coupling connects the feeder to the gear drive.

Design of the 0.5-m dissolver-centrifuge system platform has been completed. A stainless steel drip pan protects the area of the floor under the platform. The deck of the platform is fabricated of stainless steel in the area of the tanks and main piping. This stainless steel decking has raised curbs and contains a drain pipe to drain leaks from the decking down to the drip pan below.

5.2.1.3. Dissolver Heating and Cooling Requirement. A heating and cooling analysis for the dissolver was performed to include the steam requirement for the startup, cooling requirement, and cooling transient. It was found that steam would be required in both bottom and side dissolver jackets if the heatup time to boiling were to be limited to 1 hr. Cooling of the solution to ambient temperature can easily be accomplished within 1 hr with sufficient sparging and side jacket cooling alone. Accidental pressurization of the dissolver due to steam ingress through a ruptured bottom jacket was found to be less than 0.034 atm (1/2 psig) with the designed condenser and loop seal arrangement, by the addition of a flow restricting orifice limiting steam flow to the bottom jacket to 45 kg/hr (100 lb/hr) [at 6.1 atm (90 psig)]. Back-pressurization of the dissolver due to plugging of the condenser loop seal or the off-gas treatment units downstream can be avoided by installing a pressure relief valve for the vessel and a pressure-switch cutoff for the steam and sparge gas supply to the vessel.

5.2.1.4. Status of Equipment. The detail design of major equipment was completed on July 1, and all of the system vessels are in the procurement

cycle. The Thorex and product tanks are on order from a commercial supplier, with delivery expected in September 1977. The Sharples centrifuge was delivered in June. The system platform is being fabricated by a local contractor, with completion estimated by early September 1977. All other vessels and the bunker support frame are being fabricated in-house by the General Atomic Component Manufacturing Division. A number of subassembly items for the dissolver vessel have been fabricated. Materials for the repulp vessel have been ordered, and the vessel is in the planning stage. The solids feed bunkers and blowback assemblies are in manufacturing planning. The feed bunker support frame is in fabrication. Completion of the above items being manufactured by the Component Manufacturing Division is expected by about October 1, 1977.

5.2.2. Pilot Plant Heel Dissolution

5.2.2.1. Introduction. Heel operation takes advantage of the rapid dissolution rate during the first portion of leaching of ThO_2 kernels. By interrupting the normal cycle before complete dissolution is achieved, it may be possible to shorten the combined dissolution time for several batches. For sol-gel ThO_2 kernels, the point of interruption appears to be about 60% to 80% dissolution. To pinpoint the optimal parameters, a series of eight test segments were planned to be carried out in bench-scale glassware and 0.13-m and 0.20-m pilot plant dissolvers. Bench-scale tests have been completed and were reported earlier (Ref. 5-1). Pilot plant scale Tests 1 through 3 will establish the operating parameters. Tests 4 and 5 will verify these parameters using the centrifuge to separate the heel from the product. Tests 6 through 8 will investigate steam jet decanting of the mother liquor from the heel. To date, Tests 1 and 2 have been completed.

As previously reported (Ref. 5-2), based on the results of Tests 1 and 2, a threefold increase in the rate of dissolution using heel operation instead of batch dissolution was estimated. A 40% heel for sol-gel ThO_2 dissolution with a 5- to 6-hr dissolution time was selected for Test 3 in the 0.13-m dissolver.

5.2.2.2. Results. During this reporting period, three runs were made in the 0.13-m dissolver with 40% sol-gel ThO_2 (BISO) heel. The objective was to attain 1M thorium concentration in the mother liquor within 6 hr of dissolution at boiling point.

Preliminary analysis of data from these runs indicates an improvement in dissolution rate is possible in the 0.13-m dissolver with 40% heel operation, although the increase is not as large as that indicated in bench-scale tests. However, it appears the objective can be met. These run data will be reported in detail after data reduction and correlation are complete.

5.3. BENCH-SCALE TESTS ON VOLATILITY OF RUTHENIUM AND IODINE DURING ThO_2 DISSOLUTION

5.3.1. Introduction

Bench-scale studies were initiated during the quarter to assess the degree of ruthenium and iodine volatility during dissolution of thorium in Thorex solution. Use was made of tracer Ru-103 and I-131 . Distributions were calculated from gamma spectrometric data obtained from samples generated during the bench-scale tests.

5.3.2. Experimental Results

The volatility of ruthenium in simulated batch dissolution of full-burnup, 1-yr, four-segment fuel was found to be directly dependent on dissolver nitric acid concentration. A value of ~0.65% ruthenium volatility was obtained during an 8-hr run with boiling concentrated nitric acid in the dissolver. Much lower ruthenium volatility values were observed in samples with lower initial nitric acid, i.e., 0.017% during preparation of 1M thorium-Thorex solution (final nitric acid concentration = 9M) and 0.013% during 8-hr boiling of ruthenium "spiked" Thorex solution only. Sparge gas (N_2) flow rate was found to have a marked effect on ruthenium volatility.

The equipment shown in Fig. 5-2 was used for all experiments reported herein. An individual run consisted of boiling Thorex or acid solution in the dissolver following addition of tracer and normal ruthenium (in an amount simulating full-burnup, 1-yr, four-segment fuel) and, in some experiments, ThO_2 . Upon conclusion of the run, the traps were carefully disassembled and packaged for gamma ray Ru-103 analysis. The solid material used to remove Ru-103 from the N_2 gas sparge stream was activated charcoal. Sample dilutions were made as required with activated charcoal or water to bring Ru-103 activity within the range of the gamma spectrometric technique. In most runs, a N_2 sparge gas flow rate equivalent to $0.4 \text{ m}^3/\text{hr}$ ($14 \text{ ft}^3/\text{hr}$) in the 0.13-m pilot plant dissolver was used. However, some runs were performed with an equivalent $4.2 \text{ m}^3/\text{hr}$ ($7 \text{ ft}^3/\text{hr}$) N_2 gas sparge rate.

Data obtained for the volatility of ruthenium from the dissolver during the dissolution of ThO_2 to a final mother liquor concentration of 1M are given in Table 5-1. The only variable in these tests was sparge gas flow rate, and the effect of N_2 sparge rate is apparent from comparison of the data in the table (increased ruthenium volatility with the higher sparge rate).

The largest percentage of ruthenium volatility occurred in a test in which concentrated nitric acid containing ruthenium (in the absence of thorium) was boiled for 8 hr. Data from this experiment are presented in Table 5-2.

Four experiments were conducted during the quarter to assess the magnitude of iodine volatility during dissolution of ThO_2 in Thorex to 1M thorium concentration. A tracer isotope, I-131, was used to determine iodine distribution in samples generated for gamma spectrometry. Data reduction is complete for one experiment, and a value of 1.72% iodine volatility was calculated for dissolution of simulated full-burnup, 1-yr, four-segment fuel. The data are presented in Table 5-3. The system shown in Fig. 5-2 was used for the iodine volatility studies.

Fig. 5-2. Bench-scale conical dissolver

TABLE 5-1
RUTHENIUM DISTRIBUTION DATA FOR PREPARATION
OF 1M THORIUM/THOREX SOLUTION

Test	Sample	Total Activity Due to Ru-103 Constant Geometry (CPM)	Total Percent Ru Volatilized
A	Th-Thorex product	5,456,640	0.017
	Charcoal trap	935	
B	Th-Thorex product	5,200,840	0.006
	Charcoal trap	336	

- (a) Experimental conditions:
8-hr dissolution of ThO₂ in boiling Thorex
(ruthenium "spiked") to a final thorium concen-
tration of 1M.
- Test A - N₂ flow rate equivalent to 14 ft³/hr
(0.4 m³/hr)² in the 0.13-m pilot plant dissolver.
- Test B - N₂ flow rate equivalent to 7 ft³/hr
(0.2 m³/hr)² in the 0.13-m pilot plant dissolver

TABLE 5-2
RUTHENIUM DISTRIBUTION DATA FOR
CONCENTRATED NITRIC ACID SOLUTION (a)

Sample	Total Activity Due to Ru-103 Constant Geometry (CPM)	Total Percent Ru Volatilized
Th-Thorex product	5,821,820	0.65
Charcoal trap	37,784	

- (a) Experimental conditions:
8-hr boiling of concentrated nitric acid
(ruthenium "spiked").
- N₂ flow rate equivalent to 14 ft³/hr (0.4 m³/hr)
in the 0.13-m pilot plant dissolver.

TABLE 5-3
IODINE DISTRIBUTION DATA FOR PREPARATION OF
1M THORIUM/THOREX SOLUTION(a)

Sample	Total Activity Due to Ru-103 Constant Geometry (CPM)	Percent Iodine Volatilized
Th-Thorex product	39,261,300	
Charcoal trap	675,719	1.72

(a) Experimental conditions:

8-hr dissolution of ThO_2 in boiling Thorex (iodine "spiked") to a final thorium concentration of 1M.

N_2 flow rate equivalent to 14 ft³/hr (0.4 m³/hr)
in the 0.13-m pilot plant dissolver

High volatility of iodine in the dissolver is desired to keep the iodine collectable at one point in the process. Otherwise iodine will spread throughout the Acid-Thorex plant and require iodine retention devices on all vessel off-gas systems. These low iodine volatility results are disappointing and may be due to oxidation of iodine and iodide to iodate. Dissolution tests and sparge tests will continue in an effort to achieve high iodine separation from the Acid-Thorex dissolver system.

REFERENCES

- 5-1. "Thorium Utilization Program Quarterly Progress Report for the Period Ending February 28, 1977," ERDA Report GA-A14304, General Atomic Company, March 1977.
- 5-2. "Thorium Utilization Program Quarterly Progress Report for the Period Ending November 30, 1976," ERDA Report GA-A14214, General Atomic Company, December 1976.

6. SOLVENT EXTRACTION

6.1. SUMMARY

Three solvent extraction feed adjustment runs (Runs 31, 32, and 33) were completed during the quarter. The purpose of these runs was to test equipment modifications and flowsheet requirements for intercycle concentration. A schematic of feed adjustment equipment is shown in Fig. 6-1. In Run 31, the evaporator-stripper was operated at solution temperatures up to 120°C. No significant data were collected in Run 31 owing to pressurization of the vessel.

For Run 32, the ceramic packing in the tower was replaced with 5/8-in. stainless steel Pall rings. Some pressurization of the evaporator-stripper occurred with this tower packing. The 5/8-in. packing was replaced with 1-in. stainless steel Pall rings. The 1-in. Pall rings were used for tower packing for the remainder of Run 32 and Run 33. No vessel pressurization occurred with the 1-in. rings in the tower.

The evaporator-stripper was operated at solution temperatures up to 120°C in Run 32 and up to 125°C in Run 33. An acid-deficient product was not achieved in Run 32 or 33. A hydrogen ion to thorium mole ratio of 0.25 was achieved in Run 33.

Seven solvent extraction runs were completed during the quarter. Runs 62 and 63 were used to determine the extraction efficiency of the Robatel centrifugal contactor using an aqueous feed stream containing thorium. A flow schematic is shown in Fig. 6-2. The SEPHIS computer code for the Thorex flowsheet was used to calculate theoretical values which were compared with the analytical results from the pilot plant operation.

Fig. 6-1. Piping and instrumentation diagram for continuous feed adjustment equipment

STREAM	STREAM NO.	Th (g/l)	HNO ₃ (M)
1AF	1	80-175	1.35
1AX	2	---	[30% TBP]
1AA	3	---	13.0
1AP	4	---	---
1AW	5	---	---
1CX	6	---	0.01
1CP	7	30-50	---
1CW	8	---	[30% TBP]
100	9	---	[30% TBP]
10S	10	---	[0.2M Na ₂ CO ₃]
10W	11	---	WASTE

Fig. 6-2. Flowsheet schematic for solvent extraction Runs 62 and 63

The efficiency of the centrifugal contactor for uranium alone is near 100% (Ref. 6-1). However, the determination in the thorium system is less conclusive owing to (1) the uncertainties in the SEPHIS code for the Thorex system under conditions of high solvent loading and (2) the presence of a third phase (second organic phase) when the thorium concentration in the organic phase exceeds about 30 to 35 g/l. The end stream thorium concentration varied erratically, apparently due to the third phase. As a consequence, the Robatel contactor is not recommended.

Runs 68 and 69 were used to test the flowsheet for the first two cycles of the Purex (fissile particle) HRDF flowsheet. The Robatel centrifugal contactor and four pulsed columns in the solvent extraction pilot plant were used for these runs. The flowsheet for this operation is shown in Fig. 6-3.

A new configuration for the centrifugal contactor and pulsed columns was used for Runs 68 and 69. In Run 68, significant startup difficulties were experienced, primarily as a result of the new configuration. The centrifugal contactor also became plugged at startup.

In Run 68, uranium losses were controlled at a low level. However, higher than expected losses occurred via the 6AW stream owing to difficulties with the 6AA stream location and flow. Good overall zirconium-niobium decontamination was achieved. In Run 69, dibutyl phosphate (DBP) was added to the centrifugal contactor to simulate solvent degradation. The DBP addition reduced zirconium-niobium decontamination in the first cycle (5A, 5S, 5B), but the overall (two-cycle) decontamination was comparable to that in Run 68. Low uranium losses were also achieved in Run 69.

Runs 70 and 71 were similar to Runs 68 and 69 except that the Robatel centrifugal contactor was not used (see Fig. 6-4). A different configuration was also used for the pulsed columns. The change was made to give the 5A column an increased length.

STREAM	STREAM NO.	RELATIVE FLOW RATE	U (g/l)	HNO ₃ (M)
5AF	1	100	12	2
5AX	2	60	[30 % TBP]	
5AS	3	25	--	1
(5A-DBP)	(4)	(1.0)	[30 % TBP]	
5AW	5	--		
5AP	6	--		
5SR	7	--		
5SP	8	--		
5BX	9	50	--	0.01
5BP	10	--		
5BW	11	--		
6AA	12	2	--	13
6AX	13	45	[30 % TBP]	
6AS	14	20	--	2 (Fe ⁺⁺ = 0.05M)
6AP	15	--		
6AW	16	--		
6BX	17	40	--	0.01
6BP	18	(41)	29	
6BW	19	--		

Fig. 6-3. Test flowsheet for two-cycle Purex solvent extraction - Runs 68 and 69

STREAM	STREAM NO.	RELATIVE FLOW RATE	U (g/l)	HNO ₃ (M)
5AF	1	100	11	2.4
5AX	2	70	[30 % TBP]	
5AS	3	20		1.0
5AW	4	---	---	
5AP	5		---	
5BX	6	55	---	0.01
5BP	7		---	
5BW	8		---	
6AA	9	2.5	---	13.0
6AX	10	40	[30 % TBP]	
6AS	11	15	---	2.4 (Fe ⁺⁺ = 0.05)
6AP	12		---	
6AW	13		---	
6BX	14	40	---	0.01
6BP	15	42	25	1.0
6BW	16		---	

Fig. 6-4. Flowsheet schematic for Purex two-cycle system - Runs 70 and 71

In Run 70, flooding points were determined for the 5A and 6A columns. Recovering normal operation of the 5A column after flooding was difficult. Problems were also experienced with control of the 5B column interface. The overall decontamination factor for zirconium-niobium was about 8400. A detectable uranium loss occurred from the 6A column. The run was terminated when the 6AF pump failed.

In Run 71, flooding points were determined for the 5B and 6B columns. The run progressed well, with no significant operating problems. The decontamination factor for zirconium-niobium was more than twice that achieved in Run 70. Uranium losses were low throughout the run.

Run 73 utilized the five pulsed columns in the solvent extraction pilot plant to test the first cycle of the Thorex flowsheet for the HRDF (see Fig. 6-5). The configuration of the columns was similar to that used in earlier tests (Ref. 6-2). This run was made primarily to confirm earlier results since some doubts had been raised as a result of analytical problems. The run lasted about 6 hr. Of particular interest was the efficiency of the partition system for separation of uranium and thorium. The crossover of uranium to the 1BT stream and thorium to the 1CU stream was very low.

In Run 73, the operation of the solvent extraction system proceeded without significant incident. Some local flooding occurred in the scrub section of the 1A column, and some temporary flooding occurred in both the 1BS and the 1C columns. The 1BX-1BS system was started up with a 0.5M HNO_3 solution in the columns to reduce potential startup losses of uranium. The visual distribution of uranium in the 1BS column during startup was similar to that in previous runs. However, the amount of uranium in the 1BT stream samples was below the lower detection limit for analysis. A thorium loss of about 2% occurred via the 1AW stream. This loss was attributed to the high aqueous to organic flow ratio in the 1A column. Uranium losses from the 1A column were below 0.1%. The zirconium-niobium decontamination was markedly better for uranium than for thorium, as in previous runs.

STREAM	STREAM NO.	RELATIVE FLOW	COMPOSITION		
			U (G/L)	Th (G/L)	HNO ₃ (M)
1AF	1	100	35	348	1.0
1AS	2	104			0.01
1AX	3	1000		(30% TBP)	
1AW	4	262		(FISSION PRODUCTS)	
1AA	5	32			13.0
1SP	6	1000	3.5	35	
1AIS	7	26			5.0
1BX	8	600			0.2
1BT	9	600	Trace	102	
1BS	10	179		(30% TBP)	
1BU	11	1180	2.98		
1CX	12	593			0.01
1CU	13	593	5.93		
1CW	14	1180		(30% TBP)	
1WW	15	59		(NPH)	
1WS	16	59		(NPH)	
1WU	17	593	5.93		
2AF	18	6	233		
2AX	19	118		(5% TBP)	
2AW	20	41		(WASTE)	
2AS	21	34			2.0
2AP	22	118	11.8		
2CX	23	59			0.01
2CW	24	118		(ORGANIC)	
2CU	25	59	23.6		
2WW	26	6		(NPH)	
2WS	27	6		(NPH)	
2WU	28	59	27.5		
U PRODUCT	29	6	233		

Fig. 6-5. Partition flowsheet for Thorex solvent extraction system - Run 73

Bench-scale tests are being continued on the volatility of ruthenium from the feed adjustment step. Ruthenium volatility values of 0.6% and 1.4% were measured for batch and continuous feed adjustments, respectively.

A conductivity probe has been calibrated for use in continuous measurement of nitric acid in the overheads on the pilot plant system.

Some bench-scale studies are being made on fluoride volatility from simulated HRDF high-level aqueous wastes. Batch concentration of 1AW solution resulted in volatilization of approximately 15% of the fluoride. However, continuous operation yielded fluoride volatilization equilibrium values of about 5%.

6.2. SOLVENT EXTRACTION FEED ADJUSTMENT

6.2.1. Introduction

The continuous solvent extraction feed preparation process consists of continuously feeding an acidic thorium nitrate stream to a boiler pot of boiling, concentrated, acid-deficient thorium nitrate solution. Excess acid from the feed stream is boiled off (steam stripped), leaving more acid-deficient thorium nitrate in the boiler pot. The acid-deficient thorium nitrate product is continuously or semicontinuously removed from the boiler pot and is then diluted with water to prevent solidification upon cooling.

A continuous feed adjustment system has the advantage of providing a uniform condensate flow and composition, thereby facilitating operation of the downstream waste processing system. Also, since the solvent extraction process is inherently continuous, there is an additional incentive to have a continuous feed adjustment step for the dilute, intercycle thorium stream.

Bench-scale demonstration of the feasibility of the process for continuous feed adjustment has been completed. Pilot plant testing is being done (1) to determine necessary equipment items for commercial-scale operation, in particular, for the difficult task of concentrated product removal, and (2) to determine scale-up effects from the bench-scale work.

6.2.2. Results

The feed used in Runs 31, 32, and 33 was typical of the 1BT stream which feeds the intercycle concentrator. Table 6-1 contains the typical steady-state concentrations of the feed, distillate, and product streams for Run 32, and Table 6-2 contains the stream concentrations for Run 33.

6.2.3. Discussion

6.2.3.1. Run 32. In this run, the operation of the evaporator-stripper was tested with 5/8-in. Pall rings in the off-gas tower. These stainless steel rings had been used to replace the ceramic saddles that were used in previous runs. The evaporator-stripper vessel became pressurized with the 5/8-in. Pall rings in the tower. These rings were then replaced with 1-in. Pall rings. No further problems with vessel pressurization occurred after the installation of the 1-in. Pall rings.

In Run 32, the temperature in the evaporator-stripper was controlled at 115°C and then at 120°C. An acid-deficient product was not achieved at these temperatures even with large relative additions of stripping water. At 115°C, the average feed rate was 0.2 l/min. Stripping water was added at 0.45 to 0.5 l/min. At 120°C, the feed rate was added at 0.35 l/min and stripping water was added at 0.29 l/min.

6.2.3.2. Run 33. In this run, the evaporator-stripper temperature was controlled at 125°C. The average feed rate was about 0.42 l/min, and the stripping water rate was about 0.21 l/min. An acid-deficient product was not achieved at these operating conditions. The H^+ to thorium mole ratio

TABLE 6-1
 REPRESENTATIVE STREAM ANALYSES FOR SOLVENT EXTRACTION
 FEED ADJUSTMENT RUN 32

	g moles H^+ / g mole Th	H^+ (<u>M</u>)	Th (<u>M</u>)	Al (<u>M</u>)	F^- (<u>M</u>)	Density at 25°C (g/cm ³)
Feed	2.64	0.58	0.22	0.03	3×10^{-3}	1.15
Product (after dilution)	0.43	0.612	1.42	0.19	6.8×10^{-2}	1.60
Overheads		0.15		<1 ppm		1.01

TABLE 6-2
 REPRESENTATIVE STREAM ANALYSES FOR SOLVENT EXTRACTION FEED ADJUSTMENT RUN 33^(a)

	g moles H ⁺ / g mole Th	H ⁺ (M)	Th (M)	Al (M)	F ⁻ (M)	Density at 25°C (g/cm ³)
Initial pot solution (before dilution)	0.40	0.78	1.96	0.23	7×10^{-2}	1.79
Feed	2.73	0.6	0.22	0.02	7×10^{-3}	1.11
Product (after dilution)	0.26 (0.30)	0.42 (0.59)	1.63 (2.00)	0.18 (0.21)	3.3×10^{-2} (3.0×10^{-2})	1.66 (1.76)
Overheads		0.46		<1 ppm	8×10^{-5}	1.01

(a) Values in parentheses correspond to a second set of analytical results.

in the product was less than that in Run 32. This improvement was obtained with a lower stripping water to feed ratio. Results from Run 32 and Run 33 compare substantially with equilibrium data collected on the bench scale.

In Run 33, the packing in the tower was 1-in. Pall rings, as in the latter part of Run 32. No pressurization of the evaporator-stripper vessel occurred under any of the operating conditions used in Run 33.

Product was semicontinuously removed during Run 33 to control the volume in the boiler pot.

6.3. SOLVENT EXTRACTION

6.3.1. Introduction

Runs 62 and 63 were used to test the extraction efficiency of the Robatel centrifugal contactor using an aqueous feed stream containing thorium. The flowsheet schematic for this operation is shown in Fig. 6-2. Two selected concentrations of thorium in the feed were used for these runs. Several flow rates were used for each concentration. The solvent used was 30% tributyl phosphate (TBP) in normal paraffin hydrocarbon (NPH).

Two pulsed columns were also used as part of the operation of the runs. One column was used for thorium stripping and the other for solvent washing.

The SEPHIS computer code for the Thorex flowsheet was used to calculate theoretical values, which were compared with the analytical results from the pilot plant operation.

Runs 68 and 69 were used to test the flowsheet for the first two cycles of the Purex (fissile particle) HRDF flowsheet. Plutonium partitioning was simulated in the second cycle. The Robatel centrifugal contactor and four pulsed columns in the solvent extraction pilot plant

were used for these runs. Uranium was extracted in the centrifugal contactor. One column was used for scrubbing and one for uranium stripping for the first cycle. For the second cycle, one column was used for the extraction-scrub operation and one for the uranium strip operation. The plutonium partition column was simulated by the addition of ferrous nitrate to the aqueous scrub stream to the extraction-scrub column (6A) in the second cycle. The flowsheet is shown in Fig. 6-3. No plutonium was used in this operation. Radioactive zirconium-niobium was used as a tracer to permit decontamination measurement throughout the system.

Runs 70 and 71 were similar to Runs 68 and 69 except that the Robatel centrifugal contactor was not used. These runs used four pulsed columns to test two cycles of the Purex (fissile particle) flowsheet shown in Fig. 6-4. Each cycle consisted of an extraction-scrub step and the uranium strip step using two columns. The plutonium partition column was simulated by the addition of ferrous nitrate to the aqueous scrub stream to the extraction-scrub column (6A) in the second cycle. No plutonium was used in this operation. Zirconium was added to the 5AF feed to duplicate the expected concentration in HRDF fuel. Radioactive zirconium-niobium was used as a tracer to permit decontamination measurement throughout the system.

Run 73 was used to test the first cycle of the Thorex flowsheet for the HRDF (Fig. 6-5). This run utilized the five pulsed columns. The extraction-scrub step was followed by partition, partition-scrub, and uranium strip steps. The solvent was washed in the fifth pulse column. Zirconium and radioactive zirconium tracer were added to the 1AF feed.

6.3.2. Results and Discussion - Runs 62 and 63

Table 6-3 contains the stream analyses and flow rates for Runs 62 and 63. Table 6-4 contains data on losses and other calculated data. Table 6-5 gives a description of the centrifugal contactor and pulsed columns used for the runs. Table 6-6 compares the pilot plant results with SEPHIS code calculations.

TABLE 6-3
STREAM AND SAMPLE DATA FOR SOLVENT EXTRACTION RUNS 62 AND 63

1AF (Stream 1) (a)			1AX (Stream 2) Flow [30% TBP] (ml/min)	1AA (Stream 3) Flow (13M HNO ₃) (ml/min)	1AP (Stream 4) Th (g/l)	1AW (Stream 5)	
Flow (ml/min)	Th (g/l)	HNO ₃ (M)				Th (g/l)	HNO ₃ (M)
Run 62							
331	78.39	1.34	878	31	9.84	0.093	1.71
502	78.39	1.34	1143	30	14.8	0.066	1.34
502	78.39	1.34	955	25	20.4	0.038	1.34
321	78.39	1.34	528	25	36.8	5.45	1.78
319	78.39	1.34	689	25	30.4	0.066	1.42
Run 63							
271	166.71	1.35	830	21	38.4	0.027	1.70
249	166.71	1.35	574	30	16.58	31.81	2.61
285	166.71	1.35	710	26	120.6	0.482	2.28
267	166.71	1.35	603	24	29.8	2.92	2.37
317	166.71	1.35	618	23	36.8	20.60	2.20

(a) See Fig. 6-4. 1AF stream enters stage 1. 1AA stream enters stage 6. 1AX stream enters stage 8.

TABLE 6-4
CALCULATED LOSS AND FLOW RATE DATA FOR SOLVENT EXTRACTION RUNS 62 AND 63

Contactor Description	Purpose	Total Flow (l/min)	Contactor Operating rpm	Aqueous to Organic Ratio	Thorium Loss (%)	Temp. (a) (°C)
Run 62						
1A centrifugal	Extraction	1.240	1200	0.412	0.12	24.7
		1.675	1200	0.465	0.08	25.6
		1.482	2000	0.552	0.05	26.0
		0.874	1200	0.655	6.95	26.7
		1.033	1200	0.499	0.08	26.7
Run 63						
1A centrifugal	Extraction	1.122	1200	0.352	0.02	25.0
		0.853	1200	0.486	19.08	25.6
		1.021	1200	0.438	0.29	26.0
		0.894	1200	0.483	1.75	27.2
		0.958	2000	0.550	12.36	29.4

(a) Average of 1AW and 1AP temperatures.

TABLE 6-5
DESCRIPTION OF CENTRIFUGAL CONTACTOR AND COLUMN CARTRIDGE USED IN SOLVENT EXTRACTION RUNS 62 AND 63

Unit	Purpose	Diameter (mm)	Total Height of Mixing Area (m)	Other			
1A contactor	Extraction	180	0.32	8 stages with 0.4-liter total holdup per stage			
1C column	Uranium strip	76	5.8	Plates			Plate Spacing (mm)
				Nozzle Direction	Hole Size (mm)	Percent Free Area	
				Up	4.8	23	Graded ^(a)
10 column	Solvent wash	51	5.5	Down	3.2	23	51

^(a) Graded cartridge is, from the bottom, 2.6 m with 100-mm spacing, 0.5 m with 76-mm spacing, and the remainder with 51-mm spacing.

TABLE 6-6
COMPARISON OF 1AW AND 1AP CONCENTRATIONS OBTAINED IN PILOT PLANT TESTS
AND SEPHIS CALCULATIONS

Corresponding Input Flow Rate ^(a) (ml/min)	Output Stream	Pilot Plant Results		SEPHIS Eight-Stage Calculation	
		Th (g/l)	HNO ₃ (<u>M</u>)	Th (g/l)	HNO ₃ (<u>M</u>)
1. 878 (1AX) 362 (1AF+1AA)	1AP	9.84	--	29.52	0.26
	1AW	0.093	1.71	0.01	1.72
2. 1143 532	1AP	14.8	--	34.43	0.22
	1AW	0.066	1.34	0.01	1.52
3. 955 527	1AP	20.4	--	40.93	0.19
	1AW	0.038	1.34	0.12	1.56
4. 528 346	1AP	36.8	--	43.67	0.18
	1AW	5.45	1.78	5.39	1.92
5. 689 344	1AP	30.4	--	36.26	0.21
	1AW	0.066	1.42	0.01	1.76
6. 830 292	1AP	38.4	--	54.44	0.21
	1AW	0.027	1.70	0.01	1.60
7. 574 279	1AP	16.58	--	68.55	0.12
	1AW	31.81	2.61	6.26	2.36
8. 710 311	1AP	120.6	--	66.57	0.14
	1AW	0.482	2.28	0.03	2.01
9. 603 291	1AP	29.8	--	68.56	0.12
	1AW	2.92	2.37	9.206	2.07
10. 618 340	1AP	36.8	--	68.76	0.12
	1AW	20.60	2.20	30.32	1.92

(a) See Table 6-3 for the composition of each stream.

Runs 62 and 63 provided for a determination of the operating efficiency for thorium extraction in the centrifugal contactor. As shown in Table 6-3, a variety of flow rates and flow ratios were tested.

The agreement between the results from the SEPHIS code calculations and the analyses for the end stream samples from the runs was not good. One reason for the differences is the uncertainties of the code. The development of the code used correlations which are applicable at conditions of lower solvent loading than used in these tests. Also, the code does not take into account the three phases which are present at higher thorium concentrations. After thorium concentrations of 30 to 35 g/l are exceeded in the organic phase, a second organic phase is formed. This third phase complicates theoretical calculations as well as operational characteristics.

The third phase is composed primarily of thorium and TBP and has a density which is nearly equal to that of the aqueous phase. In these runs only marginal separation of this third phase from the aqueous phase was achieved in the contactor. Although aqueous phase losses were low in comparison with the SEPHIS results, the third phase had the apparent effect of causing a fluctuating unsteady condition as indicated by the erratic 1AP stream thorium values.

Except for one 1AP value, all the analyzed 1AP thorium concentrations were less than the SEPHIS predicted values. The accuracy of determining the thorium concentration in the 1AP stream is limited because of the necessity of sampling the 1AP stream, which under most conditions is a two-phase system. The consistently low thorium analyses are partially due to a possible bias in sampling of the 1AP stream, which caused a disproportionately greater amount of the lighter phase in the samples.

Under conditions of near saturation of the solvent in Run 63, very erratic thorium values were obtained for the 1AP stream. These variations are due to the cyclical nature by which the third phase was built up in the

contactor and then released. Part of the release of the third phase may have occurred via the 1AW stream, as shown by the 31.81 g/l loss in Run 63. Sampling, except on a continuous basis, would not detect these losses. A cyclical loss of thorium via the 1AW stream would also explain the consistently low 1AP analyses in both of these runs.

For the first cycle of the Thorex flowsheet, an operation with the thorium loading of the solvent in the third-phase region is desirable to permit significant fission product decontamination. In a pulsed column this desired solvent loading can be achieved without any undesirable or uncontrolled losses of thorium. In the centrifugal contactor, these same conditions produce unpredictable and significant thorium losses.

The pulsed columns for stripping thorium and washing the solvent operated without incident in these runs.

A Robatel centrifugal contactor is not recommended for thorium TBP-NPH extraction because of the uneven operation occurring due to the second organic phase. A compound pulse column where the scrub section is attached to the top of the extraction section is the recommended contactor for this service.

6.3.3. Results - Runs 68 and 69

Tables 6-7 and 6-8 contain the stream analyses and flow rates for Runs 68 and 69, respectively. Tables 6-9 and 6-10 contain the loss data and operating conditions for the two runs. Table 6-11 gives a description of the centrifugal contactor, pulsed columns, and column cartridges.

6.3.4. Discussion - Run 68

In Run 68, the two cycles of the Purex flowsheet were tested for the first time. Each of the pulsed columns was in a relative configuration which was new. The configuration of the centrifugal contactor relative to the columns was also new.

TABLE 6-7
STREAM ANALYSES AND FLOW RATES FOR SOLVENT EXTRACTION RUN 68^(a)

Stream	Stream No.	U (g/l)	HNO ₃ (M)	ZrNb (CPM)	Flow (ml/min)	Relative Flow	Other
5AF	1	12.52	2.14	72,172	690 (725) (698)	100 (100) (100)	
5AX		--	[30% TBP]		436 (481) (576)	63 (66) (83)	
5AP		25.61 (18.67) (20.90)	0.33 (0.30) (0.32)	1,677 (1,639) (1,460)			
5AW		<2 x 10 ⁻⁴ (<2 x 10 ⁻⁴) (<2 x 10 ⁻⁴)	1.94 (2.00) (1.94)	58,764 (57,504) (55,781)			
5AS		--	1.09		174 (173) (190)	25 (24) (27)	
5SR		2.61 (2.61) (2.28)	1.51 (1.56) (1.71)	3,410 (3,474) (4,609)			
5SP		21.03 (21.38) (21.79)	0.24 (0.21) (0.20)	43.9 (50.5) (60.2)			
5BX		--	0.012		542 (319) (360)	79 (44) (52)	
5BP		14.99 (23.19) (25.22)	0.13 (0.13) (0.12)	15.5 (25.1) (33.4)			

TABLE 6-7 (Continued)

Stream	Stream No.	U (g/l)	HNO ₃ (M)	ZrNb (CPM)	Flow (ml/min)	Relative Flow	Other
6AA			13.0		4.3 (3.1) (2.5)	0.6 (0.4) (0.4)	
6AS		--	1.98		164 (124) (123)	24 (17) (18)	Fe ⁺⁺ = 0.051
6AW		2.5 x 10 ⁻² (1.0 x 10 ⁻²) ^(b) (2.0 x 10 ⁻³) ^(b)	1.36 (0.91) (0.93)	63.3 (51.4) (39.7)			
6AX			[30% TBP]		299 (301) (315)	43 (42) (45)	
6AP		30.08 (23.58) (23.20)	0.33 (0.32) (0.33)	11.0 (8.9) (9.9)			
6BX		--	0.01		360 (378) (352)	52 (52) (50)	
6BP		19.46 (22.75) (19.36)	0.22 (0.22) (0.26)	9.3 (11.5) (9.6)			
5BW		-- -- --	-- -- --	12.9 (19.42) (22.63)			

(a) Values in parentheses resulted from a second and third set of operating conditions.

(b) Samples taken after 6AA flow rate was established.

TABLE 6-8
STREAM ANALYSES AND FLOW RATES FOR SOLVENT EXTRACTION RUN 69^(a)

Stream	Stream No.	U (g/l)	HNO ₃ (M)	ZrNb (CPM)	Flow (ml/min)	Relative Flow	Other
5AF	1	11.87	2.35	98,467	724 (734) (774)	100 (100) (100)	
5AX			[30% TBP]		465 (488) (471)	64 (66) (61)	
5AP		18.83 (19.92) (18.39)	0.23 (0.22) (0.22)	1,613 (50,639) (107,921)			
5AW		1.4×10^{-3} (1.4×10^{-3}) (1.4×10^{-3})	2.05 (2.15) (2.13)	86,329 (54,439) (29,061)			
5AS			0.98		181 (170) (186)	25 (23) (24)	
5A-DBP			[30% TBP]		-- (11) (27)	-- (1.5) (3.5)	DBP = 25 g/l
5SR		1.76 (1.82) (1.69)	1.73 (1.73) (1.79)	4,484 (11,971) (29,022)			
5SP		18.44 (18.37) (17.16)	0.089 (0.096) (0.096)	49.5 (589) (77,366)			
5BX			0.014		412 (369) (504)	57 (50) (65)	

TABLE 6-8 (Continued)

Stream	Stream No.	U (g/l)	HNO ₃ (M)	ZrNb (CPM)	Flow (ml/min)	Relative Flow	Other
5BP		20.57 (21.88) (22.60)	0.29 (0.27) (0.27)	39.4 (56.8) (6,679)			
6AA		--	13		16 (14) (17)	2 (2) (2)	
6AS		--	2.05		155 (155) (157)	21 (21) (20)	Fe ⁺⁺ = 0.055M
6AW		1.6 x 10 ⁻³ (2.2 x 10 ⁻³) (2.3 x 10 ⁻³)	1.03 (1.02) (1.02)	39.8 (24.9) (937.5)			
6AX			[30% TBP]		335 (316) (330)	46 (44) (43)	
6AP		23.97 (23.45) (25.79)	0.22 (0.21) (0.22)	13.0 (12.5) (14.2)			
6BX			0.014		279 (281) (286)	39 (38) (37)	
6BP		21.03 (27.29) (28.37)	0.45 (0.58) (0.49)	12.9 (14.9) (16.5)			

(a) Values in parentheses resulted from a second and third set of operating conditions.

TABLE 6-9
PERCENT LOSS, ZrNb DECONTAMINATION FACTOR, AND OPERATING DATA FOR SOLVENT EXTRACTION RUN 68^(a)

Contactor	Purpose	Volume Velocity (gal/hr/ft ²)	$\bar{V}_a$ (cm/sec)	$\bar{V}_o$ (cm/sec)	Continuous Phase	Aqueous to Organic Ratio	ZrNb DF U Basis	Uranium Loss (%)	Pulse Frequency (cpm)	Temp. (°C)
5A centrifugal	Extraction	--	--	--	--	1.98 (1.87) (1.54)	88.0 (65.7) (82.5)	<10 ⁻² (<10 ⁻²) (<10 ⁻²)		
5S pulse column	Scrub	443 (475) (557)	0.143 (0.142) (0.156)	0.358 (0.395) (0.473)	Organic	0.40 (0.36) (0.33)	31.4 (37.2) (25.3)	-- (84) (84)	84 (84) (84)	Ambient Ambient Ambient
5B pulse column	Strip	316 (258) (302)	0.198 (0.116) (0.132)	0.159 (0.176) (0.209)	Aqueous	1.24 (0.66) (0.63)	2.0 (2.2) (2.1)	-- (73) (73)	73 (73) (73)	~50 (~50) (~50)
6A pulse column	Extraction-scrub	734 (543) (582)	0.584 (0.366) (0.399)	0.245 (0.248) (0.259)	Organic	2.38 (1.48) (1.54)	2.8 (2.9) (3.1)	0.21 (0.05) (0.11)	80 (80) (80)	Ambient Ambient Ambient
6B pulse column	Strip	479 (494) (485)	0.295 (0.311) (0.290)	0.246 (0.247) (0.258)	Aqueous	1.20 (1.26) (1.12)	0.77 (0.75) (0.86)	-- (73) (73)	73 (73) (73)	~45 (~45) (~45)
							Overall DF 12,062 (11,404) (11,625)			

^(a)Values in parentheses resulted from a second and third set of operating conditions.

TABLE 6-10
PERCENT LOSS, ZrNb DECONTAMINATION FACTOR, AND OPERATING DATA FOR SOLVENT EXTRACTION RUN 69^(a)

Contactor	Purpose	Volume Velocity (gal/hr/ft ²)	$\bar{V}_a$ (cm/sec)	$\bar{V}_o$ (cm/sec)	Continuous Phase	Aqueous to Organic Ratio	ZrNb DF U Basis	Uranium Loss (%)	Pulse Frequency (cpm)	Temp. (°C)
5A centrifugal	Extraction	--	--	--	--	1.95	96.8	0.01		24 ^(b)
		--	--	--	--	(1.81)	(3.26)	(0.01)		(25)
		--	--	--	--	(1.90)	(1.41)	(0.01)		(25.6)
5S pulse column	Scrub	470	0.149	0.382	Organic	0.39	31.9	--	83	23.3
		(486)	(0.139)	(0.410)		(0.34)	(79.3)	--	(83)	(26)
		(497)	(0.152)	(0.409)		(0.37)	(1.3)	--	(83)	(26.7)
5B pulse column	Strip	283	0.151	0.170	Aqueous	0.89	1.4	--	70	51
		(280)	(0.135)	(0.181)		(0.74)	(12.4)	--	(70)	(52)
		(324)	(0.184)	(0.182)		(1.01)	(15.3)	--	(70)	(52)
6A pulse column	Extraction-scrub	667	0.479	0.275	Organic	1.74	3.5	0.01	80	Ambient
		(621)	(0.448)	(0.264)		(1.70)	(4.9)	(0.01)	(80)	Ambient
		(733)	(0.557)	(0.271)		(2.05)	(536.7)	(0.02)	(80)	Ambient
6B pulse column	Strip	446	0.229	0.275	Aqueous	0.83	0.88		68	~45
		(434)	(0.231)	(0.259)		(0.89)	(0.98)		(68)	(~45)
		(448)	(0.236)	(0.270)		(0.87)	(0.95)		(68)	(~45)
							Overall DF			
							13,524			
							(15,533)			
							(14,263)			

(a) Values in parentheses resulted from a second and third set of operating conditions.

(b) Average of 1AW and 1AP outlet temperatures.

TABLE 6-11
DESCRIPTION OF CENTRIFUGAL CONTACTOR AND COLUMN CARTRIDGE FOR SOLVENT EXTRACTION RUNS 68 AND 69

Unit	Purpose	Diameter (mm)	Total Height of Mixing Area (m)	Other			
5A centrifugal contactor	Extraction	180	0.32	8 stages with 0.4-liter holdup per stage			
				Plates			Plate Spacing (mm)
				Nozzle Direction	Hole Size (mm)	Percent Free Area	
				Down	3.2	23	51
				Up	4.8	23	Graded ^(a)
				Down	3.2	23	51
5S column	Scrub	51	5.5	Down	3.2	23	51
5B column	U-strip	76	4.6	Up	4.8	23	Graded ^(a)
6A column	Extraction- scrub	51	6.7	Down	3.2	23	51
6B column	U-strip	51	5.2	Up	4.8	23	51

^(a) Graded cartridge is, from the bottom, 2.6 m with 100-mm spacing, 0.5 m with 76-mm spacing, and the remainder with 51-mm spacing.

The centrifugal contactor was apparently plugged at startup, which caused the aqueous stream to leave the contactor via the normal 5AP route to the 5S column. The introduction of this aqueous system into the 5S column caused still further problems since the aqueous solution was returned directly to the 5A contactor. The 6A and 6B columns flooded at startup.

The entire system was shut down, and the centrifugal contactor was thoroughly flushed with nitric acid. The system was then restarted. The centrifugal contactor had been successfully flushed, and no further operational problems occurred with the 5A-5S system. The 6AA pump system was not initially operable. Temporarily, the 6AA acid was added about 2.4 m (8 ft) below the 6AF addition point. An addition point about 0.3 m (1 ft) above the 6AF addition point was used later in the run. After this flow was established, the acid addition below the 6AF was stopped.

Detectable uranium losses occurred via the 6AW stream in early samples. These losses were the result of the problems with the 6AA stream. After the 6AA stream flow was established, the 6AW uranium losses were significantly reduced.

In Run 68, the overall decontamination factor for radioactive zirconium-niobium was about 12,000. Most of the decontamination was accomplished in the 5A-5S system. Some decontamination occurred in both the 5B and 6A columns.

Ferrous nitrate was added to the 6AS stream to test the system for simulated plutonium partitioning. No plutonium was used in these runs. However, the presence of the ferrous ion tested the chemical requirements for plutonium reduction. The presence of the ferrous ion in the 6AW stream substantiated that the chemical requirements were met.

6.3.5. Discussion - Run 69

Run 69 was similar to Run 68 except that DBP was added to the 5A contactor in the latter part of the run to test the effects of solvent degradation. The 6AA stream was added to the 5BP stream prior to its addition to the 6A column.

Some startup problems occurred but were quickly remedied, and no other significant operational difficulties occurred during the run. One of the startup problems involved the 6AA stream pump. Air which had accumulated in the hydraulic head was removed to restore normal operation. The 6A and 6B columns were flooded at startup. Stable operation was restored by a reduction in the pulse frequency for each column. After the columns stabilized, the normal pulse frequency was reestablished.

Uranium losses in Run 69 were typically low. Uranium losses did occur via the 5BW stream after the addition of DBP to the system.* The DBP which followed the organic phase through the first cycle (5A, 5S, 5B) reduced the uranium stripping efficiency in the 5B column. The DBP changed the dispersion characteristics of the discontinuous organic phase, which caused larger droplets to form and a reduction in the stripping rate for uranium. The distribution coefficient (ratio of equilibrium concentration in the organic phase to the concentration in the aqueous phase) of uranium was apparently increased by the presence of DBP in the stripping column.

Zirconium-niobium decontamination in the first cycle was reduced when DBP was introduced since it increased the extraction of zirconium-niobium in the 5A-5S system. The overall decontamination factor actually increased with DBP present owing to the large increase in decontamination in the 5B column and the 6A column. The complex formed between the zirconium-niobium and DBP prevented most of the zirconium-niobium from stripping with the

*This conclusion is based on the visual observation of the uranium color distribution in the 5B column.

uranium in the 5B column. The zirconium-niobium was carried from the column via the 5BW stream with the DBP which followed the organic phase through the first cycle.

In the second cycle, all of the zirconium-niobium decontamination occurred in the 6A column. The concentration of zirconium-niobium in the 6AP stream leaving the column was about the same regardless of the concentration in the 6AF (5BP) stream. The organic phase uranium loading in the column had been increased in Run 69 to improve the decontamination under the conditions with the highest zirconium-niobium concentration. However, the decontamination achieved in the 6A column appeared to be a function of the concentration of zirconium-niobium in the 6A column feed. The overall decontamination factor in Run 69 was about 14,000.

6.3.6. Results - Runs 70 and 71

Tables 6-12 and 6-13 contain the flow rates and stream analyses for Runs 70 and 71, respectively. Tables 6-14 and 6-15 contain the loss data and other calculated data for the two runs. Table 6-16 gives a description of the pulsed columns and column cartridges.

6.3.7. Discussion - Run 70

In Run 70, the flooding points for the 5A and 6A columns were determined. After flooding, normal operation of both columns was restored. The recovery from flooding of the 6A column was reasonably rapid. However, the recovery of the 5A column took a significantly longer period. The 5A column had a longer scrub section and thus held up a larger amount of aqueous phase during flooding. During the recovery from flooding, the 5AS stream was shut off until the aqueous phase in the scrub section had been depleted. The flood and recovery in the 5A column were typical of the extraction-scrub columns.

TABLE 6-12
STREAM ANALYSES AND FLOW RATES FOR SOLVENT EXTRACTION RUN 70

Stream	Stream No.	Uranium (g/l)	HNO ₃ (M)	ZrNb (CPM)	Flow (ml/min)	Relative Flow	Other
5AF	1	10.69	2.38	107,572	731	100	Zr = 383 ppm
5AS	3		0.69		123	17	
5AX	2		[30% TBP]		[510] ^(a)	70	
5AW	4	2.2 x 10 ⁻³	2.13	87,560			
5AP	5	15.19	0.228	334			
5BX	6		0.024		395	54	
5BP	7	21.5	0.458	384			
5BW	8	1.7 x 10 ⁻³	0.017	116			
6AA	9		13		[19] ^(a)	2.6	
6AS	11		2.38		102	14.0	Fe ⁺⁺ = 0.05M ^(b)
6AX	10		[30% TBP]		275	38	
6AW	13	0.22	1.34	384			
6AP	12	16.51	0.45	28			
6BX	14		0.017		280	38	
6BP	15	17.44	0.93	21			
6BW	16	1.3 x 10 ⁻³	0.017	16			

(a) Estimated from material balance.

(b) As Fe(NO₃)₂.

TABLE 6-13
STREAM ANALYSES AND FLOW RATES FOR SOLVENT EXTRACTION RUN 71^(a)

Stream	Stream No.	Uranium (g/l)	HNO ₃ (M)	ZrNb (CPM)	Flow (ml/min)	Relative Flow	Other
5AF	1	10.6	2.08	91,487	690 (723)	100 (100)	Zr = 383 ppm
5AS			1.02		205 (196)	30 (27)	
5AX			[30% TBP]		464 (449)	67 (62)	
5AW		4 x 10 ⁻⁴ (4 x 10 ⁻⁴)	1.79 (1.81)	67,798 (70,675)			
5AP		16.8 (16.7)	0.187 (0.17)	192 (212)			
5BX			0.017		377 (358)	55 (50)	
5BP		21.5 (21.4)	0.19 (0.21)	230.8 (227)			
5BW		6 x 10 ⁻³ (6 x 10 ⁻³)	0.017 (0.017)	55.2 (57.4)			
6AA			13		25 (22)	3.6 (3)	Fe ⁺⁺ = 0.05M
6AS			1.89		131 (138)	19 (19)	
6AX			[30% TBP]		264 (186)	38 (26)	
6AW		5 x 10 ⁻³ (0.02)	1.18 (1.21)	155 (112)			
6AP		25.8 (32.2)	0.204 (0.187)	11.0 (13.8)			
6BX			0.017		261 (270)	38 (37)	
6BP		23.1 (26.2)	0.28 (0.26)	11.2 (11.2)			
6BW		3 x 10 ⁻³ (2 x 10 ⁻³)	0.014 (0.017)	0.6 (0.5)			

^(a)Values in parentheses resulted from a second set of operating conditions.

TABLE 6-14
PERCENT LOSS, ZrNb DECONTAMINATION FACTOR, AND FLOODING DATA FOR SOLVENT EXTRACTION RUN 70

Column	Purpose	Volume Velocity (gal/hr/ft ²)	$\bar{V}_a$ (cm/sec)	$\bar{V}_o$ (cm/sec)	Flooding Frequency (cpm)	Continuous Phase	Aqueous to Organic Ratio	Uranium Loss (%)	ZrNb DF U Basis	Flooding Frequency (%)	Temp. (°C)
5A	Extraction- scrub	992	0.703	0.420	131	Organic	1.6745	0.02	458	67	Ambient
5B	U-strip	292	0.144	0.186	119	Aqueous	0.7745	0.01	1.2	72	~49
6A	Extraction- scrub	563	0.411	0.227	103	Organic	1.8145	1.5	10.5	83	Ambient
6B	U-strip	403	0.230	0.226	77	Aqueous	1.018	0.01	1.4 Overall DF 8357	94	~38

TABLE 6-15
PERCENT LOSS, ZrNb DECONTAMINATION FACTOR, AND FLOODING DATA FOR SOLVENT EXTRACTION RUN 71^(a)

Column	Purpose	Volume Velocity (gal/hr/ft ²)	$\bar{V}_a$ (cm/sec)	$\bar{V}_o$ (cm/sec)	Flooding Frequency (cpm)	Continuous Phase	Aqueous to Organic Ratio	Uranium Loss (%)	ZrNb DF U Basis	Flooding Frequency (%)	Temp. (°C)
5A	Extraction-scrub	988 (995)	0.736 (0.756)	0.382 (0.370)	131 (131)	Organic	1.929 (2.047)	0.01 (0.01)	755 (680)	61 (61)	Ambient
5B	U-strip	272 (261)	0.138 (0.131)	0.170 (0.164)	119 (120)	Aqueous	0.8125 (0.7973)	0.04 (0.04)	1.1 (1.2)	84 (83)	~50
6A	Extraction-scrub	579 (512)	0.438 (0.426)	0.217 (0.153)	103 (104)	Organic	2.019 (2.785)	0.04 (0.14)	25.2 (24.7)	78 (77)	Ambient
6B	U-strip	382 (332)	0.215 (0.223)	0.217 (0.153)	77 (79)	Aqueous	0.989 (1.452)	0.01 (0.01)	0.9 (1.0)	88 (86)	~38
									Overall DF 17,801 20,190		

^(a) Values in parentheses resulted from a second set of operating conditions.

TABLE 6-16
DESCRIPTION OF COLUMNS AND CARTRIDGES FOR SOLVENT EXTRACTION RUNS 70 AND 71

Column	Purpose	Diameter (mm)	Total Height of Mixing Area (m)	Nozzle Direction	Hole Size (mm)	Free Area (%)	Plate Spacing (mm)
5A	Extraction- scrub	51	6.7	Down	3.2	23	51
5B	U-strip	76	4.6	Up	4.8	23	Graded ^(a)
6A	Extraction- scrub	51	5.5	Down	3.2	23	51
6B	U-strip	51	5.2	Up	4.8	23	51

(a) Graded cartridge is, from the bottom, 2.6 m with 100-mm spacing, 0.5 m with 76-mm spacing, and the remainder with 51-mm spacing.

The run was terminated after failure of the 5BP (6AF) pump. Only one set of end stream samples had been taken. A full recovery from the flooding tests had not been effected when the samples were taken. Some transient conditions still existed. However, only small uranium losses were observed except for a 1.5% loss via the 6AW stream.

Good zirconium-niobium decontamination was obtained in the 5A column. The arithmetic decontamination factor for the 5A column was 458. The overall decontamination factor for the four columns was about 8400. This compares with a decontamination factor of 14,000 in Run 69, in which the Robatel centrifugal contactor and four columns were used.

Some problems were experienced with controlling the 5B column interface. The control valve on the 5BP stream which controlled the interface was apparently leaking. However, this problem was not serious enough to significantly affect the run.

Ferrous nitrate was added to the 6AS stream to test the system for simulated plutonium partitioning. No plutonium was used in these runs. However, the presence of the ferrous ion tested the chemical requirements for plutonium reduction.

6.3.8. Discussion - Run 71

Run 71 proceeded very smoothly. Flooding points were determined for the 5B and 6B columns. Normal operation was quickly restored following the flooding tests.

Uranium losses were controlled at a low level throughout the run. The most significant losses occurred via the 6AW stream. The worst loss via the 6AW stream was 0.14%.

Very good zirconium-niobium decontamination was obtained in Run 71. For the two sets of samples in Table 6-13, the overall decontamination

factors were about 18,000 and 20,000, respectively. These factors are better than any of those reported for other runs with this flowsheet (Runs 68, 69, and 70). Decontamination in both the 5A and 6A columns was greatly improved from Run 70. These columns were stable throughout Run 71 as compared with the flooding tests which were carried out in Run 70. In Run 71, additional time permitted the system to reach a stable, steady-state condition before sampling started. The aqueous to organic flow ratios in both the 5A and 6A columns were significantly higher in Run 71, which increased the effectiveness of scrubbing of zirconium-niobium. These aqueous to organic flow ratios were comparable to those used in Runs 68 and 69.

6.3.9. Results and Discussion - Run 73

Table 6-17 contains the flow rates and stream analyses for Run 73. Table 6-18 contains loss data and other operating data for the run. Table 6-19 contains descriptions of the pulsed columns and column cartridges.

In Run 73, a pulsed column configuration was used which was similar to that used in earlier runs. This configuration was chosen specifically to allow reconfirmation of previous data in light of the difficulties which had occurred with some analytical results of the 1BT and 1CU samples.

The 1BX and 1BS columns were filled with a 0.5M HNO_3 solution for startup. The purpose of this solution was to reduce potential uranium losses from the partition system during startup. No uranium losses occurred from the partition system which exceeded the level of parts per million. Visually, the profile of uranium in the 1BX-1BS system was not different from that observed in previous runs. There was a yellow color (indicating the presence of uranium) in the top of the 1BS column throughout the run, which was also typical of previous runs.

The 1BS column flooded at startup. To recover normal operation, the pulse frequency for the column was reduced and the 1BS flow rate was

TABLE 6-17
STREAM ANALYSES AND FLOW RATES FOR SOLVENT EXTRACTION RUN 73^(a)

Stream	Stream No.	U (g/l)	Th (g/l)	HNO ₃ (<u>M</u>)	ZrNb (CPM)	Flow (ml/min)	Relative Flow	Other
1AF	1	51.7	321	0.849	214,023	96 (124)	100 (100)	Zr = 1.81 g/l
1AS	2			0.95		178 (183)	185 (148)	
1AA	5			13		35 (44)	36 (35)	
1AX	3			[30% TBP]		950 (976)	990 (787)	
1BX	8			0.188		695 (707)	724 (570)	F ⁻ = 0.005 <u>M</u>
1BS	10			[30% TBP]		203 (208)	211 (168)	
1CX	12			0.01		575 (581)	599 (469)	F ⁻ = 0.001 <u>M</u>
1OS						105 (122)	109 (98)	0.27 <u>M</u> Na ₂ CO ₃
1AW	4	7 x 10 ⁻⁴ (1.8 x 10 ⁻³)	1.80 (1.97)	2.21 (2.50)	68,424 (73,141)			Zr = 375 ppm (Zr = 375 ppm)
1AP (1SP)	6	4.84 (5.27)	53.6 (39.9)	0.10 (0.12)	655 (863)			
1BU	11	3.76 (4.36)	1 x 10 ⁻³ (1 x 10 ⁻³)	0.034 (0.034)	4.4 (4.32)			
1BXT		1.19 (0.98)	50.8 (52.9)	0.39 (0.39)	1,231 (1,274)			

TABLE 6-17 (Continued)

Stream	Stream No.	U (g/l)	Th (g/l)	HNO ₃ (<u>M</u>)	ZrNb (CPM)	Flow (ml/min)	Relative Flow	Other
1BT	9	$<4 \times 10^{-4}$ ($<4 \times 10^{-4}$)	33.8 (45.8)	0.368 (0.365)	1,241 (1,423)			
1BSU		3.12 (3.66)	20.1 (19.1)	0.11 (0.09)	19.3 (14.7)			
1CU	13	8.14 (8.89)	2×10^{-4} (6×10^{-4})	0.017 (0.017)	7.07 (6.01)			
1CW	14	2×10^{-4} (5×10^{-4})	2.1×10^{-3} (2.2×10^{-3})	0.018 (0.018)	0.28 (0.18)			
1OW		1×10^{-3} (1.6×10^{-3})	1.5×10^{-3} (1.9×10^{-3})	-- pH = 10.9	5.56 (3.36)			
100		8×10^{-4} ($<2 \times 10^{-4}$)	9×10^{-4} (3×10^{-4})	[30% TBP]	0.32 --			

(a) Values in parentheses resulted from a second set of operating conditions.

TABLE 6-18
PERCENT LOSS, HETS, ZrNb DECONTAMINATION FACTORS, AND OPERATING DATA FOR SOLVENT EXTRACTION RUN 73^(a)

Column	Purpose	Volume Velocity (gal/hr/ft ²)	$\bar{V}_a$ (cm/sec)	$\bar{V}_o$ (cm/sec)	Flooding Frequency (cpm)	Continuous Phase	Aqueous to Organic Ratio	HETS (ft)	Percent Loss		ZrNb DF		Flooding Frequency ^(b) (%)	Temp. (°C)
									U	Th	U Basis	Th Basis		
1A	Extraction	915 (965)	0.254 0.289	0.782 0.803	136 (134)	Organic	0.325 (0.360)	-- --	<0.01 (0.01)	1.80 (2.24)	30.6 (25.3)	54.6 (30.8)	51 (52)	Ambient
1BX	Partition	597 (611)	0.254 0.259	0.422 0.433	82 (82)	Aqueous	0.603 (0.597)	4.2 (Th) 4.2	-- --	0.01 (0.01)	115.6 (165.3)	-- --	77 (77)	Ambient
1BS	Partition-scrub	653 (665)	0.572 0.582	0.167 0.171	84 (84)	Aqueous	3.42 (3.40)	<2 (U) <2	<0.01 (<0.01)	-- --	-- --	0.33 (0.70)	69 (69)	Ambient
1C	U-strip	558 (570)	0.210 0.212	0.422 0.433	96 (95)	Aqueous	0.499 (0.491)	3 (U) 3	<0.01 (0.01)	-- --	1.35 (1.47)	-- --	75 (76)	47 (47)
10	Solvent wash	915 (949)	0.086 0.100	0.950 0.974	119 (118)	Organic	0.091 (0.103)	-- --	-- --	-- --	-- --	-- --	71 (71)	~40 (~40)
											Overall DF			
											4766 (6123)	18.2 (21.5)		

^(a) Values in parentheses resulted from a second set of operating conditions.

^(b) The pulse amplitude in each case is 1 in. (25 mm).

TABLE 6-19
COLUMN AND CARTRIDGE DESCRIPTION FOR SOLVENT EXTRACTION RUN 73

Column	Column Diameter (mm)	Cartridge Height (m)	Plates			Plate Spacing (mm)
			Nozzle Direction	Hole Size (mm)	Free Area (%)	
1A Extraction-scrub	51	6.7	Down	3.2	23	51
1BX	76	5.8	Up	3.2	23	Graded ^(a)
1BS	51	5.2	Up	4.8	23	51
1C	76	4.6	Up	4.8	23	Graded ^(a)
10	51	5.5	Down	3.2	23	51

(a) Graded cartridge is, from the bottom, 2.6 m with 100-mm spacing, 0.5 m with 76-mm spacing, and the remainder with 51-mm spacing.

decreased. After restoration of the normal pulse frequency and flow rate, some additional flooding was experienced in the 1BS column. The column operated near the cyclic flooding region throughout the run.

Localized flooding (an aqueous continuous zone) occurred in the top of the 1A-1S column about 45 min after startup. This aqueous continuous region was present throughout the remainder of the run. Countercurrent flow in this region of the 1A-1S column was not significantly impeded. This scrub section flooding was typical of that observed in some previous runs. Recent LWR scrub section tests indicate this flooding may have been caused by too low a pulse frequency.

Some cyclic flooding also occurred in the 1C column later in the run. The flooding was easily alleviated with a slight adjustment of the column pulse frequency.

The only other problem which occurred during the run was the loss of the 1AF feed stream for about 5 min. The operation of the system was not significantly affected by the loss of feed. The loss of feed had no apparent effect toward reducing the flooding in the top of the 1A-1S column despite the fact that the flooding is sometimes associated with high organic phase loading.

Because problems had been experienced with 1BT uranium analyses, a number of standard samples were submitted with the run samples. The correlation of the analyses of these standard samples was good. Therefore, the reliability of the analyses of the 1BT samples for Run 73 was considered good.

The values for the uranium in the two 1BT samples from the run were both below the lower detection limit of 4×10^{-4} g/l. For the first 1BT sample (Table 6-17), the uranium concentration was less than 12 parts of uranium per million parts of thorium. For the second 1BT sample, the

concentration was less than 9 parts per million parts of thorium. The requirement for the 1BT stream of 20 parts per million parts of thorium is met by this flowsheet and operation.

The thorium present in the 1CU stream in the two samples from the run was low: 25 and 68 parts of thorium per million parts of uranium, respectively. However, a significantly greater amount of thorium was analyzed in the 1BU stream: 266 and 229 parts per million parts of uranium, respectively. The reliability of the aqueous 1CU samples is apparently better than that of the organic samples, such as the 1BU and 1CW samples. The 1CW sample had analyzed thorium values which exceeded those possible based on the 1BU values. Whether the 1CU values or the 1BU values are used, the uranium product specification of less than 500 ppm thorium was comfortably met by this operation.

The zirconium-niobium decontamination was adequate for this operation. The zirconium-niobium decontamination for uranium was over 250 times greater than that for thorium. However, decontamination of thorium exceeded the minimum decontamination factor requirement of 5 by about a factor of 4. The scrub section of the 1A-1S column was short -- only 2.7 m (9 ft). A longer scrub section which is typical of this operation should improve zirconium-niobium decontamination.

Low losses were achieved throughout the system except for the thorium losses via the 1AW stream. A relatively high aqueous to organic flow ratio -- 0.32 to 0.36 -- was used in the 1A column. An aqueous to organic ratio of 0.25 had previously been used for the 1A column operation. The pulse frequency of the 1A column was operated at a low level to control the local flooding. This lowered pulse frequency reduced the extraction efficiency in the 1A column and contributed to the thorium losses.

6.4. BENCH-SCALE INVESTIGATIONS

6.4.1. Fluoride Volatility - Concentration of 1AW Waste Stream

6.4.1.1. Introduction and Summary. Previously reported work (Ref. 6-1) dealt with the volatility of fluoride during batch and continuous feed adjustment operation with simulated Thorex dissolver solution. In the present effort, fluoride volatility during concentration of 1AW solvent extraction waste stream was studied.

In Thorex process applications, it will be necessary to concentrate the 1AW solvent extraction stream prior to waste storage. During this operation, some fluoride volatilization will occur and F^- will be present in the condensate which is ultimately routed to nitric acid recovery. The presence of fluoride ion in the condensate potentially limits the uses of the recovered nitric acid and increases the corrosion rate in the acid fractionator.

A bench-scale study has been performed to determine the extent of F^- volatility during batch and continuous concentration of simulated 1AW process stream solutions. Batch concentration of 1AW solution resulted in the volatilization of ~15% of the fluoride charged to the boiler; however, continuous operation yielded fluoride volatilization equilibrium values of ~5%. Since aluminum is known to form complexes with F^- , the effect of 1AW aluminum concentration was studied. An increase in 1AW aluminum concentration to twice the normal flowsheet value resulted in lower fluoride volatility. In the presence of increased 1AW $[Al^{+3}]$, calculated fluoride losses of ~2% were observed for both batch and continuous operations. This decrease in fluoride volatility will probably not justify doubling the aluminum concentration in the waste unless required for calcination.

6.4.1.2. Experimental and Results. Simulated 1AW waste feed solutions containing $2M\ H^+$, $0.05M$ or $0.10M\ Al^{+3}$, $0.025M\ F^-$, and $0.6\ g/l\ Zr$ (to represent fission products) were prepared and used for all experiments

described herein. Measured volumes of each feed solution were charged to the boiler pot for batch concentration. Continuous operation was accomplished through the use of a metering pump to enable controlled addition (1.4 ml/min to 4.0 ml/min) of 1AW feed to ~100 ml of boiler pot solution. Samples of overhead distillates were taken during concentration and submitted to the General Atomic Analytical Chemistry Department for F^- and H^+ measurements. Data received were used to calculate the percent fluoride volatilized for each experiment.

Data from the batch 1AW concentration experiments are contained in Table 6-20. Tables 6-21, 6-22, and 6-23 contain the results from continuous 1AW experiments.

6.4.1.3. Conclusions. Fluoride volatilization occurred in all experiments performed. Continuous concentration of 1AW waste is preferred, since reduced fluoride losses to the condensate were measured for this operational mode. Apparently, the 1AW feed flow rate is not a critical parameter with regard to F^- volatility during 1AW waste concentration.

Significant reductions in F^- losses were observed for experiments using 1AW feed containing twice the flowsheet $[Al^{+3}]$ specification. This decrease in fluoride volatility will probably not justify doubling the aluminum concentration in the waste unless required for waste calcination.

Additional experiments are being planned in which part of the waste stream feed introduction or a small flow of aluminum nitrate solution will be through a steam stripping tower, since it may be possible to achieve lower F^- volatility in this manner.

6.4.2. Ruthenium Volatility During Feed Adjustment

6.4.2.1. Introduction and Summary. In previous work (Ref. 6-1) a quantitative assessment of ruthenium volatility during feed adjustment was not possible owing to lack of sensitivity in the ruthenium analytical

TABLE 6-20
BATCH 1AW WASTE STREAM CONCENTRATION - DATA SUMMARY^(a)

Feed $[Al^{+3}]$ Conc. (M)	Distillate Collection Rate (ml/min)	Volume Dist. (ml)	F ⁻ Conc. Dist. (mg/ml)	Percent Fluoride Volatile	Total Percent F ⁻ Volatile
0.05 ^(b)	1.78	106	0.007	0.59	15.38
		100	0.165	13.87	
		36	0.011	0.92	
0.10 ^(c)	2.02	106	0.006	0.47	1.88
		100	0.010	0.80	
		38	0.007	0.61	

(a) Initial volume of 1AW feed solution = 250 ml.

(b) Normal flowsheet $[Al^{+3}]$.

(c) Twice normal flowsheet $[Al^{+3}]$.

TABLE 6-21
CONTINUOUS 1AW WASTE STREAM CONCENTRATION - DATA SUMMARY 1^(a)

Feed $[Al^{+3}]$ (M)	Feed Flow Rate (ml/min)	Dist. Volume (ml)	Conc. F^- in Dist. (mg)	Cumulative Percent F^- in Dist.
0.05 ^(b)	1.40	70	0.69	0.85
		72	1.03	1.50
		90	1.50	2.04
		76	1.88	2.60
		70	1.60	2.95
		70	1.86	3.29
		74	1.97	3.56
		62	1.65	3.74
		70	1.86	3.91
		86	2.29	4.09
		195	5.19	4.38
		190	7.58	5.00
		72	2.16	5.07

(a) Initial volume of 1AW feed solution = 100 ml. Volume of boiler pot solution was kept constant at 100 ml during course of run.

(b) Normal 1AW flowsheet $[Al^{+3}]$.

TABLE 6-22
CONTINUOUS 1AW WASTE STREAM CONCENTRATION - DATA SUMMARY 2^(a)

Feed $[Al^{+3}]$ (M)	Feed Flow Rate (ml/min)	Dist. Volume (ml)	Conc. F^- in Dist. (mg)	Cumulative Percent F^- in Dist.
0.05 ^(b)	4.0	240	3.9	2.41
		240	7.8	4.25
		280	6.9	4.55
		240	8.7	5.22
		247	5.6	5.14
		240	5.0	5.03
		68	1.6	5.02

(a) Initial volume of 1AW feed solution = 100 ml. Volume of boiler pot solution was kept constant at 100 ml during course of run.

(b) Normal 1AW flowsheet $[Al^{+3}]$.

TABLE 6-23
CONTINUOUS 1AW WASTE STREAM CONCENTRATION - DATA SUMMARY 3^(a)

Feed $[Al^{+3}]$ (M)	Feed Flow Rate (ml/min)	Dist. Volume (ml)	Conc. F^- in Dist. (mg)	Cumulative Percent F^- in Dist.
0.10 ^(b)	2.40	183	2.12	1.59
		210	3.11	2.21
		178	2.60	2.45
		220	2.55	2.48
		220	2.09	2.40
		155	1.41	2.35

(a) Initial volume of 1AW feed solution = 100 ml. Volume of boiler pot solution was kept constant at 100 ml during course of run.

(b) Twice normal 1AW flowsheet $[Al^{+3}]$.

method. Feed adjustment samples from recent experiments were submitted for ruthenium analysis by an improved atomic absorption (AAS) method currently being used in the Analytical Chemistry Department (Ref. 6-3). Ruthenium was detected in all samples, and reduction of data obtained now permits an assessment of ruthenium volatility during feed adjustment based on the AAS method.

The volatility of ruthenium during a batch feed adjustment of dissolver solution was found to be 0.61%. The volatility of ruthenium during a continuous feed adjustment operation with diluted dissolver solution containing a 10-fold increase in expected ruthenium concentration was found to be 1.43%.

6.4.2.2. Experimental and Results. Thorex dissolver solution containing ruthenium in a Ru/Th weight ratio of 1.4×10^{-3} * was evaporated to 135°C and steam stripped to $-[H^+]$. A packed tower was used to prevent mechanical entrainment of ruthenium to the overhead.

Following batch operation, a continuous feed adjustment was performed with a diluted dissolver solution (200 ml dissolver solution to 2800 ml final volume) containing 900 mg ruthenium (Ru/Th weight ratio = 1.92×10^{-2}).

Data are presented in Table 6-24 for ruthenium overhead concentrations during batch and continuous feed adjustment operations. Ruthenium volatility values of 0.61% and 1.43% were measured for batch and continuous feed adjustments, respectively.

6.4.2.3. Conclusions. The ruthenium feed adjustment volatility levels calculated using data from the improved AAS method are substantially lower than the maximum values calculated from earlier x-ray fluorescence data

*This ratio was chosen to represent dissolver solution resulting from dissolution of full-burnup, 1-yr, four-segment fuel and assumed complete ruthenium dissolution.

TABLE 6-24
RUTHENIUM VOLATILITY DURING FEED ADJUSTMENT OF DISSOLVER SOLUTIONS - DATA SUMMARY

Operational Mode ^(a)	Overhead Sample	Sample Volume (ml)	mg Ru/ Spl. Vol.	Max. Total Ru ^(b) Volatilized/Mode (mg)	Total Ru ^(b) Charged to Boiler Pot Volatilized/Mode (mg)
Batch	Evap. 1	99	0.34	0.51	0.61
Batch	Evap. 2	42	0.02		
Batch	Stm. str. 1	50	0.05		
Batch	Stm. str. 2	51	0.10		
Continuous	OH-1	232	1.18	13.25	1.43
	OH-2	138	0.55		
	OH-3	178	0.52		
	OH-4	215	0.62		
	OH-5	198	0.40		
	OH-6	195	0.78		
	OH-7	319	1.28		
	OH-8	358	2.61		
	OH-9	163	2.30		

(a) Batch operation consisted of evaporating 200 ml of dissolver solution to 135°C, followed by steam stripping to $-[H^+]$. Continuous operation consisted of continuous addition of 1 part dissolver solution to 13 parts water to the $-[H^+]$ boiler pot at 135°C.

(b) Determined by analysis of overhead samples. Feed rate = 3.85 ml/min; temperature = 135°C.

(Ref. 6-1). Since the concentration of ruthenium measured in several of the overhead samples is near the detection limit (1 $\mu\text{g Ru/ml}$ or 1 ppm) reported for the method, future feed adjustment runs will be made with Ru-103 tracer added to allow accurate ruthenium measurements at levels <1 ppm.

6.4.3. Calibration of Conductivity Instrumentation

6.4.3.1. Discussion and Summary. For process control purposes, a continuous measurement of nitric acid content in overhead distillates is desirable. Instrumentation for the in-line measurement of overhead HNO_3 conductance has been obtained and may be useful on heavy metal concentrator or denitrator equipment. This instrumentation would permit process monitoring and provide information for optimization and possible adjustment of experimental parameters during the course of a run.

The recently obtained Beckman Solubridge conductivity indicator has been calibrated using nitric acid solutions of known molarity. The prepared nitric acid solutions covered the range of 0.005M to 1.58M (this includes the equilibrium distillate $[\text{H}^+]$ range required for continuous production of acid-deficient denitrator pot products at temperatures from 115° to 140°C; see Ref. 6-4).

A cursory study of the effect of formic acid on measured nitric acid conductance values indicated little interference over nitric acid concentrations ranging from 0.005M to 1.58M. The effect of formic acid is more pronounced at lower nitric acid concentrations; however, the error is estimated to be $\sim 5\%$ for a nitric acid solution of 0.02M in the presence of 0.24M formic acid. Since the effect of formic acid ($\text{HCOOH } K_a = 1.8 \times 10^{-4}$ at 25°C) on nitric acid conductance measurements is small, suitable values should also be attainable when formic acid is used to assist denitration during feed adjustment.

6.4.3.2. Experimental. All conductance measurements reported herein were made with a Beckman Solubridge conductivity indicator (Model RIC5), temperature compensator (Model T/C KQX9-S), and conductivity cell (Model CEL-TFH50). Standard nitric acid solutions were prepared, and specific conductance and potentiometric values (unit output) were taken following immersion of the cell and compensator probes.

6.4.3.3. Results. A calibration plot of specific conductance versus nitric acid molarity was constructed from the data given in Table 6-25.

6.4.3.4. Conclusions. Conductance measurement appears useful in estimating the nitric acid concentration of overhead distillates even in the presence of formic acid. Installation and evaluation of the conductance instrumentation on the pilot plant concentrator/denitrator are planned.

REFERENCES

- 6-1. "Thorium Utilization Program Quarterly Progress Report for the Period Ending May 31, 1977," ERDA Report GA-A14441, General Atomic Company, June 1977.
- 6-2. Reddick, G. W., "Solvent Extraction in HTGR Reprocessing - Interim Development Report," ERDA Report GA-A13835, General Atomic Company, April 1977.
- 6-3. Schwab, M. R., et al., "Ruthenium Determination by Atomic Absorption Spectroscopy," Atomic Absorption Newsletter 10, No. 1, 15 (1971).
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TABLE 6-25
CONDUCTANCE INSTRUMENTATION CALIBRATION DATA

Solution Make-up	Molarity HNO ₃	Molarity HCOOH	Specific Conductance (micromhos/cm)	Potentiometric Output (mV)
D.I. H ₂ O	--	--	0	0.8
5 ml 1 <u>M</u> HNO ₃ /1	0.005	--	1,000	1.4
20 ml 1 <u>M</u> HNO ₃ /1	0.02	--	4,000	3.4
100 ml 1 <u>M</u> HNO ₃ /1	0.10	--	19,000	12.9
15 ml 15.8 <u>M</u> HNO ₃ /1	0.24	--	45,000	25.6
50 ml 15.8 <u>M</u> HNO ₃ /1	0.79	--	139,000	51.8
100 ml 15.8 <u>M</u> HNO ₃ /1	1.58	--	250,000	66.5
500 ml 15.8 <u>M</u> HNO ₃ /1	7.9	--	435,000	78.5
50 ml 15.8 <u>M</u> HNO ₃ +50 ml 23.6 <u>M</u> HCOOH/1	0.79	1.18	138,000	50.3
20 ml 1 <u>M</u> HNO ₃ +10 ml 23.6 <u>M</u> HCOOH/1	0.02	0.24	4,000	3.9
100 ml 1 <u>M</u> HNO ₃ +50 ml 23.6 <u>M</u> HCOOH/1	0.10	1.2	19,000	13.0

7. DRY SOLIDS HANDLING

7.1. SUMMARY

Qualification testing of components and systems was completed using simulated feed materials. Additional system verification will take place during the forthcoming sequential operations of the head-end pilot plant. Installation of new support structures was completed for the crusher product bunker and the primary burner product bunker. Both bunker systems are fully operational; each bunker's weigh system is being monitored for performance improvements due to the structural changes.

A pneumatic transport test apparatus was completed during the quarter and will be used for various study programs during the next fiscal year. The test apparatus is currently being utilized for testing various samplers.

7.2. INTRODUCTION

As described in the Experimental Plan, the development work is divided into several stages:

1. Cold laboratory development.
2. Hot laboratory development.
3. Cold engineering development.
4. Hot engineering development.
5. Cold prototype development.
6. Procedure development.

During the quarter, progress was made in development stage 3.

7.3. COLD ENGINEERING DEVELOPMENT

7.3.1. Qualification Testing

The activities during the past year have been concerned primarily with qualification testing. Such testing has been defined as the partial verification of the design under simulated conditions. Complete verification of the design will take place during sequential operation of the head-end pilot plant. The qualification testing is divided into two phases. Phase I is concerned with component testing and Phase II with system testing.

The solids handling system is divided into six subsystems (see Fig. 7-1):

- Subsystem No. 1. Crusher product removal system.
- Subsystem No. 2. Primary burner feed system.
- Subsystem No. 3. Primary burner product removal system.
- Subsystem No. 4. Particle classifier feed system.
- Subsystem No. 5. Particle crusher feed system.
- Subsystem No. 6. Secondary burner product removal system.

7.3.2. Component Qualification

Common to each solids handling subsystem are certain components, the performance of which needs to be determined prior to the qualification of each complete subsystem. The component testing described in the Solids Handling Activity Plan (AP520101B, December 9, 1976) is concerned with:

- 1. In-bunker filters.
- 2. Weigh systems.
- 3. Bunkers.
- 4. Feeders.

Fig. 7-1. Solids handling subsystems

A summary of the experimental results of the component testing is presented below. More detailed information on much of the component testing is given in previous quarterly reports.

7.3.3. Results of Component Qualification Testing

7.3.3.1. In-Bunker Filters. Results of the component testing of in-bunker filters may be found in Section 7.4.1.1.3 of Ref. 7-1 and in Section 7.4.2.1.2 of Ref. 7-2.

7.3.3.2. Weigh Systems

7.3.3.2.1. Description

The solids handling bunkers are equipped with an elaborate weigh system supplied by Orbitran, Inc. The system consists of three subsystems (A, B, and C), each having the capacity for four bunkers of three load cells each. The following diagram illustrates the present bunker/weigh cell layout:

One-half of the entire weigh system is utilized for monitoring the solids handling bunkers, the other half being utilized for various other pilot plant systems. This discussion pertains to that portion of the weigh system which is directly related to the solids handling system.

7.3.3.2.2. Test Procedure

The maximum design basis weight was placed on each bunker individually for load cell calibration (except for the crusher product bunker, which has a design basis capacity beyond the presently available calibration weight total of 385 kg). The following design basis weights were placed on and removed from each bunker several times in order to adjust the electronic weigh systems for accuracy and repeatability:

<u>Bunker</u>	<u>Maximum Design Load (kg)</u>
Crusher product	544 ^(a)
Primary burner feed	107
Primary burner product	272
Classifier feed	215
Particle crusher feed	64
Secondary burner product	64

(a) 385 kg maximum available calibration weight.

7.3.3.2.3. Acceptance Criteria

The following accuracies were obtained for the individual weigh cell system:

<u>System</u>	<u>Accuracy</u>	<u>Repeatability</u>
Crusher product bunker	<±0.5% of available load	<±0.1% of available load
Primary burner feed bunker	<±0.5% of design load	<±0.1% of design load

<u>System</u>	<u>Accuracy</u>	<u>Repeatability</u>
Primary burner product bunker	<±0.5% of design load	<±0.1% of design load
Classifier feed bunker	<±0.5% of design load	<±0.1% of design load
Particle crusher feed bunker	<±0.5% of design load	<±0.1% of design load
Secondary burner product bunker	<±0.5% of design load	<±0.1% of design load

These accuracies satisfy the criteria laid down in the Activity Plan, except for the crusher product bunker system, which satisfies the criteria up to the 385-kg available load. Acquisition of additional calibration weights is in progress.

Major efforts have been expended in rework and improvement of the weigh systems, both mechanical and electrical.

7.3.3.3. Bunkers. Results of bunker testing are given in Section 7.4.1.1.2 of Ref. 7-1.

7.3.3.4. Feeders

7.3.3.4.1. Test Procedure

For bunkers with no feeding devices, the Activity Plan requires that the flow rate be compatible with the capacity of the downstream conveying system. Since the secondary burner product bunker is at the end of the head-end, this does not apply. The primary burner product bunker feeds material which is recycled to the burner feed bunker or transported to the classifier. The flow rate into the conveying lines from the burners to the bunkers forms an integral part of the system performance and will be reported as part of the system testing. The compatibility of the flow rate out of a bunker into the conveying line downstream from the bunker can be completely verified only during testing of that downstream system. Nevertheless, some indication is required during component testing. It is

reasonable to assume that flow rates of less than 1 kg/s can be transported by a conveying system with a maximum available pressure drop of 12 in. Hg, and this amount is the provisional acceptability criterion. In the event that the minimum outlet results in too large a flow rate, design changes can be contemplated.

7.3.3.4.2. Results

As noted above, the flow rate out of the secondary burner product bunker is not relevant.

Crushed graphite has been discharged from the crusher product bunker at approximately 0.23 kg/s. A similar rate was observed for the primary burner product bunker.

TRISO fuel particles were discharged through a 0.025-m orifice at 0.36 kg/s, whereas BISO fuel particles are estimated to have a discharge rate of up to 1.04 kg/s through the same 0.025-m orifice. Segregation may occur with burned-back BISO/TRISO fuel mixtures, in which case the flow rate through a 0.025-m orifice (1.04 kg/s for BISO particles alone) may prove too high for the available system pressure drop. For this reason, a rotary feeder valve, similar to that used beneath the primary burner feed bunker, will be placed beneath the primary burner product hopper to throttle the discharge of fuel particles should segregation cause system overloading.

Feed rates up to 1000 g/min were achieved with the classifier feeder using TRISO fuel particles. With a 94% BISO/6% TRISO fuel particle mixture, feed rates up to 1800 g/min were observed. A maximum feed rate of approximately 1700 g/min was observed with a 78% BISO/22% TRISO mixture.

The primary burner feeder capacity was measured at different speed settings for crushed graphite and for simulated fresh feed. See Section 7.4.2.1.2 of Ref. 7-2.

7.3.3.4.3. Acceptance Criteria

The classifier feeder acceptance requirement of 1500 g/min was attained with the simulated feed material of 78% BISO/22% TRISO fuel particles. However, segregation of the fuel particle types caused the feed rate to fall below the 1500 g/min value near the end of the run, when virtually all the remaining feed was TRISO particles.

The primary burner feeder provides satisfactory performance except for feed rate accuracy. The feed rate accuracy criterion of $\pm 5\%$ is considered to be an impractical goal for a rotary feeder valve using nonhomogeneous feed. The feeder performance is acceptable for primary burner operations, and hence no design alternatives are currently being considered.

The Activity Plan calls for a flow rate such that the total system pressure drop is ≤ 40.5 kPa. Feed rates into all systems have been verified with actual feed material where available and otherwise with simulated feed materials. Modifications of equipment and operating procedures were required in some instances to assure acceptable feed rates. The primary burner outlet was equipped with a variable position knifegate valve to handle feed rate variations due to changes in burner feed materials and product temperatures.

Feed rates from the fissile and fertile classifier bunkers into subsystems No. 4 (classifier feed system), No. 5 (particle crusher feed system), and No. 6 (secondary burner product removal system) are controlled by rotary feeder valves added to account for varying types of feed materials which could be deposited in either bunker.

The secondary burner outlet feed rate is controlled by intermittent actuation of the secondary burner outlet valve. A continuously open outlet valve was found to provide too great a feed rate for the system to handle. System modifications are in progress to allow continuous feed from the burner. See Ref. 7-1 for further discussion of this subject.

7.3.4. System Qualification

The goal of system qualification is to ensure that each subsystem can convey material at a rate compatible with upstream and downstream operations so that particle breakage is minimal. This involves establishing the conveying characteristics of each system by measuring pressure drops at different solids flow rates over a range of gas velocities. The determination of the saltation point, i.e., where the suspension collapses and particles fill up the line, is also required. The ability to restart, after plugging the line, by use of the blower only should also be investigated.

The solids handling test rig is equipped with a variable speed blower, which is being used for system qualification. Once the optimal conditions for a given system have been determined, a fixed speed blower, suitably set, can be used.

The final part of system qualification, which is the measurement of fuel particle breakage during sequential operation, can be performed only during pilot plant operation with loaded fuel elements.

7.3.5. Results of System Qualification Testing

7.3.5.1. Crusher Product Removal System (Subsystem No. 1). The crusher product removal system tests consisted of transporting the crushed graphite product directly from the outlet of the UNIFRAME size reduction system through the transport tubing and into the crusher product bunker. The variable speed blower from the test rig was used so that transport gas velocities could be varied. The product feed rate varied because of the operating characteristics of the crushing system. However, the feed rate was steady enough to achieve approximate correlation between predicted and observed values of system pressure drop.

The mean feed rate is determined by dividing the total weight of feed by the total transport test time. For a given feed rate, the pressure drop

across the solids transport portion of the system ($P_2 - P_1$) can be calculated in relation to the transport gas velocity using the pressure drop formula published by the Engineering Equipment Users Association (EEUA) (Ref. 7-3). From previous experiments, it was found that one-half the EEUA value closely correlates with GA's laboratory results.

The calculated pressure drop across the system can be compared with the experimental pressure drop by observing the pressure drop ($P_2 - P_1$) for a particular velocity and feed rate.

A diagram of the piping for subsystem No. 1 is shown in Fig. 7-2. Pressures and pressure drops were measured past the inlet (P_1), at the bunker (P_2), across the in-bunker filters (ΔP_3), at the end of the system (P_4), at the inlet to the variable speed blower (P_5), in front of the orifice plate (P_6), and across the orifice plate (ΔP_7). The temperature at the exit from the variable speed blower was also measured.

The inlet gas velocity is averaged between ambient pressure and P_2 to obtain the mean gas velocity ($\bar{V}_G$) to provide direct comparison between predicted and observed values for the pressure drop across the crusher product removal system. The results of one such exercise in comparing predicted and observed pressure drops in crusher product removal system tests are shown in Fig. 7-3. An average feed rate of 0.15 kg/s was used in the calculations. Correlation of observed and predicted values within $\pm 30\%$ is considered acceptable for pneumatic conveying of solids. The data are representative of several crusher tests utilizing scrap graphite as the feed material.

During simulated blower failure experiments, transport could be restarted consistently if feed to the system was stopped immediately following the blower failure. If feed was continued for any length of time with the blower off, restart was not always successful. A small, high-pressure gas jet located at the entry to the transport system was useful in restarting a plugged system.

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Fig. 7-2. Crusher product removal system

Fig. 7-3. Observed versus predicted pressure drop results for crusher product removal system using crushed graphite fed at ≈ 0.15 kg/s

7.3.5.2. Primary Burner Feed System (Subsystem No. 2). Data were collected during operation of the primary burner feed system in support of primary burner tests. Two different types of feed materials were used: (1) simulated burner feed consisting of 81% crushed graphite, 13% coated BISO fuel particles, and 6% coated WAR fuel particles, and (2) coated TRISO fuel particles. The simulated feed mixture was used for burner startup and the TRISO fuel was used for later bed addition.

The methods of predicting pressure drop across a pneumatic conveying system were outlined in the previous section. The difference between this system and the crusher product removal system previously discussed is the system geometry. The primary burner feed system is shown schematically in Fig. 7-4.

The experiments consisted of pneumatically conveying a known weight of feed material from an entry point beneath the crusher product bunker through the transport tubing and into the primary burner feed bunker. The feed rate was not dependent upon the operation of another system, as was the case with subsystem No. 1, enabling a more consistent feed with the average being representative of the instantaneous rate.

The conveying experiment using the simulated burner feed mixture involved feeding the mixture at 0.23 kg/s into a gas stream of average velocity ranging from 15.4 m/s (on the verge of saltation) to 30.0 m/s. Figure 7-5 compares predicted and observed values of the conveying line pressure drop ($P_2 - P_1$) over the tested range of gas velocities. The agreement between calculated and measured ΔP values is within $\pm 20\%$, except at the low-velocity end where saltation effects and instrument error begin to produce major inaccuracies.

The coated TRISO fuel particle feed experiment was conducted at 0.27 kg/s and average velocities of 22.6 to 33.4 m/s. The feed rate is considered more accurate than that for the simulated feed mixture, since the TRISO feed is much more homogeneous than a mixture containing a large percentage of crushed graphite, which varies greatly in particle size. The

Fig. 7-4. Primary burner feed system

Fig. 7-5. Observed versus predicted pressure drop results for primary burner feed system using simulated primary burner feed at 0.23 kg/s feed rate

test results are in close agreement with the calculated values, as shown in Fig. 7-6.

7.3.5.3. Primary Burner Product Removal System (Subsystem No. 3). The experiments on the primary burner product removal system were dependent upon primary burner activities. Initial tests utilized crushed graphite as the feed material. During the crushed graphite discharge, there was a reducing cone (included angle = 15° , outlet = 0.0475 m) below the outlet ball valve (0.0254 m). Flow rates from 0.65 to 0.86 kg/s were observed for graphite which had been fluidized during burner heatup tests and which had lost considerable amounts of fines. The crushed graphite left in the feed hopper was then fed to the burner and discharged directly. Flow was somewhat erratic and stopped altogether on occasion owing to the presence of fines. Rates of 0.73 to 0.57 kg/s were observed, depending on the amount of gas passing through the plenum. It can be concluded that flow through the vertex of the burner plenum was limiting.

For the discharge of fuel particles, the original 0.05-m gate valve was installed, with a small (0.0191-m diameter) orifice immediately below it. The flow rate of 0.16 kg/s was obtained with no gas flowing through the burner. When gas flowed into the burner through the plenum, the rate increased to 0.20 kg/s. This small increase indicates some aeration, but no fluidization.

The flow rates of simulated fuel (1.0 kg/s for the material used for heatup tests, 0.84 kg/s for material in the feed hopper which went straight through the burner) followed the same pattern as those of crushed graphite. Although the flow rate of the feed still containing fines fluctuated, no blockages occurred.

Results of these initial experiments at ambient temperature are plotted in Fig. 7-7. The primary burner product removal system is shown schematically in Fig. 7-8.

Fig. 7-6. Observed versus predicted pressure drop results for primary burner feed system using coated TRISO fuel particles fed at 0.27 kg/s

Fig. 7-7. Predicted versus observed pressure drops for pneumatic conveying of cold material in the primary burner product removal system

Fig. 7-8. Primary burner product removal system

The first attempt at hot fuel particle discharge was made with the original outlet configuration (0.05-m knifegate outlet valve, below which was a reducing cone with an included angle of 25° and a 0.254-m outlet). The flow rate out of the bed, which was at 700°C, was so large that the blower relief valve opened and the line plugged.

The second attempt was made with an orifice (0.0157-m diameter) below the knifegate valve. The bed was initially at ambient temperature and was heated up in stages. Batches of around 85 kg were successfully dumped at 200°, 400°, and 700°C. The pressure drops were observed, as was the temperature of the gas inside the bunker. The balance of material in the bed was discharged at 190°C, and the temperature then decreased to 120°C.

Flow rates between 0.16 and 0.23 kg/s were observed. The elevated temperature of the bed in the burner meant that the fluidizing gas was sufficiently heated to fluidize. The pressure below the bed, which can be measured only before discharge, was 150 kPa with the complete bed, decreasing to 139 kPa prior to the hottest dump.

As expected, the gas temperature in the product bunker increased steadily throughout each dump (see Fig. 7-9). The increase in gas temperature caused an increase in velocity and consequently an increase in pressure drop. The power setting on the variable speed blower was steadily reduced so that the pressure drop did not increase inordinately.

At the beginning of each dump, there was an initial surge of material, as reflected by the pressure drop. However, the pressure drop soon became steady, and then began to increase again.

After passing through the in-bunker filters, which act as a heat sink, the conveying gas did not get hot during the dump. Following a dump, however, after the filters had warmed up, the gas did get hot.

Fig. 7-9. Temperature of gas in product bunker throughout each dump

In Fig. 7-10, the pressure drops observed during the conveying of hot particles are compared with predictions based on ambient temperatures and with predictions based on calculated temperatures.

Even though Fig. 7-10 demonstrates the need to consider the effect of temperature, as expressed in gas velocity and density, it can be seen that the EEUA method (modified for high temperature) is not very successful at predicting the pressure drop (see Ref. 7-1 for a more detailed discussion of temperature effects). The phenomena occurring in the system are complex, and this simple method requires an additional safety margin to take account of an increased pressure drop when conveying material at elevated temperatures.

The variable position knife-gate valve was installed beneath the primary burner prior to primary burner Test B. The primary burner removal system was successfully operated utilizing the variable position knife-gate valve to control the feed rates during burner Tests B and C. Simulated burner feed (fresh feed) was transported at ambient temperature, and FSV TRISO particle burner product was conveyed both at 700°C and at ambient temperatures during Test B. Burned-back BISO/TRISO fuel was transported at 600°C during Test C.

7.3.5.4. Particle Classifier Feed System (Subsystem No. 4). Subsystem No. 4 may be divided into two distinct transport systems: the primary burner product classifier feed loop (Fig. 7-11) and the classifier recycle loop (Fig. 7-12). Uncoated fertile BISO fuel particles were used for feed material as pressure drop data were collected during pneumatic conveying across each transport loop.

The measured data and the calculated results for the classifier feed loop are compared in Fig. 7-13. A similar comparison of results for the classifier recycle loop is shown in Fig. 7-14. The lesser accuracy in correlation between calculated and measured pressure drop results for the

Fig. 7-10. Measured and predicted conveying line pressure drops in primary burner product removal system with heated fuel particles

Fig. 7-11. Classifier feed system

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Fig. 7-12. Classifier recycle system

Fig. 7-13. Observed versus predicted pressure drop results for classifier feed system using uncoated BISO fuel particles fed at 0.05 kg/s

Fig. 7-14. Observed versus predicted pressure drop results for classifier recycle system using uncoated BISO fuel particles fed at 0.23 kg/s

classifier feed loop is attributable to the small feed rates of 0.05 and 0.06 kg/s as compared with the 0.23 kg/s feed rate of the classifier recycle stream.

7.3.5.5. Particle Crusher Feed System (Subsystem No. 5). The particle crusher feed system tests consisted of transporting uncoated TRISO fuel particles from the fissile classifier hopper to the particle crusher feed hopper, shown schematically in Fig. 7-15. The variable speed blower was used to vary the transport velocities over a wide range. The minimum feed rate determined by the clearances in the rotary feeder valve operating at zero rpm was used for the tests. Results of the observed versus predicted pressure drop analyses are shown in Fig. 7-16. The low feed rate of 0.07 kg/s explains the less favorable correlation obtained. Future tests will study the system at higher feed rates, where the analysis methods are more representative.

7.3.5.6. Secondary Burner Product Removal System (Subsystem No. 6). The secondary burner product removal system may be divided into two subsystems: (1) the BISO transport loop and (2) the secondary burner product removal system (see Fig. 7-17). The BISO system tests consisted of transporting uncoated BISO fuel particles from the fertile classifier hopper to the secondary burner product bunker. The variable speed blower was used to vary the transport velocities. The feed rate of 0.19 kg/s was determined by the clearances in the rotary feed valve at zero rpm. Figure 7-18 compares the predicted and observed pressure drop results for the system tests.

The variable speed blower was also used for secondary burner product removal system tests. An outline of the test results is as follows:

Heatup, 8/17/76: BISO particle heatup test. At ambient temperature, 50 kg of fully coated BISO particles were successfully conveyed with the fixed speed blower at 2100 rpm.

Fig. 7-15. Particle crusher feed system

Fig. 7-16. Observed versus predicted pressure drop results for particle crusher feed system using uncoated TRISO fuel particles fed at 0.07 kg/s

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Fig. 7-17. BISO transport loop and secondary burner product removal system

Fig. 7-18. Observed versus predicted pressure drop results for conveying uncoated BISO fuel particles in the BISO transport loop of the secondary burner removal system (feed rate = 0.19 kg/s)

Run 1, 9/15/76: FSV TRISO fertile ash. Approximately 45 kg were discharged at ambient temperatures. The variable speed blower was set at 65% power. The underpressure at the blower exceeded 40 kPa, the relief valve opened, and the line plugged. The small amount left in the burner was removed at 100% power.

Run 2, 10/5/76: As for run 1, but 100% power was used. The line plugged. The remaining material (23 kg) was removed with the fixed speed blower (0.5 kg/s, $\Delta P = 37$ kPa).

Run 3, 12/8/76: As for run 1, but the fixed speed blower was resheaved from 2100 to 2550 rpm, the underpressure relief valve was reset to 50 kPa, and the bunker top was stiffened. The 46-kg batch was successfully conveyed to 0.69 kg/s at a ΔP of 47 kPa, which remained steady.

Run 4, 1/18/77: The burner product was at a temperature of 140°C. Transport was interrupted when a bursting disc, intended as a backup, ruptured. The line plugged and transport could not be restarted. After cleaning, and blinding off the bursting disc, the remainder was transported.

Run 5, 2/3/77: The burner product was at 100°C. Shortly after discharge began, the ΔP exceeded 48-1/2 kPa and the relief valve opened. The secondary burner outlet valve was closed promptly. The line cleared itself, presumably since only modest quantities of product were present, and transport was resumed. The ΔP never became higher than 47 kPa and dissipated quite quickly.

Following run 5, the secondary burner product removal system was redesigned. The redesigned system will be implemented during the next fiscal year. However, the present system operated satisfactorily for all subsequent burner runs by jogging the secondary burner outlet valve open and shut, providing short but dense bursts of feed to the system. Additional tests will be conducted when the redesigned system is installed.

7.3.6. Component Testing and Improvement

Progress in component testing and/or component improvement has been achieved with weigh systems, samplers, and a new conveying system test apparatus.

7.3.6.1. Test Apparatus. During the reporting period, construction of a new test apparatus was completed. The test unit, shown in Fig. 7-19, consists of a 0.23-m³ capacity feed hopper with a 0.10-m rotary feeder valve below it leading to 0.05-m nominal transport tubing of optional length and routing. The transport tubing has a conveying air inlet filter at one end and extends along an optional path, arriving at a 0.23-m³ product hopper 4.25 m above. The product hopper has a bag filter with an exhaust line at the top leading to a blower and a gate valve below separating the product bunker from the feed bunker. The system forms a closed conveying loop allowing experiments to be conducted over and over without adding material or handling the material being studied.

Numerous experiments are planned for the new test loop. The major studies currently under consideration include testing of samplers (both gravity line and conveying line types), erosion of conveying tube walls, effects of line geometries on system performance, effects of system geometries on material holdup, and testing of candidate prototype components.

7.3.6.2. Samplers. In order to ascertain the degree of success of each unit operation of the head-end pilot plant, it is necessary to take samples. Several commercially available samplers have been acquired for testing and possible use as automatic, in-line samplers for the pilot plant. Initial bench tests (discussed in previous quarterly reports), in general, produced favorable results. It was decided to install several different samplers in the pilot line prior to the initial sequential operation of the plant to maximize the information to be obtained from pilot plant operation. Figure 7-20 displays the planned locations for samplers.

Fig. 7-19. Pneumatic transport test apparatus

Fig. 7-20. Sampler locations prior to sequential operations

Inhomogeneity of the dry particulate solids found in the pilot plant operations, as well as the inherent inaccuracy in any sampling operation of such solids, excludes the use of samplers for accountability or criticality purposes. The purpose of samplers is therefore to monitor the performance of individual process stages by measuring such properties as carbon content, particle size distribution, and fuel particle breakage.

Testing and evaluation of the standard samplers have progressed during the past months, as reported in previous quarterly reports along with detailed descriptions of the individual samplers (Refs. 7-1 and 7-4). More recent work has dealt specifically with the sampling of material while it is moving through a pneumatic transport line. (Previous work dealt solely with gravity discharge lines.)

The completion of a new pneumatic transport test apparatus, discussed in the previous section, has enabled transport line sampling experiments to commence. During the present quarter, work was performed using the Model RTA (retractable tube with air ejection) sampler from Quality Control Equipment Company. The RTA sampler consists of a movable, slotted tube with a small air supply tube located coaxially inside. The tubes extend into the conveying line, collecting a sample from the flowing material stream. The tube then retracts automatically and ejects the sample with a pulse of air from the inner tube. The period, frequency, and air pulse intensity are all adjustable to control sample size and total quantity collected. The RTA sampler is shown in Fig. 7-21.

The RTA sampler was checked out, and preliminary experiments were conducted with crushed graphite. Crushed graphite was pneumatically conveyed through the test apparatus, during which time the RTA sampler was activated. The sample size distribution was analyzed and compared with the size distribution of the transported material. The results were encouraging, but show a tendency for the sampler to take a somewhat coarser sample distribution than the distribution of the bulk feed. The results are shown in Fig. 7-22. Additional tests are planned with crushed graphite and with

Fig. 7-21. Retractable tube with air ejection (RTA) sampler

Fig. 7-22. Size distributions of samples taken by cross-cutting and TS samplers

mixtures of feed to better quantify the RTA sampler performance prior to installation in the pilot line.

7.3.6.3. Weigh Systems. Major structural changes to the weigh systems of two solids handling bunkers were completed during the quarter. The original support structures (Fig. 7-23) of the crusher product bunker and the primary burner product bunker were replaced with stronger, more rigid structures (Fig. 7-24). There were three reasons for the change: weigh cell accuracy, the addition of samplers in the systems, and structural stability.

Bunker support stability was threatened by the conservative design of bunker volumes. As shown in Table 7-4 of Ref. 7-1, the actual bunker volumes for the two systems under consideration are considerably larger than the design volumes. The actual volume is 34% larger than the design volume for the crusher product bunker and 108% larger for the primary burner product bunker. Analyses showed the original bunker support structures to have a marginal load-bearing capability for such increased loads. Increasing the height of the support structures to accommodate the addition of samplers to each system further degraded the load-bearing capabilities.

Under such marginal loading conditions, deflection of bunker supports under load was observed to adversely affect weigh system accuracy. As the bunker supports deformed elastically under load, external connections to the bunker such as metal bellows began exerting resisting forces against the bunker, causing erroneous weigh cell readings.

In summary, new supports were required to increase the safe bunker load capacities, increase weigh system accuracies, and facilitate the addition of samplers beneath each bunker. Preliminary indications verify the intended purpose of the new bunker supports. Preliminary weigh cell calibrations show reductions in error of approximately one-half. Weigh

Fig. 7-23. Bunker and support cage [63.50-cm (25 in.) diameter bunker]

35-135-207~14

Fig. 7-24. New bunker support structures

system accuracy and repeatability will be studied closely over the next few months to quantify the difference in performance due to the more rigid bunker supports.

REFERENCES

- 7-1. "Thorium Utilization Program Quarterly Progress Report for the Period Ending February 28, 1977," ERDA Report GA-A14304, General Atomic Company, March 1977.
- 7-2. "Thorium Utilization Program Quarterly Progress Report for the Period Ending November 30, 1976," ERDA Report GA-A14214, General Atomic Company, December 1976.
- 7-3. Engineering Equipment Users Association (EEUA), Pneumatic Handling of Powdered Materials, Constable and Company, Ltd., London, 1963.
- 7-4. "Thorium Utilization Program Quarterly Progress Report for the Period Ending May 31, 1977," ERDA Report GA-A14441, General Atomic Company, June 1977.

8. GASEOUS EFFLUENT TREATMENT

The draft of the conceptual design package has been completed and internally reviewed and is undergoing revision before final release. It contains:

DDI 565001	Design Document Index: Engineering Scale Off-Gas Treatment System
DC 565001	Design Criteria: Engineering Scale Off-Gas Treatment System
SD 565101	System Description: Burner Off-Gas System
SD 565201	System Description: Dissolver Off-Gas System
PF 565101	Process Flow Diagram: Burner Off-Gas System
PF565201	Process Flow Diagram: Dissolver Off-Gas System
PF 565202	Process Flow Diagram: Dissolver Off-Gas System (Alternative Process)
PI 565001	Piping & Instrumentation Diagram: Combined System (BOG & DOG)
CI 565001	Control & Instrumentation Diagram: Combined System (BOG & DOG)
CI 565101	Control & Instrumentation Diagram: Burner Off-Gas System
CI 565201	Control & Instrumentation Diagram: Dissolver Off-Gas System

All the reactor vessels (adsorption beds and catalytic reactors) and most of the auxiliaries have been sized, and work on a detailed design of the off-gas system will be in progress during the next two quarters.

During this reporting period, some gas analyzers (NO_x , CO, and I_2 analyzers), CO/HT oxidation catalysts (pellets, monoliths, and wire meshes), and molecular sieves (AgX, AgZ, 3A, and 5A) were received. The rest of the molecular sieves and gas analyzers on order are due during the next quarter.

A visit was made to W. R. Grace & Company and Engelhard Industries, the suppliers of molecular sieves and catalysts to GA and the industry experts in the adsorption and catalysis fields, to discuss the off-gas system design and requirements. Valuable information has been obtained from these suppliers and is being incorporated into the design.

9. PLANT MANAGEMENT

9.1. SUMMARY

Information previously generated in the Maintainability and Reliability Study is being integrated with the Reprocessing Plant Operating Mode Simulation Study. A failure mode analysis for dry head-end systems has been initiated. The final draft of the HRDF Head-End Remote Maintenance Report has been completed and is in review. General Atomic personnel attended HET Project planning meetings and the ERDA/Contractor Conceptual Design Review meeting during the quarter. Principal agreements resulting from the review meeting were (1) to begin reprocessing equipment design on October 1, 1977, (2) to delay facility design and construction one year to FY-80, and (3) to use the Conceptual Design Report as the HETF design basis, with the incorporation of medium-enriched uranium (MEU) fuel (~20% enriched fissile, proliferation-resistant fuel). In support of the HET Project planning effort, HETF-Reprocessing system Technical Summaries were prepared, an initial evaluation of the impact on the HETF due to LEU and MEU/Th fuel cycle cases was prepared, and a compilation of "Outstanding Items for Title I Resolution" from the HETF Conceptual Design Report was prepared in conjunction with UCC-ND.

9.2. MAINTAINABILITY AND RELIABILITY

9.2.1. Introduction

Work on Phase I of this study was initiated in January 1977. The objective of the Phase I study is to establish preliminary availability allocation requirements for the HRDF reprocessing facility and its major process systems, based on the HRDF Recycle Plant overall availability requirement. A specific near-term objective is to develop a methodology for establishing availability requirements for major systems.

During the preceding quarter, the availability requirements for major equipment systems in the dry head-end were calculated and plotted as a function of probable upper and lower bounds of system reliability and maintenance downtime. Wet head-end systems capacity data and functional diagrams were prepared.

9.2.2. Activity

Information previously generated is being applied to and interfaced with the Reprocessing Plant Operating Mode Simulation Study. A failure mode analysis for dry head-end systems has been initiated. Effort on this study is being held to a low level because of personnel commitment to higher-priority commercialization activities.

9.3. HOT ENGINEERING TEST REPROCESSING PRELIMINARY DESIGN

9.3.1. HET Project

General Atomic staff members participated in planning meetings with UCC-ND and ERDA WPR staff members to develop task plans, budget requirements, and staffing plans for the HETF-Reprocessing detail design effort.

On July 27-28, 1977, a meeting was held at GA to review developing plans, schedules, and costs for the HET Project. The meeting was attended by GA and UCC-ND project members.

The ERDA/Contractor HET Project Conceptual Design Review meeting was attended at ORNL on August 9-10, 1977. The HET Project Conceptual Design Report was discussed regarding criteria/requirements, facility and equipment features, cost estimate, Schedule 44 for Line Item Funding, alternate facility configurations, impact of MEU fuel, and project plans and schedules. Principal agreements reached with ERDA WPR staff were:

1. Facility design and construction for Line Item Funding will be delayed one year to FY-80.

2. Reprocessing equipment detail design will be started October 1, 1977, and will be carried out on HTGR Fuel Recycle Program operating funds.
3. The HETF planning and design basis will be the Conceptual Design Report, with such revision as required for MEU fuel (approximately 20% enriched fissile, proliferation-resistant fuel).

In support of the HET Project planning effort, several supportive tasks were completed during this reporting period. HETF-Reprocessing system Technical Summaries relating the requirements, equipment, components, and technical issues were prepared for the following systems and subsystems:

System 1100	Fuel Size Reduction
System 1200	Primary Burning
System 1300	Particle Classification and Material Handling
System 1400	Particle Crushing and Secondary Burning
System 1500	Dissolution and Feed Adjustment
System 1600	Solvent Extraction
System 1800	Product Handling
Subsystem 1921	Burner Off-Gas Treatment
Subsystem 1931	Dissolver Off-Gas Treatment

An initial evaluation summary was prepared describing the effect on the HTGR HETF of alternate LEU and denatured fuel cycle cases being considered for the HTGR, LWR, and FBR. The impact on the HETF head-end, aqueous separation, and refabrication operations was summarized for each fuel cycle with regard to the technical issues, process, equipment, and support systems that could be assessed.

A listing and completion schedule of the HETF CDR "Outstanding Items for Title I Resolution" initially prepared by UCC-ND was reviewed.

Comments, including some additional items, were compiled and forwarded to UCC-ND.

Two support tasks are currently in progress. The HETF Level 2 Reprocessing Facility Requirements Document, submitted under the HTGR Fuel Recycle Program, is being reviewed for comment. Calculations of the FSV spent fuel element composition (HEU) are being performed for verification of ORNL ORIGEN calculations used to prepare the Environmental Impact Statement for the HETF.

9.4. HRDF REMOTE MAINTENANCE

9.4.1. Introduction

Study activities for the HRDF Head-End Remote Maintenance Report were completed during the quarter. The final report will provide a review of prior maintenance experiences throughout the nuclear industry and will also include a recommended strategy to be adopted for the HRDF head-end. The final draft of the head-end report has been completed and is now undergoing editorial and internal technical review. Publication is anticipated in September 1977.

9.4.2. Activity

It was concluded from a review of the prior maintenance experience that any maintenance strategy adopted for the HRDF should be a combination of maintenance methods. These methods must be applied consistent with process operation, plant availability, and safety requirements, as well as personnel exposure limits and cost effectiveness. Owing to the current state of the HRDF development program, many of the parameters controlling plant layout, equipment configuration, equipment placement, and process operation have not been defined to the degree necessary to permit establishment of detailed maintenance requirements. However, there is an opportunity to include overall maintenance considerations in the equipment

and facility design and development programs before other considerations have imposed difficult maintenance constraints.

It is thus incumbent to establish clear direction with regard to maintenance strategy to avoid unnecessary redirection or redesign in the future. The maintenance strategy recommended for the HRDF was presented in the form of design guidelines which should be applied in conjunction with other equipment and facility design considerations.

It must be recognized that maintenance will be a dominating factor in the design of the equipment and facilities for the HRDF. Therefore, the guidelines provided must be given priority over other mechanical design considerations consistent with process operational requirements and plant safety where a compromise is necessitated. Plant availability requirements must be satisfied in order for the HRDF to be successful. A tradeoff between equipment reliability and maintenance must be achieved to provide the most cost effective means of meeting these requirements.

The guidelines will be used to form the basis for development of detailed maintenance design criteria later in the program. The HRDF remote maintenance report also includes recommendations for future development programs which must be carried out in conjunction with equipment reliability and operations testing to identify detailed maintenance requirements and develop specialized maintenance equipment, techniques, and procedures.

9.4.3. Maintenance Strategy

The HRDF maintenance study has shown that overall head-end maintenance should encompass a centralized remote maintenance facility in conjunction with remote removal and replacement of process cell equipment. This means that all process cell equipment must be designed to permit remote disconnection and removal from its operating position for transfer to the remote maintenance cell. This is true for both preventive maintenance

operations and replacement of failed equipment. Spare components will be remotely installed in the process cell to replace the defective component, and operation will be commenced with the shortest possible downtime. No decontamination should be required for these operations. Repairs to failed equipment will be performed "off-line" in designated maintenance cells. No remote repairs or "contact" maintenance will be performed in the process cells. The degree to which component disassembly is provided to facilitate remote removal will be determined during final equipment design.

Equipment removal and replacement will utilize a bridge-mounted "stacker" crane (similar to those used in warehouses) for in-cell handling of all equipment modules. A bridge-mounted manipulator provided with portable lights and television will be used in conjunction with the stacker crane to operate remote disconnects which will be provided on all piping and electrical lines. The manipulator can also handle removal and replacement of small components should this be required. During periods of normal process operation, the stacker crane, manipulator, and accessory gear will be "parked" in a shielded area to minimize continuous exposure to the high radiation levels anticipated in the process cell. Maintenance of these items will be provided through access to the maintenance cell. High maintenance components or those sensitive to high radiation levels should be placed in a shielded corridor running around the outside of the process cell, where possible. Access of these items to the process equipment will be through penetrations in the shielded wall. Maintenance of these components will be accomplished in the same fashion as used for the large process equipment, except that a wall-mounted manipulator equipped with lights and television will be used for the remote removal and replacement operations. If equipment which is of a high maintenance nature or sensitive to high radiation exposure must be located in the process cell, the use of installed spares and appropriate radiation shielding must be furnished.

In order to evaluate this recommendation it must, of course, be compared with the alternates available. These include:

1. Contact maintenance.
2. In-cell remote maintenance.

There are variations on both of these approaches, but basically they represent the two major alternatives. These two alternatives have the following advantages and disadvantages relative to the proposed strategy:

● Advantages

1. Downtime is minimized.
 - a. Repairs, if required, will be performed "off-line" in the maintenance cell.
 - b. No time-consuming decontamination of the process cells is required.
 - c. Equipment not involved with the failure can continue to operate during the removal and replacement operations.
2. Specialized in-cell equipment is minimized.
 - a. Special maintenance equipment will be provided in the maintenance cell which can service more than one process cell, thus eliminating duplication and avoiding the use of "portable" equipment. Also, exposure of this equipment to the continual high radiation environment of the process cell is avoided.
 - b. Through-the-wall manipulators can be eliminated in the process cell by using a wall-mounted type.

3. Cell viewing requirements are reduced.
 - a. Television mounted on the handling equipment will replace expensive shielded windows.
 - b. Portable lights will replace extensive permanent cell lights. This also reduces cell heating caused by large fixed lights.
 - c. Viewing "behind" equipment will be eliminated or greatly reduced.
4. Process cell space required for maintenance is minimized.
 - a. The need for clear space all around equipment to permit maintenance access is eliminated.
 - b. Side removal permits vertical stacking of equipment. This minimizes pipe runs and permits use of gravity flow.
5. The variety of handling equipment is minimized.
 - a. Use of a "stacker" crane tends to "universalize" handling of equipment by standardizing interfaces.

● Disadvantages

1. A large spare parts inventory is required.
 - a. Direct replacement of components prior to attempting to effect repairs requires a large inventory of spare parts. However, this can be reduced by standardizing as much as practical so that one spare can serve multiple uses.

2. Close-tolerance placement of equipment is necessary.

- a. Replacement equipment must be matched to a full-scale mock-up of the jig which duplicates the "as-built" cell and equipment conditions. Initial cell construction requires close-tolerance pipe and support location.

3. A large maintenance cell is required.

- a. Since essentially no maintenance is performed in the process cell, a large well-equipped remote maintenance cell will be required.

Complete details of the prior maintenance experience contained in the final report further reinforce the validity of the recommended maintenance strategy.

9.4.4. Maintenance Development Program

9.4.4.1. Reliability Program. Once the maintenance equipment has been designed, a program should be developed and implemented to demonstrate the reliability of this equipment. This demonstration will have two principal objectives: (1) to establish reliability goals for the maintenance equipment, and (2) to qualify this equipment with respect to these goals by testing.

9.4.4.2. Reliability Goals. The reliability goals for the maintenance equipment should be based on the results of three other independent studies, two of which are already identified by the Experimental Plans for Task 200 of the HTGR Fuel Recycle Development Program.

First, the availabilities of maintainable process equipment must be allocated. This allocation is presently planned as the result of the Maintainability/Reliability Study 006 (Subtask 260). Initially, this

allocation will relate availability to reasonable limits of maintainability and reliability for a given system configuration, feed type, and operating cycle. Subsequently, the results of the HRDF Reprocessing Plant Simulation Study 008 (Subtask 600) will narrow the range of allocated availabilities. The results of these studies, together with estimated failure rates for the process equipment, will determine the average allowable downtime for each major maintenance action.

Required mean repair times (MTTR) for the maintainable process equipment will be estimated either from analysis of prototypical cold pilot plant maintainability data or from motion-and-time studies of maintenance procedures for HRDF process equipment. The difference between the average allowable downtime and the required MTTR for each maintainable process equipment item will be an indication of the maximum allowable downtime for the maintenance equipment (if administrative and supply downtimes for the process equipment are ignored).

Finally, the mean downtime (MDT) for the maintenance equipment will be estimated based on motion-and-time studies of its maintenance procedures after this equipment is designed. Once all of this information has been obtained, it will be possible to estimate the required reliability (or mean failure rate) of each generic type of maintenance equipment (cranes, connectors, lift mechanisms, etc.).

9.4.4.3. Reliability Testing. Tests to verify that the maintenance equipment will meet the established reliability goals will be devised based on standard techniques of reliability sampling and statistical control, such as those embodied in a number of military standards. Time sample sizes for these tests can be estimated for various confidence levels. Upper and lower control limits can be established as a basis for acceptance or rejection of the test results.

The maintenance equipment will be designed to deal with the probable-failure mechanisms of process equipment.

The test program planned for the maintenance equipment must consider the following additional factors: (1) the availability, cost, and required amount of test equipment; (2) the availability of space and skilled technical personnel; (3) the ratio of the number of hours of reliability testing per item to the expected number of hours of useful life; (4) the cost of necessary refurbishing of the maintenance equipment after extensive reliability testing; and (5) the ratio of the combined total testing time per item, consisting of such tests as debugging, production, reliability, and special tests, to the expected number of hours of useful life.

9.4.5. Procedure Development

Detailed maintenance procedures must be developed in conjunction with process and maintenance equipment design and development.

Detailed maintenance requirements must be determined to:

- Confirm process equipment configurations.
- Determine maintenance equipment requirements.
- Establish equipment availability.
- Identify equipment cell arrangement requirements.
- Identify spare parts requirements.
- Determine maintenance facility requirements.
- Confirm the cost effectiveness of the maintenance approach.
- Establish maintenance manpower requirements.
- Identify maintenance equipment development requirements.

Prototypical process and maintenance equipment configurations must be proven by actual demonstration of chosen maintenance techniques. This should be accomplished by performing the required maintenance operations under simulated conditions which closely duplicate actual cell conditions. These operations should be carried out in accordance with maintenance procedures which would be "qualified" to confirm suitability and completeness.

After completion of generic maintenance operations, overall HRDF maintenance programs can be formulated into an integrated maintenance plan.

9.4.6. Test Facility Requirements

As indicated above, design, development, and demonstration of maintenance equipment and procedures must be carried out in conjunction with prototypical equipment development. Facilities currently available for cold pilot plant development and testing must be expanded to accomplish these goals.

It is necessary to simulate to the maximum extent practical actual process cell constraints in order to perform meaningful developmental tests. Previously, demonstrations were done on selected components or equipment features within existing facility constraints. This approach is not acceptable for prototypical development. Also, specific design features were not demonstrated on the basis that they represented technology which had been previously demonstrated. This approach must be weighted carefully for future programs, since it is vital to develop an "integrated" process and maintenance system. It may appear obvious that a particular configuration or idea is workable based on someone else's experience or previous demonstration; however, it is essential that this be integrated into GA's situation and adequately demonstrated. In GA's situation, one could assume that the Grayloc flange, for example, being a proven component, can be used throughout the system and that no development or demonstration is required. This would be a dangerous assumption. Unique features of the head-end equipment, process operations, or maintenance might dictate the use of other types of flanges, modifications to the flange, or special handling equipment and procedures. These may become apparent only if attempts are made to perform actual remote maintenance demonstrations.

It is recommended, therefore, that the cold pilot plant facility be expanded and provided with equipment requisite to permit full-scale prototypical remote maintenance demonstrations. Specific requirements

cannot be established without a detailed study performed in conjunction with other process development plans. However, it is possible to set down basic guidelines for structuring the development facility requirements.

Based on the maintenance strategy recommended, the following capabilities should be demonstrated and appropriate facilities and equipment provided for their accomplishment:

- Remote removal of all process equipment.
- Remote reinstallation of all process equipment.
- Remote disassembly of all process equipment.
- Remote reassembly of all process equipment.
- Remote replacement of equipment components.
- Remote disconnection of typical process lines.
- Remote testing techniques.
- Remote inspection techniques.
- Remote decontamination and clean-up techniques.
- Remote adjustment or calibration techniques.
- Remote repair techniques.
- Remote handling capabilities.
- Remote viewing capabilities.
- Remote tool operation.
- Remote service techniques.

Some of these capabilities can be simulated (e.g., decontamination) to the degree necessary to verify technical and operational feasibility and suitability. Others can be done on a limited basis (e.g., testing or inspection) to establish feasibility of techniques, accessibility, and identification of special equipment or procedures required for performance.

Facility-related equipment should include:

- Cell envelope simulation.
- Simulated viewing windows.
- Remote television, three-dimensional type.

- Equipment racks.
- Simulated cell arrangement of equipment.
- Bridge-mounted manipulators.
- Remotely operated crane capability.
- Stacker handling crane with manipulators.
- Through-the-wall master-slave manipulator.
- Lighting simulation.
- Simulated pass-through for equipment (air locks).
- Typical remote service connections.
- Typical remote jumpers.
- Maintenance mock-up jigs.
- Storage rack and stands.
- Glove box simulation.

Process remote operational features requiring demonstration (e.g., remote sampling) should also be considered so that these capabilities can be accommodated.

10. HET FUEL SHIPPING

There was no activity in this area during the present reporting period.

11. HTGR RECYCLE DEMONSTRATION FACILITY

11.1. SUMMARY

Flowsheets for the HRDF Thorex and Purex solvent extraction systems were reviewed and updated to reflect the current process development status. Transmittal of the revised flowsheet drafts and corresponding material balance streams to ORNL/UCC-ND is scheduled for August 31, 1977. A topical report (Ref. 11-1) covering HRDF reprocessing yields and material throughput was released for publication on July 22, 1977. A GASP computer simulation language has been chosen as being most suitable for modeling operating modes in an HRDF reprocessing plant. Development of a simple model of the fuel element crushing system is in progress. General Atomic personnel participated in a review of HRDF plant and storage facility options and recycle cost estimates being developed in the RAMCO HTGR Commercialization Studies.

11.2. REPROCESSING FLOWSHEET REVIEW AND MATERIAL BALANCE

11.2.1. Introduction

This study is part of the continuing technology assessment to ensure that (1) the proposed HRDF flowsheet incorporates recent technology development improvements and new design data and (2) supporting technical programs are apprised of flowsheet design issues requiring resolution. The updated reprocessing flowsheet is intended to become a baseline document for HRDF design definition and to provide guidance for technical development activities.

During the preceding quarter, approved draft copies of the head-end process and off-gas treatment system sections of the topical report were transmitted to ORNL for use in their Commercialization Study activity.

11.2.2. Activity

Flowsheets for the Thorex and Purex solvent extraction systems were revised and updated to reflect the current process development status. The revised process flowsheets and pertinent material balance tables underwent internal technical review. The reviewers' comments are being incorporated into the flowsheets and material balance tables, and draft copies are scheduled for transmittal to ORNL by August 31, 1977.

11.3. REPROCESSING YIELDS AND MATERIAL THROUGHPUT

11.3.1. Introduction

This study defines the basis for material balances to accompany the HRDF reprocessing flowsheets prepared as guidance to the development program. Head-end, off-gas, and solvent extraction material balances have been prepared on the basis defined. Results are given in Ref. 11-1.

11.3.2. Activity

Detailed material balances for Purex and Thorex solvent extraction resulted in the product streams shown in Table 11-1. The final report (Ref. 11-1) was released for publication on July 22, 1977.

11.4. SIMULATION OF REPROCESSING PLANT OPERATING MODES

11.4.1. Introduction

Work on this subtask accelerated in June with the addition of a full-time programmer to the staff. The objective of this effort is to develop a computerized simulation model of an HTGR fuel reprocessing plant. Specific problems to be studied and reported using the model are:

TABLE 11-1
SOLVENT EXTRACTION PRODUCT

	From Thorex Solvent Extraction to Refabrication	From Thorex Solvent Extraction to Th Storage	From Purex Solvent Extraction to Refabrication
Average liquid flow (l/day) ^(a)	40	624	17
Heat load (W/l) ^(b)	0.1	0.002	Negl.
Activity ^(c)			
From fission products (Ci/l)	Negl. ^(d)	0.007	Negl.
From U (Ci/l)	2.2	Negl.	0.05
From Th (Ci/l)	Negl.	0.002	Negl.
Uranium (g/l)	233	0.04	470
Thorium (g/l)	Trace ^(d)	464	Negl.
Other heavy metal (g/l)	--	Negl.	Trace
Fission products (g/l)	Negl.	Trace	Trace
HNO ₃ (M)	0.25	0.5	0.2

(a) Exclusive of rework material. Based on 263 equivalent operating days per year; 10,000 spent standard makeup fuel elements processed per year.

(b) Decay heat.

(c) Based on activity level for Th, U, or fission products given in Ref. 11-2. Does not account for decay time during processing.

(d) "Trace" designates concentration in the range of 10^{-5} . "Negl." designates concentration $\geq 10^{-6}$.

1. The effects of equipment reliability on system performance.
2. The dependence of system performance on the level of surge capacities available.
3. The effects of batch versus continuous operation on system performance.

The model will be used to develop HRDF system availability requirements.

11.4.2. Activity

A GASP training course was attended by a staff representative. This language has been chosen as the most suitable for modeling a combination of batch and continuous operating mode systems (Ref. 11-3).

A GASP simulation language is on the GA computer system, and several runs have been made in order to become familiar with the language. A simple model of the fuel element crushing system is expected to be complete early in September.

11.5. HTGR COMMERCIALIZATION

11.5.1. Introduction

Contributions to commercialization studies during the quarter included review of selected portions of the RAMCO Gas-Cooled Reactor Commercialization Study.

11.5.2. Activity

The R. M. Parsons Company updated cost estimates for a commercial recycle plant, the HRDF plant, and various storage facility options were

reviewed in a meeting at RAMCO, Hartford. Revisions resulting from the meeting were reviewed, and comments were submitted to RAMCO along with comments on the Recycle Costs section of the RAMCO Study report.

REFERENCES

- 11-1. Holder, N., and L. Abraham, "Reprocessing Yields and Material Throughput - HTGR Recycle Demonstration Facility," ERDA Report GA-A14320, General Atomic Company, August 1977.
- 11-2. Hamilton, C. J., et al., "HTGR Spent Fuel Composition and Fuel Element Block Flow," ERDA Report GA-A13886, General Atomic Company, July 1, 1976.
- 11-3. Pritsker, A. Alan, The GASP IV Simulation Language, John Wiley & Sons, N.Y., 1974.

12. NONPROLIFERATION ASSESSMENT FOR THORIUM FUEL CYCLES

12.1. SUMMARY

As part of the nonproliferation study, Level 1 functional flow diagrams were prepared for specified processing areas in HTGR, LWR, HWR, GCFR, and LMFBR thorium fuel cycle options. Fuel material descriptions for discrete processing steps in HTGR fuel cycle options were included.

12.2. ALTERNATE FUEL CYCLES/NONPROLIFERATION STUDY

12.2.1. Introduction

This study provides support to ORNL as lead contractor for an evaluation of the nonproliferation aspects of the thorium fuel cycle for various reactor types. The objectives of the evaluation are to determine the potential for proliferation of nuclear weapons through diversion of fissile isotopes in thorium-based fuel cycles, to evaluate the economics of various thorium fuel cycles, and to determine the development required to attain commercial application of the thorium fuel cycles of interest. General Atomic is providing support in assessment of the functional area of spent fuel reprocessing for HTGRs and other thermal reactors, as well as fast reactors, operating on thorium fuel cycles. The functional area of fresh fuel fabrication for HTGRs is also included in GA's scope of work.

During the preceding quarter, specification trees and Level 0 functional flow diagrams were prepared for (1) reprocessing of HTGR, HWR, LMFBR, and GCFR fuels and (2) fabrication of HTGR fuels. Candidate fuel cycle cases were defined and described, then condensed into representative processing cases as far as practical in order to reduce the ultimate

nonproliferation assessment effort to a concise matrix. Proliferation-critical functions and the relative proliferation attractiveness of process fuel materials were indicated diagrammatically. A brief representative assessment of the state of the art was included. A letter report covering this phase of the work was completed and transmitted to ORNL.

12.2.2. Activity

In accordance with revised alternate fuel cycle matrix guidance issued by ERDA/WPR, Level 1 functional flow diagrams were prepared covering reprocessing of specified HTGR, LWR, HWR, GCFR, and LMFBR Th/U fuel options, plus two add-on HTGR options. Level 1 diagrams were also generated for the corresponding HTGR fresh fuel fabrication options. State-of-the-art assessments and indication of proliferation-critical functions were included with the functional flow diagrams. Fuel material descriptions for each Level 1 step of fresh fuel processing and spent fuel reprocessing for HTGR alternate fuel cycles were summarized in tabular form. The descriptions included fuel enrichment, composition, form, mixture where applicable, and range of radioactivity level. The GA Phase I input to the Alternate Fuel Cycles/Nonproliferation Study was compiled as an informal report and transmitted to ORNL.

APPENDIX A

TOPICAL REPORTS PUBLISHED DURING THE QUARTER

Rode, J. S., "A Design Evaluation of the 40-cm (16 inch) Primary Burner System," ERDA Report GA-A14452, General Atomic Company, June 1977.

Wilbourn, R. G., "Safety Aspects of Solvent Nitration in HTGR Fuel Reprocessing," ERDA Report GA-A14372, General Atomic Company, June 1977.

Holder, N., and L. Abraham, "Reprocessing Yields and Material Throughout - HTGR Recycle Demonstration Facility," ERDA Report GA-A14320, General Atomic Company, August 1977.

Mosen, A. W., "Analytical Chemistry Department Annual Report - 1976," ERDA Report GA-A14500, General Atomic Company, August 2, 1977.

Rode, J. S., "A Design Evaluation of the 20-cm (8 inch) Secondary Burner System," ERDA Report GA-A14510, General Atomic Company, August 1977.

APPENDIX B
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