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Vitrification of Radioactive High-Level Waste by Spray Calcination and In-Can Melting

**M. S. Hanson
W. J. Bjorklund**

July 1980

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CALCINATION AND IN-CAN MELTING

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SUMMARY

After several nonradioactive test runs, radioactive waste from the processing of 1.5 t of spent, light-water-reactor fuel was successfully concentrated, dried and converted to a vitreous product. A total of 97 L of waste glass (in two stainless steel canisters) was produced. The spray calcination process coupled to the in-can melting process, as developed at Pacific Northwest Laboratory, was used to vitrify the waste. An effluent system consisting of a variety of condensation and scrubbing steps more than adequately decontaminated the process off gas before it was released to the atmosphere.

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INTRODUCTION

The research, development and demonstration of the technology for solidifying waste from the processing of light-water-reactor (LWR) spent fuel has been ongoing at the Pacific Northwest Laboratory (PNL). The feasibility of solidifying radioactive wastes was demonstrated in the Waste Solidification Engineering Prototypes (WSEP) program between 1966 and 1970. The waste used in the WSEP program was simulated, power-reactor waste consisting of Hanford reactor-fuel-processing wastes and nonradioactive chemicals.

Although the WSEP program proved that the technology for solidifying high-level radioactive wastes existed, the solidification process needed to be demonstrated using actual power-reactor wastes to show that the LWR fuel cycle could indeed be closed. Therefore, the Nuclear Waste Vitrification Project (NWVP) was initiated to demonstrate that actual LWR high-level waste (HLW) could be immobilized by the vitrification process. The project was divided into two phases: the preparation of the HLW from the spent fuel (which will not be discussed in this report) and the subsequent vitrification of the HLW. The overall project is discussed by Wheelwright (Wheelwright et al. 1979).

In the NWVP, HLW that was recovered from approximately 1.5 t of LWR spent fuel was solidified by vitrifying it into borosilicate glass. The process used was similar to that used in the final stages of the WSEP program (McElroy et al. 1972)--spray calcination in conjunction with in-can melting (SC/ICM). Changes in the hardware were made, including the installation of a new calciner vessel, a vibrator and filters, and a combination condenser-scrubber. However, with some minor exceptions, the remainder of the equipment was unchanged from that used in the WSEP program. This report describes the SC/ICM vitrification process, the preparations for vitrifying radioactive HLW, and the two SC/ICM runs in which two canisters of vitrified HLW glass were produced.

THE SPRAY CALCINATION/IN-CAN MELTING VITRIFICATION PROCESS: AN OVERVIEW

The SC/ICM vitrification process can be divided into three interconnected systems: 1) the spray calciner (SC), 2) the in-can melter (ICM), and 3) the effluent system. The majority of the equipment for each of these systems is located in a shielded, radiochemical processing cell. Operations are controlled remotely using master-slave manipulators and cranes. Each system, its individual components and their functions, is described below.

SPRAY CALCINER

The SC, shown in Figure 1, is a 14-in.-OD, vertically mounted right cylinder with 3/8-in.-thick walls. The length of the calciner spray chamber from the top flange to the cone is 6 ft 5-3/4 in. The chamber is constructed of 310 stainless steel, which was chosen because of its excellent resistance to corrosion and air oxidation at high temperatures. The spray chamber walls are heated to about 750°C by an external, 3-zone, resistance-heated furnace that surrounds the cylinder. The furnace is rated at 20 kW per zone and is fired by a 240-V, 90-A power supply that provides a single phase to each zone.

The HLW is pneumatically atomized into the top of the spray chamber with a commercially available, internal-mix spray nozzle that has been modified to accept a ceramic insert to control erosion in the nozzle orifice. The nozzle also has a bellows-sealed, remotely operable cleanout needle, which can be used to remotely unplug the nozzle orifice. Compressed air is used to atomize the liquid waste. The feed rate is controlled by a pneumatic control valve coupled to a magnetic flowmeter as shown in Figure 2. The feed pump is a 2-hp, centrifugal, stainless steel pump.

As the atomized waste falls through the calciner spray chamber, heat is transferred to the waste from the chamber walls via radiation and convection. The waste is concentrated, dried and oxidized in seconds. The calcined waste, or calcine, is typically less than 0.5% moisture and 1% nitrate. A side-mounted, air-operated vibrator is periodically operated to prevent a buildup of solids on the chamber walls.

FIGURE 1. Spray Calciner Coupled to ICM

FIGURE 2. Spray Calciner Feed System

About 15% to 50% of the calcine is entrained by the water vapor and the atomizing air passing through the spray chamber. Sixteen, porous, sintered stainless steel filters are used to separate the calcine from the gases that pass to the process off-gas system. The filters are 2 in. in dia by 3 ft long and have a nominal pore size of 65 μm . The average filter decontamination factors^(a) are in the range of 10^3 to 10^4 (Hanson and McElroy 1977),

(a) Decontamination factor here is defined as the ratio of the mass of calcine entering the calciner to the mass of the material leaving the sintered stainless steel filters.

which means that more than 99.9% of the solids carried to the filters are de-entrained. The solids accumulated on the filters are periodically removed, to prevent an excess pressure drop, by directing short pulses of compressed air backwards through the filters. The calcine removed from the filters joins that passing directly through the spray chamber and into the calciner cone and is discharged into the ICM.

Glass-forming material (frit) is metered into the SC cone at a rate proportional to the calcine production rate. The frit feeder is a variable-speed, weigh-belt solids feeder. The feeder is located outside of the radiochemical cell in the service gallery, where it is protected from the radiochemical environment and is accessible for routine contact maintenance. As the frit is discharged from the feeder, it is conveyed by gravity through the cell wall into the calciner cone via a 2-in.-dia pipe. To assure smooth gravity flow, the pipe is sloped at no more than a 45° vertical angle from the feeder to the calciner cone. To provide multiple protection against the possibility of accidentally discharging radioactive materials into the service gallery, the frit-conveyor pipe has an air break and an automatically operated air-lock system, all located in a shielded niche in the cell wall (see Figure 3). The air lock consists of a pair of valves that are positioned in series in the frit-conveyor pipe below the air-break funnel. The valves are operated simultaneously with a timer controller and a pneumatic-cylinder actuator. The valves are operated so that when one valve is open the other is closed. When in operation, the frit feeder discharges continuously into the pipe, through the air-break funnel and into the air-lock system. When the upper air-lock valve is open, frit accumulates in the chamber between the two valves. When the lower valve is opened, the accumulated frit is discharged to the calciner cone and more frit begins to accumulate behind the upper valve.

IN-CAN MELTER

In the ICM process (Blair 1976), the final storage canister is used as the melting crucible to vitrify the mixture of calcine and glass-forming frit. The canisters that were used in this project were constructed of 8-in., schedule-40S, 304L stainless steel pipe (see Figure 4). Standard, butt-weld

FIGURE 3. Frit-Addition System

pipe caps were used to form the bottom of the canisters. The canisters were equipped with 18 thermocouples located in three areas: at the centerline, at mid-radius and at the canister wall. The temperatures were then recorded in the three areas axially every 13 in. along the canister's length.

The ICM furnace, shown in Figures 1 and 5, is a 6-zone, resistance-heated furnace. Each of the 6 zones is 14 in. high and has 15 kW of power. The furnace is fired by a 95-V, 50-A, 3-phase AC power supply. The design operating temperature for the furnace is 1050°C. A 15-in.-OD by 1/4-in.-wall Hastelloy-X[®] susceptor tube is used in the furnace. Since the waste is

[®] Tradename of Carborundum, Inc.

FIGURE 4. Waste Canister

FIGURE 5. In-Can Melter Furnace

self-heating due to radioactive decay, the furnace must be cooled to maintain canister wall temperatures within material limits. Each of the furnace zones has a 2.5-kW cooling capacity at 100 scfm, using air as the cooling medium.

EFFLUENT SYSTEM

The effluent system is a major part of the waste solidification process. In this project the effluent system had three major purposes: 1) decontamination of process off-gases, 2) concentration of aqueous HLW feed for the calciner, and 3) determination of the effluents resulting from the solidification process. A flow diagram of the effluent system is shown in Figure 6. The system is, in general, the same as that used in the WSEP program (McElroy et al. 1972), except that the solidifier condenser was replaced with a quench-scrubber column. The use of the effluent system to concentrate HLW feed is discussed under the section entitled "Waste Preparation/Feed Makeup." In this section, the major pieces of equipment and their functions are briefly described.

Quench Scrubber

The quench scrubber, tank (TK) 111, is a 10-in.-ID, schedule-20, 304L stainless steel column packed with 1-in., 304L stainless pall rings. Two liquid-distribution weirs, one near the top of the column and one at the mid section of the column, are used to collect and distribute scrub solution and condensed liquids to the pall-ring packing. A tank at the bottom of the column is used to contain scrub solution. An overflow line that drains to the evaporator maintains a constant liquid level in this tank. The scrub solution from the tank is fed to the recycling pump. The recycled scrub solution is cooled by a water-cooled heat exchanger and is then fed to both the lower and upper weirs. A weak acid stream consisting of fractionator distillate is also fed to the upper weir to scrub the incoming off gas.

A steam jet assembly installed on the down stream side of the scrubber provides vacuum control in the SC. A condenser cools the steam and off gas as it is ejected from the steam jet.

FIGURE 6. Effluent Control System

Evaporator

The evaporator (TK) 113 has two functions: 1) to concentrate HLW before it is spray-calcined and 2) to concentrate condensate during SC/ICM operation. The evaporator is a tube, thermosiphon type that is designed to operate with a maximum-to-minimum operating volume ratio of 18:1 and has an overall heat-transfer rate of $320 \text{ Btu}/(\text{h})(\text{ft}^2)(^\circ\text{F})$. The tube bundle has process solution outside the tubes to minimize choking caused by vapors near the outlet. The evaporator is operated continuously during SC/ICM operation. It is constructed entirely of commercially pure titanium for improved corrosion resistance.

At the maximum boil-up rate of 530 L/h, the boiling-rate-to-cross-section-area ratio is $1600 \text{ lb}/(\text{h})(\text{ft}^2)$. Although the design pressure for the tube bundle is 125 psig, an operating limit of approximately 38 psig was used to eliminate the possibility of red oil reaction. Maximum design boil-up capacity was established by the need for recycled-condensate-to-feed ratios of up to 7:1 while processing feed at a maximum rate of 50 L/h. Condensates are recycled to control the acidity of the evaporator overheads at less than about 0.5M, which improves retention of ruthenium in the waste. The downcomer size was based on the minimum diameter that would permit the shielded, air-purged liquid level tubes and the specific gravity tubes to operate reasonably well. There is a jacket around the reboiler for auxiliary heating with low-pressure steam if needed. Design pressure for the jacket is 50 psig.

The evaporator tower has four stages of aerosol de-entrainment: 1) a simple chevron baffle, 2) dry impingement caps, 3) an upper section for high-efficiency separators, and 4) a mist eliminator. At the maximum boil-up rate, the impingement velocities of stages 1, 2, and 3 are 36, 51 and 85 ft/s, respectively, which is sufficient to remove nearly 100% of the aerosols 25, 7, and 4 μm in dia, respectively. The nominal, evaporator tower pressure drop at maximum capacity is 12 in. H_2O .

Acid Fractionator

The all-titanium acid fractionator (TK 115) consists of a packed distillation column on the top of a standard reboil tank. The top and bottom

spheroid heads are partially reinforced with an extra thickness of titanium plate for added strength. Special features include a tube bundle in the reboiler, a de-entraining sieve plate with bypass provisions in the column, and an integral reflux condenser. The fractionator was designed to accommodate liquid feed with 0.8M HNO_3 and an overhead fraction of 0.01M HNO_3 .

The tube bundle is identical to the one in the evaporator, except that it is shorter and has a shroud to control the boiling pattern. The liquid level in the tank is held constant during the run by controlling reboiler steam flow. Maximum design boil-up capacity, 310 L/h, was based on maximum, nitric-acid distillation rates from the waste evaporator under conditions with partially condensed feed.

The rectifying section of the fractionator column is packed with 1-in.-dia, titanium raschig rings. The exhaust section has a similarly packed section, which is surmounted by a de-entraining sieve plate and an integral reflux condenser. The impingement velocity of the vapors through the 3/16-in.-dia holes of the sieve plate is 150 ft/s at maximum capacity. Minimum velocity to restrict weeping to reasonable amounts is 20 to 25 ft/s. The sieve plate has an alternate, external drain and has chemical-addition piping. The reflux rate is controlled manually. At maximum boil-up capacity, the total column pressure drop is 8 in. H_2O .

Scrubber

The process off-gas scrubber (TK 118) is a 1300-L tank surmounted by a 1-in., raschig-ring-packed scrubbing column. A recirculation pump recycles the scrub solution at rates up to 40 L/min. Gas flows up to 6000 L/min are acceptable.

Miscellaneous Service Tanks

Four stainless steel tanks are available. Each has a volume of about 1000 L. All have agitators or pumps, coils and jackets and are fully instrumented.

MATERIAL BALANCE

The overall theoretical material balance for the SC/ICM system is shown in Figure 7.

FIGURE 7. Overall Material Theoretical Balance for the Design Basis of 10 L/h

- (a) "76-199", -20 +80 mesh with -325 mesh silicon metal
 (b) Steam = 8.8 scfm; atomizing air and NO_x = 6 scfm; inleakage = 4.35 scfm.
 (c) Includes steam to vacuum jet.
 (d) From SC operation only.
 (e) 0.25-s pulse every 10 to 30 min.
 (f) Varies during course of run.
 (g) Includes ~20 scfm from air jet.

PREPARATIONS FOR THE RADIOACTIVE RUNS

In preparation for processing radioactive HLW, a number of nonradioactive SC/ICM runs, both outside and inside the radiochemical cell, were completed. In addition, various operational tests were conducted on the in-cell effluent system. Lastly, actual radioactive HLW was prepared for vitrification by concentrating it and adding nonradioactive chemicals to obtain the required formulation. These preparations are described in detail below.

NONRADIOACTIVE OPERATIONS

The nonradioactive operations that were completed in preparation for processing radioactive HLW consisted of: 1) two SC/ICM runs in equipment located outside of the radiochemical cell, 2) operation of the evaporator/fractionator system and the integrated effluent system inside the radiochemical cell with nonradioactive solutions, and 3) two operations of the SC/ICM system inside the radiochemical cell with nonradioactive, simulated HLW.

Simulated-HLW Operations Outside Radiochemical Cell

Two runs were made with simulated HLW in a nonradioactive laboratory using SC/ICM equipment that is functionally equivalent to the SC/ICM equipment inside the radiochemical cell. The objectives of the operations were to verify operability with the chosen feed formulation, verify the quality of the glass, and get an indication of potential volatilization problems. The composition of the simulated-HLW feed is given in Table 1. The operating parameters for the two nonradioactive runs are given in Table 2.

Visual examination of the glass product from run 1 revealed that there was a layer of sodium molybdate on the surface of the glass and that the glass was grainy. The connecting section between the calciner and the melter was also partially plated with RuO_2 . To alleviate these problems, Si metal was added with the frit during run 2 to act as a reducing agent. Visual inspection of the glass product from run 2 indicated a large improvement in quality over run 1. Only small traces of molybdates were found on top of the glass, and no Ru was found in the connecting section.

TABLE 1. Composition of Simulated HLW

<u>Element</u>	<u>Calcine Oxide Form</u>	<u>Gram Oxide/L</u>	<u>Chemical</u>	<u>Quantity, g/L</u>
Na	Na ₂ O	9.72	NaNO ₃	27.81
Cr	Cr ₂ O ₃	0.83	Cr(NO ₃) ₃ •9H ₂ O	4.37
P	P ₂ O ₅	0.97	H ₃ PO ₄ (75%)	1.79
Sr	SrO	0.74	Sr(NO ₃) ₂	1.51
Zr	ZrO ₂	3.57	ZrO(NO ₃) ₂ •2H ₂ O (1.96M Zr solution)	14.5 (ml/L)
Cd	CdO	0.06	Cd(NO ₃) ₂ •4H ₂ O	0.144
Te	TeO ₂	0.51	TeO ₂	0.51
Ba	BaO	1.15	Ba(NO ₃) ₂	1.96
Rh	Co ₃ O ₄	0.44	Co(NO ₃) ₂ •6H ₂ O	1.59
Ni, Pd	NiO ^(a)	1.62	Ni(NO ₃) ₂ •6H ₂ O	6.29
Fe	Fe ₂ O ₃	19.64	Fe(NO ₃) ₃ •9H ₂ O	99.62
Ru	RuO ₂	2.09	Ru nitrate solu- tion (0.13M Ru in 11M HNO ₃)	122 (ml/L)
Rb	K ₂ O ^(a)	0.29	KNO ₃	0.62
Cs	Cs ₂ O	2.03	CsNO ₃	2.81
Mo	MoO ₃	3.75	MoO ₃	4.68
Tc	MoO ₃ ^(a)	0.93		
Y	RE ₂ O ₃ ^(b)	16.19	RE ₂ (NO ₃) ₃ •6H ₂ O	42.6
La				
Ce				
Pr				
Nd				
Pm				
Eu				
Gd				
U				
Ni				
TOTAL		64.53		

(a) Substitution.

(b) Rare earth oxide mixtures.

TABLE 2. Operating Conditions for Simulated-HLW Operations
Outside Radiochemical Cell

<u>Operating Parameter</u>	<u>Run 1</u>	<u>Run 2</u>
SC temperature, °C	750	750
ICM temperature, °C	1000	1050
Average SC feed rate, L/h	20	20
Waste-oxide-to-glass-frit ratio	1:24	1:2.1
HLW concentration, L/MTU	851	851
ICM additives	None	1.5 wt% Si
HLW H ⁺ concentration, <u>M</u>	5	5

An analysis of the off gas, based on the condensate from the calciner off gas and the scrub solution, revealed the amount of ruthenium and cesium lost to the effluent system (see Table 3). Although the losses to the effluent system appear to be higher for run 2, in actuality they were lower than for run 1 since no plateout was seen. Based on the quality of the glass and the lower potential for volatilization, it was decided that the Si metal additive would be used for the subsequent runs in the radiochemical cell.

TABLE 3. Ruthenium and Cesium Losses in Simulated-HLW Operations

<u>Species</u>	<u>Run Number</u>	<u>Percent In Condensate</u>	<u>Percent Collected In Scrub Solution</u>
Ru	1	0.018	0.0037
Ru	2	0.084	0.006
Cs	1	0.01	0.0013
Cs	2	0.013	0.004

Evaporator and Fractionator Tests

For these tests, which were conducted in the radiochemical cell, the evaporator and fractionator, and their auxiliary systems, were started, operated and shut down twice. The primary objectives were to train personnel, leak-check the equipment, verify service-gallery piping connections, check

in-cell piping, and check pump and agitator operation. The tests were also designed to simulate the HLW concentration operations. The tests were successfully completed in three phases:

1. The evaporator was started containing 1000 L of 1.5M HNO_3 to simulate diluted HLW and was operated with an evaporation rate of 120 L/h for 4 h. The volume of liquid in the fractionator was held constant at 1000 L of 10M HNO_3 .
2. Nitric acid (2M) was pumped to the evaporator from the feed tank at a rate of 180 L/h using the HLW-feed pump system. The evaporator was operated at a boil-off rate of 180 L/h to maintain a constant volume of 500 L to represent acid stripping and further waste concentration.
3. Nitric acid (2M) was pumped to the evaporator at 60 L/h and acid strip water was introduced at 120 L/h. The liquid volume was again held constant.

The evaporator and fractionator systems proved operable with only minor problems uncovered. The HLW concentration procedure also proved viable.

Integrated Effluent System Test

The objectives of the effluent system test were:

- check scrubber service connections in service gallery
- check in-cell piping for continuity and leaks
- verify that the quench-scrubber instrument system was operational
- check vacuum-boost steam jet operation
- obtain pressure data throughout the off-gas system at a variety of air flow rates
- check to see that quench-scrubber samplers were operational
- determine the maximum flow rate attainable through the quench scrubber/evaporator/fractionator/condensate-tank loop
- determine the operating relationships between the quench-scrubber steam jet and an additional, air-operated vacuum jet

- obtain operating experience with the system
- verify operating procedures.

Testing the effluent system involved the operation of the entire system. Since the SC/ICM was not operating, the off-gas blowers were used to pull cell air through a ball valve on the quench-scrubber inlet and through the remainder of the off-gas system. The evaporator and the fractionator were operated during the test. Condensate from the fractionator condenser, which was collected in the condensate tank, was pumped to the quench scrubber for use as scrub solution. The effluent system was started up, shut down, restarted, and then operated until all components reached steady state. The adjustments and tests made are listed in the run chronology (see Appendix). The system vacuum effects are shown in Table 4.

The results of the effluent system tests can be summarized as follows:

- The quench scrubber was operational and functioned as designed.
- The maximum boil-up rate obtained in the fractionator was 365 L/h. (This boil-up rate was the limiting step in the condensate-recycle loop.)
- A satisfactory vacuum was maintained throughout the system, while air was passed through the system at more than 20 scfm with both the evaporator and the fractionator boiling near maximum rates.
- It was determined that the air jet is necessary for maintaining vacuum control during SC operation.
- Venting the storage vault tanks through an auxiliary, rather than the primary, off-gas blower and closing the cross-tie between the blowers significantly increased the system vacuum.
- The pumping capacity of the fractionator condensate tank pump was much higher than necessary to feed the quench scrubber; in fact, the condensate tank could be pumped out in a short period of time if the liquid levels were not watched carefully or the control valves were not properly positioned.

- The fractionator column cooling coil could be used to help balance the condensate-recycle loop, although this may affect fractionation.
- The effluent system was fairly vacuum tight; there was only about 3 cfm of inleakage to the system.
- It made no difference in system operation whether the recycled scrub solution was fed to the upper or lower weir in the quench scrubber.

TABLE 4. Integrated Effluent System Vacuum Effects

Effluent System Components	Data Set						
	1	2	3	4	5	6	7
Quench scrubber outlet pressure, in. H ₂ O	-2.8	-2.8	-3.6	-3.6	-4.0	-8.4	-8.0
Quench scrubber ΔP , in. H ₂ O	1.6	2.0	3.0	3.0	1.6	2.0	1.4
Evaporator pressure, in. H ₂ O	-0.8	-0.4	-3.6	-4.0	-4.8	-8.0	-7.2
Fractionator pressure, in. H ₂	-3.2	-2.8	-6.0	-5.2	-7.2	-10.4	-9.2
Scrubber pressure, in. H ₂ O	-23.6	-22.0	-14.8	-6.8	-3.6	-4.0	-0.8
Air jet ΔP , in. H ₂ O (flowrate, scfm)	2.0 (16)	2.0 (16)	2.8 (17)	3.2 (17.5)	4.0 (20.5)	4.0 (20.5)	3.6 (17.7)
Steam jet setpoint, %	30	30	30	30	30	30	30
Steam jet bypass valve setpoint, % open	100	100	50	50	50	50	50
#1 vent jet air supply setpoint, %	0	0	40	75	75	75	80
#1 vent jet bypass valve setpoint, % open	100	100	50	50	0	0	0

Nonradioactive SC/ICM Run 1

The objectives of the first nonradioactive operation of the entire SC/ICM vitrification system inside the radiochemical cell were to:

- verify operation of SC/ICM system
- train personnel
- obtain pressure data throughout the system at predicted operating parameters
- precoat the new, sintered-metal filters in the calciner.

This test run was conducted using the operating parameters anticipated for the subsequent radioactive runs, with one exception. A canister that had been partially filled in the WSEP program was used to contain the glass product; thus, the lower zones of the ICM furnace were not operated in order not to melt the WSEP glass during this run. Simulated HLW was made up and transferred to the feed tank and was then fed to the SC/ICM.

After completing a prestart checkoff and a vacuum test of the system, startup was begun. Frit was added to the frit feeder and the necessary acid was added to the evaporator and fractionator. The evaporator and fractionator were brought to a boil, frit was added to the melter to cover the WSEP glass, and the power was turned on to the SC and ICM furnaces. When the calciner was brought to operating temperature (750°C), it was not possible to maintain a vacuum in the calciner. A systematic check of the piping and flanges revealed no leaks; it was then noticed that the calciner pressure was cycling in frequency with but was lagging the calciner zone-3 temperature. The power to zone 3 was turned off, and as zone 3 cooled the calciner vacuum increased (see Figures 8 and 9). This indicated that the leak was in the area surrounding zone 3; tightening the bolts on the main flange that connects the calciner chamber to the cone reduced the leak to an acceptable level.

When the leak was reduced to an acceptable level, the furnaces were reheated and startup continued. A fail-safe system that interlocks the HLW feed pump to the atomizing air was tested and found to be acceptable. While testing the air-operated calciner vibrator, the air-inlet block connector to

FIGURE 8. Spray Calciner Pressure During Leak Testing

FIGURE 9. Spray Calciner Zone Temperature During Leak Testing

the vibrator broke. The decision was made to continue the run without the vibrator. The quench-scrubber condensate recycle loop was started, and the entire effluent system was brought to steady state. Water was initially fed to the calciner, and after a few minutes a switch was made to simulated-HLW feed.

Initially some problems were encountered in controlling the feed rate, but these were soon corrected. The feed pump was on for 4 h, and a total of 79.4 L of simulated-HLW feed were calcined; the average feed rate was 19.85 L/h. Shutdown followed. The operating parameters for the run are given in Tables 5 and 6. The run successfully fulfilled the objectives of proving the operability of the SC/ICM and providing staff training.

TABLE 5. Nonradioactive Run-1 Operating Parameters

Parameter	Material/Value
Feed type	Simulated HLW (see Table 1)
Total time of SC/ICM operation	4 h
Feed consumed	80 L
Calcine produced	5 kg
Frit type used	76-101 (-20 +80 mesh) with 2.25 wt% Si metal
Atomizing air flow rate	6 scfm
Spray calciner filter Δ pressure	5.4 in. H ₂ O maximum
Feed flow rate	15 to 40 L/h
ICM furnace temperatures	
Zone 1	644°C
Zone 2	644°C
Zone 3	630°C
Zone 4	630°C
Zone 5	280°C (power off)
Zone 6	70°C (power off)

TABLE 6. Nonradioactive Run-1 Operating Temperatures

Spray Calciner	Temperature, °C	
	Prior to Feeding	2 h after Feeding
Furnace		
Zone 1		
Point 1	750	775
Point 2	775	800
Zone 2		
Point 1 ^(a)	660	680
Point 2	775	800
Zone 3		
Point 1	670 to 725	730 to 800
Point 2	670 to 725	700 to 775
Thermowell		
4 in. from top	570	400 (350 at shutdown)
22 in. from top	775	610 (510 at shutdown)
44 in from top	780	600 (530 at shutdown)

(a) Thermocouple near hole on furnace for vibrator

Nonradioactive SC/ICM Run 2

The objectives of the second in-cell run were to:

- verify SC/ICM system operability
- adjust and test the air-operated vibrator
- run at 15 L/h for 24 to 30 h using simulated HLW feed
- determine optimal operating parameters for the effluent system during SC/ICM operation
- determine operating relationships between the quench-scrubber jet and the vent jet during SC/ICM operation
- familiarize personnel with sampling procedures

- test gas and liquid samplers during operation
- train personnel in the handling of samples
- obtain further operating experience with the SC/ICM system.

During nonradioactive run 2, the effluent system was again operated to simulate subsequent radioactive runs. The evaporator and fractionator were boiling, and the condensate from the fractionator was collected and continuously pumped to the quench scrubber to serve as scrub solution and strip water. The vent jet and the quench-scrubber jet were operated as necessary to maintain a sufficient vacuum throughout the system. The condensate from the SC off gas and the quench-scrubber jet was collected in the fractionator condensate tank.

The operating conditions for nonradioactive run 2 are shown in Tables 7, 8, and 9, and the run chronology is given in the Appendix.

TABLE 7. Nonradioactive Run-2 Operating Parameters

Parameter	Material/Value
Feed type	Simulated HLW (see Table 1)
Total time of SC/ICM operation	30 h
Feed consumed	337 L
Calcine produced	22 kg
Frit type used	76-101 (-20 +80 mesh) with 2.25 wt% Si metal (-325 mesh)
Atomizing air flow rate	7.2 scfm
Nozzle back pressure	43 psig
Spray calciner filter Δpressure	1.5 in. H ₂ O
Feed flow	
Controller	20% to 25% valve opening
Feed rate (from magnetic flowmeter)	12 to 15 L/h
Tank dropout rate	11.2 L/h (average)
ICM furnace temperatures	
Zone 1	1035°C
Zone 2	1035°C
Zone 3	1035°C
Zone 4	1030°C
Zone 5	700°C
Zone 6	300°C (power off)

TABLE 8. Nonradioactive Run-2 Operating Temperatures

<u>Spray Calciner</u>	<u>Temperature, °C</u>		
	<u>Prior to Feeding</u>	<u>2 h after Feeding</u>	<u>3 h after Feeding</u>
Furnace			
Zone 1			
Point 1	750	750	775
Point 2	780	785	790
Zone 2			
Point 1 (a)	625	620	600
Point 2	775	775	775
Zone 3			
Point 1	700	700	680
Point 2	700	660	650
Thermowell			
4 in. from top	550	275 to 330	275 to 330
22 in. from top	750	450 to 445	450 to 445
44 in. from top	750	405 to 505	405 to 505
Filters			
Front	300	250	230
Back	305	--	--
Off gas	255	250	230
Cone	430	350	305
SC/ICM connecting section	180	160	160

(a) Thermocouple near hole on furnace for vibrator.

TABLE 9. Nominal Operating Parameters for Effluent System--
Nonradioactive Run 2

Parameter	Value
Spray calciner pressure	-14 in. H ₂ O
Noncondensable gas flow rate	17.5 scfm
Quench scrubber	
Vessel pressure	-8.9 L
Column outlet pressure	-16.4 in. H ₂ O
Condensate tank pressure	-14.4 in. H ₂ O
Evaporator	
Specific gravity	1.11
Volume	610 L
Pressure	-8.4 in. H ₂ O
Liquid temperature	104.25°C
Vapor temperature	94.5°C
Evaporator condenser pressure	-10 in. H ₂ O
Evaporator to fractionator conductivity	0.7M H ⁺
Fractionator	
Pressure	-10 in. H ₂ O
Specific gravity	1.22
Volume	1115 L
Temperature	94.5°C
Fractionator condenser to evaporator flow	0 L/h
Fractionator distillate receiver	
Volume	233 to 817 L
Pressure	-7.6 in. H ₂ O
Preheater pressure	-38 in. H ₂ O
Scrubber	
Volume	554 L
Pressure	-9.2 in. H ₂ O

The results of run 2 can be summarized as follows:

- The SC and ICM furnaces functioned as designed.
- The HLW feed system functioned as designed.
- The air-operated vibrator was adjusted; however, it had to be manually operated (required turning switch for solenoid valve on and off several times) due to high pressure drops across the solenoid valve, the wall penetration tailing, and the disconnectors on the vibrator piping. The vibrator was actuated approximately every 30 min.
- The filter blowback system functioned as designed.
- Cooling individual zones on the ICM was difficult to do without affecting the other zones.
- The feed-control valve opened only 20% to 25% because of controller problems, which resulted in a feed rate of only 15 L/h with the recirculation valve wide open. The feed control valve should ideally open 60% to 80%.
- The thermowell in the SC chamber provided valuable information on probable wall scaling.
- Noncondensable gas flow through the system, as measured upstream of the packed scrubber, was up to 26.8 scfm prior to the run. However, because an adequate vacuum could be attained by the use of the quench-scrubber jet, the decision to continue the run was made. After feed to the calciner was started, the noncondensable gas flow slowly decreased until it leveled out at about 17.5 scfm. Both vacuum jets had to be closed down as the run progressed. The buildup of solids was as expected; a pressure drop of 2.4 in. H₂O was reached as the system lined out.
- Blowback pulses affected the pressure in the SC and quench scrubber, but did not affect the evaporator or systems downstream. The pulses did not show up as significant noncondensable gas flow.
- The fractionator condensate tank could easily be pumped dry during startup, unless closely controlled.

- During normal operation, the amount of liquid in the condensate tank increased 52 L/h. As necessary, the liquid in the condensate tank was jetted to low-level vault tanks for storage, before it was evaporated following the run.
- There was a vacuum loop in the drain of the high-efficiency particulate air (HEPA) filter preheater and the scrubber. To prevent problems, the preheater pressure must remain higher than -30 in. H₂O.
- Liquid lost from the packed scrubber averaged about 4.5 L/h. Water was added as required to maintain a constant level of approximately 500 L.
- Operating a single liquid sampler increased the noncondensable gas flow rate about 3 scfm.
- Not all of the samplers operated. The O-rings on some of the liquid samplers needed to be replaced and the tygon tubing initially used would not support a sufficient vacuum.

In summary, the two nonradioactive runs inside the radiochemical cell demonstrated that the SC/ICM vitrification system was operable and provided needed training for subsequent radioactive runs.

WASTE PREPARATION/FEED MAKEUP

The spent fuel from which the HLW was generated came from the storage pool at Nuclear Fuel Service's West Valley plant. General data on the spent fuel are given in Table 10. Note that two different wastes were used for the two radioactive runs. Waste from the low-burnup fuel was used for run 1, and waste from both fuels was used for run 2. The constituents of the HLW used in the two radioactive runs are given in Tables 11 and 12. Inert, nonradioactive chemicals were added to the HLW waste to simulate the presence of intermediate-level wastes, corrosion products, degraded solvent, etc. The inert additives were the same for the two runs and are listed in Table 13.

The processing of 1024 kg of U-equivalent spent fuel produced a volume of 8370 L of HLW or about 8175 L/MTU. Since it was desired to feed the spray calciner with HLW at 851 L/MTU, a nearly 10/1 concentration was necessary. The processing scheme for concentrating the HLW is shown in Figure 10.

TABLE 10. Spent-Fuel Average Data

Parameter	Low-Burnup Fuel	High-Burnup Fuel
Reactor	Point Beach	Point Beach
Fuel type	Pressurized water reactor	Pressurized water reactor
Configuration	14 x 14 (179 rods)	14 x 14 (179 rods)
Initial enrichment	2.27 wt% ^{235}U	3.04 wt% ^{235}U
Final enrichment	0.85 wt% ^{235}U	0.94 wt% ^{235}U
Discharge date	October 1972	April 1974
Burnup	20,500 MWd/MTU	29,282 MWd/MTU
Average power	29 MW	30 MW
Separation	Dec. 1978 - Jan. 1979	Feb. - Mar. 1979

The concentration of the HLW went well, with the exception of two minor problems. The first problem occurred approximately 21 h into the concentration process for run 1. At this point, an increase in the pressure drop across the evaporator tower indicated that the Brinks[®] mist eliminator in the top of the de-entrainment tower of the evaporator was becoming plugged. The plugging is thought to have been caused by foaming of the HLW in the evaporator and/or by the formation of acid-insoluble, dibutyl-phosphate (DBP)-iron complexes. An attempt to unplug the mist eliminator by flushing it with HNO_3 and Na_2CO_3 was only partially successful. The amount of liquid in the evaporator was lowered from 1000 to 600 L to increase the head space in the tank, the boil-off rate was lowered, and Antifoam B[®] was added at 5 ppm (by volume). However, the pressure drop remained high, and it was necessary to remove the mist eliminator and replace it with a stainless steel, mesh-type mist eliminator. The plugging problem did not reoccur for the remainder of the concentration process.

The second problem also occurred during run 1. A neoprene diaphragm in a diaphragm-operated control valve that was used to control the flow of strip water from the fractionator condenser to the evaporator ruptured. For the remainder of the run it was necessary to transfer the strip water in 100-L batches with a steam jet. The diaphragm-operated control valve was replaced.

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Table 11. Waste Constituents for Radioactive SC/ICM Run 1^(a)

Element	Grams	Moles	Oxide Formed	g Oxide
Fission Products				
Ge	0.22	0.003	GeO ₂	0.3
As	0.05	0.0003	As ₂ O ₃	0.07
Se	31.7	0.40	SeO ₂	44.5
Rb	203.0	1.19	Rb ₂ O	222.0
Sr	503.0	5.74	SrO	594.8
Y	284.0	1.60	Y ₂ O ₃	360.7
Zr	2280.0	24.99	ZrO ₂	3079.8
Mo	2130.0	22.20	MoO ₃	3195.9
Tc	530.0	2.68	Tc ₂ O ₇	829.8
Ru	1350.0	13.36	RuO ₂	1777.8
Rh	288.0	1.40	Rh ₂ O ₃	355.2
Pd	838.0	7.88	PdO	964.0
Ag	41.7	0.19	Ag ₂ O	44.8
Cd	45.3	0.40	CdO	51.7
In	1.0	0.009	InO	1.1
Sn	31.5	0.27	SnO ₂	40.0
Sb	7.7	0.03	Sb ₂ O ₃	9.2
Te	348.0	2.73	TeO ₂	435.3
Cs	1530.0	5.76	Cs ₂ O	1622.0
Ba	975.0	7.10	BaO	1089.0
La	782.0	2.81	La ₂ O ₃	917.1
Ce	1520.0	10.85	CeO ₂	1867.0
Pr	738.0	0.87	Pr ₆ O ₁₁	891.6
Nd	2520.0	8.74	Nd ₂ O ₃	2939.0
Pm	22.1	0.08	Pm ₂ O ₃	25.7
Sm	571.0	1.90	Sm ₂ O ₃	662.2
Eu	95.3	0.31	Eu ₂ O ₃	110.4
Gd	54.3	0.17	Gd ₂ O ₃	62.6
Tb	1.2	0.002	Tb ₄ O ₇	1.4
Dy	0.8	0.002	Dy ₂ O ₃	0.9
Subtotal	17.7 x 10 ³			22.20 x 10 ³
Actinides				
U ^(b)	4850.0	6.79	U ₃ O ₈	5719.0
Np	252.0	1.06	NpO ₂	286.0
Pu	38.4	0.16	PuO ₂	43.5
Am	84.3	0.35	AmO ₂	95.5
Cm	4.6	0.01	Cm ₂ O ₃	5.1
Subtotal	5.23 x 10 ³			6.15 x 10 ³
TOTAL	22.95 x 10 ³			28.35 x 10 ³

(a) Based on 1 MTU.

(b) Addition of depleted U required to achieve this value.

Table 12. Waste Constituents for Radioactive SC/ICM Run 2(a)

Element	Grams	Moles	Oxide Formed	g Oxide
Fission Products				
Ge	0.33	0.005	GeO ₂	0.5
As	0.07	0.0005	As ₂ O ₃	0.1
Se	4.65	0.06	SeO ₂	6.5
Rb	307.0	1.80	Rb ₂ O	335.7
Sr	758.0	8.65	SrO	896.4
Y	422.0	2.37	Y ₂ O ₃	535.9
Zr	3330.0	36.51	ZrO ₂	4498.0
Mo	3100.0	32.31	MoO ₃	4651.0
Tc	759.0	3.83	Tc ₂ O ₇	1188.0
Ru	1920.0	19.0	RuO ₂	2528.0
Rh	369.0	1.79	Rh ₂ O ₃	455.0
Pd	1210.0	11.37	PdO	1392.0
Ag	53.7	0.25	Ag ₂ O	57.7
Cd	69.1	0.61	CdO	78.9
In	1.2	0.01	InO	1.4
Sn	44.8	0.38	SnO ₂	56.9
Sb	11.4	0.05	Sb ₂ O ₃	13.6
Te	502.0	3.93	TeO ₂	627.9
Cs	2240.0	8.43	Cs ₂ O	2375.0
Ba	1420.0	10.34	BaO	1585.0
La	1140.0	4.10	La ₂ O ₃	1337.0
Ce	2230.0	15.91	CeO ₂	2739.0
Pr	1080.0	1.28	Pr ₆ O ₁₁	1305.0
Nd	3680.0	12.76	Nd ₂ O ₃	4293.0
Pm	37.0	0.13	Pm ₂ O ₃	43.0
Sm	803.0	2.67	Sm ₂ O ₃	931.2
Eu	150.0	0.49	Eu ₂ O ₃	173.7
Gd	91.2	0.29	Gd ₂ O ₃	105.1
Tb	1.58	0.002	Tb ₄ O ₇	1.9
Dy	0.92	0.003	Dy ₂ O ₃	1.9
Subtotal	25.7 x 10 ³			32.2 x 10 ³
Actinides				
U ^(b)	4800.0	6.72	U ₃ O ₈	5660.0
Np	426.0	1.80	NpO ₂	483.5
Pu	40.6	0.17	PuO ₂	46.0
Am	297.0	1.23	AmO ₂	336.4
Cm	11.4	0.02	Cm ₂ O ₃	12.5
Subtotal	5.58 x 10 ³			6.54 x 10 ³
TOTAL	31.3 x 10 ³			38.7 x 10 ³

(a) Based on 1 MTU.

(b) Addition of depleted U required to achieve this value.

TABLE 13. Inert Chemicals Added to HLW

<u>Compound</u>	<u>Amount Added, kg/MTU</u>	
	<u>Run 1</u>	<u>Run 2</u>
NaNO_3	23.67	34.91
$\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	84.55	124.71
$\text{Cr}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	3.72	5.49
$\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	1.55	1.99
H_3PO_4 (75%)	1.52	2.24

The nominal operating parameters for the two concentration operations are given in Table 14. The run-1 chronology, shown in the Appendix, indicates the many times the system was up and down while attempting to diagnose and correct the problems during the concentration operation. The chronology for the second concentration operation is also in the Appendix.

A gamma-energy analysis was done on a HLW sample after it was concentrated. Table 15 lists the activities of the isotopes that were analyzed. The HLW was also visually examined following concentration and was found to contain a large amount of solids, as shown in Figures 11 and 12. The solids are believed to be the waxy, DBP-iron complexes that plugged the evaporator mist eliminator.

FIGURE 10. Processing Scheme for Concentration of HLW

(a) Gage pressure.

TABLE 14. Nominal Operating Parameters for HLW Concentration Runs

Parameter	Value	
	Concentration Run 1	Concentration Run 2
Noncondensable gas flowrate	4 to 7.5 scfm	5 scfm
Evaporator		
Specific gravity	1.01 to 1.23	1.02 to 1.25
Volume	570 L	650 L
Pressure	-9.6 in. H ₂ O	-11.6 in. H ₂ O
Liquid temperature	105°C	106.5°C
Vapor temperature	106°C	108°C
Evaporator condenser pressure	-16 in. H ₂ O	-13.2 in. H ₂ O
Evaporator condenser to fractionator conductivity	0.4M H ⁺	0.4M H ⁺
Fractionator		
Temperature	97.5°C	102°C
Pressure	-14.4 in. H ₂ O	-14. in. H ₂ O
Specific gravity	1.23	1.25
Volume	1028 L	1012 L
Fractionator condenser to evaporator flow (strip water)	180 L/h	115 L/h
Tower temperature	102°C	109.5°C
Fractionator distillate receiver		
Volume	140 to 950 L	20 to 970 L
Pressure	-8 in. H ₂ O	-4 in. H ₂ O
Preheater pressure	-29.6 to -13.2 in. H ₂ O	-18 in. H ₂ O
Scrubber		
Volume	500 L	520 L
Pressure	-21.6 in. H ₂ O	-22.4 in. H ₂ O

TABLE 15. Gamma-Energy Analysis

Isotope	Activity, Ci/L	
	Run-1 HLW	Run-2 HLW
⁹⁵ Nb	1.24×10^{-3}	1.97×10^{-3}
¹⁰³ Ru	4.41×10^{-4}	0.20
¹⁰⁶ Ru	4.26	9.14
¹²⁵ Sb	0.30	0.26
¹³⁴ Cs	9.39	20.41
¹³⁷ Cs	59.33	76.13
¹⁴¹ Ce	2.13×10^{-4}	5.68×10^{-4}
¹⁴⁴ Ce	2.85	7.43
¹⁵⁴ Eu	1.94	2.83

FIGURE 11. Solids in HLW Following Concentration

FIGURE 12. A Close-Up of Solids in HLW Following Concentration

RADIOACTIVE-WASTE SOLIDIFICATION PROCESS

When the preparations described in the previous section were completed, two SC/ICM runs were made inside the radiochemical cell with actual radioactive waste. Two canisters of HLW glass were produced with no major problems. The two, radioactive SC/ICM runs are detailed below.

RADIOACTIVE SC/ICM RUN 1

The procedures for starting radioactive run 1 were as follows. Following the initiation of air flow through the atomizing nozzle at about 2 scfm, the power was turned on to the SC and ICM furnaces. When the SC/ICM reached operating temperatures (see Table 16), the equipment was held at these temperatures for approximately 1.5 h to ensure total heat up. Air flow was maintained throughout the heat-up period to prevent thermal shocking of the nozzle after heat up. During the heat-up period (6 h for the ICM and 6.5 h for the SC), the effluent system was started. The effluent system was brought to operating temperatures and allowed to reach steady-state conditions. The atomizing air flow was then increased to 7.2 scfm at 43 psig, and about 10 to 15 in. of H₂O vacuum was maintained in the SC. The filter blowback air was turned on

TABLE 16. Operating Temperatures for SC/ICM Run 1

<u>Zone</u>	<u>Temperature, °C</u>
SC furnace	
1	750
2	750
3	770
ICM furnace	
1	1035
2	1035
3	1040
4	1045
5	1050
6	1050

(up to the solenoid valves) at 80 psig. The filter blowback timer was set to pulse the filters sequentially for 1/4 s every 20 min.

With the feed control valve closed and the recycle valve open, the feed pump was started and feed was recirculated. The feed recycle valve was then closed to attain a feed-line pressure of 25 psig. At this time, the frit feed system was started. Frit was added to the canister at a rate of 1.33 kg/h for about 7 min.

After the frit feed system was started, the water flow to the calciner was started and was slowly increased to 10 L/h. After 5 min of water flow, the water was valved off and the HLW feed was valved into the calciner. The HLW flow rate was increased to about 10 L/h. The noncondensable off-gas flow through the effluent system immediately after HLW feed was initiated was about 21 scfm. The air-operated vibrator on the calciner was set with an air-line pressure of 80 psig and was manually operated every 20 min.

After 22 h of continuous operation, a gamma-energy scan of the canister indicated that it was half full. At this point, 356 L of waste feed had been processed and 47 kg of frit had been added.

At one point during the run, the feed rate, as measured by the magnetic flowmeter, became very erratic (see Figure 13). It appeared as though the nozzle was becoming plugged; however, using the cleanout needle had no effect. Visual inspection of the nozzle through a shielded viewing window revealed that a nut holding the cleanout needle to the actuating cylinder had dropped off, allowing the needle to partially drop into the nozzle. The feed to the calciner was turned off, and water was flushed through the nozzle. A manipulator was then used to retract the needle and hold it. When this was accomplished the feed to the calciner was restarted, and although the feed flow continued to be erratic, no further problems due to the needle were encountered during the run. The total feed-off time was 28 min.

After the feed flow to the calciner was restarted, the equipment ran well for 46.7 h until the pump heel in the feed tank was reached. Since the pump heel in the tank is of considerable volume (100 L), nitric acid was added to

FIGURE 13. Feed Rate Showing Effects of Nozzle Plugging

maintain the liquid level above at a pumpable level. Nitric acid (4.5M) was added in two separate 75-L additions, once when the pump heel was initially reached and again when the pump heel was reached a second time.

When the feed tank pump heel was reached the second time after 52.5 h of feeding, the feed to the calciner was terminated for the run. The air-operated vibrator and the filter blowback system were manually activated 10 to 15 times to get as much calcined waste as possible out of the calciner and into the waste canister. The frit feeder feed rate was increased to 2 kg/h and was operated for 0.5 h to insure that there was enough frit to mix with any excess calcined waste that might have fallen from the filters or calciner chamber after the feed was terminated.

Following the termination of feed to the SC, the power to the calciner furnace was shut off. The ICM furnace was left at operating temperature for 1 h after the last frit was added to insure that all the frit and calcined waste melted. The power to the ICM furnace was turned off 12.7 h following final termination of feed to the SC.

After the ICM furnace was turned off, the canister walls were quenched from 1050⁰ to 550⁰C using forced air. The forced air came from the furnace blower, which was valved to each of the six zones of the canister. Thus, each zone could be cooled separately using different air flow rates as needed. When some of the zones reached 550⁰C, the furnace was restarted and used in conjunction with the blower to maintain the temperature at 550⁰C until all the zones were at 550⁰C. The blower was then shut off, and the canister was allowed to air-cool in the furnace. The cooling curve is shown in Figure 14. As can be seen from the figure, mechanical difficulties were encountered with the blower.

When the walls of the canister reached 550⁰C and the blower was shut off, the shutdown of the off-gas system was started; this signaled the termination of the first run. A detailed chronology of run 1 is given in the Appendix. Radioactive run 1 exceeded the overall theoretical material balance for the 10-L/h design basis by 57%, demonstrating an average feed rate of 15.7 L/h.

Following the termination of the run, a gamma-energy scan was performed on the calciner cone; extremely high activities were noted. Because of this unusual occurrence, the nozzle was removed and the interior of the calciner was visually inspected. The inspection revealed an accumulation of calcined waste on the walls of the calciner cone. A material balance investigation (see Table 17) indicated that some of the material on the calciner walls was nonradioactive calcine and frit. However, the exact quantities could not be determined since the moisture content of the calcine was not known. The material was mechanically removed from the calciner before radioactive SC/ICM run 2.

There are two possible explanations for the buildup of material in the calciner; both may be contributing factors. The first is that an extremely high feed rate was realized during the first in-cell, nonradioactive run when the magnetic flowmeter was operated for a brief period on the 0- to 200-L/h scale instead of the 0- to 50-L/h scale. The high feed rate could have caused a buildup of uncalcined waste. The other possibility is that the nozzle was atomizing poorly for the nonradioactive runs and then the tip fell off during

FIGURE 14. Cooling Curve for Run-1 Waste Canister

(a) Not available.

TABLE 17. Material Balance for Radioactive SC/ICM Run 1

Feed
Concentration: 833 MTU
Acid concentration: 4.4M H ⁺
Solids content: 66 g oxide/L
Feed processed: 806 L (undiluted equivalent)
Average feed rate: 15.7 L/h
Heat content: 1.12 W/L
Activity: 313 Ci/L
Canister
Net glass weight: 116 kg
Net calcine added: 22.3 kg
Net frit added: 93.7 kg
Estimated density: 2.8 g/cm ³
Fill level: 1.37 m
Glass volume: 41.3 L
Decay heat: 380 W
Activity: 1.05 x 10 ⁵ Ci

the latter portion of radioactive run 1. A visual inspection of the calciner feed nozzle revealed that the nozzle tip (air cap) was missing. The nozzle air cap apparently vibrated loose during run 1. The air cap had not been tack-welded to the body of the nozzle. The estimate of the time that the air cap fell off is supported by the fact that the gamma-energy analysis indicated that no radioactive calcine buildup occurred during the major portion of radioactive run 1. In addition, the glass in the upper portion of canister 1 appeared less dense (i.e., frit rich) than expected.

During radioactive run 1, the effluent system operated very well. The nominal operating parameters for the effluent system are given in Table 18. The effluent system maintained a satisfactory vacuum in the calciner, while handling a noncondensable gas flow ranging from 12 to 27 scfm. The vacuum in the SC varied from a maximum of -27.6 in. H₂O to 0 in. H₂O. The zero

TABLE 18. Nominal Operating Parameters for the Effluent System--
Radioactive SC/ICM Run 1

Parameter	Value
Noncondensable gas flowrate	18.75 scfm
Spray calciner pressure	-17.2 in. H ₂ O
Quench scrubber	
Vessel volume	9.3 L
Column outlet pressure	-20 in. H ₂ O
Condensate tank pressure	-10 in. H ₂ O
Evaporator	
Specific gravity	1.0575
Volume	565 L
Pressure	-11 in. H ₂ O
Liquid temperature	103.5°C
Vapor temperature	102°C
Evaporator condenser pressure	-12 in. H ₂ O
Evaporator condenser to fractionator flow	57.7 L/h
Evaporator to fractionator conductivity	0.43M H ⁺
Fractionator	
Temperature	97.5°C
Pressure	-11.2 in. H ₂ O
Specific gravity	1.25
Volume	1008 L
Fractionator condenser to evaporator flow	0 L/h
Fractionator coil temperature	96°C
Fractionator distillate receiver	
Volume	100 to 885 L
Pressure	-4.8 in. H ₂ O
Preheater pressure	-26 in. H ₂ O
Scrubber	
Volume	499 L
Pressure	-12.4 in. H ₂ O

vacuum occurred during a cycle of filter blowbacks in which pulses of air were directed backwards into the filters to remove solids accumulated on the filter. The blowbacks generally decrease the calciner vacuum by 8 to 13.2 in. H_2O . The lowest vacuum in the calciner when a blowback was not taking place was -5.2 in. H_2O , which occurred near the beginning of the run. Erratic feed flow also caused a wide variation in the calciner vacuum; during one period the vacuum fluctuated between -27.2 and -14 in. H_2O because of variations in the feed flow. The typical calciner operating pressures during run 1 are shown in Figure 15.

The pressure drop across the SC's sintered stainless steel filters was fairly constant at 2.55 in. H_2O , although during a filter blowback the pressure drop increased to 7.5 in. H_2O . However, even at the maximum pressure drop of 7.5 in. H_2O , the effluent system was easily able to maintain a sufficient vacuum in the calciner.

FIGURE 15. Typical Calciner Operating Pressures Versus Time

The quench scrubber operated as designed. The pressure drop across the scrubber was low, the maximum being 4 in. H_2O . The steam jet was easily able to increase the vacuum from 4 to -18 in. H_2O . The off gas from the calciner, which exited the filters at temperatures from 175° to $275^{\circ}C$, was easily cooled to 30° to $40^{\circ}C$ in the quench scrubber.

The evaporator was initially heeled with 522 L of 5M HNO_3 to bring the effluent system to steady-state acid concentrations in a shorter time. During the course of the run, the evaporator was operated continuously to boil the condensates from the SC and the quench-scrubber steam jet. The amount of liquid in the evaporator was held constant during the run. The HNO_3 molarity varied from 2.4 to 6.5 with a final molarity of 3.46. The temperature of the liquid in the evaporator was generally about $103.5^{\circ}C$, with the vapor temperature at $102^{\circ}C$. The vapor temperature did drop to about $96^{\circ}C$ when the non-condensable gas flow increased. At the termination of the run, the amount of liquid in the evaporator was 576.5 L.

The acid fractionator was initially heeled with 1012 L of 10M HNO_3 , again to bring the system to steady-state acid concentrations in a shorter time. The fractionator was also operated during the entire run, and the amount of liquid was held constant. The acid concentration ranged from 10.25M to 15.5M with the final molarity at 14.5. The final amount of liquid in the fractionator was 1023.7 L.

Both liquid and gas samplers were used to obtain samples of effluents from various points in the effluent system during run 1 (see Figures 16 and 17). Liquid samples were taken every 15 to 20 min. However, operating the gas sampler was extremely time consuming and interfered with taking liquid samples. Because of the time it took to take gas samples, only 29 liquid and 7 gas samples were obtained during the run.

RADIOACTIVE SC/ICM RUN 2

The startup procedures for run 2 were basically the same as for run 1. The canister for run 2 contained about 8 kg of material removed from the calciner following run 1, and 16 kg of frit were mixed with this material to

FIGURE 16. Typical Liquid Sampler

provide for glass formation. To initiate run 2 the atomizing air was turned on to cool the nozzle, and then the SC and ICM furnaces were started. When the furnaces reached operating temperatures, the noncondensable off-gas flow through the system was 24 scfm, which was even higher than during run-1 start-up. Attempts were made to decrease off-gas leakage by tightening connections that were most likely to leak. The bolts that fasten the calciner chamber to the cone, the canister to the cone, and the flange to the top of the evaporator were tightened with an impact wrench. This resulted in a small decrease in leakage (down to 22 scfm), which meant that a sufficient vacuum could be pulled on the calciner. In addition, because it had been noted on the previous runs that leakage decreased after HLW was fed to the calciner, startup was continued.

FIGURE 17. Typical Gas Sampler

When the furnaces were up to operating temperatures (see Table 19), and the effluent system was operating, the feed pump was started and the pressure was set as described for run 1. However, when water was turned on to flush the nozzle, no flow could be obtained. The cleanout needle was activated but there was still no flow; thus, the feed pump was shut down and the nozzle was removed. A deposit could be seen on the retaining cap, and the nozzle appeared plugged. A new nozzle was installed and startup procedures were continued. While the nozzle was being replaced, the furnaces were kept at operating temperatures and the effluent system was kept running. When the nozzle was in place, the frit feeder was started at 3 kg/h. Water flow was established to the new nozzle, which was followed by HLW feed.

After 3.3 h of feed flow, it was discovered that a section of line from the frit feed funnel to the frit feed line that had been removed to calibrate the feeder had not been replaced. During this time approximately 8 kg of frit

TABLE 19. Operating Temperatures for Radioactive SC/ICM Run 2

<u>Zone</u>	<u>Temperature, °C</u>
SC Furnace	
1	800
2	760
3	775
SC Thermowell	
1	250 to 300
2	400 to 430
3	420 to 470
ICM Furnace	
1	1050
2	1050
3	1070
4	1040
5	1045
6	1065

did not reach the canister. The section of line was replaced, and the frit feed rate was increased to compensate for the missing frit. Later it was noticed that the air cylinder that actuates the frit feeder air lock was not working properly. A connection between the cylinder and the air lock had disconnected. Thus, the feed to the calciner was shut off and the feed system was flushed with water. Then the frit feeder was stopped, and the air lock was repaired. After a feed-off time of 1.5 h, the HLW feed to the SC was restarted.

For the next 14.6 h the SC/ICM system operated routinely until a momentary drop in the atomizing-air pressure tripped the feed fail-safe alarm, which shut down the feed pump. The feed fail-safe alarm is an electronic tie between the atomizing-air pressure and the feed pump. If for any reason the air pressure drops below a preset value, the feed pump is stopped to prevent any HLW from inadvertently flowing to the outside of the radiochemical cell via the atomizing-air line. When the feed fail-safe alarm tripped, an inspection of the

SC/ICM instrument system indicated that all systems were normal; therefore, feed was restarted. Total feed-off time was 5 min. The operation of the SC/ICM system was routine for the remainder of the run.

When the canister was filled with glass to the 6-ft level, as detected by a gamma-energy scan, the HLW feed was turned off and water was flushed through the feed lines. At this point, 429 L of HLW had been fed to the calciner, and 78.75 kg of frit had been mixed with the HLW. After the HLW feed was stopped, the SC was vibrated and the filters were repeatedly blown back while the frit feeder remained operating. Water to the nozzle was turned on and off a couple of times to attempt to thermally shock the calciner chamber to release any calcined waste sticking to the chamber walls. The frit feeder was shut down; the total amount of frit added was 83.7 kg. Since the feed tank was not pumped down near the pump heel during this run, no dilution of the HLW was necessary as was done in run 1.

The canister was allowed to heat-soak for about 2.5 h before the power to the furnaces was turned off and the cooldown phase begun. As in run 1, the canister walls were quenched to 550°C using forced air and then were allowed to cool in air (see Figure 18). When the entire canister reached 550°C, the effluent system was shut down, signaling the termination of run 2. The material balance for the second radioactive run is given in Table 20. The overall theoretical material balance (see Figure 17) was exceeded by 17% in SC/ICM run 2.

As in run 1, the effluent system operated very well. The nominal operating parameters are given in Table 21. The pressure in the SC varied between -26 in. H₂O and 5.2 in. H₂O. However, the 5.2 in. H₂O resulted from a momentary pressurization caused by a filter blowback. The normal range was between -13.6 in. and -7.2 in. H₂O. The blowback of the filters dropped the calciner vacuum by up to 9.6 in. H₂O. The pressure drop across the sintered stainless steel filters remained constant at 3.75 in. H₂O, except during filter blowbacks when the pressure drop momentarily increased to 10.5 in. H₂O.

The quench scrubber functioned well, cooling the calciner off-gas to 27°C. The pressure drop across the scrubber averaged about 4 in. H₂O. The

FIGURE 18. Cooling Curve for Run-2 Waste Canister

(a) not available.

TABLE 20. Material Balance for Radioactive
SC/ICM Run 2

Feed
Concentration: 760 MTU
Acid concentration: 6.6M H^+
Solids content: 86.15 g oxide/L
Feed processed: 429 L
Average feed rate: 12.7 L/h
Heat content: 2.05 W/L
Activity: 526 Ci/L

Canister
Net glass weight: 145 kg
Net calcine added: 36.96 ^(a)
Net frit added: 83.7 ^(a)
Estimated density: 2.7 g/cm^2
Fill level: 1.75 m
Glass volume: 52.9 L
Decay heat: 1012 W
Activity $2.64 \times 10^5 \text{ Ci}$

(a) 8.16 kg of calcine and 16.33 kg of frit were in the canister before run 2 from cleaning out the SC following run 1.

evaporator was initially heeled with 6.23 L of 5M HNO_3 . During the course of the run, the acid concentration varied between a minimum 1.7M and a maximum 5.75M . When the run ended, the evaporator contained 519 L of 5.5M acid. The acid fractionator was initially heeled with 993 L of 10M HNO_3 . During the run, the acid concentration ranged from 9.8M to 13M .

Based on the problems with collecting gas samples in run 1, the gas samples were abandoned in favor of gathering more liquid samples (which will provide all of the necessary data). A total of 76 liquid samples were obtained during run 2.

TABLE 21. Nominal Operating Parameters for Effluent System--
Radioactive SC/ICM Run 2

Parameter	Value
Spray calciner pressure	-10.4 in. H ₂ O
Noncondensable gas flowrate	23.0 scfm
Quench scrubber	
Vessel volume	9.3 L
Column outlet pressure	-14.4 in. H ₂ O
Condensate tank pressure	-5.2 in. H ₂ O
Evaporator	
Specific gravity	1.08
Volume	522 L
Pressure	-7.6 in. H ₂ O
Liquid temperature	102°C
Vapor temperature	100.5°C
Evaporator condenser pressure	-15.6 in. H ₂ O
Evaporator to fractionator flow	62.5 L/h
Evaporator condenser to fractionator conductivity	NA
Fractionator	
Temperature	99°C
Pressure	-14 in. H ₂ O
Specific gravity	1.23
Volume	882 L
Fractionator condenser to evaporator flow	28 L/h
Fractionator coil temperature	97.5°C
Fractionator distillate receiver	
Volume	184 to 928 L
Pressure	-2.8 in. H ₂ O
Preheater pressure	-28 in. H ₂ O
Scrubber	
Volume	775 L
Pressure	-9.2 in. H ₂ O

EFFLUENT DISTRIBUTION

Determining the distribution of materials in the effluent system was not a goal of this project. Sample analyses will be reported at a later date. However, the projected radiochemical balance is shown in Figure 19. This projection is based on the empirical values found in earlier operations of this effluent equipment (McElroy et al. 1972).

The gross activities released from the process to the environs via the building stack are given in Table 22. In the process of concentrating and solidifying all of the waste, more than 1.03×10^6 Ci of beta-gamma and 4.14×10^3 Ci of alpha materials were processed. Discounting the activity releases from all other sources in the building, the effluent system was shown to have the worst-case decontamination factors^(a) given in Table 23.

Requirements for the control of nitrogen oxides (NO_x) by the effluent system are based on two sets of existing regulations, one on opacity and one on concentration. The opacity regulation, as set by the State of Washington, requires that the stack plume be of no more than 20% opacity. (In the building stack, the visible NO_x threshold is approximately 25 ppm.) The concentration limit, set by the Environmental Protection Agency, has two stipulations:

1. The annual average concentration of NO_x in the environs must be no more than 100 g/m^3 (0.05 ppm).
2. The 24-h average concentration of NO_x in the environs must be no more than 250 g/m^3 (0.125 ppm).

A dilution factor from the stack to the environs of approximately 1760 makes the annual average concentration limit 88 ppm and the 24-h limit 220 ppm (as measured in the stack).

The NO_x opacity was evaluated by a Certified Plume Evaluator. The opacity observations were made from a distance of 600 ft. At no time during the operation was a plume observed to exceed 5% opacity (5% is the visible opacity threshold).

(a) Decontamination factor is defined here as the ratio of the activity of the material entering the process to that of the material exiting the process.

FIGURE 19. Vitrification and Off-Gas System Radiochemical Balance(i)

- (a) Effluent curie/feed curie, ratio of total curies gained of component to total curies of component in feed per run.
 (b) Average concentration in gas; low and high signify low- and high-burnup waste.
 (c) Particulate assumed to be a homogeneous mixture of fission products, excluding gases and volatile species.
 (d) $DF = \frac{\text{curies/unit time in feed}}{\text{curies/unit time in overheads}}$
 (e) Evaporator DF = 5; condenser DF = 10.
 (f) Fractionator DF = 7.5; condenser DF = 10.
 (g) Assumes 25 scfm for 2A jet and no sparge air.
 (h) No detectable gain.
 (i) Dissolver off gas not included in balance.

	1	A	2	B	3	4	C	5	6	7	8	D	9	10	11	12	13	E	14	F	15
	HLW FEED	CALCINER OFF GAS	SPRAY CALCINER	QUENCH SCRUBBER (TK-111)	QS CONDENSER OFF GAS	CONDENSATE AND OVERFLOW	EVAPORATOR (TK-113)(e)	EVAPORATOR OFF GAS	EVAPORATOR BOTTOMS	TK-113 CONDENSER OFF GAS	EVAPORATOR CONDENSATE	FRACTIONATOR (TK-115)(f)	FRACTIONATOR OFF GAS	FRACTIONATOR BOTTOMS	TK-115 CONDENSER OFF GAS	FRACTIONATOR CONDENSATE	SCRUBBER BOTTOMS	SCRUBBER (TK-118)	SCRUBBER OFF GAS(g)	FINAL POC FILTRATION	OFF GAS TO VENT
Ru FEED RATIO ^(a)	1.0	1E-2		2E-5	1E-2		4E-6	1E-2	4E-7	3.6E-6		5.3E-8	3.9E-6	5.3E-9	4.8E-8	1E-5		5.3E-10		2.1E-11	
Ru CONC $\mu\text{Ci}/\text{cm}^3$, LOW ^(b)		3.5E-2		1.1E-4	3.83		2.2E-5	136	2.2E-6	1.4E-3		3.0E-7	2.6E-2	3.0E-8	1.8E-5	6.7E-2		9.9E-10		3.9E-11	
Ru CONC $\mu\text{Ci}/\text{cm}^3$, HIGH ^(b)		1.3E-1		4.1E-4	14.03		8.2E-5	496	8.2E-6	5.1E-3		1.1E-6	9.8E-2	1.1E-7	6.7E-5	2.5E-1		3.6E-9		1.4E-10	
PART FEED RATIO ^(a)	1.0	5E-4		5E-7	5E-4		1E-7	5E-4	1E-8	9E-8		1.3E-9	9.9E-8	1.3E-10	1.2E-9	1.2E-9		2.6E-13		1.7E-14	
PART CONC $\mu\text{Ci}/\text{cm}^3$, LOW ^(b,c)		4.7E-2		7.4E-5	5.09		1.5E-5	178	1.5E-6	9.2E-4		1.9E-7	1.8E-2	1.9E-8	1.2E-5	2.1E-4		1.3E-11		8.4E-13	
PART CONC $\mu\text{Ci}/\text{cm}^3$, HIGH ^(b,c)		1.3E-1		2.0E-4	13.52		3.9E-5	477	3.9E-6	2.4E-3		5.1E-7	4.5E-2	5.1E-8	3.2E-5	5.7E-4		3.4E-11		2.2E-12	
EQUIP Ru DF ^(d)		100		5E2		5E1					7.5E1						1E2		25		
EQUIP PART DF		2E3		1E3		7.5E1					1E2						5E2		15		
SS-12 Ru FEED RATIO	1.0	1.9E-2					1.9E-2					1.4E-3		1.9E-6		3.1E-5	E-8		4E-10		
SS-12 Ru CONC $\mu\text{Ci}/\text{cm}^3$								8.7E-7					5E-7				4E-8				
SS-12 PART FEED RATIO	1.0	1E-4					1.4E-3					2.4E-6		2E-10		NDG ^(h)	3.2E-11		2E-12		
SS-12 PART CONC $\mu\text{Ci}/\text{cm}^3$								4.8E-5					7E-7				2E-8				
SS-12 Ru DF		53						613								3.1E3		25			
SS-12 PART DF		1E4						3.1E6								NDG ^(h)		16			
ASSUMED VOLUME OR FLOW					310 L/h		500 L		310 L/h		1000 L		310 L/h	1000 L							

TABLE 22. Gross Activity Released to Stack

Operation	Activity Released, Ci			Activity Concentration, Ci/cm ³		
	Alpha	Beta	Gamma	Alpha	Beta	Gamma
Radioactive Run 1						
HLW Concentration (a)	1.25×10^{-7}	3.48×10^{-7}	(b)	1.56×10^{-15}	4.27×10^{-15}	(b)
SC/ICM (a) Operations	4.1×10^{-8}	4.6×10^{-8}	(b)	1.5×10^{-15}	1.6×10^{-15}	(b)
Radioactive Run 2						
HLW Concentration (a)	1.7×10^{-8}	7.8×10^{-8}	(b)	6.4×10^{-16}	2.7×10^{-15}	(b)
SC/ICM Operations	1.8×10^{-8}	7.3×10^{-8}	5.16×10^{-8}	6.5×10^{-16}	2.7×10^{-16}	1.89×10^{-15}

(a) During SC/ICM operations, the spent fuel dissolution system was in operation, contributing an undetermined amount to the activities shown.

(b) Data not available.

TABLE 23. Worst-Case Effluent System Decontamination Factors

<u>Operation</u>	<u>Decontamination Factor</u>	
	<u>Alpha</u>	<u>Beta-Gamma</u>
Radioactive Run 1		
HLW concentration	7×10^7	(a)
SC/ICM operations	2×10^{10}	(a)
Radioactive Run 2		
HLW concentration	1×10^{11}	(a)
SC/ICM operations	7×10^{10}	2×10^8

(a) Data not available.

Stack sampling conducted during the operation also reflected very low NO_x emissions, generally in the area of 1 to 2 ppm in the stack. For example, during radioactive SC/ICM run 1 on 2-20-79 at 11:55, a stack sample showed a NO_x concentration of 1.26 ppm.

PROJECT EVALUATION

In summary, the overall operations were highly successful; the SC/ICM vitrification process worked well. Two canisters containing vitrified HLW from power-reactor fuel were safely produced, with only a few minor problems with hardware. Since the total operating time for the SC, including the two nonradioactive and the two radioactive runs, was only 133 h, some hardware problems were to be expected. In addition, the effluent system functioned as expected. No radioactive or hazardous chemicals in excess of the allowable were released. Neither the operating staff nor the general public was exposed to hazardous chemicals via the environs. This project has proven that HLW from a power-reactor fuel cycle can be safely processed into a vitreous state that is ready for disposal.

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APPENDIX

The chronologies for the following runs are included in this Appendix.

- integrated process off-gas system operation
- nonradioactive SC/ICM run 2
- HLW-concentration run 1
- HLW-concentration run 2
- radioactive SC/ICM run 1
- radioactive SC/ICM run 2.

INTEGRATED PROCESS OFF-GAS SYSTEM OPERATION

11:45 TK-111 Purex[®] valve open 3/4
11:25 P-116 on^(a)
13:17 TK-111 Purex valve closed to 1/2
13:40 TK-111 Purex valve closed
16:40 steam off to TK-113 and TK-115
17:25 H₂O to TK-115 coil off
18:00 steam on to TK-113 and TK-115
18:05 steam to TK-115 increased 10%
18:20 TK-113, TK-115 boiling
18:30 steam to TK-113 raised to 50% loading; H₂O to T-115 coil to 22%
18:35 2A rack ball valve opened to 100%
19:05 steam to TK-115 increased 10%
19:45 B-102 on; crosstie closed
19:55 H₂O to TK-111
20:10 B-102 intake valve opened
20:30 P-116 feed to TK-111 closed to 10%
21:00 H₂O to T-115 coil to 11%
21:15 TK-11 Purex valve open 3/4; P-116 feed to TK-111 to 7%
21:40 H₂O to T-115 coil to 116%

[®] Registered tradename of Purex Corp.

(a) See Figures 5, 6, 9, 16 and 20 for definitions of equipment abbreviations, such as P-116, B-102, T-111, etc.

23:00 #1 vent jet open 20%
23:15 #1 vent jet open 75%; TK-111 purex valve to 1/4 open
23:25 #1 vent jet bypass closed
23:30 P-111 feed to TK-111 changed from lower to upper weir
23:40 TK-111 jet off; TK-111 Purex valve closed
01:00 Shutdown

NONRADIOACTIVE SC/ICM RUN 2

11/8/79

15:20 atomizing air on; SC furnaces on
17:15 ICM furnaces on
19:20 TK-113 startup
22:00 P-116 started
22:50 #1 vent jet on

11/19/78

00:10 SC furnace to temperature (750°C)
01:40 #1 vent jet bypass closed
02:00 adjusted vibrator (ran manually)
02:10 adjusted atomizing air up to 7 scfm flow
02:40 TK-111 jet on
02:45 fail-safe system tripped
06:55 cooling blower on to zones 5 and 6
07:30 cooling blower off
09:00 TK-111 jet off
09:40 TK-111 jet on
09:45 100 L H₂O to TK-118
10:20 frit feed on at 1.28 kg/h
10:40 cooling blower on to zones 5 and 6
11:10 SC feed on
11:15 cooling blower off
11:30 adjusted frit feed to 1.92 kg/h; vibrator on for 15 s (was operated every hour)

12:75 cooling blower on (throttled down)
13:40 cooling H₂O on on TK-114
14:00 cooling blower off on zone 5 and 1/2 open on zone 6; first filter
blowback
15:30 box used in checking frit feed flow accidentally left in; thus, no
frit went down into the canister; frit flow restarted
15:50 cooling blower off
18:40 filter blowback (filter blowback was energized every hour)
22:00 cooling blower on
23:15 cooling blower off
22:30 jetted TK-116 to TK-101

11/10/78

02:00 added more simulated feed solution to TK-114
04:00 first sampler operated
05:30 jetted TK-116 to TK-101
06:25 cooling blower on
10:00 reduced TK-111 jet setting
13:00 replaced SC feed controller (working on automatic)
15:00 TK-113 gas sampler on
17:05 feed off; H₂O on (started shutdown)
20:30 TK-113 steam off
21:30 TK-111 jet off
24:00 drained E-115 to TK-113

HLW-CONCENTRATION RUN 1

1/31/79

17:40 TK-113 steam on
17:55 TK-115 steam on
18:42 TK-113 boiling
19:22 TK-115 boiling
20:14 P-114 feed on

2/1/79

04:35 added 55 L H₂O to TK-118
15:40 P-114 off because of vacuum problem in TK-113
16:05 TK-113 and TK-115 off
19:32 TK-113 steam on
20:09 TK-113 boiling
20:10 TK-115 steam on
20:32 TK-113 and TK-115 steam off to idle at 90°C

2/2/79

01:35 TK-113 steam on
01:50 TK-113 steam on
05:15 T-113 trays flushed
08:55 #1 vent jet on
09:10 TK-113 boiling
10:10 TK-113 trays flushed with 4 L of 2M HNO₃
10:23 TK-113 trays flushed with 20 L of 2M HNO₃
11:46 P-114 on
12:35 P-114 off
13:07 T-113 trays flushed with 50 L 2M HNO₃
15:45 P-114 on

2/6/79

16:15 TK-113 and TK-115 steam on
17:00 TK-113 boiling; P-114 on
17:40 TK-113 and TK-115 off; P-114 off
22:10 flushed demister with 1.96M HNO₃
22:25 TK-113 and TK-115 steam on; P-114 on

2/7/79

02:15 Tk-113 off of boil; P-114 off
03:25 flushed demister
03:10 TK-113 stam on
03:50 P-114 on
07:00 TK-113 steam off; P-114 off

07:10 flushed demister
07:25 TK-113 steam on; P-114 on
11:40 TK-113 and TK-115 steam off; P-114 off
12:30 TK-113 jetted to TK-114
13:19 TK-112 jetted to TK-113
13:45 TK-113 and TK-115 steam on
17:40 TK-113 and TK-115 steam off
17:45 flushed demister with 200 L of 5M HNO_3
18:55 flushed demister with 100 L of 5M HNO_3
19:30 TK-113 and TK-115 steam on
21:20 TK-113 and TK-115 steam off
23:40 flushed demister with 100 L of 5M HNO_3

2/8/79

01:55 TK-114 jetted to TK-112
02:35 TK-113 jetted to TK-114
05:00 flushed demister with 500 L of 5M HNO_3
07:00 TK-113 and TK-115 steam on; recycling all condensates
10:00 TK-113 and TK-115 steam off
15:45 flushed demister with Na_2CO_3
17:45 pumped 300 L from TK-114 to TK-113
19:50 pumped 300 L from TK-114 to TK-113
20:00 TK-113 and TK-115 steam on
21:45 TK-113 and TK-115 boiling
22:00 P-114 on

2/9/79

01:00 P-114 off; TK-113 and TK-115 steam off
17:50 jetted TK-113 to TK-114
18:04 jetted TK-114 to TK-112

2/10/79

07:45 removed demister and installed new one

2/12/79

01:14 jetted 446 L HLW from TK-112 to TK-113
05:30 TK-113 and TK-115 steam on

07:00 P-114 on

12:35 P-114 feed decreased from 180 to 45 L/h and strip water flow from E-115 to TK-113 on at 210 L/h

17:10 added Dow Corning Antifoam

2/14/79

13:00 TK-113 and TK-115 steam off; out of HLW

2/16/79

05:45 jetted TK-113 to TK-114

08:30 TK-113 and TK-115 boiling; P-114 on

2/17/79

16:30 #1 vent jet on

18:00 E-115 to TK-113 strip water DOVC began malfunctioning

18:10 no strip water available

18:30 P-114 off; TK-113 and TK-115 steam off

2/18/79

05:15 TK-113 and TK-115 steam on

08:30 TK-113 and TK-115 steam off

09:15 TK-115 steam on

11:00 TK-113 steam on

13:30 began jetting strip water from TK-112 to TK-113

2/19/79

03:35 TK-113 and TK-115 steam off

05:00 jetted TK-113 to TK-114

06:45 flushed TK-113 to TK-114 with 35 L H₂O

HLW CONCENTRATION RUN 2

3/12/79

06:00 jetted TK-112 to TK-113

09:05 TK-113 and TK-115 steam on

10:14 P-114 on
10:45 started adding antifoam
21:00 P-114 flow back to 0 L/h; E-115 to TK-113 strip water flow on at
120 L/h

3/13/79

13:30 P-114 off to reduce TK-113 volume
20:00 P-114 on

3/14/79

22:20 TK-113 and TK-115 steam off

3/17/79

02:35 TK-113 and TK-115 boiling
02:40 P-114 on
03:30 E-115 to TK-113 strip water on

3/18/79

05:00 A-114 off
07:20 P-114 off
18:05 Tk-113 volume = 555 L; TK-113 and TK-115 steam off
21:00 12 L of U solution added (5 kg U in 12 L 5M HNO_3)
22:00 Cold chemicals (124 L) added to TK-113
22:05 TK-113 and TK-115 steam on
23:25 TK-113 pressurized; TK-113 and TK-115 steam off

3/19/79

01:00 TK-113 and TK-115 steam on
03:30 TK-113 and TK-115 steam off; TK-113 plugging; demister appeared
plugged
06:45 TK-113 jetted to TK-114

2/19/79

10:30 started heat-rise test on TK-114
11:20 turned on atomizing air at 2 scfm
11:55 started heat up on ICM and SC furnaces

14:40 1A rack started (TK-113 and TK-115)
18:00 ICM furnace at temperature
18:25 SC furnace at temperature
19:53 frit feeder on
20:00 H₂O on to SC
20:05 feed on 10 L/h
20:25 filter blown back every 20 min
20:30 increased power to SC furnace
20:35 SC chamber vibrated approximately every 20 min
23:00 frit feed rate increased to maintain a 2:1 ratio of frit to
calcined waste (using tank drop-out data)--minor changes made on
frit feed rates throughout the run.

2/20/79

11:00 TK-118 sample obtained
11:05 noticed small temperature drop in zone-5 canister centerline
thermocouple for a few minutes (indicated melt level)
11:20 TK-113 sampled
11:40 TK-116 samples
12:25 E-111 sampled
17:93 vacuum improved after gas sampling was finished
18:15 gamma scan indicated canister half full;
a 10⁰ temperature drop on zone-4 canister centerline temperature
was noticed.

2/21/79

03:40 filter chamber thermocouple started
05:10 added 100 lb of frit into frit feed hopper
06:00 erratic feed rate--tried cleanout needle, but did not have any
effect
06:20 closed P-114 recirculation valve to obtain smoother feed flow
09:00 had problems with SC cleanout needle--increased O-114 recirculation
09:29 feed flow to SC completely stopped--changed to H₂O
09:50 frit feeder turned off

09:54 P-116 back on--was turned off by fail-safe system switch
09:57 feed back on--using manipulator to keep cleanout needle in the up position
11:30 feed flow still erratic
14:47 turned temperature down on the ICM furnace to 950⁰C in zones 4, 5 and 6
19:15 added 75 L of 4.5M HNO₃ to TK-114 to get as much HLW as possible, due to pump heel
22:30 added second batch of 75 L of 4.5M HNO₃ to TK-114

2/22/79

01:04 feed off; H₂O on; vibrator active and filter blowback manually operated
01:16 frit feeder set to 2 kg/h
01:45 frit feeder off
02:00 power to SC furnace off
02:45 shut off ICM furnace
03:22 blower 1 on--not operable; blower has ruptured seal
03:55 blower 1 operating
04:26 zones 1 and 2 of ICM valved out from cooling blower
04:35 cooling blower not operating
06:00 cooling blower back on
06:05 zones 1 and 2 of ICM furnace turned on to maintain temperature at 550⁰C
06:28 zones 3 and 6 of ICM furnace valved out from cooling blower
08:30 heating zones 5 and 6 of ICM furnace down to 650⁰C due to sharp temperature drop of the canister wall
09:00 having problems with cooling blower--cannot get any cooling air to zones 5 and 6 of ICM
09:10 turned atomizing air down to 2 scfm
10:45 cooling blowers not operating--will let furnace cool off at natural rates
15:35 turned atomizing air off to SC
15:40 shutdown of remaining equipment

RADIOACTIVE SC/ICM RUN 2

3/20/79

23:25 filled frit hopper with frit

23:40 turned on ICM furnace

3/21/79

02:00 vacuum-checked system

02:10 started SC furnace

03:10 having problems with SC furnace zone 3 (firing board)

03:55 started calibration of frit feeder

06:53 ICM furnace at operating temperature

07:35 finished calibrating frit feeder

07:40 SC furnace at operating temperature

09:00 - 14:00 conducted the following to decrease leakage from the system:

1. tightened bolt on SC barrel--no change
2. tightened canister bolts--no change
3. tightened evaporatory column flange--no change
4. reduced stream to quench scrubber--better vacuum and leakage decreased

15:50 turned P-114 pump off; having symptoms of plugged nozzle--cleanout needle activated without any change

15:30 removed SC feed nozzle assembly--found deposit on steel retaining cap

20:40 finished installing a new SC feed nozzle assembly

21:00 started frit feeder at a setting of 3 kg/h

21:15 H₂O on to the SC

21:37 water off and HLW feed on

22:00 SC chamber vibrated (SC vibrated approximately every 30 min)

3/22/79

00:56 found that a line section from the frit feeder funnel to the frit feed line that was removed to calibrate frit feeder was not reconnected--approximately 8 kg of frit did not reach the canister

03:16 increased frit feed rate to 15 L/h
03:53 noticed frit feeder air lock was not working properly (air cylinder unscrewed from air-lock shaft)--turned HLW feed and frit feeder off
05:10 frit feeder on
05:20 HLW feed on--could not get any flow using flush water
09:30 lowered air pressure to frit feeder air-lock cylinder from 90 to 65 psig
11:30 lowered air pressure on frit feeder air-lock cylinder to 40 psig
14:55 momentary drop in atm air pressure cuasing feed pump to trip off--instruments indicated everything was normal; fail-safe system switched to off position and restarted P-114 and P-116; cleanout needle activated to clean nozzle
15:00 feed back on and on fail-safe position
22:40 noticed that when the vibrator was activated there was a fluctuation in the feed rate on the feed rate recorder

3/23/79

01:30 decreased HLW feed rate (to 8 L/h) and frit feed rate to allow contents in canister, at zone 2 level, to reach normal operating temperature
01:45 decreased temperature in zones 5 and 6 on the ICM
02:35 changed frit feed rate from 2.5 to 2.0 kg/h
05:38 increased HLW feed rate from 10 to 15 L/h
07:10 gamma scan indicated that the 6-ft 0-in. level was reached
07:15 HLW feed off; water on
07:25 frit feeder off
09:20 started frit feeder; vibrated the SC chamber; activated filter blowback several times
11:00 going into shutdown mode
11:30 SC furnace off
13:21 turned on cooling blower to cool canister
14:30 canister wall temperature is at 550°C
22:50 ICM furnace turned off--temperature on entire canister was at 550°C

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