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Technical Summary Nuclear Waste Vitrification Project

E. J. Wheelwright
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L. K. Holton
E. R. Irish
D. H. Siemens

May 1979

Prepared for the U.S. Department of Energy
under Contract EY-76-C-06-1830

Pacific Northwest Laboratory
Operated for the U.S. Department of Energy
by Battelle Memorial Institute

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PNL-3038

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Available from
National Technical Information Service
United States Department of Commerce
5285 Port Royal Road
Springfield, Virginia 22151

Price: Printed Copy $____*; Microfiche $3.00

*Pages	NTIS Selling Price
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Pacific Northwest Laboratory
Richland, Washington 99352

DOE-RL Richland, WA

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ACRONYMS/ABBREVIATIONS

ALATEP	as low as technically and economically practical
AMU	aqueous makeup
ATP	Acceptance Test Procedure
BWR	Boiling Water Reactor
CTH	cask transfer hood
d	day
DAS	data acquisition system
DF	arithmetic decontamination factor
DOE	Department of Energy
ERDA	Energy Research and Development Administration
h	hour
HA	extraction column, solvent extraction system
HAF	feed solution to the solvent extraction system
HAW	aqueous waste stream from the first solvent extraction cycle (from the HA column)
HC	stripping column, solvent extraction system
HCP	uranium-plutonium product from the HC column
HCW	waste from stripping column
HCX	HC column extractant
HEDL	Hanford Engineering Development Laboratory, operated by Westinghouse for the DOE
HEPA	high-efficiency particulate air filter
HLLW	high-level liquid waste
HLW	high-level waste
HS1 & 2	scrub column, solvent extraction system
ICM	in-can melter
ID	inside diameter
ILW	intermediate-level waste
IX	ion exchange system
L	liter
LOI	loss on ignition
LWR	Light Water Reactor
NMM	Nuclear Materials Management

NWVP	Nuclear Waste Vitrification Project
OATP	Operational Acceptance Test Procedure
OD	outside diameter
PNL	Pacific Northwest Laboratory operated by Battelle Memorial Institute for the DOE
POG	process off gas
PWR	Pressurized Water Reactor
QA	quality assurance
RL	Richland Operations Office of the DOE
RTD	resistance temperature detector
RWP	Radiation Work Procedure
SC	spray calciner
scfm	standard cubic feet/minute
SNM	Special Nuclear Materials
SOP	Safe Operating Procedure
tU	metric ton (1000 kg) uranium

ACKNOWLEDGEMENTS

The authors wish to acknowledge the technical contributions to this report made by the following people: D. E. Hadlock, D. E. Knowlton, S. J. Kostorowski, H. H. Van Tuyl, and N. G. Wittenbrock. They also appreciate the contributions of J. S. McPherson, for editing the document, and of E. M. Davie, for typing the manuscript.

TECHNICAL SUMMARY
NUCLEAR WASTE VITRIFICATION PROJECT

1.0 INTRODUCTION

1.1 BACKGROUND

The Nuclear Waste Vitrification Project (NWVP) was initiated to provide a demonstration of the vitrification of high-level liquid waste (HLLW) from spent fuel discharged from an operating LWR, a key to closing the fuel cycle and maintaining reprocessing as a technically acceptable option.

Since 1958, the Pacific Northwest Laboratory (PNL), operated by Battelle Memorial Institute for the DOE, and its predecessor, the General Electric Company, have conducted an on-going research, development, and demonstration program to provide technology for the solidification of wastes from the processing of commercial LWR fuels. Between 1966 and 1970, the feasibility of solidifying radioactive wastes was demonstrated on an engineering scale with simulated commercial wastes prepared from Hanford reactor-fuel reprocessing wastes, plus added inert chemicals.

At its inception, the NWVP encompassed two tasks of the Commercial High-Level Waste Immobilization Program: Waste Preparation, and Radioactive Demonstration of Vitrification. The project was started in April 1976 and terminated in June 1979.

1.2 OBJECTIVE OF THE NWVP

The objective of the NWVP was to demonstrate the solidification (vitrification) as a borosilicate glass, of actual high-level liquid waste from spent LWR fuel.

1.3 SCOPE OF PROJECT

The scope of the Waste Preparation task included the design, installation, and operation of a small-scale (1 tU LWR fuel/month) pilot plant, which would prepare actual HLLW for vitrification. The operational aspects of this task included: the procurement of irradiated LWR fuel; the processing operations to prepare HLLW; and other necessary operations associated with the disposition of the uraniumn waste and other fuel residues, including the plutonium associated with the fuel. The plutonium by-product was purified and converted to plutonium oxide for ease of transport and storage.

The scope of the vitrification task included the modification of the spray calciner/in-can melter system previously used (McElroy 1972) and operation of this engineering-scale (up to 1 tU/day) waste vitrification system. The HLLW was vitrified in two 20.3-cm- (8-in.-) diameter by 2.44-m- (8-ft-) long canisters.

1.4 PURPOSE OF THIS REPORT

The purpose of this report is to present in summary form an overview of the NWVP facilities and their operation and performance. Separate topical technical reports on numerous aspects of the project activities will be issued.

2.0 SUMMARY

Six pressurized-water-reactor (PWR) fuel assemblies, containing 2.3 tU from an operating light-water reactor (Point Beach), have been processed for the generation of high-level liquid waste (HLLW). A conventional Purex-type process was used for the first NWVP processing cycle so the HLLW generated would be typical of the nitric-acid, fission-product waste stream from the first extraction cycle (HAW) of a commercial plant.

Uranium and non-radioactive chemicals, normally added to the HLLW by back-cycling of waste from second and third solvent extraction cycles, were added to the dilute HLLW to produce a waste composition typical of the HLLW from a commercial plant. The waste was then concentrated tenfold to provide feed for the PNL-developed spray calciner/in-can melting (SC/ICM) process. During vitrification, the liquid waste was pumped at a rate of 10 to 15 L/h to the calciner vessel, which was heated to 750°C. The powdered calcine fell into a stainless-steel canister, which was maintained at 1050°C; this canister was attached directly to the bottom of the calciner. Glass-forming chemicals were metered into the canister simultaneously with the calcine. After the materials melted, the canister was cooled to produce a stable vitreous glass. Two 20.3-cm- (8-in.-) diameter by 244-cm- (8-ft-) high canisters containing glass were produced.

The waste preparation facility was designed to process 50 kg U/day (capacity factor = 1). This capacity equates to 1 tU/month, with a planned on-stream efficiency of 70%. The actual on-stream efficiency exceeded 90% for the first three weeks of operation, then dropped to around 50% for the remaining period of operations. The average on-stream efficiency of 62% is excellent for the first hot startup of a new processing facility. The decrease in efficiency was almost entirely due to a plugging problem in the overflow line from the solvent-extraction-product (HCP) concentrator.

The vitrification facility, constructed several years before the inception of the NWVP, was designed to operate at a capacity of 1 tU/day using waste at a concentration of 378 L/t of uranium processed. During project operations, the facility was operated with a more dilute waste for two 2-day campaigns in order to deliberately lengthen the operating time for data collection purposes. The operating runs were routine. The only processing difficulty was a partial failure of the atomizing nozzle in the spray calciner, which resulted in incomplete filling of the first canister of glass.

The plutonium by-product of waste preparation was purified from uranium and fission products by ion exchange. The plutonium nitrate-nitric acid solution was calcined directly to oxide in a screw calciner, then the oxide was sealed in containers and placed in storage. The calciner product met the specifications developed for NWVP. However, one can of oxide self-heated sufficiently, when sealed in an insulated transfer container, that residual volatile materials ruptured the can as it was being removed from the transfer container. Both the calciner temperature and oxide residence time in the calciner were increased to reduce the amount of residual volatile materials in subsequent batches of oxide. As an added precaution, the oxide was heated to 650°C for three hours in a muffle furnace before it was packaged for storage. A parametric study of the effect of calciner operating conditions on product quality and the safety of subsequent storage conditions is recommended.

All cask transfers of uranium liquid waste, sheared hulls and other fuel-assembly hardware, and other solid waste to the 200 East Area for disposal were made without incident. All off gas released to the atmosphere was continuously monitored and found to be well within the discharge limits established for the NWVP by the Environmental Assessment for the project.

3.0 FACILITIES DESCRIPTION

3.1 BACKGROUND

Because the NWVP is a demonstration of technology, there are two principal products from the project: 1) the documentation describing the "what and how" of the successful demonstration, and 2) two canisters of borosilicate glass containing the fission products from irradiated LWR fuel. A by-product of the project is purified plutonium oxide. The principal wastes from the demonstration are fuel assembly hardware, cladding hulls, a fission-product-contaminated uranium stream, a low-level-contaminated process condensate stream, gaseous wastes, failed equipment, and miscellaneous solid wastes.

The feasibility of solidifying radioactive wastes was demonstrated on an engineering scale by 1970. This was done in the 324 Building High-Level Cells; the process equipment used for these operations remained in place. Thus, the 324 Building was the logical location in which to demonstrate the vitrification of HLLW separated from actual LWR fuel.

Before the selection of the 325 Building High-Level Cells for the waste preparation task, two other alternatives were considered by PNL: 1) use of the 200-E Area Hot Semi-Works, and 2) use of additional space in the 324 Building. The Semi-Works alternative was discarded because of the hazards of moving high-level liquid waste between areas, the lack of remote maintenance capability, large capacity, and the excessive cost and time to return to safe operation. The time aspect alone made Hot Semi-Works incompatible with the program needs. The use of additional space in the 324 Building was discounted predominantly because of conflicts with the overall waste management programs, overcrowding of the facility, and personnel logistics. This latter alternative was reconsidered by both PNL and ERDA-RL during the conceptual design stage and was again rejected for these reasons. Therefore, the 325-A Building was chosen as the location for the high-level waste preparation facility.

3.2 DESIGN FEATURES

The simplified flowscheme in Figure 3.1 pictorially shows the major processing functions of NWVP and their physical relationships to each other. These functions are fuel receipt and storage, shearing, dissolving, interbuilding transfer of dissolver solution, solvent extraction, interbuilding transfer of HLLW, preparation of HLLW for vitrification, vitrification, and off-gas treatment. Subsidiary functions include ion exchange, plutonium calcination, and waste disposal. The equipment and facilities are described in Section 3.0. Operation and performance of the facilities are described in Section 4.0.

Fuel-cask unloading capability existed in the 324 Building, whereas the 325-A Building hot cells could only receive chopped fuel in a specially designed canister cask. The integrated NWVP facility was designed to reduce the level of radiation exposure received by operating personnel to as low a level as reasonably achievable. This dictated that the primary dissolver be located adjacent to the fuel shear equipment in the 324 Building and that an existing, but unconnected, underground pipeline between the two buildings be modified and connected. The pipeline was needed for transfer of off gas, dissolver

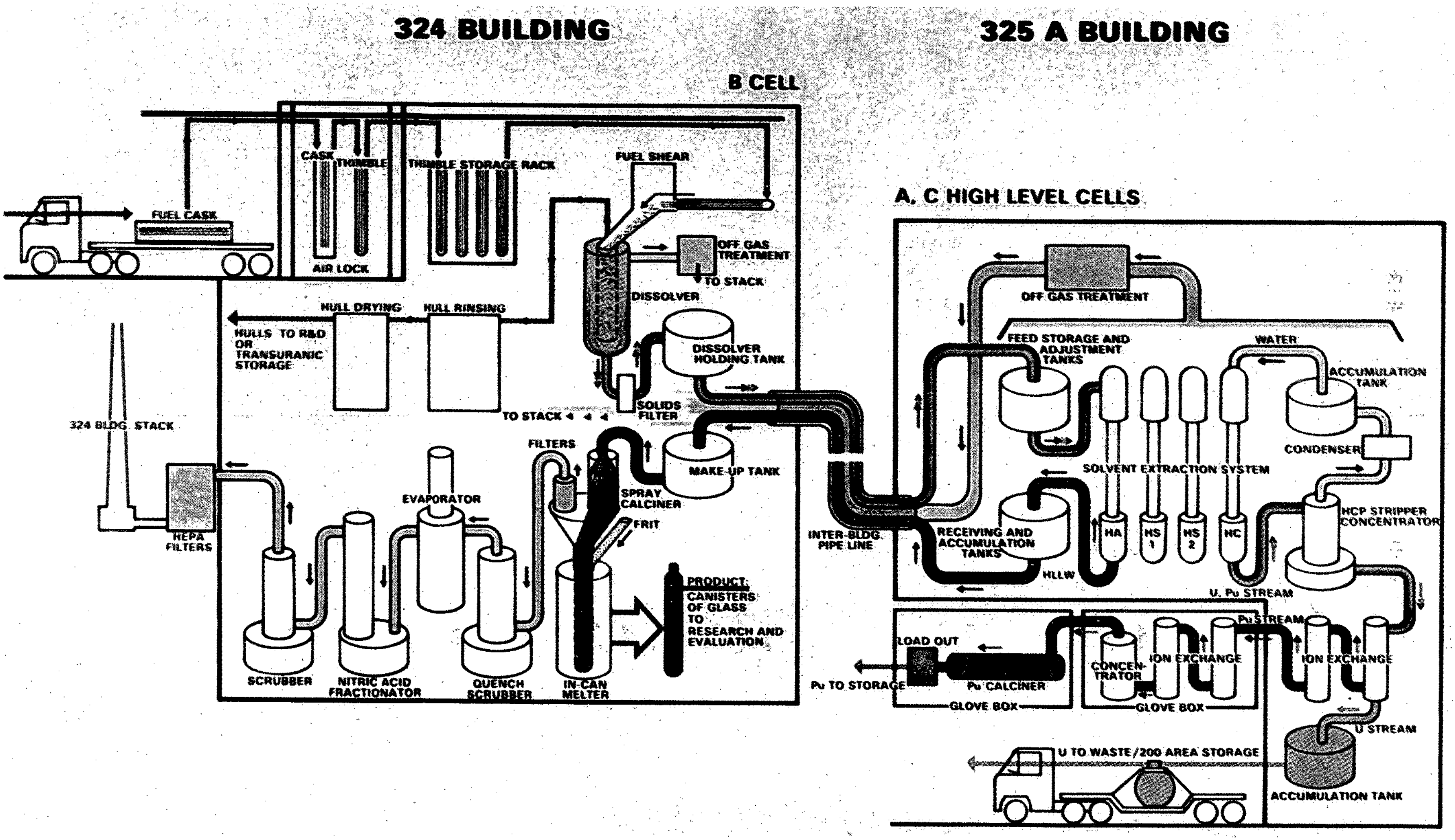

FIGURE 3.1. Nuclear Waste Vitrification Project

solution, and HLLW between the buildings. Transfer by pipeline was preferred because such transfers did not involve radiation exposure to operating personnel. However, the capability for cask transfer of chopped fuel and high-activity solutions between buildings was maintained as a backup option.

The objective of the NWVP was to demonstrate the solidification (vitrification) as a borosilicate glass, of HLLW from spent LWR fuel. Therefore, for public acceptance, the fission-product HLLW used for vitrification must not be a synthetic solution, but must be typical of the HLLW produced by existing or potential fuel reprocessing plants. Because at the inception of the NWVP such plants used a Purex-type solvent extraction system for the first co-decontamination cycle, this system was selected for NWVP. Although alternative mechanisms for counter-current contact of aqueous and organic streams were considered, fixed-plate pneumatic-pulsed columns were selected because of extensive on-plant technology, simplicity of design, and the ability of such equipment to process unclarified feed. In a Purex-type solvent extraction system, the aqueous waste stream from the extraction column (designated HAW) contains essentially all of the fission products, solids, and other nonextractable impurities present in the dissolver solution; thus, this stream becomes the HLLW from the reprocessing system.

The hot cells within the 324 and 325-A Buildings were designed for distinctly different types of operation. The 324 Building hot cells were designed with a central air lock and remotely-operated overhead bridge cranes for remote installation, maintenance, and operation of both large and small pieces of equipment. This design feature is illustrated by Figure 3.2. After fabrication, all pieces of the 324 Building equipment were set up and operated in a "cold" zone to ensure that they functioned properly and complied with design criteria. The equipment was then disassembled and remotely installed within the cell by operations personnel. The hot cell was not decontaminated for this installation.

Conversely, there is no large air lock for the 325-A Building hot cells, as shown in Figure 3.3. Small items can be moved in or out of the cells through a 33-cm- (13-in.-) dia port through each rear shielding door, but installation or replacement of large equipment requires decontamination of the cell to a level low enough for personnel entry through the cell door. This imposes restraints on equipment design and installation. All items of equipment, such as valves, pumps, or instrumentation susceptible to radiation damage or rapid failure, must be located within reach of the manipulators, must be light enough to be handled by manipulators, and must be small enough to be replaced through the 33-cm- (13-in.-) dia port. Construction craftsmen initially installed all the equipment in the cells using hands-on methods. Any failure of a large piece of equipment that cannot be by-passed will require decontamination of the cell before the equipment can be replaced.

3.3 324 BUILDING

3.3.1 Fuel Acquisition and Storage

The NWVP facilities are designed to receive and handle either pressurized water reactor (PWR) or boiling water reactor (BWR) spent fuel assemblies. Such assemblies, shipped in an NAC-1 shielded shipping cask by tractor-trailer, are received in the Truck Lock unloading facility, located in the 324 Building near the rear face of the hot-cell

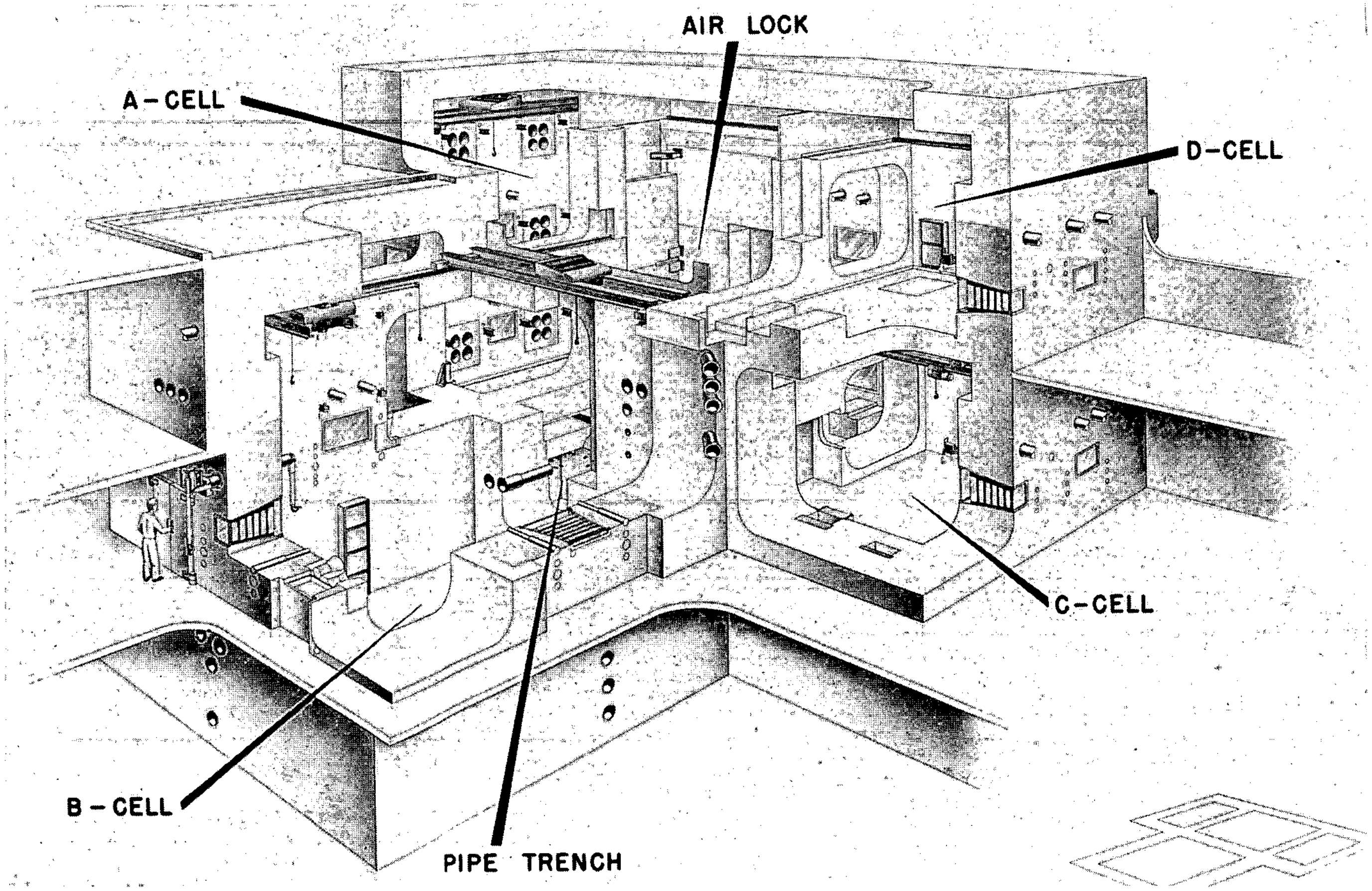

FIGURE 3.2. 324 Building Radiochemical Engineering Cells

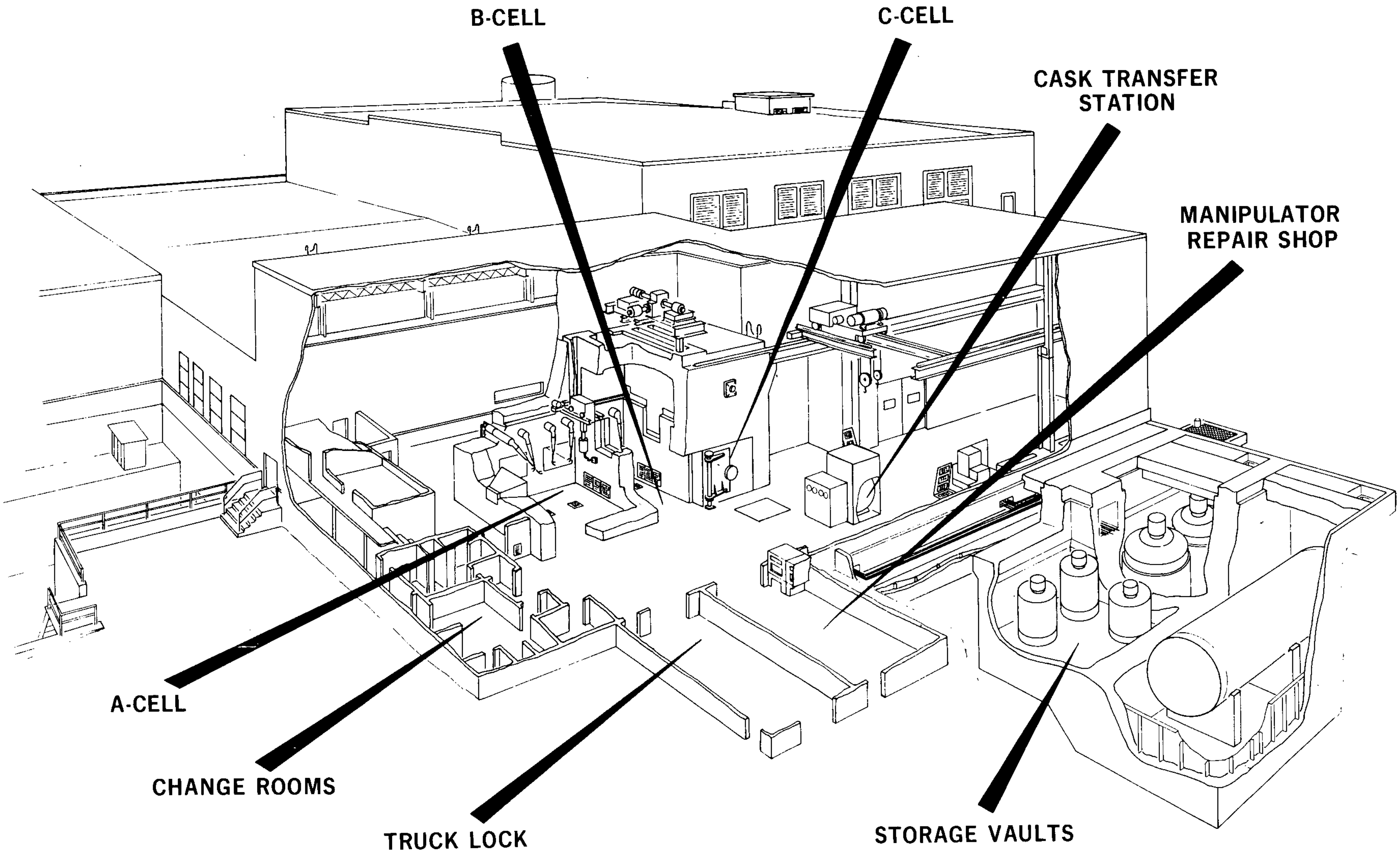

FIGURE 3.3. 325-A Radiochemistry Building

complex. Figure 3.4 shows a schematic arrangement of some of the unloading equipment, in addition to some of the processing equipment installed in B-Cell. The cask is removed from the trailer by a 30-ton bridge crane, is carried to a cradle located in the Cask Handling Area, elevated from a horizontal to a vertical position, then again moved by the crane and positioned on a track-mounted cask dolly. The cask is then draped with plastic and rolled into the shielded Air Lock; the tracks outside the Air Lock are removed, and the Air Lock door is closed. With a wall-mounted jib crane and a 6-ton bridge crane, the cask lid is removed, then the fuel assembly is extracted from the cask and placed inside a handling thimble. The fuel assembly, enclosed in the thimble, is transferred into B-Cell by the bridge crane and is placed in a fuel storage rack, designed as a mini-wet-basin. The cask is then resealed, the drapes are removed, and as necessary, the cask is decontaminated. After the Air Lock door is opened, the cask is removed and replaced on the trailer, by a reversal of the steps described above.

The fuel thimbles are square tubes 3.31 m (130.5 in.) long, closed at one end. The open end is rounded and funneled out to facilitate remote acceptance of the square fuel assembly. A lifting bail attached to the thimble permits movement of the thimble by overhead cranes. The thimble provides structural support and damage protection for the fuel assembly during handling and storage.

The fuel storage rack can hold up to nine fuel assemblies. The cooling capacity of the heat exchanger in the storage rack exceeds 45 kW. However, because such capacity was not needed for the 4- and 6-yr-cooled fuel used in the project, the cooling coils in the storage rack were not used. A sketch showing the fuel storage rack, fuel assembly table, a fuel thimble, the feeder puller, and the shear is shown in Figure 3.5.

3.3.2 Fuel Disassembly and Shearing

For disassembly and shearing, a thimble containing one PWR fuel assembly is removed from the storage rack and placed on the disassembly table, as shown in Figure 3.5. Seventeen of the 196 Zircaloy tubes in a PWR fuel assembly do not contain fuel. Their function is to hold instrumentation, tie the nozzle plates together, and hold the spacers in place. The fuel pins are not attached at either end. Disassembly is accomplished by severing each instrument tube with an inside tubing cutter and by removing the top nozzle plate. The fuel pins are then withdrawn from the assembly, leaving the tie tubes, spacers, and bottom nozzle plate in place inside the thimble.

The table for fuel assembly has multiple design features. In addition to holding the fuel thimble, it has horizontal, vertical and axial motions necessary to line up fuel rods with a feeder-puller mechanism to the shear. Linear actuators provide power for the three motions of the table.

The feeder-puller is designed to remove one to four fuel rods and to feed them into the shear. A double-acting air cylinder operates the device, which grabs, pulls, releases, and retracts in sequence with the upand downstroke of the shearing blade.

A commercial hydraulic shear(a) has been modified so that it is more resistant to the radiation environment. The pump, motor, and fluid reservoir (water/glycol) are located

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(a) W. A. Whitney Co., Rockford, IL.

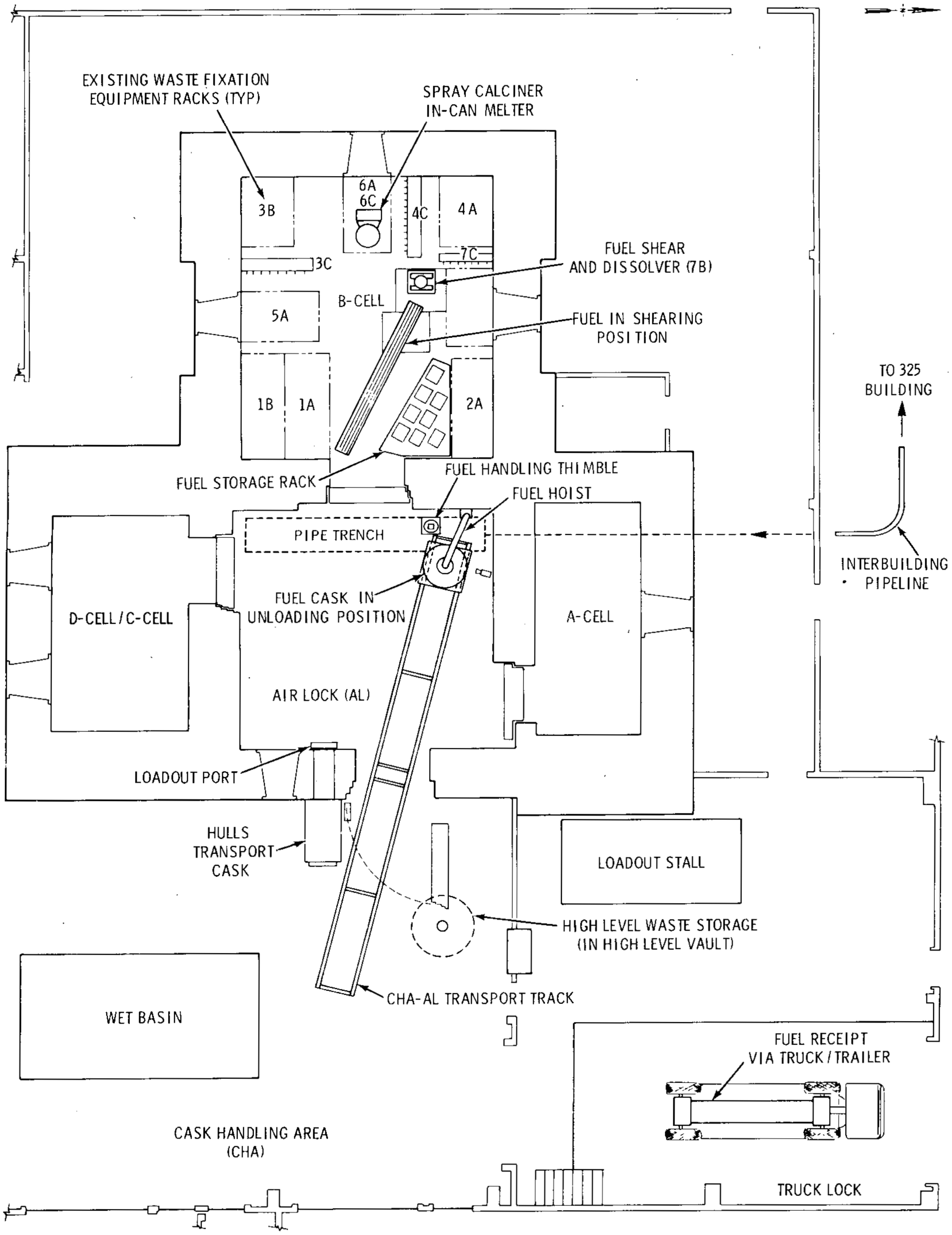

FIGURE 3.4. 324 Building Radiochemical Engineering Cells - Plan View

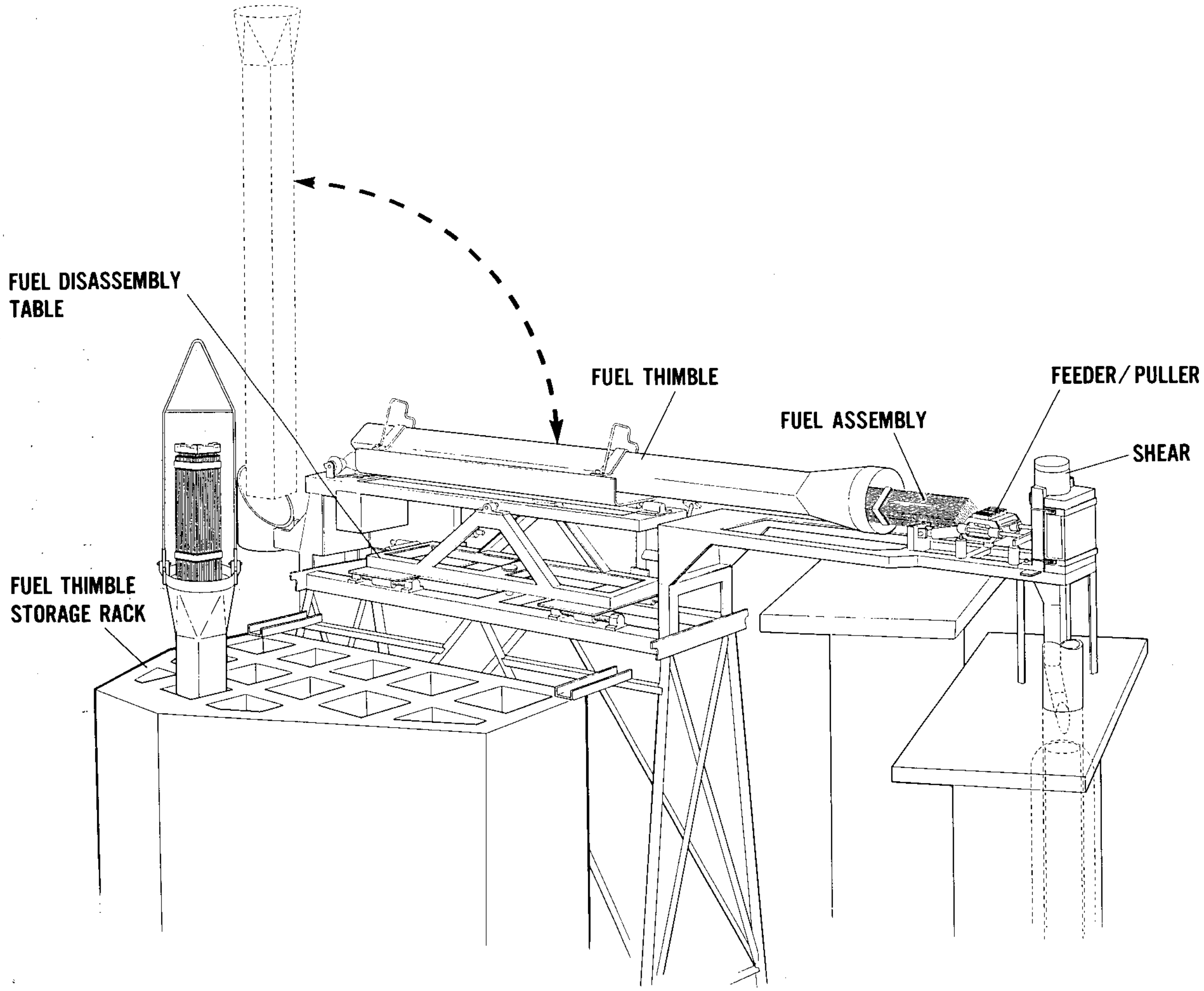

FIGURE 3.5. Fuel Disassembly and Shearing

outside the cell. The cutting mechanism has been designed so that lateral forces on the blade are balanced. Two fuel pieces are produced, 5.1 cm (2 in.) and 1.3 cm (1/2 in.) in length, with this double-balanced cut design. The cut fuel pieces drop down a chute into a basket located inside the dissolver.

3.3.3 Primary Dissolver

The major components of the primary dissolver are the dissolver vessel, with its auxiliary reservoir, and the dissolver off-gas system. The dissolver off-gas system consists of a demister, a condenser, a steam heater, and a silver zeolite bed with an auxiliary electrical heater. This equipment is shown in Figures 3.6 and 3.7. (Integration of the dissolver system, the fuel shear, the off-gas system, and the dissolver-solution holding tanks is shown later by the process flow diagram in Figure 4.1.)

The 25.4-cm- (10-in.-) dia dissolver vessel and the 30.5-cm- (12-in.-) dia auxiliary reservoir are fabricated from Schedule 40 pipe with three cross connections between them. The arrangement facilitates thermosiphon recirculation between the two vessels, thus maximizing the capacity of the dissolver while maintaining prescribed nuclear criticality safety constraints. The entire unit is designed to fit inside an existing 78.7-cm- (31-in.-) ID tank for secondary containment.

The dissolver lid seats in a water-seal-pot enclosure for maintaining a normal operating pressure of -12 to -25 cm (-5 to -10 in.) of water within the dissolver. Pressures of <-30 cm or >30 cm (<-12 in. or >12 in.) of water cannot be reached in the dissolver vessel without breaking the water seal.

Inside the dissolver are both steam and water coils, alternately wound down the inside diameter of the shell encompassing the total length of the basket. These coils are held in place and are protected from possible damage by a perforated screen. The screen (16 ga. 63% free area) also limits the diameter that fuel rods could fill, in case fuel is accidently sheared without a basket in place.

The perforated basket fits inside the dissolver. It is supported primarily from the reinforcing collar at the upper end. Some support from the bottom brackets is available, if linear expansion occurs. The basket-lifting rod extends to the top of the dissolver, where a lifting bail is located. The bail fits into slots to properly align the basket to the chopped-fuel chute, which is located just above the basket. A perforated screen on the lifting rod handle prevents any pieces greater than 0.397 cm (5/32 in.) in diameter from being carried over into the reservoir with the recirculating solution.

The top overflow to the reservoir is located about 7.6 cm (3 in.) above the chopped fuel inlet. During dissolution, the solution overflowing into the reservoir effects a seal of the fuel inlet line, thus, inleakage from the shear is reduced. The middle cross connection between the dissolver and reservoir has been installed primarily for the dissolution of a half batch of fuel.

The lower return line from the reservoir enters the dissolver at a tangential position to aid in keeping suspended any material that might settle on the bottom of the dissolver. The dissolver contains an air sparge just below the basket, four thermocouple penetrations,

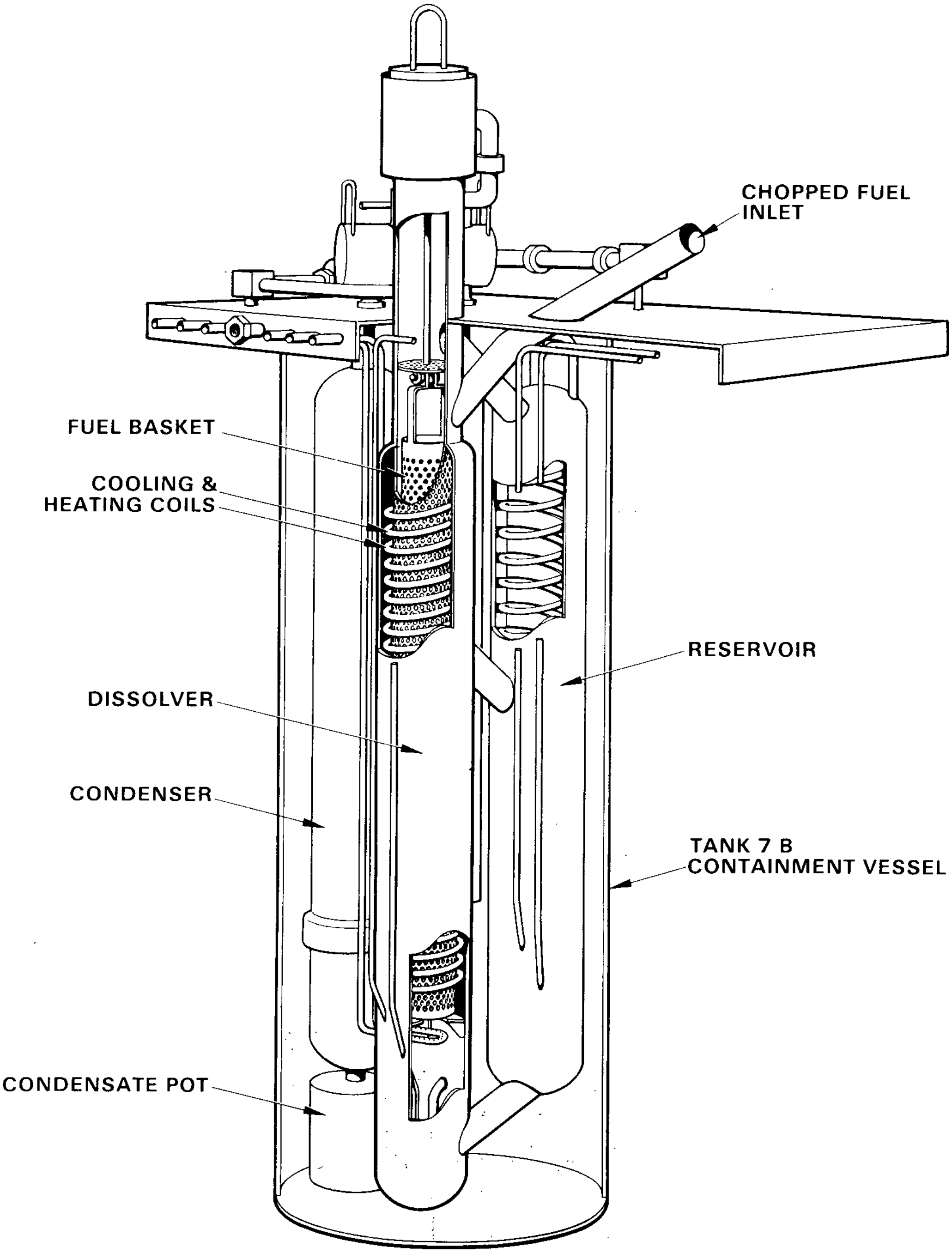

FIGURE 3.6. 324 B-Cell Dissolver System - Side View

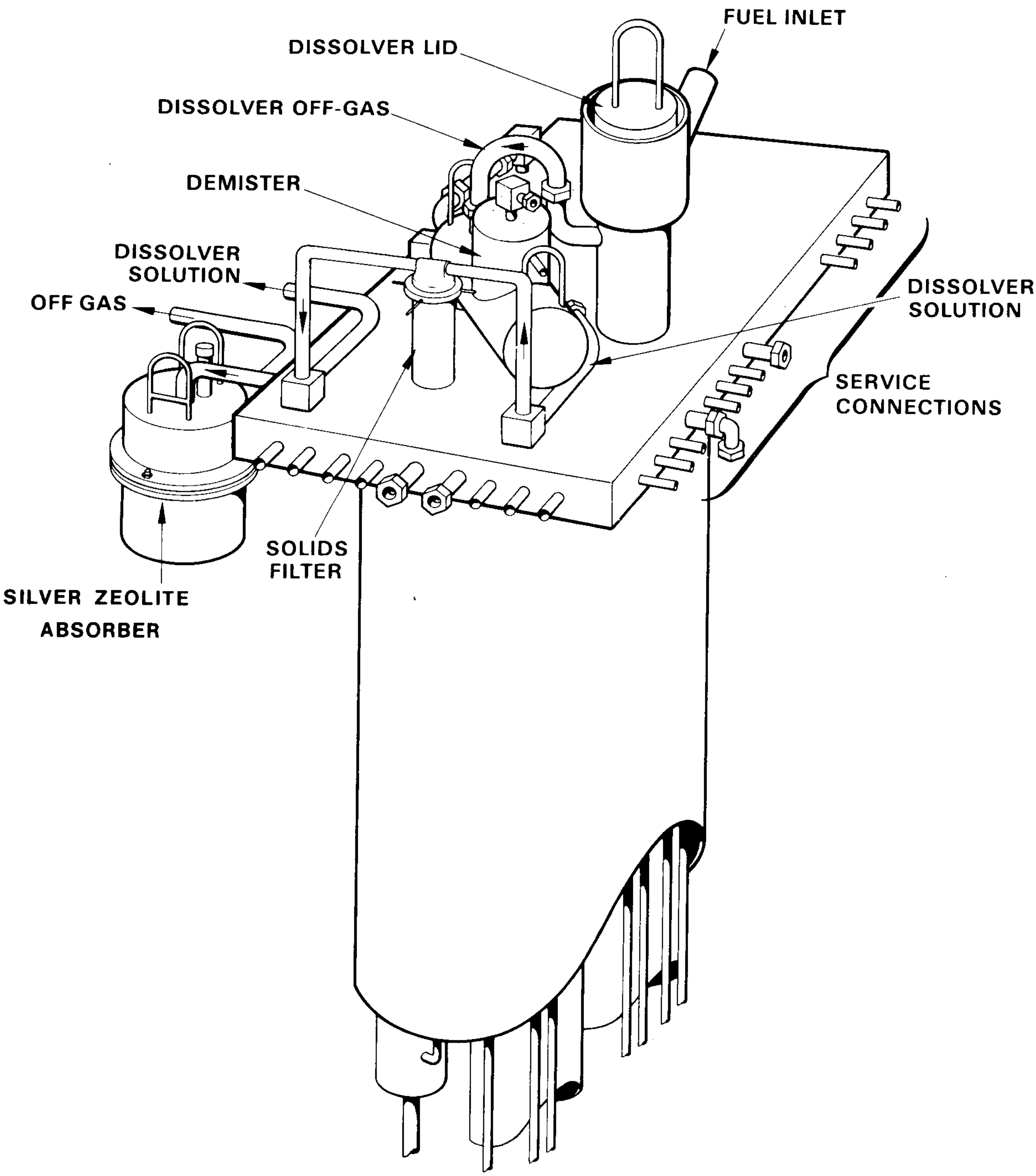

FIGURE 3.7. 324 B-Cell Dissolver System - Top View

a pressure tap, specific gravity and liquid-level dip tubes, a solution jet-out line, a chemical addition line, and a spare line extending to the bottom of the dissolver. All construction materials are 304L stainless steel.

3.3.4 Dissolver Off-gas System

The dissolver off-gas system consists of a demister, shell and tube condenser, off-gas heaters, and a silver zeolite bed.

The demister, sized for 1.13 m^3/min. (40 scfm) gas flow at 95°C, is installed in the dissolver off-gas line upstream from the condenser. The demister mesh pad has a cross sectional area of 186 cm^2 (0.2 ft^2) and a depth of 23 cm. (9 in.). Predicted performance data is 97% removal of 10 μ diameter droplets, at a gas flow of 1.42 m^3/min. (50 scfm).

The dissolver condenser is a shell and tube condenser with a heat-transfer surface area of 6.32 m^2 (68 ft^2). The condenser is sized for the maximum heat transfer area that could physically fit into the existing space envelope. It is desirable to keep the off gas at 60°C to avoid iodine retention in the condensate.

A steam-jacketed heater and an auxiliary electrical heater are used to raise the temperature of the off gas to between 100 and 130°C; thus, condensation of water in the zeolite bed is avoided and the reaction kinetics of the absorption process are increased.

The silver zeolite absorber is designed as a vertical gas-flow bed, 25 cm in diameter x 15 cm deep (10 in. in diameter x 6 in. deep) with dissolver off gas flowing down through the bed. The bed is contained within a basket for remote replacement.

3.3.5 Dissolver-Solution Filtration and Transfer to 325-A Building

Two types of solids remain suspended in the dissolver solution after dissolution: 1) Zircaloy or Zircaloy-oxide chips and fines, and 2) undissolved fuel and fission-product particles.

Dissolver solution is transferred by jet from the dissolver, through an in-line filter canister, to a holding tank (shown later in Figure 4.1). The 100 μ stainless-steel filter is designed to retain only the larger particles and to pass the finer, easily suspended particles that remain with the fission products during subsequent processing and end up in the vitrified glass. When plugged with solids, the filter canister is removed from the system, filled with cement, then surrounded by cement in a disposal bucket. These precautions are taken to avoid the potential of a Zircaloy-fines fire.

Dissolver solution in the holding tank is air-lifted to a recirculating head pot. This head pot provides a constant source of dissolver solution at an adequate elevation for vacuum transfer through the 3/8-in. section of the interbuilding pipeline from 324 Building to 325-A Building (see Section 3.5).

3.3.6 HLLW Concentration and Feed Preparation

The major components of the HLLW concentration and feed preparation facility, all located in the 324 Building, are the service tanks TK-112, TK-114 and TK-116, the evaporator (TK-113), the acid fractionator (TK-115), and the process-off-gas scrubber

(TK-118). The HLLW is transferred by a 324 Building steam jet from TK-W4 in the 325-A Building Vault to TK-106 in 324 Building. Solution is then transferred to a storage tank, TK-107, where chemical additions are made before the solution is transferred to B-Cell for concentration. (The tank-to-tank transfer of solution in B-Cell is shown later in the process flow diagram, Figure 4.3.) Each of these equipment pieces is described below.

Evaporator (TK-113)

The evaporator has three functions: 1) HLLW concentration, 2) condensate concentration during calciner operation, and 3) off-gas decontamination. The equipment is a medium-length-tube, thermosyphon-type evaporator designed to operate with a maximum-to-minimum operating volume ratio of 18:1. The evaporator is constructed entirely of commercially-pure titanium for improved corrosion resistance.

The evaporator tower consists of four stages of de-entrainment: 1) a simple chevron baffle at the bottom, 2) and 3) two stages of dry impingement caps in the center, and 4) a remotely replaceable mist eliminator at the top. At the maximum design boilup rate (500 L/h), the impingement velocities of stages 1, 2, and 3 are 11, 16, and 26 m/sec (36, 51 and 85 ft/sec), which is sufficient for nearly 100% removal of aerosols of 25, 7, and 4 μ, respectively.

Acid Fractionator (TK-115)

The all-titanium acid fractionator consists of a packed-tower distillation column surmounting a standard reboil tank. The top and bottom spheroid heads are partially reinforced with an extra thickness of titanium plate for added strength, similar to those in the evaporator. Special features of the fractionator include a remotely replaceable tube bundle in the reboiler, a de-entraining sieve plate with by-pass provisions in the tower, and an integral reflux condenser.

The rectifying section of the fractionator tower is packed with 2.5-cm-(1-in.-) dia titanium Raschig rings. The exhausting section includes a similarly packed section surmounted by a de-entraining sieve plate and an integral reflux condenser. The fractionator is designed for a liquid feed of up to $0.8\underline{M}$ HNO_3, an overhead fraction of $0.01\underline{M}$ HNO_3, and a concentrate up to $10\underline{M}$ HNO_3.

Process-Off-Gas Scrubber (TK-118)

The process off-gas (POG) scrubber is a 1300 L tank surmounted by a packed scrubbing tower. A recirculating pump recycles the liquid scrub solution to the top of the tower at rates of up to 40 L/min. Gas flows of up to 11.8 m^3/min (416 scfm) are acceptable. Gas discharged from the scrubber leaves B-Cell and enters the 324 Building vent system through the E-101 condenser, as shown in Figure 3.8.

Miscellaneous Service Tanks

Four stainless steel service tanks are available, three of which have been used. Each has a volume of about 1000 liters. All have agitators or pumps, coils, and jackets and are fully instrumented.

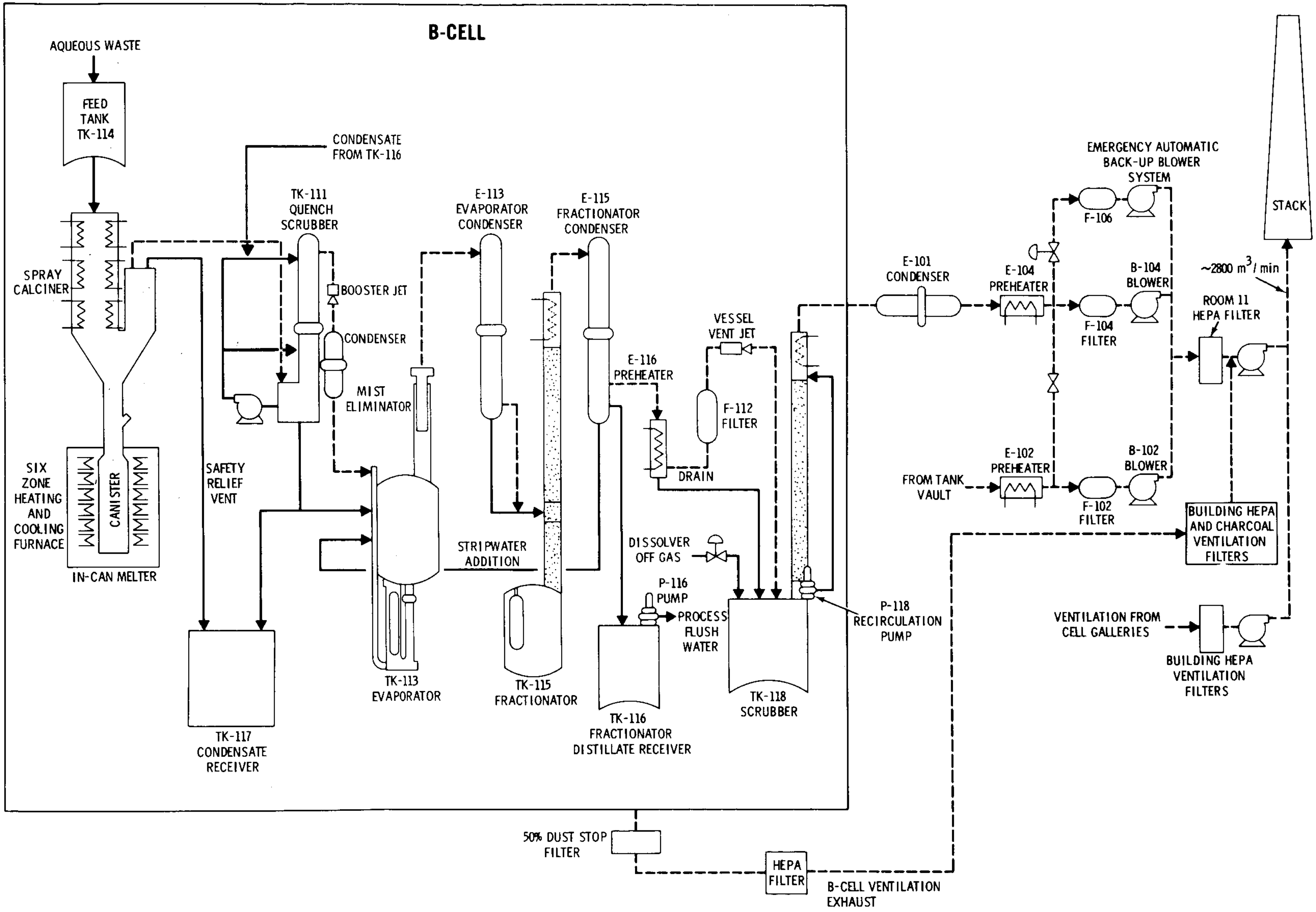

FIGURE 3.8. 324 Building B-Cell Effluent Control System

3.3.7 Waste Calcination and Vitrification

After adjustment of the chemical composition and concentration, the HLLW is pumped to the spray calciner/in-can melter, shown schematically in Figure 3.9, for conversion to glass. A photograph of the spray calciner/in-can melter, installed in B-Cell during a prior program, is shown in Figure 3.10. This system was modified for use during the NWVP by replacing the Metallic Metal Furnace with a connecting pipe, as shown in Figure 3.9 and later in Figure 4.3. In simplest terms, the spray calciner is a vertically mounted chamber, 35.6-cm in diameter by 197.5 cm high (14 in. in diameter by 77.8 in. high) that is fabricated from 310 stainless steel, chosen because of its excellent resistance to corrosion at high temperatures. The spray-chamber walls are heated to about 750°C by a surrounding 3-zone, 45 kW resistance furnace. The rate of feed-solution flow to the spray calciner is controlled by a pneumatic control valve coupled to a magnetic flow meter. Compressed air is used in a modified, commercially-available, internal-mix spray nozzle to atomize the feed solution as it enters the top of the spray chamber.

As the atomized waste solution falls through the chamber, heat is transferred to it from the walls by radiative and convective means. The waste is concentrated, dried, and oxidized to a calcine product of typically less than 0.5% moisture and 1% nitrate as it drops through the chamber. A buildup of solids on the chamber wall is prevented by periodic use of a wall-mounted, air-operated vibrator. Porous stainless-steel filters are used to remove solid particles from the off gases leaving the spray-calciner chamber. The off-gas filter unit contains sixteen 5.1-cm-OD x 91-cm-long (2-in. OD x 36-in.-long) filters with a nominal pore size of 65 μ. The calciner is operated at a nominal pressure of -12 to -25 cm (-5 to -10 in.) of water.

Glass-forming material, called frit, is metered into the lower cone of the spray calciner at a rate proportional to the rate of calcine production. The frit falls with the calcine into the receiving canister below the calciner, where the mixture is melted and then cooled to form the final waste-glass product. The in-can melter uses the final storage canister as the melting crucible to vitrify the mixture of calcine and frit. The canisters are constructed of Schedule 40, 304L stainless-steel pipe. Details of the canister are shown in Figure 3.11. The melter furnace is a six-zone resistance type. Each zone is 35.6 cm (14 in.) high and delivers 15 kW of power. The furnace operates at 1050°C.

The quench scrubber (TK-111) serves as the primary off-gas scrubber for the spray calciner. This vessel (shown in Figure 3.8) is a 25.4-cm- (10-in.-) dia Schedule 20 pipe tower, fabricated from 304L stainless steel, and packed with 2.5-cm (1-in.) pall rings of the same material. Two liquid distribution weirs, one near the top and a second at the midsection of the tower, serve to collect and redistribute scrub solution and condensate to the packing. Condensate from the fractionator distillate receiver (TK-116) is pumped to the top weir and scrubs the rising gas stream as it falls through the tower. The scrub solution plus the spray-calciner condensate from the quench section of the tower are collected in a tank at the base of the tower and recycled by a pump to either the top or the bottom of the tower, depending on operating needs. This recycled liquid is cooled by

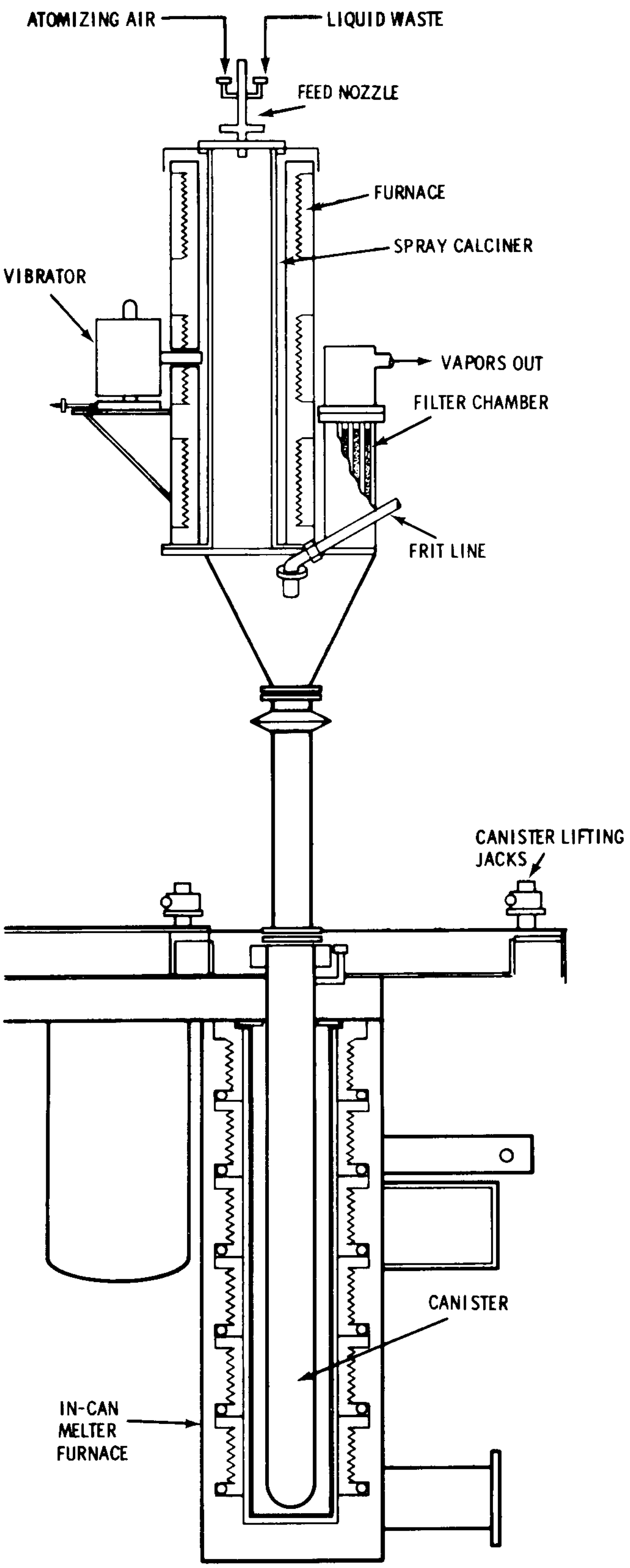

FIGURE 3.9. Schematic View of the Spray Calciner/In-Can Melter

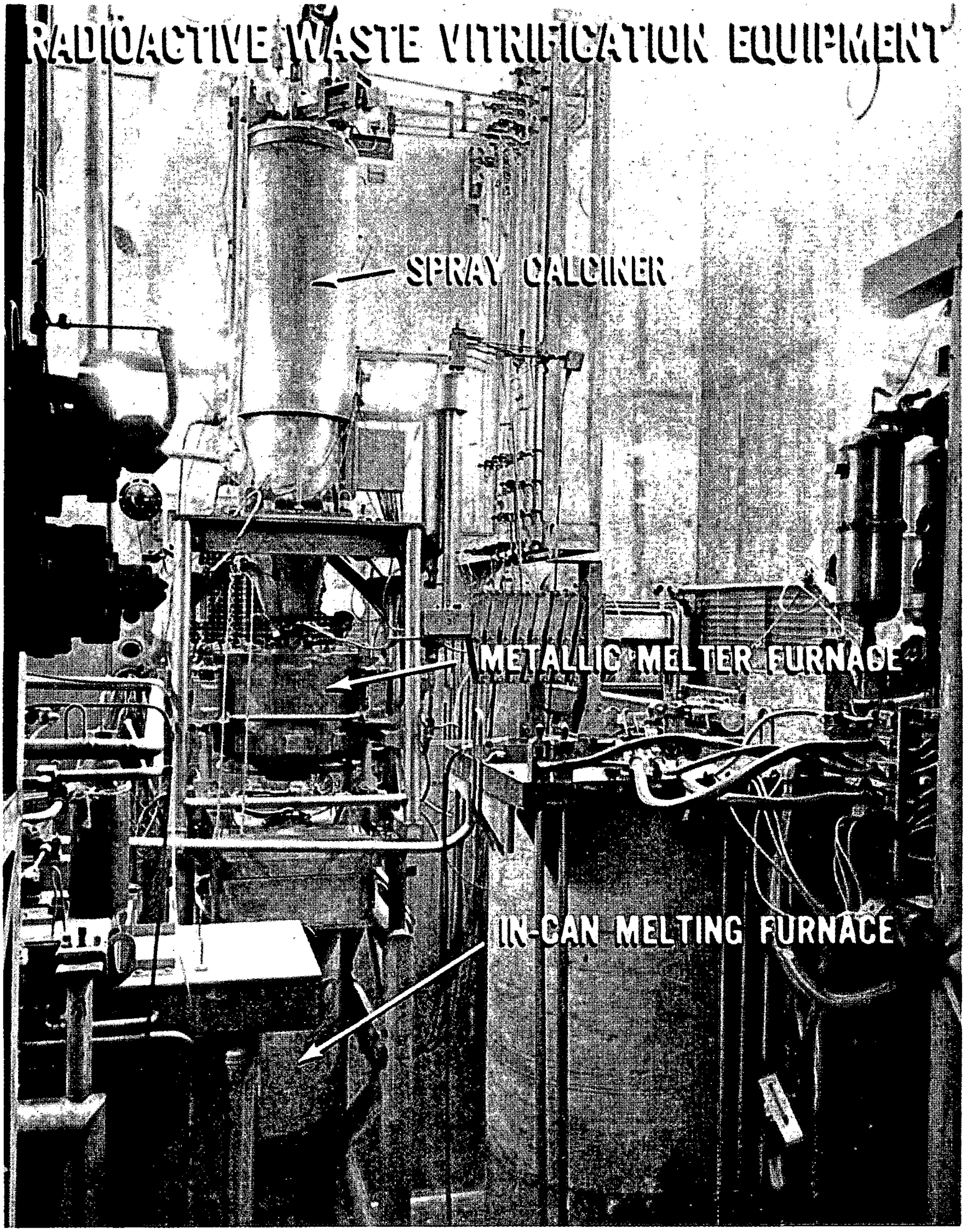

FIGURE 3.10. Photograph of the Spray Calciner/In-Can Melter Installed in 324 B-Cell

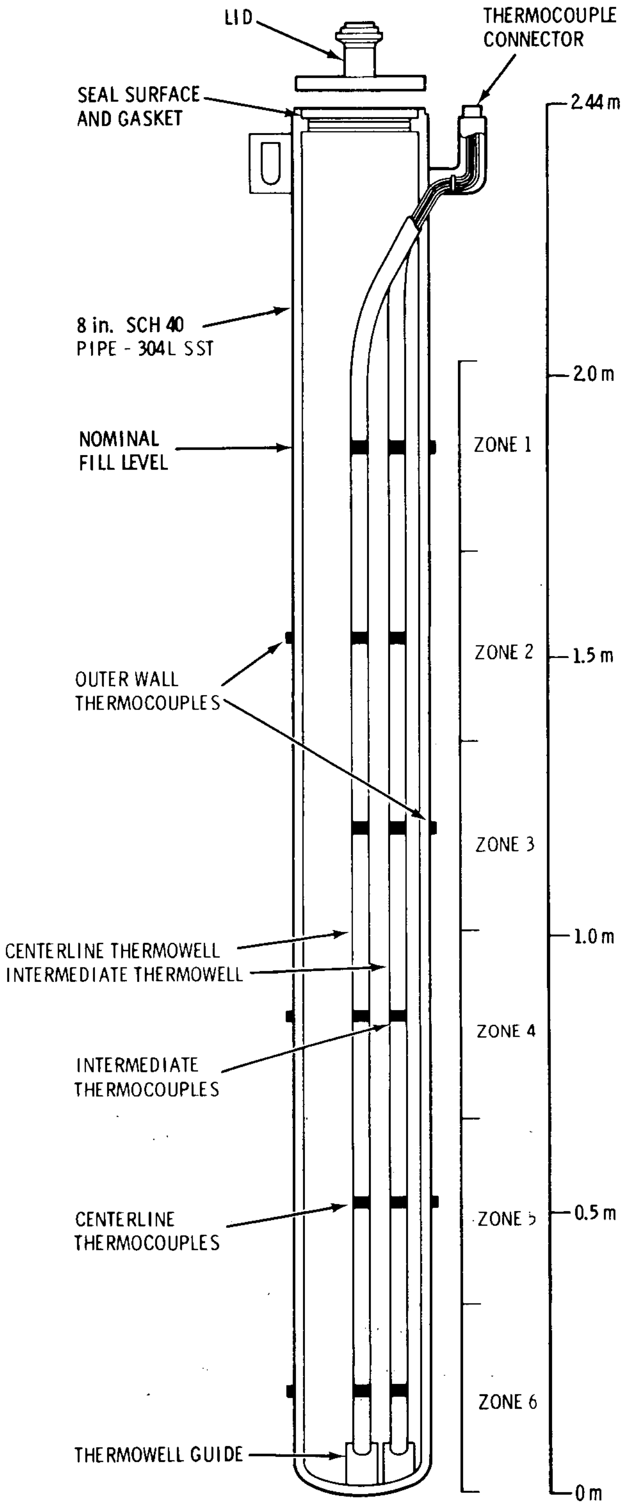

FIGURE 3.11. Schematic View of a Waste Canister

passing it through a water-cooled heat exchanger. A constant liquid level is maintained in the collection tank by an overflow weir draining to TK-113.

A steam-jet assembly, located downstream from the quench scrubber, maintains the spray calciner/in-can melter system at the prescribed negative pressure. A condenser on the jet discharge line cools the gas and condenses the steam before its entry into TK-113. The other vessels used to decontaminate the off gas from the calcination-vitrification process, TK-113, TK-115, and TK-118, were described in Section 3.3.6.

3.3.8 324 Building Ventilation System

The parts of the 324 Building ventilation system used in the NWVP were previously shown in Figure 3.8. Off gas transferred from 325-A Building through the pipeline is discharged into the 324 Building pipe trench (described later in Section 3.5), from which it enters the B-Cell ventilation system and then, after HEPA filtration, the building ventilation system (as shown in Figure 3.8). The B-Cell process off gas enters the building system through condenser E-101.

3.4 325-A BUILDING FACILITY

Integration of the 325-A Building facility into the NWVP was shown in Figure 3.1. The physical location of the major structural components within the building was shown in Figure 3.3. Dissolved fuel from the 324 Building is vacuum-transferred through the interbuilding pipeline to the 325-A Building, where the uranium and plutonium are separated from the fission products by solvent extraction. The HLLW, containing most of the fission products, is vacuum-transferred back to the 324 Building for vitrification. The uranium and plutonium are then processed through a cycle of ion exchange, during which the plutonium is partitioned from the uranium and further decontaminated from fission products. Finally, if needed, the plutonium is processed through a second cycle of ion exchange for additional decontamination; it is then calcined to oxide. The uranium and fission-product waste stream from the ion-exchange cycles is accumulated and disposed of as an intermediate level waste (ILW). A back-up dissolver system is provided in the 325-A Building for use in the event of a malfunction in the 324 Building dissolver or in the interbuilding pipeline.

The major processing components of the 325-A Building facility, therefore, include: a back-up dissolver system, a solvent extraction system, two ion-exchange systems (IX1 and IX2), the plutonium calciner, and an off-gas treatment system. Two important supporting systems are the ILW system and the process control instrumentation. The processing equipment is contained in two hot cells (A- and C-Cells) and in two lightly-shielded glove boxes. Liquid storage tanks are located below the cells and in an auxiliary vault. Chemicals are added to the processing equipment from an aqueous make-up area (AMU) adjacent to the cells and above the change rooms. These systems are briefly described in the following sections.

3.4.1 Back-up Dissolvers

During the design stages of the project, it was noted that any problem in the 324 Building dissolver or in the interbuilding pipeline would greatly delay the completion date of the project. A back-up dissolver system that could dissolve chopped fuel has been, therefore, installed in C-Cell of the 325-A Building. If the fuel cannot be dissolved in the 324 Building, it can be chopped and then transferred by cask to the 325-A Building for dissolution and processing. The back-up dissolver system consists of two 60-L steam-heated tanks into which baskets of chopped fuel can be placed. After the fuel is dissolved, the basket is removed and placed in the hulls rinse tank to remove any adhering dissolver solution. If the dissolvers are operated on a continuous basis, approximately 50 kg of uranium can be dissolved per day. The off gas from each of the dissolvers passes through a condenser and then into the C-Cell off-gas treatment system (described later in Section 3.4.5). The back-up dissolvers; process off-gas scrubber, HEPA filter, and silver zeolite bed; and miscellaneous tanks, valves and transfer jets are shown in Figure 3.12.

3.4.2 Solvent Extraction System

After the dissolver solution is transferred to the 325-A Building, it is sampled for Special Nuclear Materials (SNM) accountability and adjusted to a nitric-acid concentration of 2.5M. This solution is then fed to the solvent-extraction system, where the uranium and plutonium are separated from the fission products in a standard Purex-type, co-decontamination, pulse column battery. The solvent-extraction system consists of: 1) an extraction column (HA), in which the uranium and plutonium are co-extracted into the solvent; 2) two scrub columns (HS1, HS2) for scrubbing carried-over fission products back from the solvent, 3) a stripping column (HC), in which the uranium and plutonium are stripped from the solvent back into an aqueous phase, 4) a U-Pu (HCP) concentrator, where the volume of solution is reduced and the HNO_3 concentration is increased to 7.5M for ion-exchange processing, and 5) a solvent cleaning system, in which the solvent is washed and filtered before it is recycled to the extraction column. The four solvent extraction columns and the HCP concentrator are pictured in Figure 3.13. The design criteria of the pulse columns are summarized in Table 3.1. The solvent used is 30 vol% tri-butyl phosphate (TBP) in a normal paraffin hydrocarbon diluent. The system is designed to process 50 kg U/day, with actual throughputs ranging up to 60 kg U/day. The HLW exiting the HA column is accumulated and sampled for accountability before transfer to the 324 Building.

The solvent-extraction system is operated on a continuous-flow basis, the feed-solution (HAF) flow and all intercolumn flows, except the solvent added to the HA column, are motivated and controlled by calibrated air lifts and gravity overflow. Cold chemical additions from the AMU are metered by positive displacement pumps or by flowmeter and control-valve systems. The solvent added to the HA column is pumped through a flowmeter and a control valve. Aqueous-organic interfaces are sensed with floats and are automatically controlled by adjustments to the aqueous flow rates from the columns. Air pulsers are used in all four columns, with pulse amplitude adjustments made by varying the pressure to the pulsers. Frequency can be controlled over a range of 0 to 120 pulses/min with an electronic pulse frequency controller. Overall column density is measured by two independent methods: 1) bubbler tubes extending to the bottom of the columns, and 2) pressure transducers mounted on the side of each column. Bubbler tubes are used to measure the density of the organic phase at the top of each column. The HC column is constructed with a water jacket to allow for operations at 50 to 60°C.

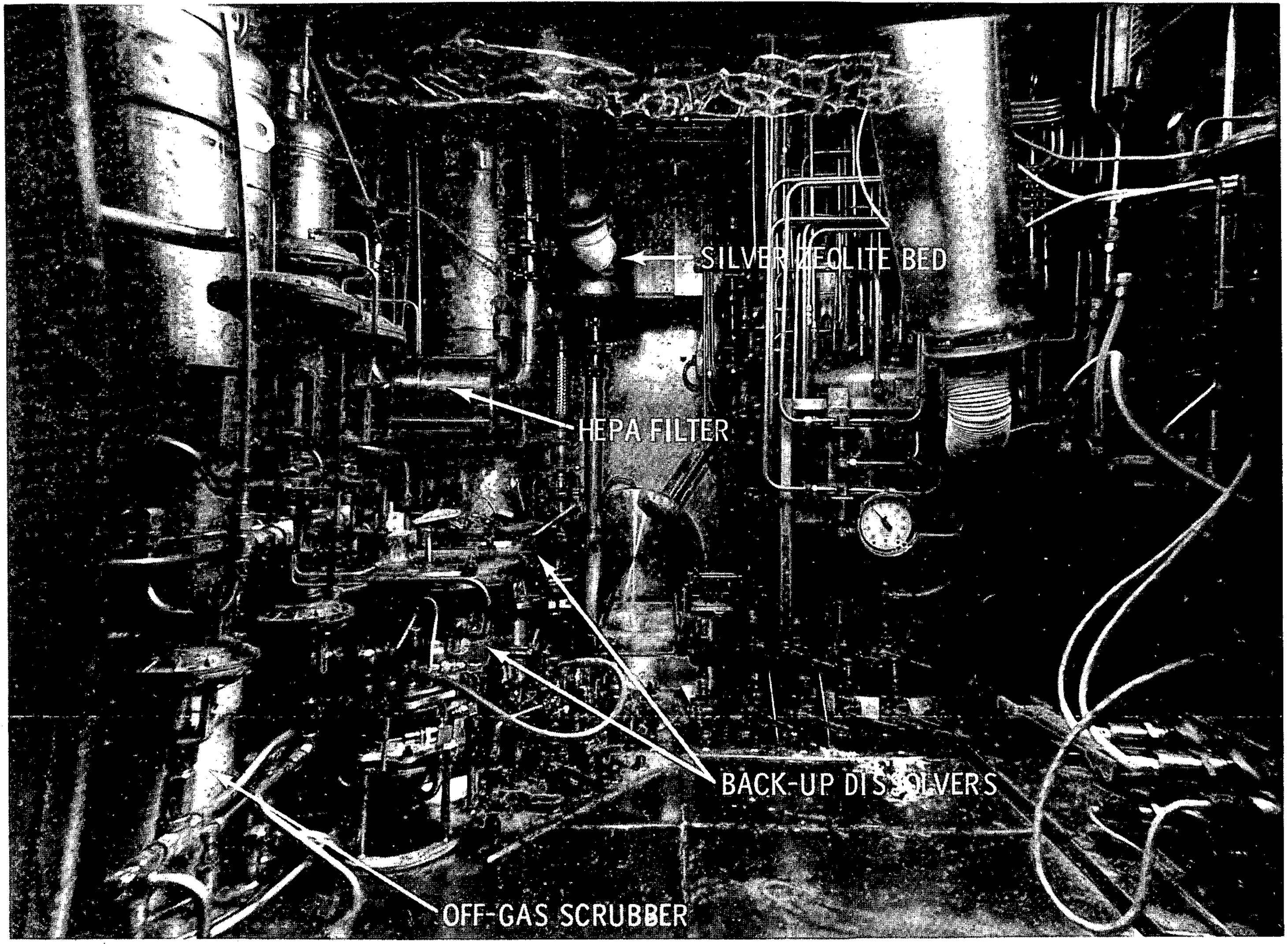

FIGURE 3.12. C-Cell Equipment Viewed Through the Cell Window, 325-A Building

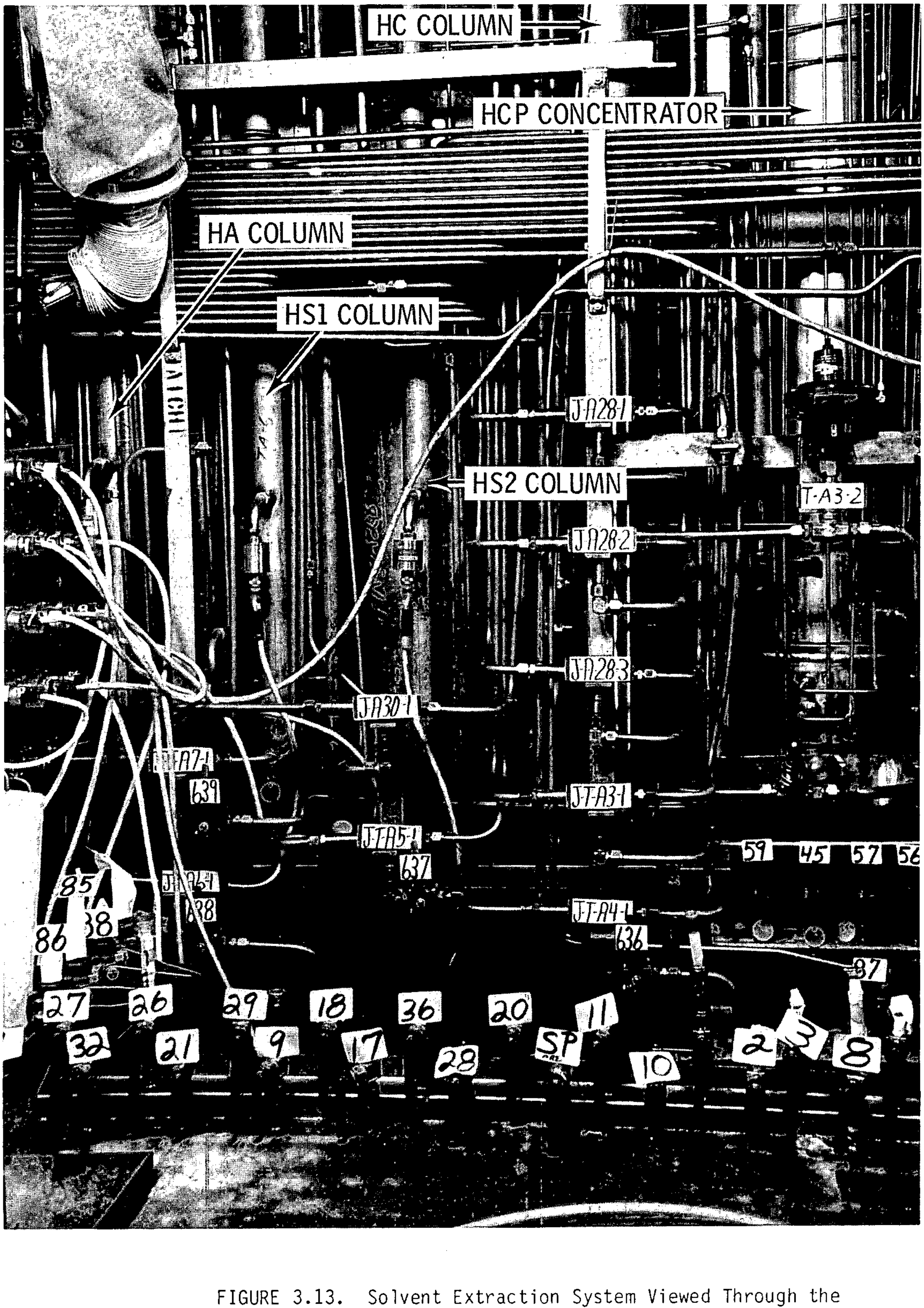

FIGURE 3.13. Solvent Extraction System Viewed Through the North Window of A-Cell, 325-A Building

TABLE 3.1. Pulse Column Design Criteria

Column	Internal Dia (cm)	Height of Plate Section (m)	Plate Spacing (cm)	Plate Hole Size (mm)	Plate Free Area (%)	Pulse Amplitude - (cm)[a]	Pulse Frequency - (cy/min)[a]	No. of Transfer Stages
Extraction (HA)[b]	4.52	3.66	5.1	3.18	23	3.1	75	4
1st Scrub (HS1)[b]	4.52	3.05	5.1	3.18	23	5.1	25	3
2nd Scrub (HS2)[b]	4.52	3.05	5.1	3.18	23	5.1	25	2
Strip (HC)[c]	7.06	3.66	5.1	3.18	23	3.1	60	3

(a) Amplitude and frequency are variable over a very large range.
(b) Continuous phase - organic.
(c) Continuous phase - aqueous.

The dilute product stream from the HC column (HCP) is prepared for the ion-exchange partitioning system by concentrating the solution to 7.5M HNO_3 in the HCP concentrator. The concentrator, fabricated from titanium, was originally designed with a counter-current-flow stripping tower to remove dissolved solvent from the HCP. However, because of restrictive distributor plates within the tower, flooding occurred well below design flow-rates. The feed to the concentrator was, therefore, added directly to the reboiler section (as shown later in Figure 4.7) and the stripping option was abandoned. Steam stripping of the solvent is still possible at flow rates up to 50% of design flow rates, but has not been attempted during the project. The product accumulated from the concentrator is sampled for SNM accountability before being fed to the ion-exchange partitioning system.

Solvent is continually recycled through the system after the degradation products are washed from it in the HO contactor. This contactor, shown in Figure 3.14, is a continuous flow turbo-mixer in which the solvent is contacted with sodium carbonate. The cleaned solvent overflows the turbo-mixer and is then pumped through a 25 μ filter back to the extraction column, with no further treatment.

3.4.3 Ion Exchange Systems

The anion exchange utilizes three operating cycles for each batch of feed solution processed:

1. Feed Cycle - Pu^{IV} is absorbed by the resin from the 7.5M HNO_3 feed solution pumped through the resin bed.
2. Scrub Cycle - Absorbed Pu^{IV} is decontaminated from uranium and fission products when 7.5M HNO_3 is pumped through the resin bed.
3. Product Elution Cycle - Pu^{IV} is removed from the resin when 0.5M HNO_3 is pumped through the bed.

Each system, IX1 installed in A-Cell and IX2 installed in a shielded glove box, includes two resin columns, as well as feed tanks, waste tanks, product tanks, pumps, control valves, and process control instruments. The IX1 system is pictured in Figure 3.14. The shielded glove box, containing the IX2 system is shown in Figure 3.15. In each system, the primary column is sized to contain 450 g plutonium per batch, approximately the amount of

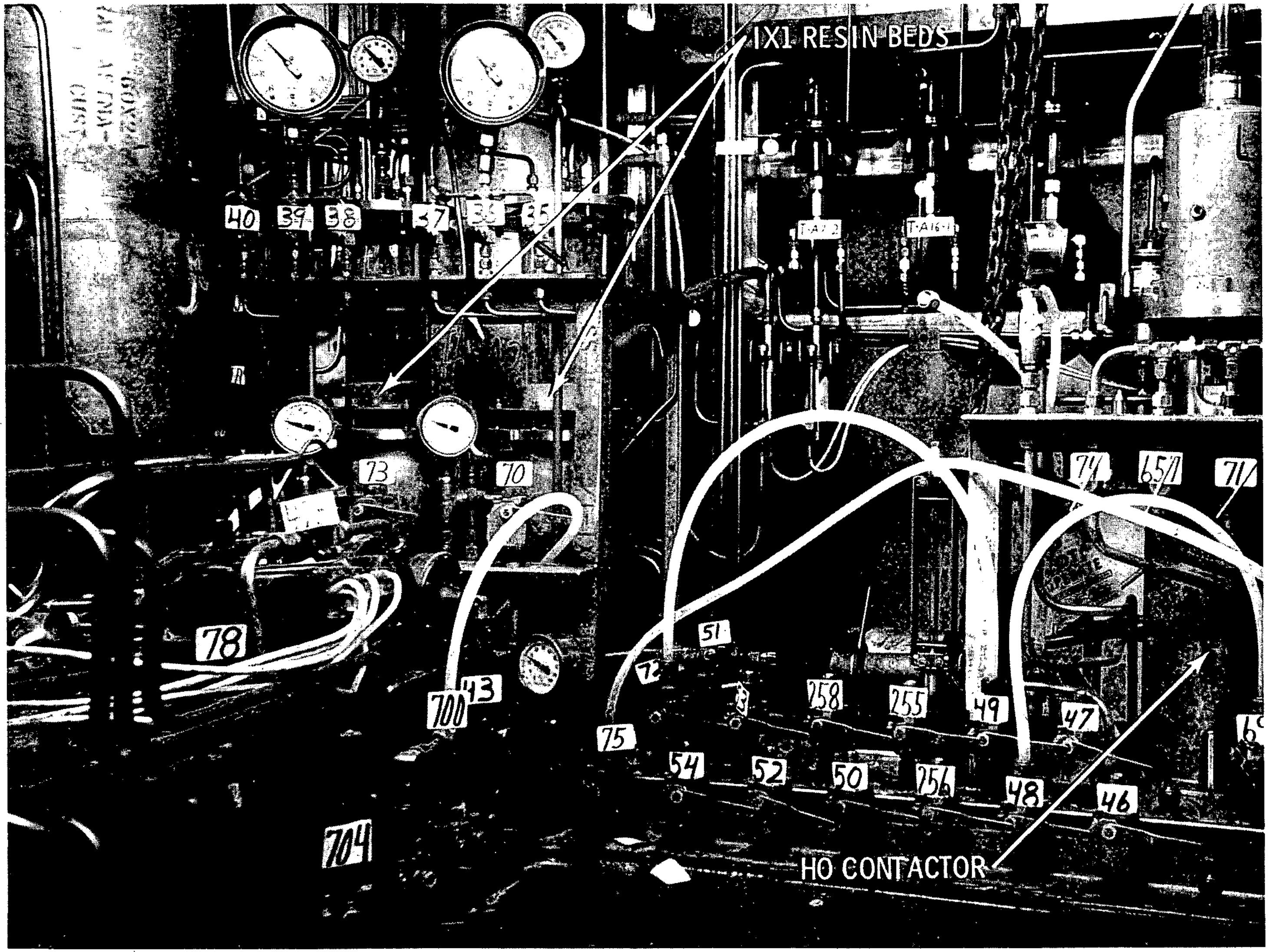

FIGURE 3.14. Ion-Exchange and Solvent Treatment Systems Viewed Through the South Window of A-Cell, 325-A Building

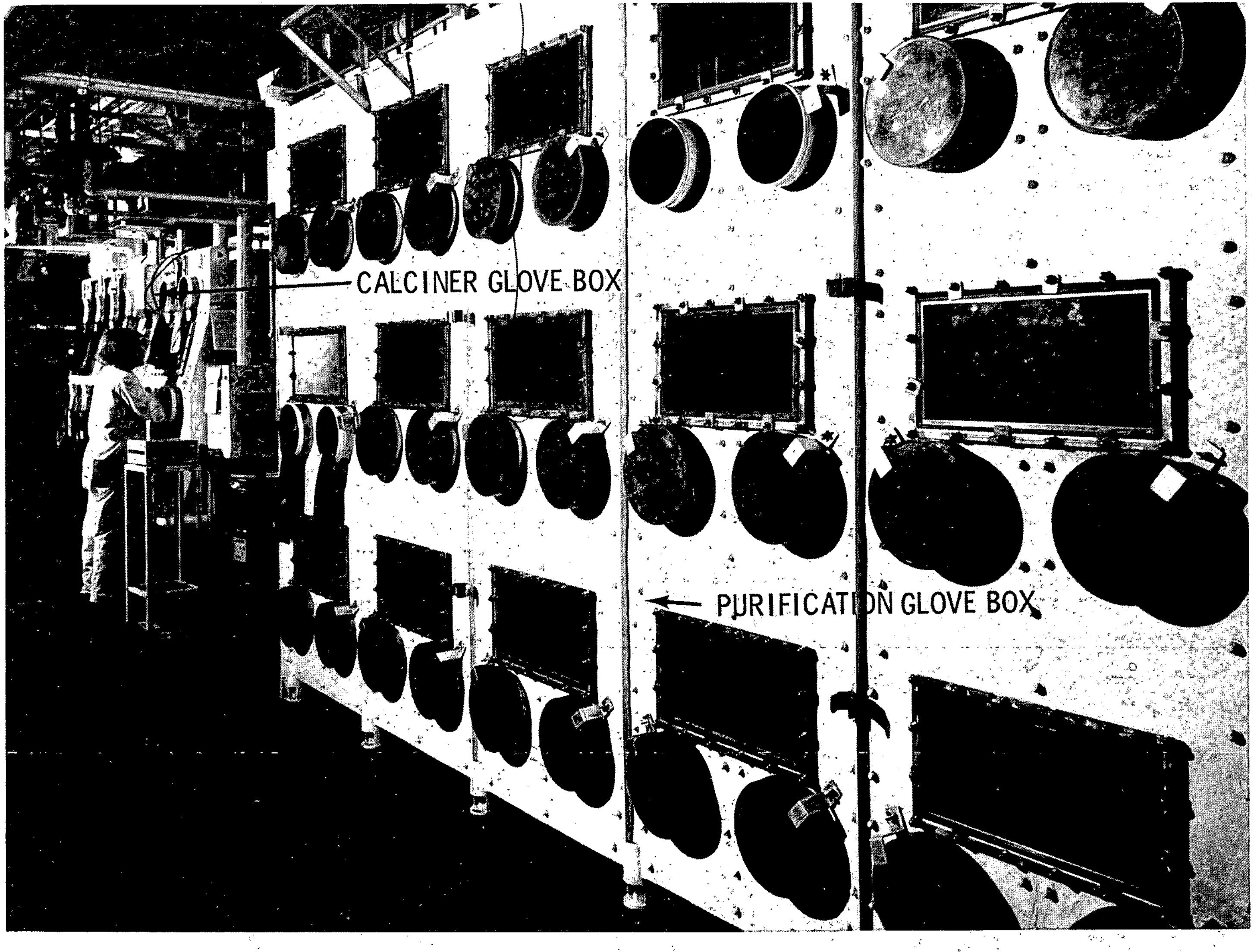

FIGURE 3.15. Lead-Shielded Glove Boxes Containing the IX2 Ion-Exchange Purification System and the Screw Calciner

plutonium contained in 50 kg of dissolved fuel. The second column is used as a backup, or tailing column. The resin columns are water-jacketed for operation at 60°C, but in operation only the primary column is heated. Each resin column in the IX1 system is 10.2-cm (4-in.) ID and contains 10.5 L of Dowex® 1, X-4 (50-100 mesh) resin. The IX2 columns have the same diameter and contain 8.0 L of the same resin.

The solutions exiting the columns during the feed and scrub cycles are sampled and analyzed for plutonium, then are reworked or discarded as ILW. The plutonium by-product stream is transferred from the IX1 system to the IX2 system or directly to the plutonium calciner. A plutonium concentrator is available to reduce the volume of feed to the calciner, if necessary.

3.4.4 Plutonium Calciner

The plutonium-nitrate product from the ion-exchange system is directly calcined to oxide in a continuous-feed screw calciner installed in the shielded glove box shown in Figure 3.15. The screw, trough, and cover plate are fabricated from titantium for minimal corrosion. The unit is designed to operate at 450 to 550°C, with a feed flowrate of 40 to 50 ml/min (1.5 g Pu/min). The 5.1-cm- (2-in.-) dia trough is 122 cm (48 in.) long. The temperature is monitored by six thermocouples bolted to the bottom of the trough and spaced at fixed distances, ranging from 15 to 25 cm (6 to 10 in.) apart. The plutonium-nitrate nitric-acid stream is fed directly to the trough at the head end of the calciner and then forced down the trough by the screw. The unit is heated by two banks of resistance heaters. These heaters are automatically controlled at a selected temperature for the product end. Air is pulled countercurrently to the plutonium flow to aid in calcination and to entrain the moisture, NO_x, and CO_2 that are driven off during the process. The off-gas stream is pulled through a sintered metal filter, a condenser, and a caustic scrub pot before it enters the normal glove-box vacuum system. The oxide product leaving the calciner is collected in a stainless steel container, cooled, weighed, and then stored in slip-lid metal cans with the lid taped in place. Accumulated batches of approximately 2.5 kg are homogenized in a V-shell blender, sampled for chemical analyses, reweighed, then stored in tape-sealed cans according to plutonium-handling guidelines. This product from the calciner represents the final SNM accountability point in the process.

3.4.5 Off-gas System

The off-gas system for the 325-A Building provides initial processing of the vessel off gases and the dissolver off gas when the backup dissolvers are in use. The gases exiting the 325 Building system are pulled to the 324 Building for further processing before they are released out the stack. The major constituent of the vessel off gas in the 325-A Building is air used in sparging, airlifting, and purging of the steam jets. The system is designed to process 0.71 m^3/min. (25 scfm) of vessel off gas.

The functions of the system are to: 1) reduce the tritiated water content to a point where no more than 15 Ci of tritium per week are released to the 324 Building system, 2) remove radio-iodine, and 3) remove particulates. The equipment used for these functions

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are a packed-tower water scrubber for tritium removal, a silver zeolite bed for iodine removal, and a high-efficiency particulate air (HEPA) filter for removal of particulates. (This equipment is pictured in Figure 3.12.)

A vacuum is applied to the system by an air jet in 324 Building, the off gas is pulled through an annulus in the interbuilding pipeline and released into B-Cell in the 324 Building, where it is subsequently processed through the 324 Building ventilation system.

The majority of the tritium and iodine are evolved during dissolution; thus, the 325-A Building off-gas system is designed under the assumption that the back-up dissolvers in C-Cell will be in use. Therefore, during normal operation, where the dissolution is accomplished in the 324 Building, the off gas from the 325-A Building that is drawn through the pipeline contains minimal contamination.

3.4.6 Intermediate-Level Waste System

The uranium-bearing streams from the ion-exchange system, along with the streams from the off-gas scrubber, the solvent-wash contactor, and the calciner off-gas condenser, are accumulated continuously in TK-A10, then batch-transferred to TK-A11. The transferred solution is sampled for SNM accountability, then discarded as ILW by one of two routes: 1) direct vacuum transfer to a 1900 L (500 gal) shielded transfer cask positioned in the Cask Transfer Hood (CTH), or 2) jet transfer to vault tank PT-2, followed by vacuum transfer at a later time to a 1900 L (500 gal) cask in the CTH. Two identical casks are used in rotation to transfer all ILW by truck from the 325-A Building to the 200 East Area for disposal by Rockwell Hanford Operations.

3.4.7 Process Instrumentation

There are two major categories of instrumentation associated with the waste preparation facility: 1) process monitoring and control, and 2) analytical measurements.

Process Monitoring and Control Instrumentation

The major functions of the process monitoring and control system are to record and control liquid flows, levels and densities; temperatures; and pressures; as well as to provide an historical data bank of these parameters.

Most in-cell batches of solution are transferred by steam jets, actuated at the control panels. Programmable controllers are used on each jet-gang valve system to cycle the solenoid valves between steam, air purge, and vent during and after each transfer.

Continuous process flows within the cells are controlled by air lifts or air-driven pumps. The air-flow rate to each air lift is monitored with an electronic rotameter and is automatically controlled by an air-operated valve. Each air lift is calibrated before it is operated to determine liquid-flow rate as a function of air-flow rate. During operations, tank drop-out rates are periodically measured to check these calibrations. Two air-driven centrifugal pumps are used in the cells. One is used to pump solvent to the bottom of the extraction column and the other to recycle solution to the top of the off-gas scrubber. Flow rates from both of these pumps are monitored with electronic rotameters and are controlled by air-operated valves.

Level and density are measured by bubbler tubes and differential pressure transducers. Levels and densities for each tank are recorded on strip charts on the control panels and are connected to an audible alarm system to signal when normal operating ranges are exceeded.

Temperatures are measured with resistance temperature detectors (RTDs) that can be remotely removed and replaced, if necessary. Pressures are measured in the same manner as densities, by dip tubes and differential pressure transducers.

In addition to the strip-chart records, records of all process parameters are kept in the data acquisition system (DAS). This computer system records all of the process parameters every ten seconds and stores the information on magnetic tape. Any parameter that is not within present limits will trigger an audible and visible alarm at the appropriate control panel. A mini-computer coupled to the system allows many automatic routine calculations and data searches.

Analytical Measurements

Most analytical measurements are made on process samples by the HEDL analytical laboratory; however, a few analyses are made in the cells and glove boxes. The in-process instrumentation includes two high-resolution gamma spectrometers, three alpha monitors, and a high-accuracy load cell for weighing the calcined oxide.

The high-resolution spectrometers are used to monitor the plutonium and gamma-emitting impurities in the effluent and product streams from the two ion-exchange systems.

Two of the alpha monitors are used to measure the plutonium content in the IX1 feed waste and scrub waste solutions. Since these solutions are transferred to tanks that are not critically safe by geometry, the monitors are calibrated to set off an alarm if more than 0.2 g/L of plutonium is present. The third alpha monitor was designed to indicate high uranium or plutonium concentrations in the organic stream leaving the strip column (HCW). However, early in operations, that flow-through monitor was blocked by an O-ring that expanded because of a reaction with the organic solvent. Therefore, no information has ever been obtained from this monitor.

The high-accuracy load cell is used as the final accountability point for plutonium.

3.5 INTERBUILDING PIPELINE

A plot plan of the 324-325 Building pipeline is shown in Figure 3.16. During a construction project several years before the inception of NWVP, the major straight section of the line, approximately 198 m (650 ft), was placed underground but not connected on either end. This section consisted of a 2-in. Schedule 40, 304L stainless-steel, welded pipe, encased within a 4-in. Fiberglas-reinforced epoxy pipe. During NWVP construction, the original 2-in. line was tested for cross-sectional clearance and then was given a pressure test. Two 304L stainless-steel, all-welded lines, a 3/8-in. Schedule 40 pipe, and a 3/4-in. Schedule 40 pipe were assembled, tested, and drawn through the buried 2-in. line. The system was then extended into each building, as shown in Figure 3.17. The pipeline runs at depths ranging from 1 to 4 m (3 to 12 ft) underground. The route of the pipeline is marked at the ground surface and is totally within the exclusion area to

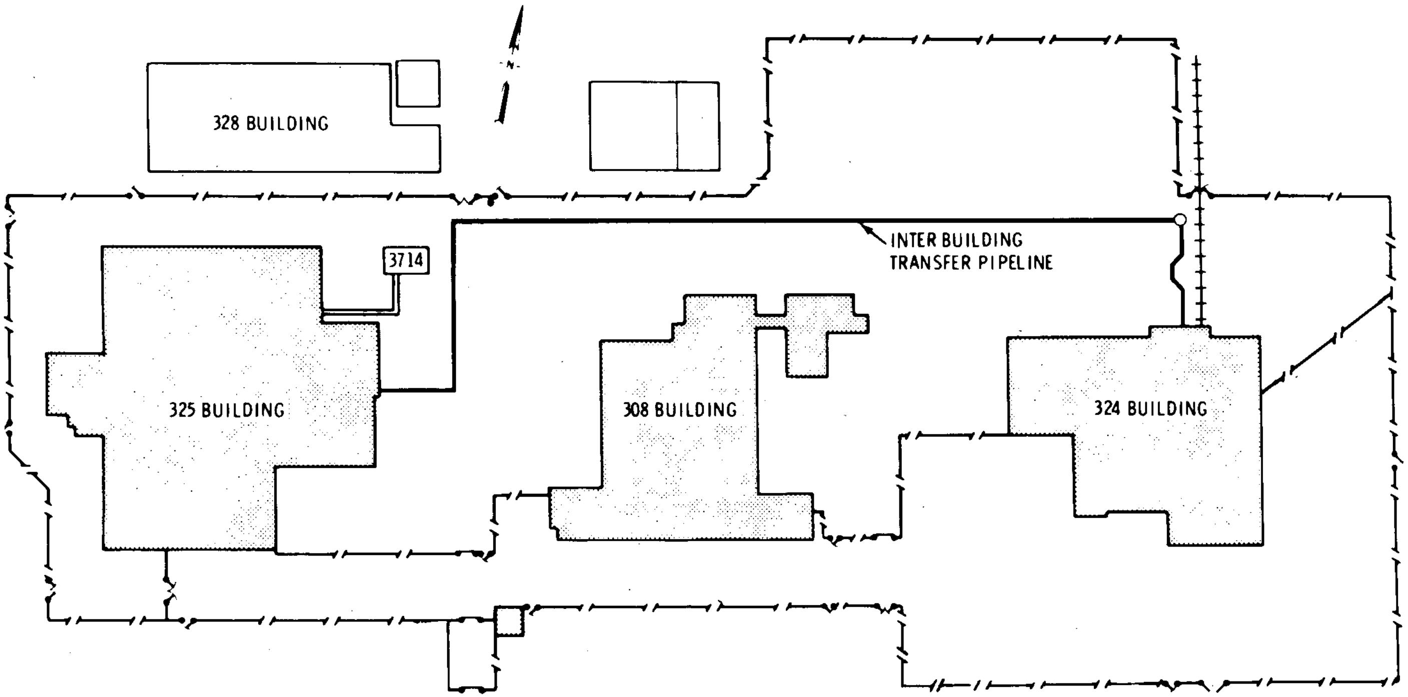


FIGURE 3.16. Plot Plan for Buildings 324 and 325

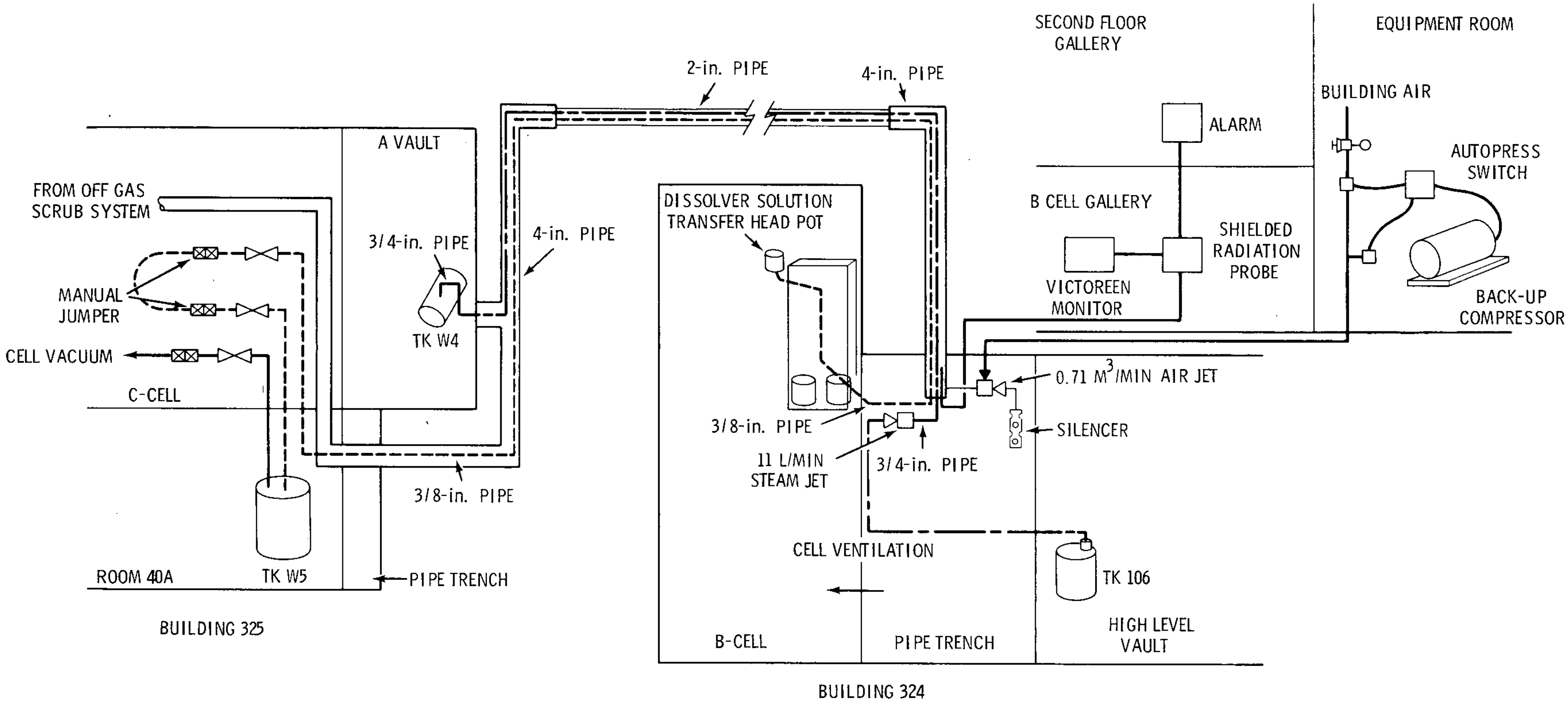


FIGURE 3.17. Interbuilding Transfer Pipeline System

prevent accidental excavation and to eliminate the potential for even a low exposure to personnel during transfer of radioactive solutions.

The 3/8-in. line connects the dissolver-solution transfer head pot in B-Cell of 324 Building with a block valve in C-Cell of 325-A Building. A polyethylene jumper, in C-Cell, is used to connect the block valve with a line extending to TK-W5 located in a vault below C-Cell. Tank TK-W5 serves as a vacuum tank for receipt of dissolver solution from 324 Building. The transfer of dissolver solution through the 3/8-in. pipeline is initiated by: 1) turning on the air lift on the line from TK-126 (the dissolver solution storage tank) to the dissolver solution transfer pot, 2) applying vacuum to TK-W5, and 3) opening the block valves in C-Cell. The vacuum applied to TK-W5 is controlled by use of an air-bleed tank. Constant communication between the two buildings is maintained by telephone during any transfer. A 25 L flush of 2.5M nitric acid, approximately the volume of the 3/8 in. pipeline, is drawn through the line after each transfer of dissolver solution to free the line from suspended solids and concentrated uranyl nitrate.

The 3/4-in. pipeline connects TK-W4 in A-Vault of 325-A Building with TK-106 in the 324 Building High-Level Vault. The solution is transferred by a steam jet in the 324 Building Pipe Trench. For NWVP purposes, this line is used to transfer HLLW from 325-A Building to 324 Building.

The encasement surrounding the two lines used for solution transfer has two functions: 1) secondary containment for the two smaller lines, and 2) a route for transfer of the process off gas from 325-A to 324 Building for discharge from the much higher stack. A 0.71 m^3/min (25 scfm) air jet in the 324 Building B-Cell Pipe Trench transfers the off gas between buildings. Exhaust gas from the jet is discharged to the pipe trench, where it flows into B-Cell and is treated with the B-Cell ventilation exhaust. A backup air compressor ensures uninterrupted flow of the exhaust gas from 325-A Building (as shown in Figure 3.17). The beta-gamma, radiation-monitoring system continuously monitors the gas stream drawn through the interbuilding pipeline to detect any leak in the 3/8-in. or 3/4-in. lines (also shown in Figure 3.17).

4.0 OPERATIONS AND PERFORMANCE OF FACILITIES

4.1 STAFFING

4.1.1 Process Engineering

A multidisciplinary Process Technology group was organized at the start of conceptual design and maintained until the end of the project. The level of effort varied with the work load, but included a dozen engineers or scientists working full time on the project, an additional dozen working only part time, and four non-PNL engineers who consulted with Process Technology when needed. The Process Technology staff were mostly senior people with considerable experience in their respective specialties and with the types of facilities to be designed, constructed and operated. In addition to their contributions during design and construction, Process Technology provided Operations with technical directions and approvals for all phases of cold testing and final hot operations.

Process Technology provided Operations with detailed Run Books,(a) reviewed and approved by project management, that contained operational procedures and specified process flowsheet conditions. Authority was granted to Process Technology to make flowsheet changes within the bounds of the Criticality Safety Specifications, the Process and Environmental Specifications, and to give detailed technical directions to Operations for day-to-day operations by a formalized Process Memo procedure. Concurrence of Operations was required for these changes.

4.1.2 Process Operations

Staff Requirements

Because of the increased complexity and production nature of the NWVP, compared with previous work conducted in the same buildings, it became necessary to recruit and train a much larger operations staff, most of whom had had no similar previous experience. The 325-A Building was operated 24 hours/day, 7 days/week (A-B-C-D shifts), with supplemental personnel on day shift. The 324 Building was also operated 24 hours/day, but only 5 days/week (X-Y-Z shift), with supplemental personnel on day shift. Table 4.1 lists the operating staff requirements.

Staff Training

Because of the relative inexperience of the operating staff, a comprehensive training program was established that included a four-week formal course of study, about 12 weeks of hands-on training in the operation and functioning of the process equipment, and qualification testing.

(a) 1. NWVP Task 5 Run Book (for 324 Building)--W. J. Bjorklund, M. S. Hanson, L. S. Romero, F. E. Haun.
2. NWVP Task 6 Run Book (for 324 Building)--W. J. Bjorklund, L. K. Holton, D. H. Siemens, D. N. Berger, T. A. Golding.
3. Run Plans, Waste Preparation Task, 325-A Building--E. R. Irish, E. J. Wheelwright.

TABLE 4.1. Operating Staff Requirements

	Totals
Operations Manager	1
SNM Specialist	1
Secretary	1
Security Clerk	1
Shift Requirements	
324 Building	
X-Y-Z Shifts	
1 Operating Specialist	3
1 Working Leader	3
4 Technicians	12
Day Shift Only	
Technicians	4
325-A Building	
A-B-C-D Shifts	
1 Operations Engineer	4
1 Lead Technician	4
4 Technicians	16
Day Shift Only	
Senior Operations Engineer	1
Operations Specialist	1
Technicians	4
	56

During the four-week course, all technicians were given generalized training in the areas of:

- NWVP orientation
- mathematics
- chemistry
- physics
- radiation safety
- radioactive materials handling
- nuclear safeguards and accountability
- criticality control.

They were given specialized training in the operation of specific equipment in the buildings to which they were assigned:

324 Building	325-A Building
Fuel handling and disassembly	Backup dissolvers
Fuel shearing and dissolution	Solvent-extraction system
Evaporator-fractionator	Ion-exchange system
Spray calciner/in-can melter	Screw calciner
Process off gas	Dissolver solution transfer and accountability system
	General glove-box operation
	Chemical makeup
	Off-gas system
	ILW and HLW systems

The hands-on training was initiated as soon as construction was completed on each operating system and was the most significant aspect of the staff training program. This training included practice of procedures needed for the safe operation and control of manipulators and cranes, sampling equipment, and the process operating systems previously enumerated.

Procedures

Acceptance Test Procedures (ATPs) were prepared for 325-A Building instrument systems to demonstrate that the equipment was properly installed. The ATP approval parties were PNL, who also acted as the test coordinator, and Vitro Engineering, who acted as a witness and was responsible for recording the test results. The construction contractor was responsible for the organization and performance of the ATPs and provided all equipment for the tests.

In the 324 Building, all previously used equipment and all new equipment were tested to some degree before installation and/or operation. The results of each test were documented and placed in the QA file. Up to four tests were performed on each piece of equipment or system, as follows:

1. Dimensional Check - size, fit, clearance, load check, etc.
2. Operational Test - cold functional test of process equipment
3. Acceptance Test Procedure - performed by construction contractor, before turnover to Operations Section
4. Design Verification Tests - detailed verification of components under actual operations of in-cell equipment.

The Operations Section of the NWVP wrote Operational Acceptance Test Procedures (OATPs) for 325-A Building equipment, which were used to verify that all equipment was properly installed and that it functioned in accordance with design specifications. All process lines were checked to be sure they were properly routed; all tanks were calibrated. All in-cell cranes, filters, pumps, jets, agitators, valves and other transfer systems (vacuum and pressure) were checked for proper operation. Cooling coils, steam coils, and hot water systems were checked for their ability to meet design requirements, and each system (i.e., concentrator, ion exchange, dissolver, etc.) was evaluated under simulated operating conditions.

Safe Operating Procedures (SOPs) were written by the Operations Section for each operation and piece of equipment required for the NWVP process. Each SOP required the approval of the Operations Section, Safety and Nuclear Materials Management (NMM), PNL Building Manager, Process Technology, Radiation Monitoring, and Quality Assurance.

Radiation Work Procedures (RWPs) were prepared by the Radiation Monitoring Section to cover all NWVP operations in the 324 and 325-A Buildings.

4.1.3 Support Services

Radiation Monitoring

Complete radiation monitoring was provided by a supervisor and nine radiation monitors assigned as follows:

1 Radiation Monitoring Supervisor
1 Radiation Protection Engineer

324 Building Radiation Monitors		325-A Building Radiation Monitors	
X-Y-Z Shifts	3	A-B-C-D Shifts	4
Day Shift	1	Day Shift	1

Craft Services

Craft Services were assigned to day-shift during operation of the NWVP and were called out as necessary on weekends and at night. The actual craftsmen assigned to the NWVP are listed below:

- 2 Foremen
- 5 Instrument Technicians
- 4 Pipefitters
- 5 Millwrights
- 3 Electricians.

Analytical Services

Analytical services were provided by Westinghouse (HEDL) Analytical Services, located in the 325 Building.

Analyses for routine process control and accountability were provided on an around-the-clock basis by the following full-time staff:

- 1 Technical Leader
- 1 Chemist
- 4 Analysts (A-B-C-D Shift)
- 1 Analyst (Day Shift).

In addition, many analyses required special equipment and personnel skills, such as mass spectrometry and amperometry. Five technical specialists were used on an as-needed basis to perform such analyses.

4.2 WASTE PREPARATION

4.2.1 324 Building Operations

Spent-Fuel Acquisition and Storage

Six PWR fuel assemblies, discharged from the Point Beach Reactor, were obtained for waste preparation processing. Table 4.2 summarizes the irradiation characteristics of the fuel assemblies.

TABLE 4.2. Fuel Assembly Data

			Pre Irradiation		Post Irradiation		
Assembly No.(a)	Discharge Date	Burnup (MWd/tU)	^{235}U (wt%)	U (kg/assembly)	^{235}U (wt%)	U (kg/assembly)	Pu (g/assembly)
A15	10-10-72	20,600	2.27	398.1	0.84	386.6	2850
A16	10-10-72	20,600	2.27	398.1	0.84	386.6	2850
A27	10-10-72	20,100	2.27	398.1	0.86	386.8	2820
B01	05-10-74	29,500	3.04	389.2	0.94	373.7	3360
B24	05-10-74	29,500	3.04	389.2	0.94	373.7	3360
B25	05-10-74	29,600	2.99	389.2	0.93	373.7	3370

(a) Zircaloy cladding, Point Beach PWR 14x14 (196 rods).

The spent fuel assemblies were received and transferred from the shipping cask to the storage rack in B-Cell without incident. Photographs of these steps are shown in Figures 4.1 through 4.4. The average time required to complete these fuel-handling operations for each of the six shipments was 14 hours. All six assemblies were received and placed in the storage rack before shearing and dissolving were started. Water was added to the basin as needed, but cooling water was not connected to the basin cooling coils.

Fuel Disassembly and Shearing

The disassembly operations were performed as planned, except the use of a circular saw was required at times in addition to the internal tubing cutter, to remove the top nozzle plate of the assemblies. Several of the PWR fuel rods contained collapsed sections along the rod. These collapsed sections were removed with an external tubing cutter and manually (with manipulators) placed in the shear chute. The noncollapsed sections were sheared automatically with the feeder-puller/shear equipment.

Fuel rods were pulled from the assembly and fed into the feeder-puller with a manipulator. In some cases a single-rod puller, attached to the fuel rod and the feeder-puller, was used to free tightly bound rods. The feeder-puller/shear mechanism worked automatically and reliably. The hydraulic hoses to the shear and a cam pin on the feeder-puller mechanism were replaced, the only maintenance performed on the system. Average time to shear a batch of 40 to 42 rods was eight hours.

The three fuel assemblies with a low burnup were disassembled and sheared first, followed by the three assemblies with a higher burnup.

Fuel Dissolution

A process flowsheet and material balance for the 324 Building dissolver is shown in Figure 4.5. During a normal batch operation, 40 to 42 rods of spent fuel, representing an average of 86.4 kg uranium, were sheared and dissolved. Twenty-six separate dissolver batches were required to process the six PWR fuel assemblies.

Spent fuel was sheared into the dissolver basket located inside the dissolver vessel (see Figures 3.5 and 3.6). Before shearing operations began, the dissolver was filled with

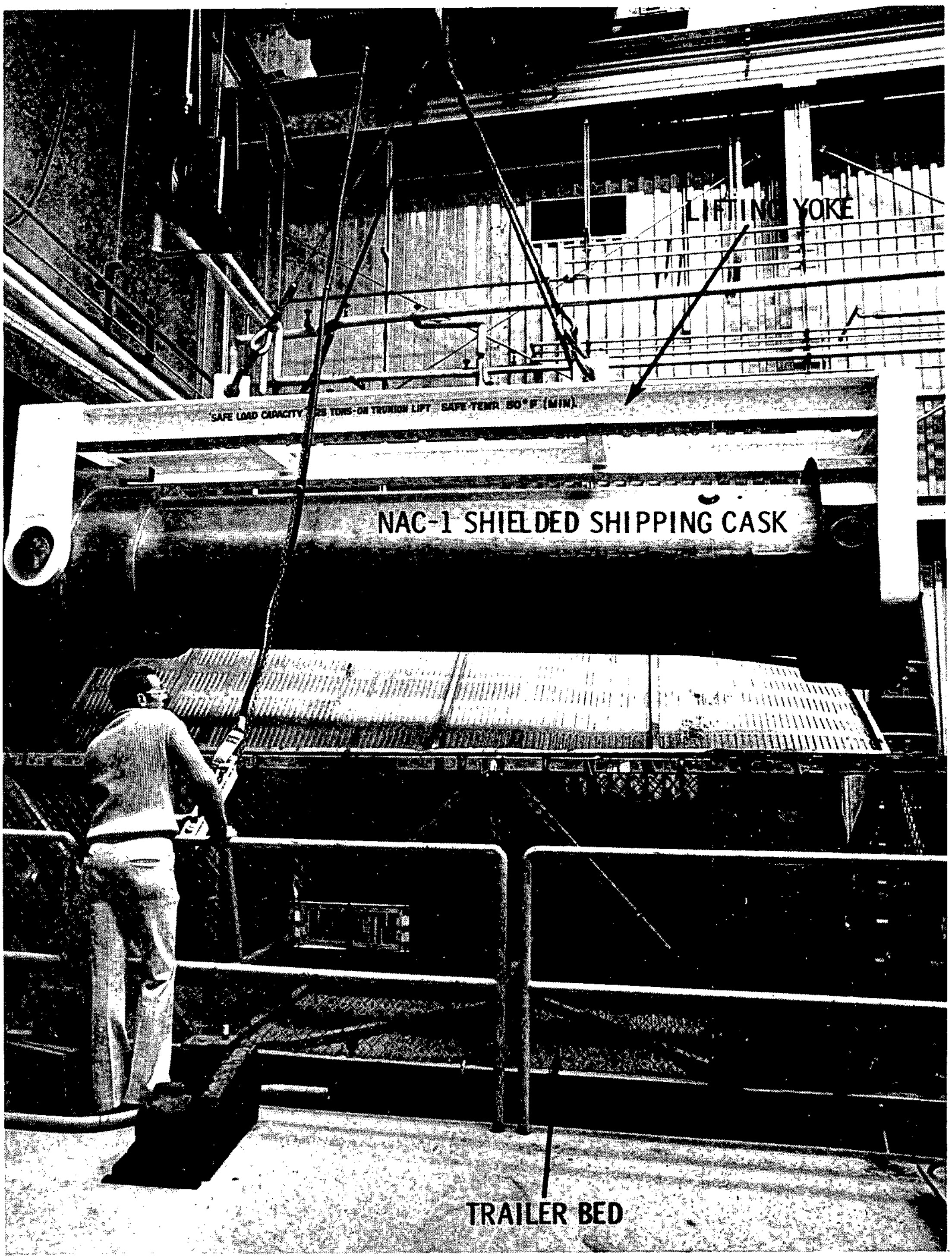


FIGURE 4.1. Removal of an NAC-1 Shielded Shipping Cask from a Tractor-Trailer in the 324-Building Truck Lock

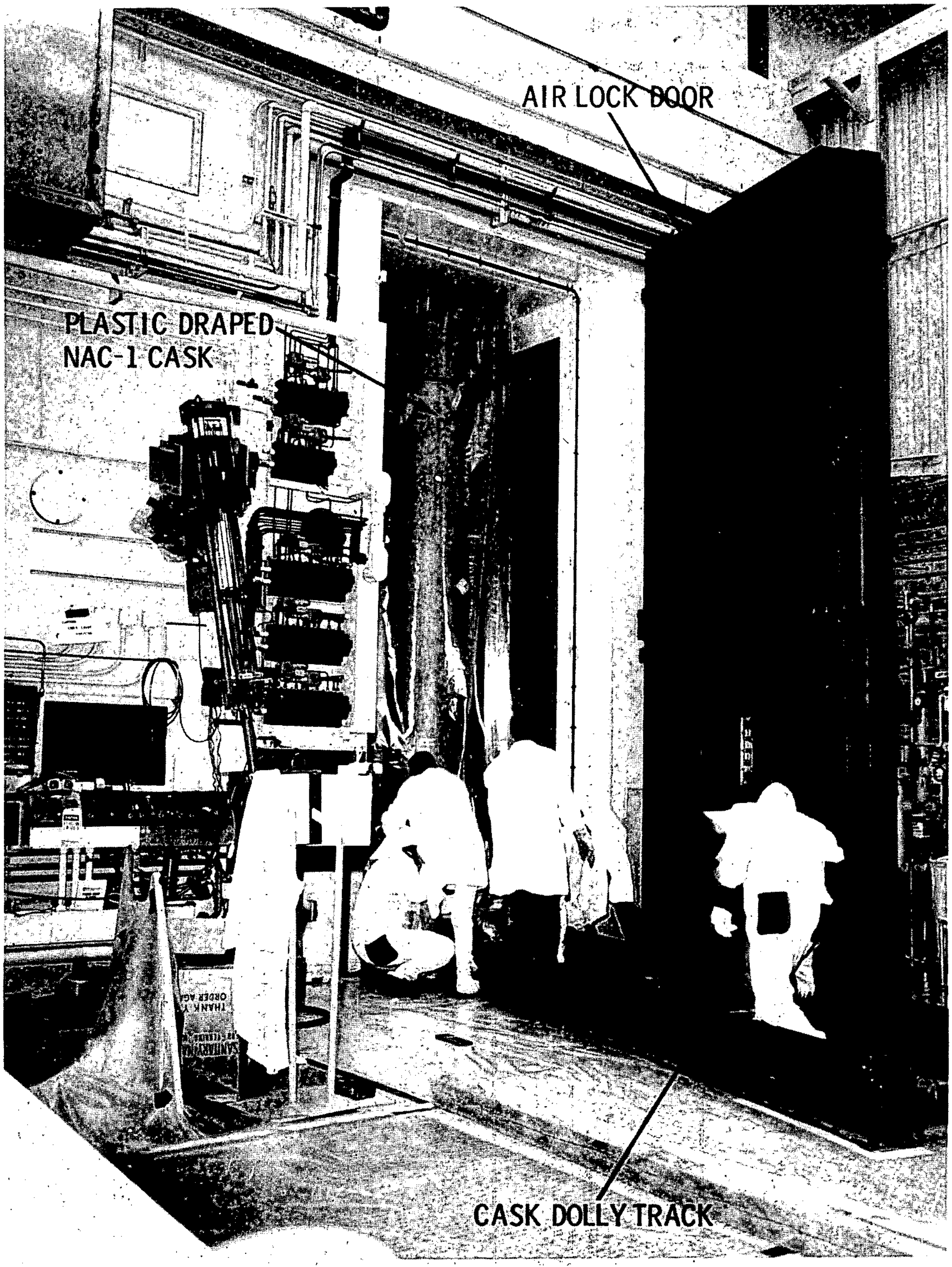


FIGURE 4.2. Transfer of Plastic Draped NAC-1 Cask into the Air Lock Using Track-Mounted Cask Dolly

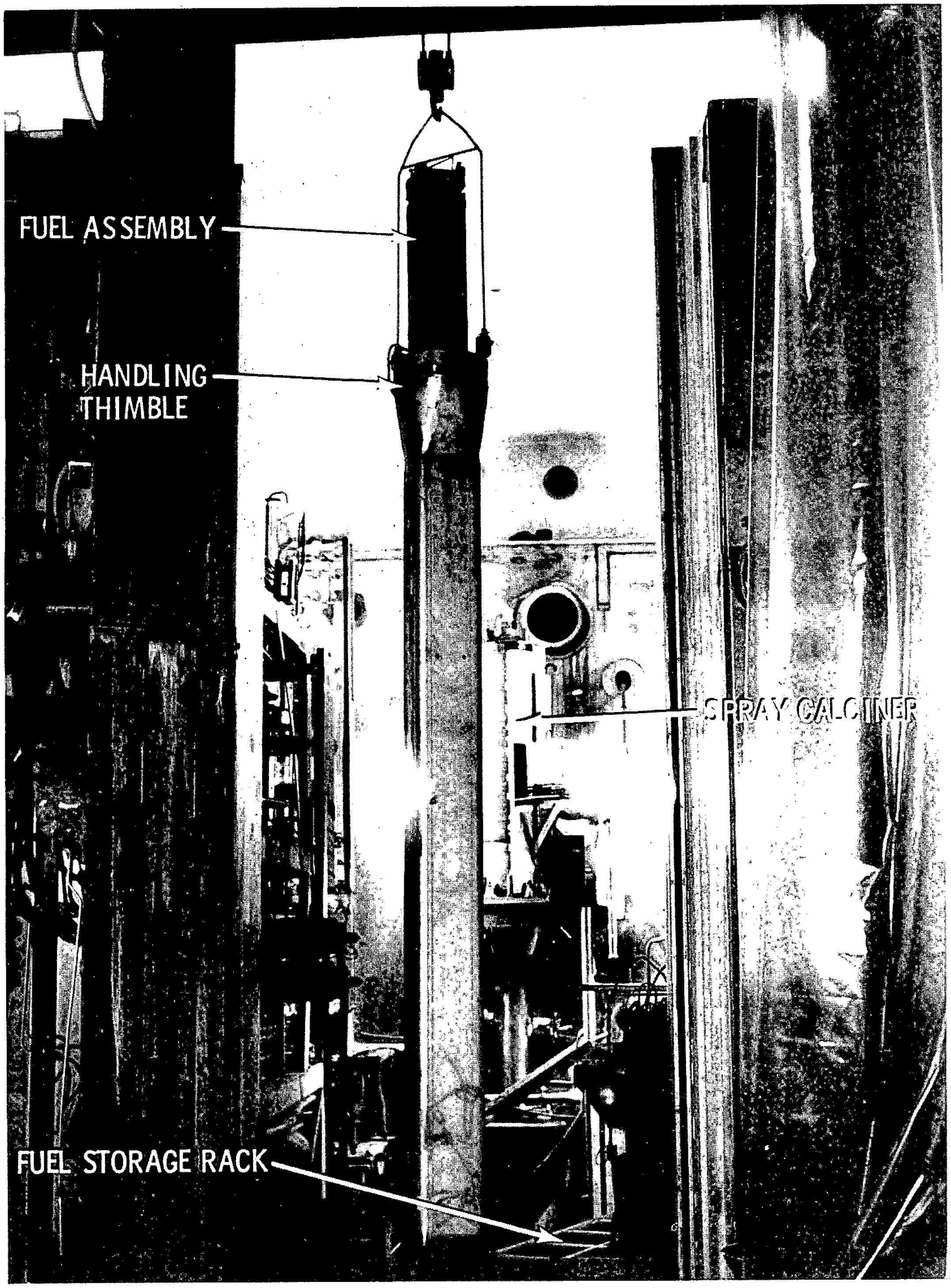


FIGURE 4.3. Transfer of a PWR Fuel Assembly from the Air Lock into B-Cell for Storage - Photographed Through Air Lock Window with B-Cell Door Open

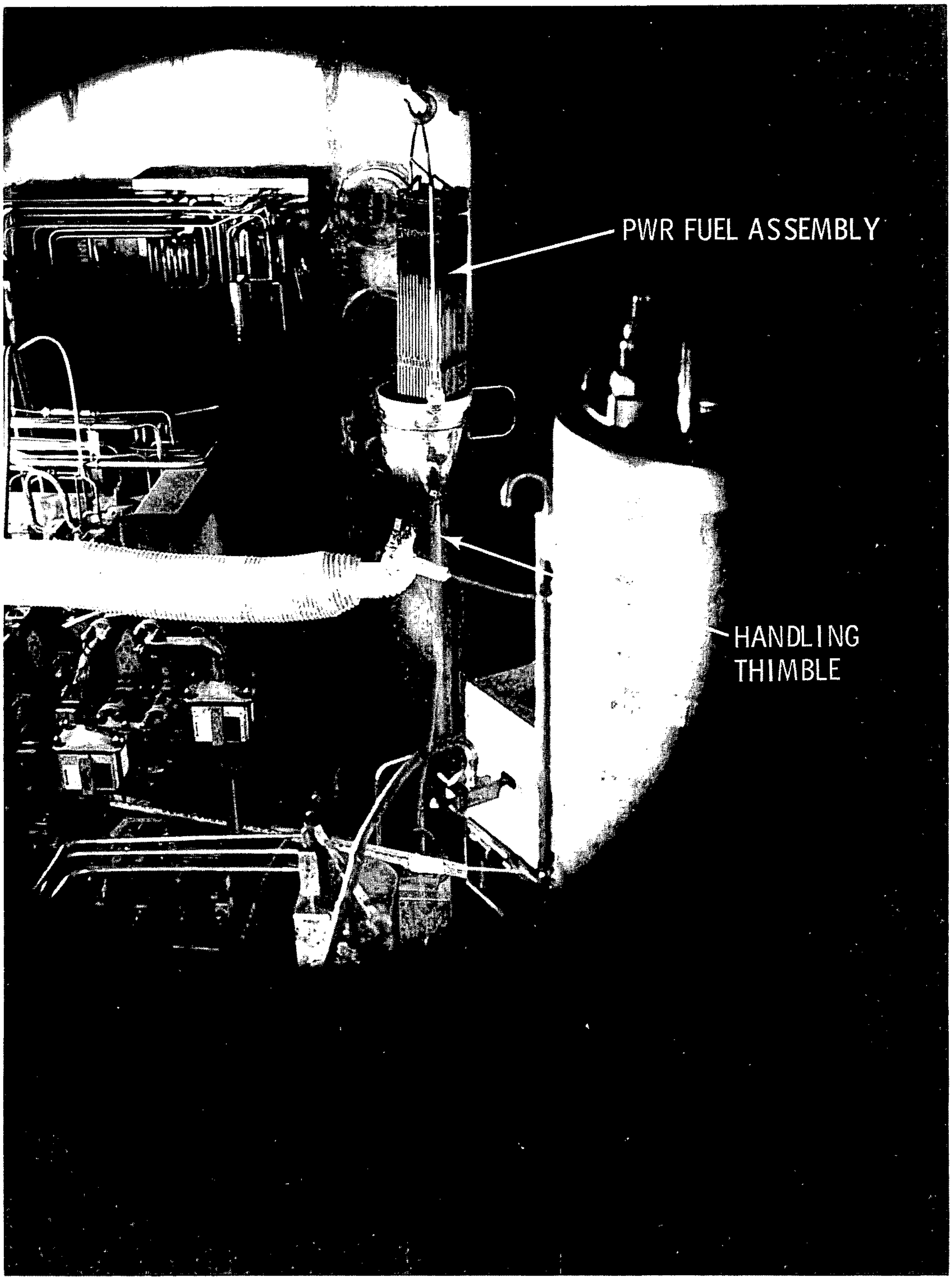
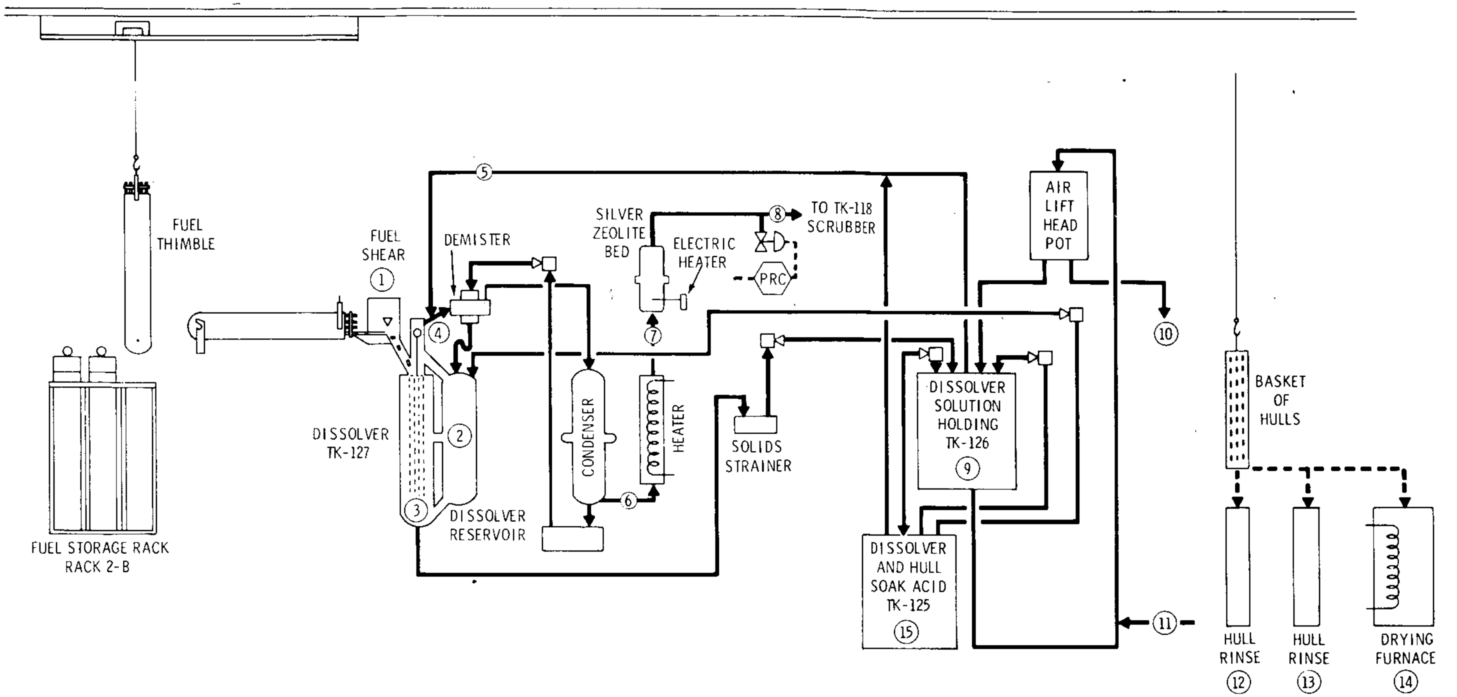


FIGURE 4.4. Transfer of a PWR Fuel Assembly from the Air Lock into B-Cell for Storage - Photographed from Cell Window



	FUEL CHARGE	DISSOLVER	AIR SPARGE	DOG (MAX)	VOG (MAX)	OFF GAS FROM CONDENSER	OFF GAS TO SILVER ZEOLITE	OFF GAS TO SCRUBBER	DISSOLVER PRODUCT	DISSOLVER SOLUTION TO 325 BUILDING	AIR LIFT SUPPLY	#1 HULL RINSE	#2 HULL RINSE	HULL DRYING FURNACE	ACID HOLDING TANK
IDENTIFICATION NUMBER	①	②	③	④	⑤	⑥	⑦	⑧	⑨	⑩	⑪	⑫	⑬	⑭	⑮
URANIUM (kg)	86.4														
URANIUM CONCENTRATE (g/L)		363							323	287					
TEMPERATURE (°C)		95		95	50	60	130	50	50			30	30	150	30
GAGE PRESSURE (mm Hg)		-10		-10				-20							
GAS FLOW (m^3/min)	0.03[a]	0.06-0.28	0.14-0.28	0.57	0.28[b]	0.85	0.85	0.85[c]	0.11		0.06				0.11
VOLUME (L)		238							267	300[d]		60	60		300
HNO_3 (M)		7.3 (INITIAL) 2.5 (FINAL)							2.2	2.2					7.3
DENSITY, @ 25°C (g/cm^3)		1.56							1.50	1.45		1.01	1.00		1.23

[a] INLEAKAGE DURING SHEARING

[b] INCLUDES VARIOUS SPARGES, AIRLIFT GAS ETC

[c] CONTROLLED INLEAKAGE FOR VACUUM ADJUSTMENT NOT INCLUDED

[d] INCLUDES ~33 L TOTAL DILUTION FROM FLUSHES

FIGURE 4.5. Process Flow Diagram of the 324 B-Cell Dissolver System

200 L of 7.3$\underline{M}$ nitric acid. After shearing operations, the dissolver acid volume was increased to 238 L by an addition of 7.3$\underline{M}$ nitric acid. The solution in the dissolver was then slowly heated to 90 to 95°C and held at that temperature. Dissolution was considered complete when the specific gravity of the dissolver solution did not change over a period of at least one hour. The dissolution was completed about four hours after the start of dissolver vessel heatup. Approximately 40% of the dissolution was completed during the shearing process before the temperature of the dissolver was increased.

Acid consumption was determined to be between 2.3 and 2.7 moles of nitric acid per mole of uranium dioxide. Average acid consumption was 2.5 moles of nitric acid per mole uranium dioxide.

The dissolver solution was cooled and transferred to the dissolver solution holding tank after dissolution was complete. The fuel hulls remaining in the dissolver basket inside the dissolver were then contacted with 200 L of 7.3$\underline{M}$ nitric acid. The hulls were leached at a temperature of 90 to 95°C for two hours. This hulls leach step ensured that no undissolved fuel remained in the fuel hulls. The hulls leach acid was used for dissolution acid in the next dissolver batch.

After cooldown, the basket containing the hulls was removed from the dissolver vessel and transferred to the hulls rinse tanks. The basket of hulls was contacted with water in two separate rinse tanks and allowed to drip dry; it was then transferred to a furnace and further dried.

Dissolver Solution Filtration

Dissolver solution was jet-transferred from the dissolver vessel (TK-127) to the dissolver solution holding tank (TK-126) after dissolution was complete. During this transfer, the dissolver solution was filtered through a 100 µ stainless steel filter. Undissolved solids were allowed to collect inside the filter housing.

Two filters were used for the transfer of 26 batches of dissolver solution. After transfer of 13 batches, the first filter was removed from the system and filled with cement for disposal even though the pressure drop through the filter had not exceeded the design criteria of 64 cm of water. After completion of the project, the second filter was removed and stored in water for possible future characterization of dissolver-solution solids.

When the dissolver solution was transferred to 325-A Building, the solution in the holding tank (TK-126) was continuously cycled by airlift to the transfer head pot. The dissolver solution was then vacuum-transferred from the head pot to 325-A Building through the 3/8-in. interbuilding pipeline.

Storage of Leached Hulls

The leached spent-fuel hulls were dried in a furnace and then packaged in 19-L (5-gal), carbon-steel, crimp-lid buckets. The buckets were closed, moved into the Air Lock and remotely loaded into shielded storage casks. The loaded casks were removed from the Air Lock, decontaminated, and shipped to the Rockwell Hanford Company for storage. The track-dolly system that was developed for moving the fuel shipping cask was used for moving the hulls casks in and out of the Air Lock.

Four buckets of hulls were loaded into each cask. The casks provide 10 cm (4 in.) of lead shielding for the hulls. The dose rate through the side of the loaded casks was 125 to 150 mrem/h. One cask was fabricated with voids in the lead shielding, resulting in a dose rate of 10 R/h when loaded with buckets of hulls. The dose rate was reduced to the normal range before transfer of the cask to Rockwell by attaching additional lead to the outside surface of the cask, over the defective areas.

4.2.2 325-A Building Operations

In discussing the performance of the waste preparation facility in the 325-A Building, two factors must be kept in mind:

- The project objective for operating the system was to produce the desired amount of "typical" high-level waste in a minimum amount of time. Efficient recovery of uranium or plutonium was not a project objective. All processing steps beyond solvent extraction were performed only to facilitate safe handling, storage, or disposal of the uranium and plutonium. There was little incentive, time, or funding to optimize the process performance. If the objective of operating the system had been to maximize the recovery of pure uranium or plutonium, the operating philosophy would have been quite different.
- Satisfactory operation of a complex process is a function of both the equipment and the skills and dedication of the operating staff. Smooth operations are always difficult to achieve and are more difficult to maintain when a project is of very limited duration and people are expecting to be out of work or transferred in a few weeks. Thus, the accomplishments of the project operations were noteable, even though not optimal.

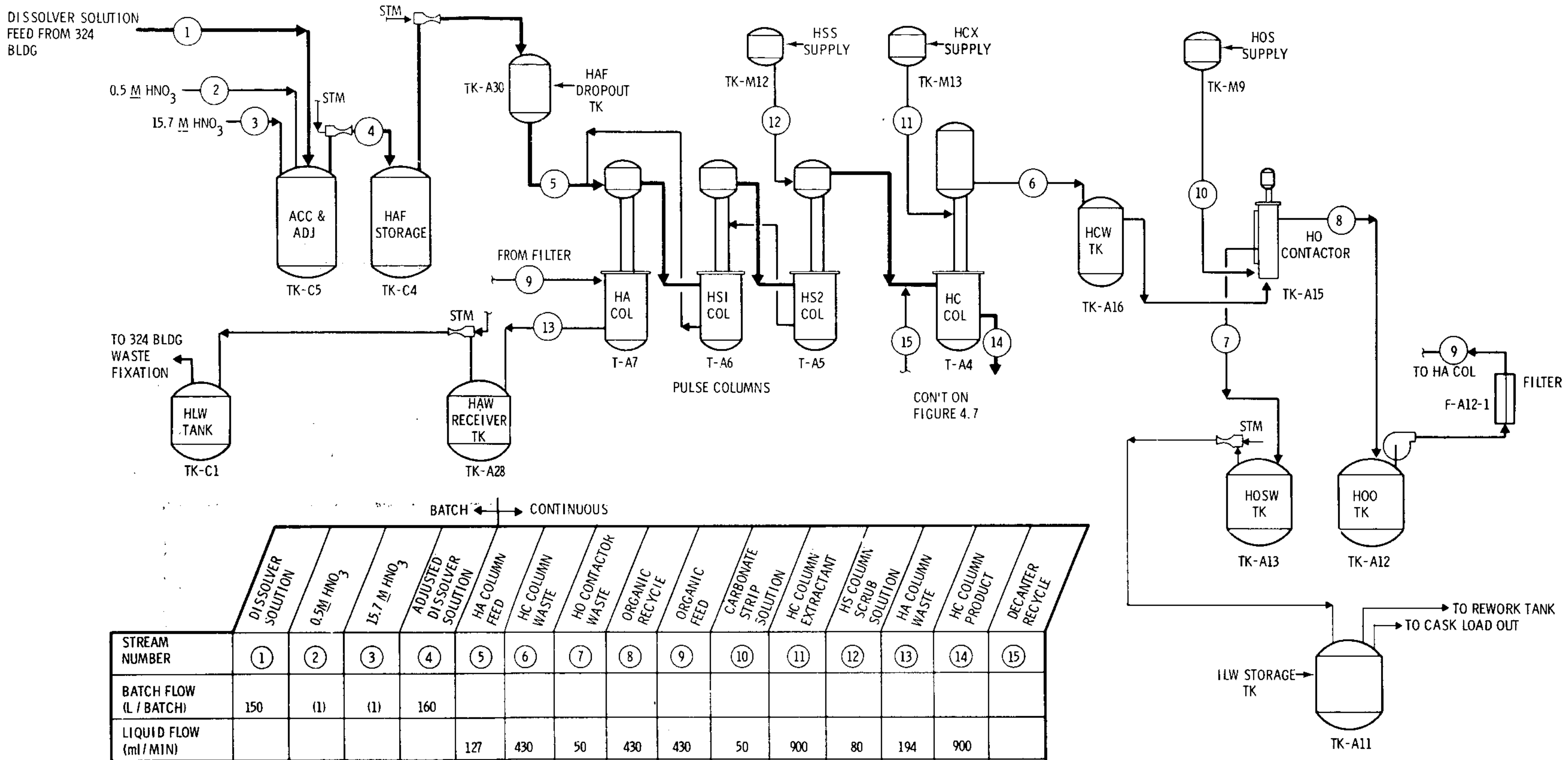
The process flowsheets for that part of the waste preparation process operated in the 325-A Building are presented in Figures 4.6 and 4.7. The overall performance of the same facility is summarized in Table 4.3. These data were prepared from a large number of analyses which were, at times, lacking in accuracy and completeness. Nevertheless, the data do give a good overview of the performance. The performance of the three major processing systems, solvent extraction, ion exchange and calcination, plus some of the supporting systems, are summarized in the following sections.

The performance of the 325-A Building process off-gas system was never specifically analyzed because the system did not have sampling capabilities. However, the overall performance of the 325 and 324 Building off-gas systems was highly satisfactory, as reported in Section 4.4 of this report.

Performance of Solvent-Extraction System

Equipment

The equipment in the solvent extraction system operated quite well, with only one serious problem during the three months of hot operations. During the first three weeks of operation, including the first hot startup, the equipment operated with no perceptible difficulty. Thereafter, the overflow line from the HCP concentrator intermittently plugged, restricting the flow rate and requiring cleanout. The average operating time between pluggings was two to three days and, since the entire solvent extraction system had to be



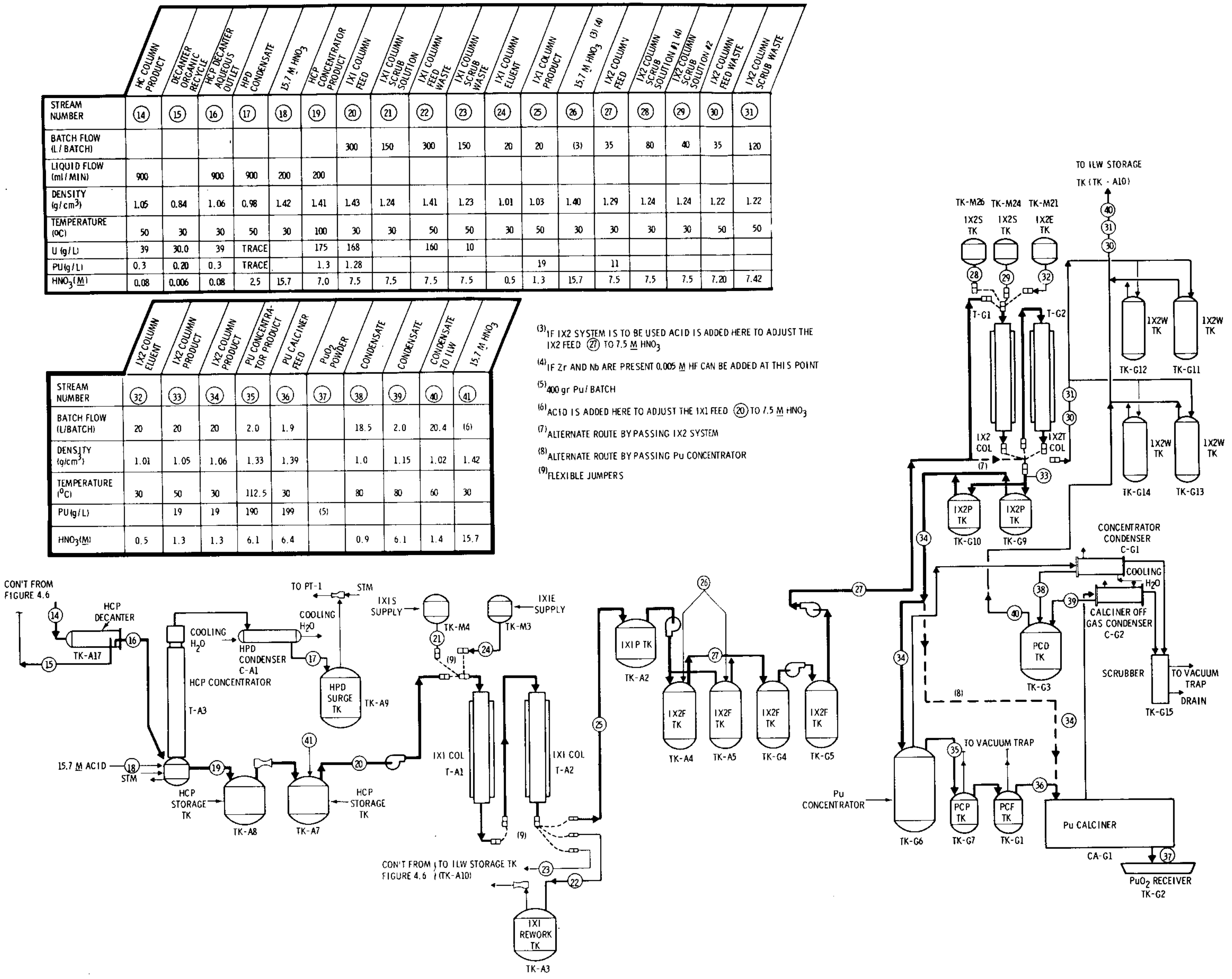
BATCH ← → CONTINUOUS

	DISSOLVER SOLUTION	0.5M HNO3	15.7 M HNO3	ADJUSTED DISSOLVER SOLUTION	HA COLUMN FEED	HC COLUMN WASTE	HO CONTACTOR WASTE	ORGANIC RECYCLE	ORGANIC FEED	CARBONATE STRIP SOLUTION	HC COLUMN EXTRACTANT	HS COLUMN SCRUB SOLUTION	HA COLUMN WASTE	HC COLUMN PRODUCT	DECANTER RECYCLE
STREAM NUMBER	①	②	③	④	⑤	⑥	⑦	⑧	⑨	⑩	⑪	⑫	⑬	⑭	⑮
BATCH FLOW (L / BATCH)	150	(1)	(1)	160											
LIQUID FLOW (ml / MIN)					127	430	50	430	430	50	900	80	194	900	
DENSITY (g / cm^3)	1.5	1.01	1.42	1.45	1.44	0.84	1.01	0.84	0.84	1.0	1.0	1.05	1.06	1.05	0.84
TEMPERATURE (°C)	40	30	30	30	30	40	40	30	30	30	30	30	30	50	30
U (g/L)	320			300	275(2)	6×10^{-3}	0.25						8×10^{-5}	39	30
PU(g/ L)	2.5			2.3	2.1(2)	TRACE	TRACE						2×10^{-5}	0.30	0.20
HNO_3(M)	2.2	0.5	15.7	2.58	2.5(2)	2×10^{-4}					0.01	2.5	2.05	0.077	0.006
TBP								30%	30%						
NPH								70%	70%						
NH_2OH-HNO_3(M) (HAN)											0.05				
Na_2CO_3 - (M)							0.23			0.25					

(1) AS NEEDED TO ADJUST HA FEED ⑤ TO 2.5 M HNO_3

(2) DILUTED TO THIS VALUE BY JET TRANSFERS

FIGURE 4.6. Process Flow Diagram for Waste Preparation--Part 1



STREAM NUMBER	HC COLUMN PRODUCT	DECANTER ORGANIC RECYCLE	HCP DECANTER AQUEOUS OUTLET	HPD CONDENSATE	15.7 M HNO_3	HCP CONCENTRATOR PRODUCT	IX1 COLUMN FEED	IX1 COLUMN SCRUB SOLUTION	IX1 COLUMN FEED WASTE	IX1 COLUMN SCRUB WASTE	IX1 COLUMN ELUENT	IX1 COLUMN PRODUCT	15.7 M HNO_3 (3) (4)	IX2 COLUMN FEED	IX2 COLUMN SCRUB SOLUTION #1 (4)	IX2 COLUMN SCRUB SOLUTION #2	IX2 COLUMN FEED WASTE	IX2 COLUMN SCRUB WASTE
	⑭	⑮	⑯	⑰	⑱	⑲	⑳	㉑	㉒	㉓	㉔	㉕	㉖	㉗	㉘	㉙	㉚	㉛
BATCH FLOW (L/BATCH)							300	150	300	150	20	20	(3)	35	80	40	35	120
LIQUID FLOW (ml/MIN)	900		900	900	200	200												
DENSITY (g/cm^3)	1.05	0.84	1.06	0.98	1.42	1.41	1.43	1.24	1.41	1.23	1.01	1.03	1.40	1.29	1.24	1.24	1.22	1.22
TEMPERATURE (°C)	50	30	30	50	30	100	30	30	50	50	30	50	30	30	30	30	50	50
U (g/L)	39	30.0	39	TRACE		175	168		160	10								
PU(g/L)	0.3	0.20	0.3	TRACE		1.3	1.28					19		11				
HNO_3(M)	0.08	0.006	0.08	2.5	15.7	7.0	7.5	7.5	7.5	7.5	0.5	1.3	15.7	7.5	7.5	7.5	7.20	7.42

STREAM NUMBER	IX2 COLUMN ELUENT	IX2 COLUMN PRODUCT	IX2 COLUMN PRODUCT	PU CONCENTRATOR PRODUCT	PU CALCINER FEED	PuO_2 POWDER	CONDENSATE	CONDENSATE	CONDENSATE TO ILW	15.7 M HNO_3
	㉜	㉝	㉞	㉟	㊱	㊲	㊳	㊴	㊵	㊶
BATCH FLOW (L/BATCH)	20	20	20	2.0	1.9		18.5	2.0	20.4	(6)
DENSITY (g/cm^3)	1.01	1.05	1.06	1.33	1.39		1.0	1.15	1.02	1.42
TEMPERATURE (°C)	30	50	30	112.5	30		80	80	60	30
PU(g/L)		19	19	190	199	(5)				
HNO_3(M)	0.5	1.3	1.3	6.1	6.4		0.9	6.1	1.4	15.7

(3) IF IX2 SYSTEM IS TO BE USED ACID IS ADDED HERE TO ADJUST THE IX2 FEED ㉗ TO 7.5 M HNO_3

(4) IF Zr AND Nb ARE PRESENT 0.005 M HF CAN BE ADDED AT THIS POINT

(5) 400 gr Pu/BATCH

(6) ACID IS ADDED HERE TO ADJUST THE IX1 FEED ⑳ TO 7.5 M HNO_3

(7) ALTERNATE ROUTE BYPASSING IX2 SYSTEM

(8) ALTERNATE ROUTE BYPASSING Pu CONCENTRATOR

(9) FLEXIBLE JUMPERS

FIGURE 4.7. Process Flow Diagram for Waste Preparation--Part 2

TABLE 4.3. Summary of Separations Process Performance

	During January (15 days)	During February (28 days)	During March (31 days)	During April	Totals for Campaign
Feed to Solvent Extraction					
Volume (L)	2,380	2,320	3,750	--	8,450
Uranium (kg)	631	729	985	--	2,350
Plutonium (g)	4,340	5,410	7,930	--	17,700
HLLW Accumulated for Transfer to 324 Building					
Volume (L)	4,780	4,220	6,790	--	15,800
Uranium (g)	1,510	510	610	--	2,630
Plutonium (g)	25.0	9.5	17.2	--	51.7
ILW Accumulated for Transfer to 200 Areas					
Volume (L)	9,320	15,700	23,100	1,670	49,800
Uranium (kg)	605	641	1,110	27	2,380
Plutonium (g)	136	325	685	28	1,170
Process Losses					
Pu in HLLW (%)	0.6	0.2	0.2	--	0.3
U in HLLW (%)	0.2	0.07	0.06	--	0.11
Pu in ILW (%)	3.1	6.0	8.6	--	6.6

shut down to clear the plug, the system was, thereafter, continually being started up and shut down. This mode of operation added to the waste losses summarized in Table 4.3.

It is believed that an organic complex formed from the degradation of TBP in the concentrator obstructed the overflow line. Since no steam stripping of solvent was possible because of restrictive distributor plates in the tower of the concentrator (see section 3.4.2), all of the soluble TBP in the HCP entered the reboiler. The volume of TBP entering the unit was 260 to 600 ml/d, determined by an analysis of the HCP stream. This agrees quite closely with the solubility calculations, which predicted about 300 ml of TBP/d entering the concentrator.

The only successful mechanism for freeing the line was to alternately pump a few liters of HNO_3 through the line, followed by hot (60 to 70^oC) $1.0\underline{M}$ sodium carbonate. Since there was no means of obtaining a sample of the material causing the problem, there still exists some uncertainty as to the exact nature of the plug. However, its apparent solubility in sodium carbonate supports the hypothesis that a uranium-phosphate solid was involved.

Time spent on this problem delayed the completion of the project by three to four weeks and reduced the efficiency (actual throughput rate/design throughput rate X 100) from 90% before inception of the plugging, to 62% for the overall project. However, since that was the only significant problem encountered in the first hot startup of a new facility, the overall performance is considered quite satisfactory.

Process

Extraction. The average waste losses from the HA Column were 0.3% for plutonium and 0.1% for uranium, as shown in Table 4.3. With the exception of one batch accumulated during the first week of hot operations, the average plutonium losses were 0.2%. As mentioned earlier, the efficiency of the system was not optimized and the continual startup and shutdown increased waste losses. At no time did the uranium in the HLW approach the imposed limit of 0.5 wt% of the uranium in the dissolver solution; therefore, no attempt was made to increase the extraction efficiency.

Stripping. The uranium and plutonium losses from the stripping column (HC) averaged 0.08% and 0.3%, respectively. The uranium and plutonium were removed from the solvent in the solvent wash contactor (HO) and discarded with the ILW. Approximately 53 g of plutonium was added to the ILW from this stream.

Fission-Product Decontamination. Because of the large backlog of analytical samples that occurred, the analyses needed to calculate fission-product decontamination factors (DFs) relative to plutonium were cancelled. However, for a two-day period during the middle of the operation, samples were taken and the following DFs were obtained for the entire solvent extraction system:

^{144}Ce ----- 3.0×10^3
^{125}Sb ----- 5.6×10^4
^{106}Ru ----- 2.4×10^2
^{137}Cs ----- 8.1×10^4
^{154}Eu ----- 1.0×10^3.

Further decontamination occurred in the ion-exchange system as discussed in the following section.

Performance of Ion-Exchange System

As stated in Section 3.4, the purpose of the A-Cell ion-exchange system (IX1) is to partition plutonium from uranium and to further decontaminate plutonium from the fission products and other impurities. The system satisfactorily performed within the design criteria. However, operating experience before the concentrator overflow line was plugged indicated that the processing rate of the 325-A Building facility was probably limited by the ion-exchange system, not the solvent extraction system, and that the limit is close to the design criteria of 50 kg U/day.

Plutonium Losses

The plutonium losses to the ILW from the IX1 system were found to be much higher than expected. Periodic analyses of the feed (IX1F), the feed cycle waste (IX1FW), and the scrub cycle waste (IX1SW) showed that the losses occurred from the feed absorption cycle and that all of the plutonium lost to waste was in the unabsorbable +6 oxidation state. The capacity of the resin beds was never exceeded. Indeed, in only one batch out of the 61 batches processed was any detectable amount of plutonium absorbed on the resin in the tailing column. Oxidation of plutonium in the HC column or the HCP concentrator was suspected of causing the high losses; therefore, on March 10th the concentration of the reducing agent, hydroxylamine nitrate (HAN) in the HC column extractant (HCX), was increased from 0.01 to 0.05 molar. The average plutonium loss of 6.6% in the three previous batches was reduced to 2.4% in the six batches following the increase in HAN. The limited amount of time before the end of the project did not permit further investigation of the effect of HAN concentration or point of addition upon plutonium losses.

Additional data on plutonium oxidation were obtained during a test intended to improve the performance of the concentrator over-flow line by reducing the potential for acid hydrolysis of TBP in the concentrator. On March 20th the acid concentration in the concentrator reboiler was reduced from 7.5 to 5.0$\underline{M}$ HNO_3. This did not decrease the frequency of plugging but did, by promoting the oxidation of plutonium in the concentrator, increase the plutonium waste losses from the IX1 system to a single-batch high of 10.5%. The waste losses returned to about 2% when the acid concentration was increased to 7.5$\underline{M}$ HNO_3.

Fission-Product Decontamination

The fission-product DF in the IX1 system was high enough that the plutonium product could be directly calcined with no further purification. Results from the gamma spectrometer for the same two days that the solvent extraction system DFs were calculated show the following plutonium decontamination factors:

^{106}Ru[a] ----- 2×10^3
^{137}Cs[a] ----- 5×10^3.

The overall plutonium decontamination factors for these two fission products were, therefore:

4.8×10^5 for ^{106}Ru, and
4.05×10^8 for ^{137}Cs.

Radiation fields in and around the glove boxes in Room 604 were quite low. The vessel containing plutonium nitrate solution eluted from the IX1 column had a maximum reading at contact through a 0.76 mm equivalent lead glove of 20 mR/h gamma. Readings of 8 mR/h gamma and 6 mrem/h neutrons were noted at the closest point to the vessel through the glove box walls. Maximum radiation readings on a can of PuO_2 (1.3 kg) of about 200 mR/h gamma were observed at contact through a 0.76 mm equivalent lead glove. Readings of 28 mR/h gamma and 20 mrem/h neutron were noted at the closest point of contact through the glove-box walls.

Glove-box Ion-Exchange System

The IX2 system was operated for only the first five days of hot operation. The decontamination achieved in the IX1 system was found to be sufficient that the glove-box ion-exchange system and product concentrator could be by-passed. Had the fuel been stored for a shorter time before processing, the ion exchange system would probably have been needed to obtain the desired purification of the plutonium product.

Ion-Exchange Resin

The Dowex 1, X-4 (50 to 100 mesh) resin was found to have adequate capacity for effective use in this application. However, after about 20 batch cycles were processed,

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[a] Gamma spectrometer calibrated for only these two energy peaks.

sufficient damage to the cross linkage had occurred that the resin particles were softened, thereby becoming deformed and causing the pressure drop to increase during the feed absorption cycle. In addition, the resin swelled more during product elution, and resettling of the bed between cycles was less effective and more difficult. Because of these problems, the resin in the IX1 columns was changed after about each 20 batches processed.

Acid Concentration

Although they were not specifically studied, fluctuations in the concentration of nitric acid in the IX1F and IX1S streams within the range 6.5 to 7.5M produced no observable effect on performance. However, the IX1E (dilute nitric acid used to elute the plutonium product from the resin) was at one time made up at 0.7M HNO_3 rather than 0.5M HNO_3. This caused an almost doubling of the volume required to remove the product from the resin bed.

Plutonium Calciner Performance

The function of the calciner is to transform the plutonium nitrate solution to a dry oxide for safe storage and handling. No plutonium-oxide purity specifications were imposed upon the project. However, a process specification was established that the loss on ignition (LOI) be <1.5 wt% when the oxide was heated at $200^{\circ}C$ for two hours.

Most plutonium calcination processes in the past transformed the plutonium nitrate to an oxalate before calcining it. Little operating knowledge of a direct denitration calciner was available. Two of the major operating characteristics of such calciners that were investigated during the project were: 1) holdup of oxide in the calciner, and 2) moisture content or LOI of the oxide produced.

Oxide Holdup in the Calciner

Before the start of operations, there was no reliable means of estimating holdup of oxide on the calciner parts. It was realized that some oxide would adhere to the walls, the screw, and the off-gas filter, but any estimate of the quantity was just a guess.

After the calciner had been operated for four weeks and 6 kg of oxide had been processed, the unit was disassembled and cleaned out. The amount of oxide remaining in the trough and on the screw was 600 g, with 80 g on the sintered metal filter. Approximately 60 g of very fine powder had also deposited on the glove-box ventilation HEPA filter. A major part of the holdup in the trough could be attributed to the flexibility of the calciner screw. As oxide became glazed onto the bottom surface of the trough, the shaft flexed and allowed the flights to turn over the top of the oxide glaze rather than removing the glaze as it was formed.

It was obvious from input-output measurements that the calciner had not reached a holdup equilibrium when cleaned out, and that the oxide was still accumulating within the calciner. Therefore, these holdup values are really less than the total oxide holdup would be if equilibrium had been reached. The short duration of the project precluded further measurements of this important parameter.

Moisture Content of Oxide Product

The feed to the calciner averaged about 19 g Pu/L dissolved in 1.0$\underline{M}$ HNO_3. During operations, the feed end of the calciner was kept between 200 and 300^{o}C, and the product end was at 450^{o}C. Before hot operations began, it was calculated that the residence time of plutonium within the calciner would be 30 minutes. The actual residence time was longer than this due to the buildup and sloughing off of oxide on the trough and screw. Accurate measurements of the residence time were not made.

The moisture content of oxide from the screw calciner varied between 0.30 wt% and 0.65 wt% (avg. = 0.44%), as shown in Table 4.4. It was assumed that these moisture content values were within the specified limits (substitution by the analytical laboratory of a specific moisture analysis for a 200^{o}C LOI was not known until later), and that at this low moisture content any pressure buildup in the storage container, from thermal or radiolytic processes, would be negligible. However, the can containing blend No 6A did build up pressure and ruptured the mechanically-sealed outer can as it was being transferred to a storage facility. After the rupture of can 6A, LOIs were determined on several of the archive samples from previously calcined batches. Table 4.4 lists LOI results on every

TABLE 4.4. Analyses of Oxide for Volatile Impurities

	Oxide Product from Screw Calciner		Oxide Calcined at 650 C for 3 Hours	Oxide Stored for 40 Days in Tape-Sealed Cans		
Storage Can No.	Moisture (a) (wt%)	LOI (b) (wt%)	LOI (wt%)	Moisture (wt%)	N_2 (c) (wt%)	LOI (wt%)
1A	0.38	1.73	--	--	--	--
2A	0.30	--	--	--	--	--
2B	0.30	--	--	--	--	--
3A	0.61	1.87	--	--	--	--
3B	0.54	1.90	--	--	--	--
4A	0.40	--	--	--	--	--
5A	0.42	2.59	--	--	--	--
5B	0.48	--	--	--	--	--
6A(d)	0.45	3.20	0.55	--	--	--
6B	0.41	2.64	0.64	--	--	--
7A	0.65	1.80	0.41	--	--	--
7B	0.51	1.94	0.32	--	--	--
8A	0.32	1.62	0.25	0.16	0.029	0.30
8B	0.37	1.54	0.31	0.26	0.037	0.46
9A	0.39	1.22	0.31	0.28	0.028	0.43
9B	0.45	1.11	0.36	0.24	0.027	0.40

(a) Moisture determined by coulometric analysis of water driven from oxide sample heated at 400^{o}C for 10 minutes.
(b) Loss on ignition (LOI) determined by sample weight loss on heating to 950^{o}C for 2 hours.
(c) N_2 determined by fusion and gas chromatography.
(d) Ruptured can.

batch processed after the can ruptured. Experiments performed as a result of the rupture have shown that a moisture content of 0.5 wt% is sufficient to rupture a sealed can, if the temperature inside the can exceeds about 140°C. The exact cause of the rupture is not, at present, known, but it seems prudent to suspect both too high a moisture content and undecomposed nitrates. Following the rupture, the calciner temperature was increased to 500°C, and the screw rotation was decreased, resulting in increased residence time.

As an added precaution, oxide processed from the screw calciner after the can ruptured was batch-blended, sampled, weighed, and then heated in a muffle furnace at 650°C for three hours. After it was cooled, the oxide was blended, sampled, and placed in tape-sealed cans for interim storage. A comparison of the LOI values before and after the 650°C calcination, given in Table 4.4, shows a five-fold decrease in volatiles by calcination to the higher temperature.

An experiment was performed to evaluate moisture absorption by the oxide during normal handling operations and storage in the glove box. Cans 8A, 8B, 9A, and 9B were tape-sealed in the glovebox and held for 40 days following calcining at 650°C, blending and sampling. These cans were then opened, and the oxide was reblended and resampled. The analytical results indicated in Table 4.4 are somewhat inconclusive because moisture analyses were not made following the 650°C calcination. The analyses do indicate that the amount of nitrate present after calcination at 650°C is small, and the moisture reabsorbed during handling operations and 40 days of storage, 0.14 to 0.20 wt%, was quite low.

Possible adverse effects during long-term storage of small amounts of moisture and/or volatiles in plutonium oxide from high-burnup fuel are not quantitatively known. The effects of self-heating of the oxide are equally important. Both volatility content and oxide temperature can be controlled by proper processing and storage conditions. These effects must be evaluated experimentally before long-term storage requirements can be defined.

Off-gas System

No problems were encountered with the off-gas system during the course of hot operations. Because the 325-A Building dissolvers were not used with irradiated fuel, the efficiency of the silver zeolite filter could not be ascertained. All other portions of the system functioned according to design.

Intermediate-Level Waste System

No significant problems were encountered with the ILW System. When cask shipments to the 200 East Area were delayed because of bad roads or for other reasons, the waste was transferred to vault tank PT-2, then removed from tank PT-2 when shipments could again be made. Approximately 700 L of ILW were generated each day during hot operations in the 325-A Building. A total of 42 cask shipments were made to the 200 East Area for disposal of the waste generated during cold testing, tracer-level testing, and full hot operations. Figure 4.8 pictures a full cask, chained to a truck bed, ready to leave the 325-A Building for the 200 East Area.

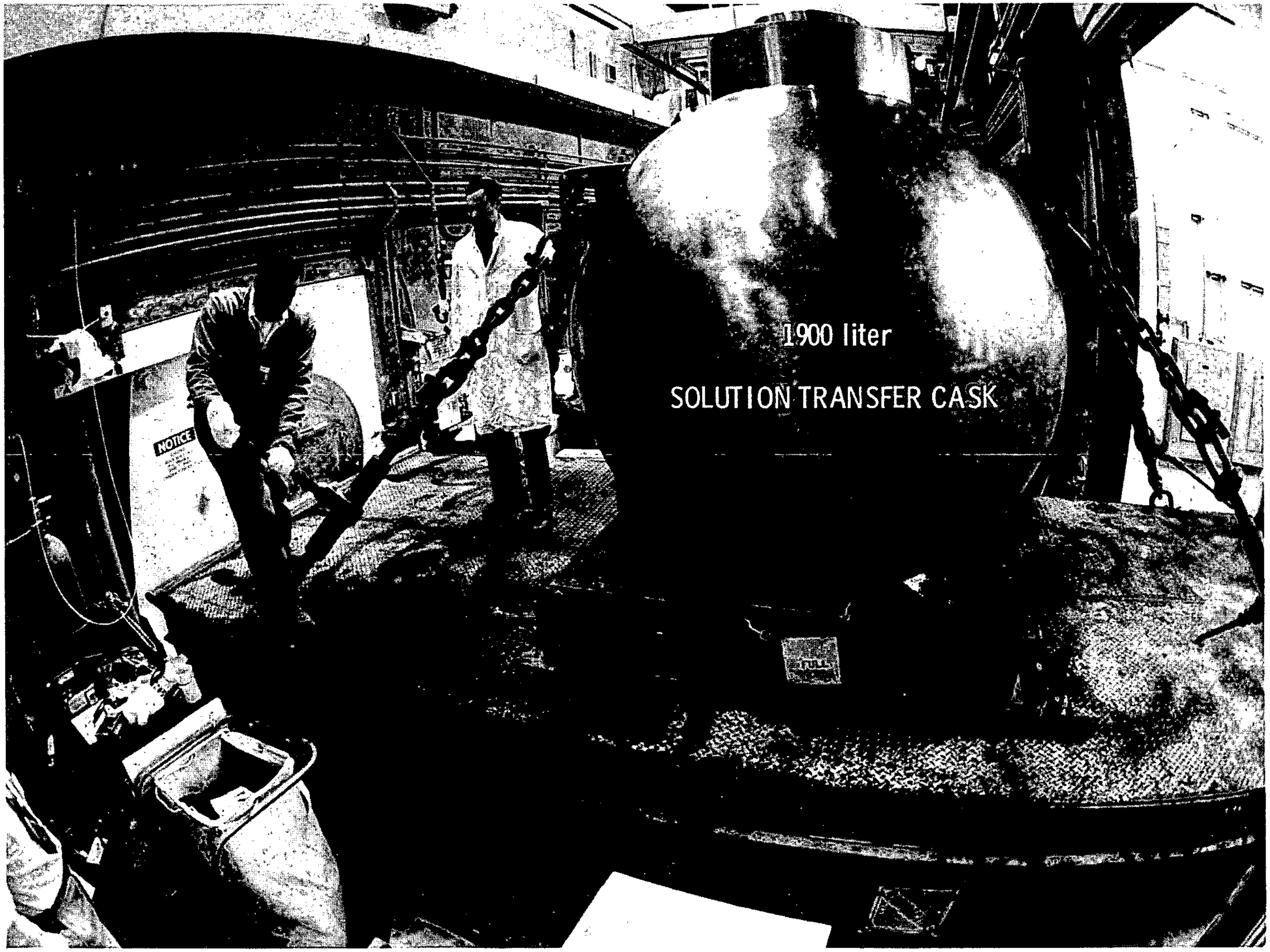


FIGURE 4.8. Preparation of Cask for Shipment of ILW to 200 East Area

Process Instrumentation

Process instruments generally performed quite well. Some minor problems were encountered because recommended limitations brought on by additional instrumentation requirements were exceeded. Some instrumentation, at first located in the chemical makeup area (AMU), suffered acid-fume damage because of leaks in some tubing fittings. The equipment was renovated and moved to a fume-free area. The processing equipment was shut down only once (less than eight hours) because of instrumentation failure. Overall, the process instrumentation caused few problems and was most beneficial to the success of the project.

4.3 WASTE VITRIFICATION

4.3.1 Feed Preparation

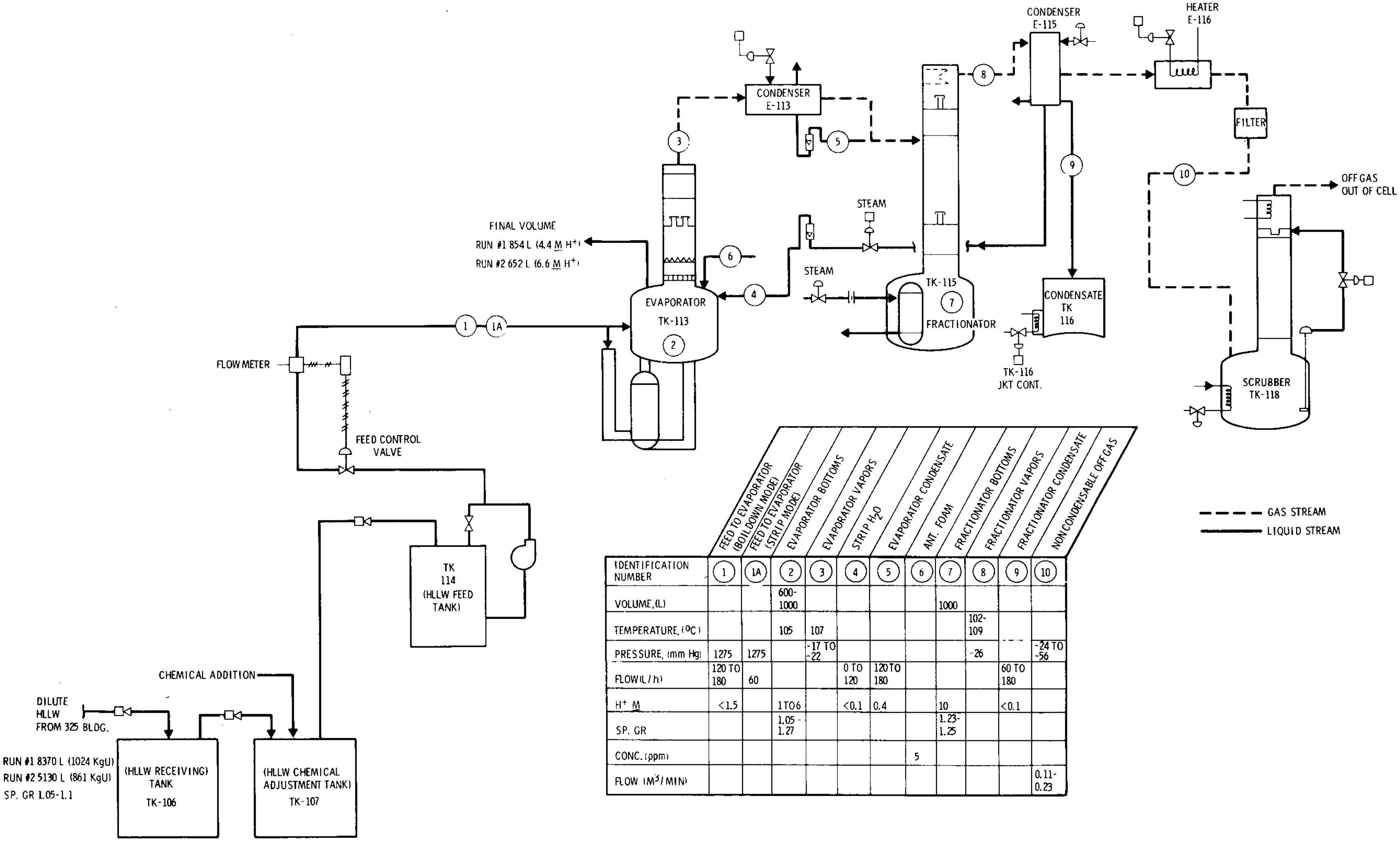
Dilute HLLW was transferred through the Interbuilding Pipeline from TK-W4 in 325-A Building to TK-106 in 324 Building. The HLLW was then transferred to TK-107, where the chemical composition was adjusted to that of typical waste by the addition of uranium and non-radioactive chemicals. The waste was then transferred to B-Cell and concentrated to the required volume and acid concentration for feed to the vitrification process. The feed preparation steps are shown schematically in Figure 4.9. A convenient expression for describing various kinds of waste is the amount of material which is associated with or which results from processing spent fuel containing one metric ton of uranium, i.e., 1 tU equivalent. Two waste-preparation/waste-vitrification runs were made. The first contained waste from only the three low-exposure fuel assemblies, and the second contained waste from both low- and high-exposure fuel assemblies.

Table 4.5 summarizes the volumes of waste solution processed for each vitrification run. The quantities of fission products and actinides, per tonne of uranium, found in fuel irradiated to these two exposures are given in Tables 4.6 and 4.7.(a) Not all of the HLLW from 325-A Building was prepared as feed for calcination (compare Tables 4.3 and 4.5). A volume of HLLW representing approximately 0.47 tU was reserved for use in other DOE programs.

The NWVP glass composition was modeled after the well-characterized PW-8a composition (Ross 1978). To achieve the desired composition, non-radioactive chemicals were added to the dilute waste before it was concentrated. The sources of most non-radioactive chemicals in typical reprocessing waste would be intermediate-level waste, corrosion products, and degraded solvent. Some specific chemicals are added to achieve the desired glass composition. Those chemicals added to the dilute waste are shown in Table 4.8.

The feed preparation required nearly a 10-fold reduction in the waste volume and acid depletion before calcination to be representative of a reprocessing plant waste feed. This was accomplished by a concentration/acid-stripping operation using the thermosyphon evaporator (TK-113). A two-step boil-down was required to control the nitric acid concentration. First, the waste was concentrated from a 1.5M HNO_3 solution to 4 to 6M HNO_3.

(a) ORIGEN computer code predictions.



	FEED TO EVAPORATOR (BOILDOWN MODE)	FEED TO EVAPORATOR (STRIP MODE)	EVAPORATOR BOTTOMS	EVAPORATOR VAPORS	STRIP H_2O	EVAPORATOR CONDENSATE	ANT. FOAM	FRACTIONATOR BOTTOMS	FRACTIONATOR VAPORS	FRACTIONATOR CONDENSATE	NONCONDENSABLE OFFGAS
IDENTIFICATION NUMBER	①	(1A)	②	③	④	⑤	⑥	⑦	⑧	⑨	⑩
VOLUME, (L)			600-1000					1000			
TEMPERATURE, (°C)			105	107					102-109		
PRESSURE, (mm Hg)	1275	1275		-17 TO -22					-26	—	-24 TO -56
FLOW (L/h)	120 TO 180	60			0 TO 120	120 TO 180				60 TO 180	
H^+ M	<1.5		1 TO 6		<0.1	0.4		10		<0.1	
SP. GR			1.05 - 1.27					1.23-1.25			
CONC. (ppm)							5				
FLOW (M^3/MIN)											0.11-0.23

FIGURE 4.9. Process Flow Diagram--Feed Preparation for Vitrification

TABLE 4.5. Volumes of Waste Processed During Feed Preparation

Feed Preparation Run No.	Average Burnup (MWd/tU)	Dilute HLLW Received in 324 Building (L)	Waste Equivalent (tU)	Waste Volume after Concentration (L)
1	20,500	8370	1.02	850
2	29,500	5130	0.86	650

TABLE 4.6. Point Beach Reactor Fuel Waste for SC/ICM Run #1 (basis 1 tU)

Element	Grams	Moles	Oxide Formed	g Oxide
Fission Products				
Ge	0.22	0.003	GeO_2	0.3
As	0.05	0.0003	As_2O_3	0.07
Se	31.7	0.40	SeO_2	44.5
Rb	203.0	1.19	Rb_2O	222.0
Sr	503.0	5.74	SrO	594.8
Y	284.0	1.60	Y_2O_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_2O_7	829.8
Ru	1350.0	13.36	RuO_2	1777.8
Rh	288.0	1.40	Rh_2O_3	355.2
Pd	838.0	7.88	PdO	964.0
Ag	41.7	0.19	Ag_2O	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_2O_3	9.2
Te	348.0	2.73	TeO_2	435.3
Cs	1530.0	5.76	Cs_2O	1622.0
Ba	975.0	7.10	BaO	1089.0
La	782.0	2.81	La_2O_3	917.1
Ce	1520.0	10.85	CeO_2	1867.0
Pr	738.0	0.87	Pr_6O_{11}	891.6
Nd	2520.0	8.74	Nd_2O_3	2939.0
Pm	22.1	0.08	Pm_2O_3	25.7
Sm	571.0	1.90	Sm_2O_3	662.2
Eu	95.3	0.31	Eu_2O_3	110.4
Gd	54.3	0.17	Gd_2O_3	62.6
Tb	1.2	0.002	Tb_4O_7	1.4
Dy	0.8	0.002	Dy_2O_3	0.9
Subtotal	17.7×10^3			22.20×10^3
Actinides				
U[a]	4850	6.79	U_3O_8	5719.0
Np	252	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_2O_3	5.1
Subtotal	5.23×10^3			6.15×10^3
TOTAL	22.95×10^3			28.35×10^3

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

TABLE 4.7. Point Beach Reactor Fuel Waste for SC/ICM Run #2 (basis 1 tU)

Element	Grams	Moles	Oxide Formed	g Oxide
Fission Products				
Ge	0.33	0.005	GeO_2	0.5
As	0.07	0.0005	As_2O_3	0.1
Se	4.65	0.06	SeO_2	6.5
Rb	307.0	1.80	Rb_2O	335.7
Sr	758.0	8.65	SrO	896.4
Y	422.0	2.37	Y_2O_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_2O_7	1188.0
Ru	1920.0	19.00	RuO_2	2528.0
Rh	369.0	1.79	Rh_2O_3	455.0
Pd	1210.0	11.37	PdO	1392.0
Ag	53.7	0.25	Ag_2O	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_2O_3	13.6
Te	502.0	3.93	TeO_2	627.9
Cs	2240.0	8.43	Cs_2O	2375.0
Ba	1420.0	10.34	BaO	1585.0
La	1140.0	4.10	La_2O_3	1337.0
Ce	2230.0	15.91	CeO_2	2739.0
Pr	1080.0	1.28	Pr_6O_{11}	1305.0
Nd	3680.0	12.76	Nd_2O_3	4293.0
Pm	37.0	0.13	Pm_2O_3	43.0
Sm	803.0	2.67	Sm_2O_3	931.2
Eu	150.0	0.49	Eu_2O_3	173.7
Gd	91.2	0.29	Gd_2O_3	105.1
Tb	1.58	0.002	Tb_4O_7	1.9
Dy	0.92	0.003	Dy_2O_3	1.1
Subtotal	25.7×10^3			32.2×10^3
Actinides				
U(a)	4800.0	6.72	U_3O_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_2O_3	12.5
Subtotal	5.58×10^3			6.54×10^3
TOTAL	31.3×10^3			38.7×10^3

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

TABLE 4.8. Inert Chemicals Added to HLLW (kg/tU)

Compound	Run #1	Run #2
$NaNO_3$	23.67	34.91
$Fe(NO_3)\cdot 9H_2O$	84.55	124.71
$Cr(NO_3)\cdot 9H_2O$	3.72	5.49
$Ni(NO_3)\cdot 6H_2O$	1.55	1.99
H_3PO_4(75%)	1.52	2.24

This was followed by continued concentration, with the addition of strip water to maintain a constant acid concentration, until the final volumes shown in Table 4.5 were achieved. The concentration procedure was the same for the two runs.

The capacity of the evaporation equipment far exceeded the requirements for the small batches of waste received from 325-A Building; therefore, frequent startup and shutdown operations were required. The feed preparation task was accomplished with only one minor problem--foaming in the evaporator. This, in turn, caused plugging in the mist eliminator in the evaporator tower. The foaming and plugging were believed to have resulted from a reaction of traces of organic with iron. The addition of an antifoaming reagent reduced the problem; however, the demister at the top of the evaporator tower was replaced twice during project operations because of excessive pressure drop.

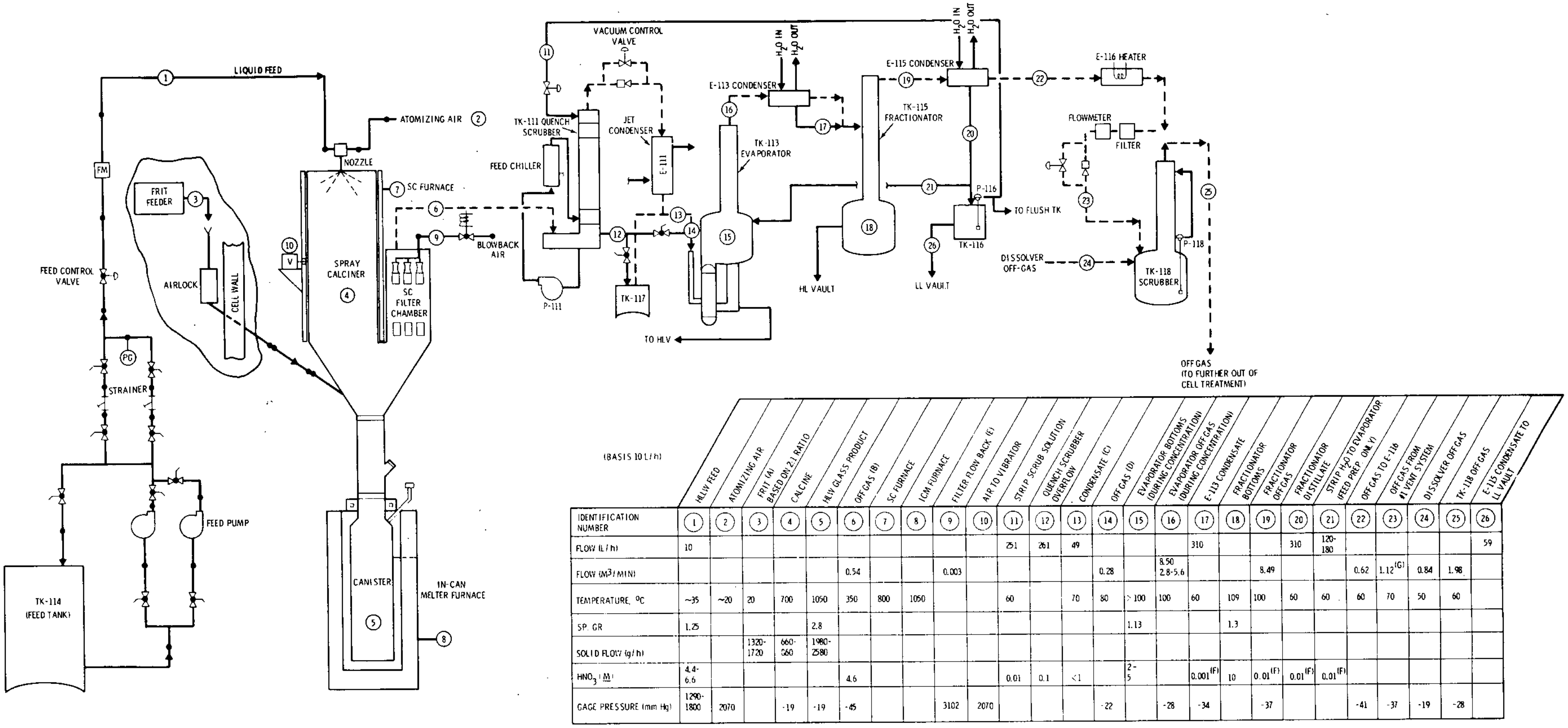
4.3.2 Vitrification

Concentrated waste from the feed preparation runs was used for the two batch operations of the spray calciner/in-can melter system (SC/ICM); two canisters of glass were produced to meet the project objective. A flowsheet and material balance for the SC/ICM process are shown in Figure 4.10. The material balance basis is 10 L/h of liquid pumped to the spray calciner from TK-114. This is the nominal flow for the B-Cell processing equipment. Specific flows for the two vitrification campaigns exceeded this basis. The agitator in TK-114 must be operated to uniformly suspend the very fine solid particles in the waste when the feed pump is in operation. As a consequence, this tank has a moderately large heel and all of the concentrated waste could not be pumped to the spray calciner (compare the uranium equivalents of waste in Tables 4.5 and 4.10).

Operating conditions for both vitrification runs are summarized in Table 4.9. Glass canister data are shown in Table 4.10. Run #1 used feed prepared from the lower burnup waste, while Run #2 had a mixture of both lower burnup and higher burnup waste feed.

The vitrification runs went smoothly, including operation of the effluent cleanup system. One equipment component failed during the first vitrification campaign, but it was not discovered until after the campaign was completed. A ceramic cap on the feed nozzle fell off sometime during Run #1. A retaining ring that should have been welded had not been welded in place, so the ring vibrated off, allowing the air cap to fall off. Without the air cap, essentially no atomization of the liquid feed occurred, resulting in a buildup of calcine on the lower cone of the spray calciner. This explained a lower-than-expected glass volume in Can #1. The material was removed from the calciner before Run #2. A small amount of this material, plus some added frit, was in the bottom of the second canister before Run #2 and formed a heel of waste glass. Data shown in previous tables includes the averaged values for material in the canister.

All other processing equipment operated well. The sintered-metal off-gas filters removed the entrained calcine dust with little further drop in pressure. Filters were blown back every 20 min, while the calciner barrel was vibrated every 30 minutes. Pumps, furnaces, frit feeders, and essentially all auxiliary equipment operated smoothly during both runs.



(BASIS 10 L/h)

	HLLW FEED	ATOMIZING AIR	FRIT (A) BASED ON 2:1 RATIO	CALCINE	HLW GLASS PRODUCT	OFFGAS (B)	SC FURNACE	ICM FURNACE	FILTER FLOW BACK (E)	AIR TO VIBRATOR	STRIP SCRUB SOLUTION	QUENCH SCRUBBER OVERFLOW	CONDENSATE (C)	OFFGAS (D)	EVAPORATOR BOTTOMS (DURING CONCENTRATION)	EVAPORATOR OFFGAS (DURING CONCENTRATION)	E-113 CONDENSATE	FRACTIONATOR BOTTOMS	FRACTIONATOR OFFGAS	FRACTIONATOR DISTILLATE	STRIP H_2O TO EVAPORATOR (FEED PREP. ONLY)	OFFGAS TO E-116	OFFGAS FROM #1 VENT SYSTEM	DISSOLVER OFFGAS	TK-118 OFFGAS	E-115 CONDENSATE TO LL VAULT
IDENTIFICATION NUMBER	①	②	③	④	⑤	⑥	⑦	⑧	⑨	⑩	⑪	⑫	⑬	⑭	⑮	⑯	⑰	⑱	⑲	⑳	㉑	㉒	㉓	㉔	㉕	㉖
FLOW (L/h)	10										251	261	49				310			310	120- 180					59
FLOW (M^3/MIN)						0.54			0.003					0.28		8.50 2.8-5.6			8.49			0.62	1.12[G]	0.84	1.98	
TEMPERATURE, °C	~35	~20	20	700	1050	350	800	1050			60		70	80	> 100	100	60	109	100	60	60	60	70	50	60	
SP. GR	1.25				2.8										1.13			1.3								
SOLID FLOW (g/h)			1320- 1720	660- 860	1980- 2580																					
HNO_3 (M)	4.4- 6.6					4.6					0.01	0.1	<1		2- 5		0.001[F]	10	0.01[F]	0.01[F]	0.01[F]					
GAGE PRESSURE (mm Hg)	1290- 1800	2070		-19	-19	-45			3102	2070				-22		-28	-34		-37			-41	-37	-19	-28	

[A] "76-199", -20 +80 MESH "WITH -325 MESH SILICON METAL

[B] STEAM 0.25 M^3/MIN
ATOMIZING AIR & NO_x 0.17 M^3/MIN
INLEAKAGE 0.11 M^3/MIN

[C] INCLUDES STEAM TO VACUUM JET

[D] FROM SPRAY CALCINER OPERATION ONLY

[E] 0.25 SEC PULSE EVERY 10-30 MIN

[F] VARIES DURING COURSE OF RUN

[G] INCLUDES ~0.57 M^3/MIN FROM AIR JET

FIGURE 4.10. Process Flow Diagram for Waste Vitrification

TABLE 4.9. Operating Conditions NWVP Runs #1 & 2

Conditions	Run #1	Run #2
Feed Concentration (L/tU)	833	760
Feed HNO_3 ($\underline{M}$)	4.4	6.6
Solids Content (g oxide/L)	66	86
Feed Processed (L)	806	430
Average Feed Rate (L/h)	15.7	12.7
Heat Content (W/L)	1.12	2.05
Activity (Ci/L)	313	526
Run Time (h)	53	33.6
Average Fuel Burnup (MWd/tU)	20,400	25,900
Onstream Factor(%)	99	96

TABLE 4.10. NWVP Canister Data - Runs #1 & 2

Conditions	Run #1	Run #2
Glass Weight (kg)	116	145
Glass Volume (L)	41.3	52.9
Fill Height (cm)	137	175
Frit to Calcine Ratio	4.2:1	2.2:1
Glass Density (g/cc)	2.8	2.7
Equivalent Uranium (tU)	0.40	0.72
Decay Heat[a] (W)	380	1012
Activity[a] (Ci)	1.05×10^5	2.64×10^5

(a) Based on ORIGEN computer code predictions.

The waste-glass canisters used in the in-can melting process were maintained at 1000 to 1050°C during the entire filling of the can. Once the cans were full, they were force-cooled with air while still in the furnace, until the wall temperatures were 550°C for all zones; this process took about two hours. The canisters were held for six hours at a wall temperature of 550°C. The furnace was then turned off and allowed to cool naturally until the can walls reached 200°C (30 hours). At this point the canisters were removed from the furnace and transferred to another cell, where the lids were to be welded in place. Quality verification tests will be performed as part of another program at PNL.

4.4 OFF GAS DISCHARGED TO THE ATMOSPHERE

In addition to ventilation air from other sources, the 325 Building stack discharges ventilation air from glove boxes, hot cells, and the storage vault in the 325-A Building complex, but not off gas from A- and C-Cell process vessels. This process off gas goes through the pipeline to the 324 Building for discharge. All ventilation air and process off gas generated in the 324 Building is discharged from the 324 Building stack.

During the NWVP, both the 324 and 325 Building stacks were sampled and analyzed for radioisotope emissions. This was done to ensure that releases would be maintained "As Low As Technically and Economically Practical" (ALATEP), and that any violation of the release limits established in the Process and Environmental Specifications would be known and corrected within the specified time period. The release limits are given in Table 4.11.

TABLE 4.11. Gaseous-Effluent Release Limits and Correction Time

	From Either 325 or 324 Building Stack	
Alpha	Level I(a)	6×10^{-14} to 6×10^{-13} μCi/cc
	Level II(a)	6×10^{-13} to 6×10^{-12} μCi/cc
	Level III(a)	$>6 \times 10^{-12}$ μCi/cc
Beta(b)	Level I(a)	3×10^{-11} to 3×10^{-10} μCi/cc
	Level II(a)	3×10^{-10} to 3×10^{-9} μCi/cc
	Level III(a)	$>3 \times 10^{-9}$ μCi/cc
Iodine-129	324 Building	2×10^{-11} μCi/cc
Tritium	324 Building	15 Ci/week
	325 Building	0.1 Ci/week
Krypton	No Release Limits Defined	

(a) Correction Time: Level I - 1 week, Level II - 1 day, Level III - 2 hours.
(b) Excluding ^{3}H and ^{85}Kr activities.

The 325 Building stack was sampled for alpha, beta, iodine-129, and tritium in the form of tritiated water (HTO). Sampling for the 324 Building stack consisted of alpha, beta, iodine-129, tritium (HTO), tritium gas (HT), and krypton-85. The results of the analyses of these samples are documented in Tables 4.12 and 4.13. The rates of ventilation air discharged from stacks during NWVP were 3.88×10^{7} m^{3}/week from the 325 Building stack, and 2.71×10^{7} m^{3}/week from the 324 Building stack.

TABLE 4.12. Weekly Emissions from the 324 Building Stack

				Tritium (Ci)	
	Alpha (Ci)	Beta (Ci)	Iodine-129 (Ci)	(HT)	(HTO)
Weekly Control Limit	1.63×10^{-6}	8.13×10^{-4}	5.4×10^{-4}	[(HT) + (HTO)] = 15.0	
Average Weekly Release	$<2.9 \times 10^{-8}$	$<1.4 \times 10^{-7}$	5.8×10^{-5}	0.023	0.61
Highest Weekly Release	$<8.9 \times 10^{-8}$	$<3.0 \times 10^{-7}$	4.9×10^{-4}	0.12	2.1

TABLE 4.13. Weekly Emissions from the 325 Building Stack

	Alpha (Ci)	Beta (Ci)	Iodine-129 (Ci)	Tritium (HTO) (Ci)
Weekly Control Limit	2.3×10^{-6}	1.2×10^{-3}	7.8×10^{-4}	0.10
Average Weekly Release	$<2.4 \times 10^{-7}$	$<7.6 \times 10^{-7}$	$<3.1 \times 10^{-6}$	<0.01
Highest Weekly Release	1.4×10^{-6}	5.8×10^{-6}	8.9×10^{-6}	0.13

4.4.1 Alpha and Beta Measurements

Alpha/beta-detection capability was present in both buildings for stack emissions before NWVP went into operation. During the period of NWVP operations, alpha/beta emission concentrations did not exceed the Level I environmental specifications. Towards the end of the project, alpha levels increased somewhat for the 325 Building stack, but did not exceed the Level I specification. It was determined from analysis that the sources of alpha were from thorium work in the 325-B Analytical Hot Cells, not from NWVP operations.

4.4.2 Iodine-129

At the start of NWVP operations, both buildings had iodine-131 detection capability, and this was expanded with the use of additional collection media for iodine-129. Iodine emission levels did not exceed the specification of 2×10^{-11} µCi/cc.

Iodine-129 released during dissolution was absorbed on the dissolver off-gas silver zeolite bed (Section 3.3). Total iodine-129 release during the shearing of 26 batches (six spent fuel bundles) was 930 µCi. A large fraction of this iodine release (860 µCi) occurred during the first two dissolver batches, during which silver Zeolon® was used as absorbent bed material. Previous accidental flooding of the bed with a $HNO_3 \cdot$ UNH solution during cold testing, followed by a wash with distilled water, may have damaged the bed and caused this poor performance.

Iodine release in the remaining 24 dissolver runs, in which silver zeolite was used as absorbent bed material, totaled 70 µCi. The batch average release, on this basis was 2.3 µCi per batch.

4.4.3 Tritium (HT, HTO)

Tritium stack sampling was installed in 324 and 325 Buildings for the NWVP operations. Tritium (HTO) was collected by the absorption of water vapor within a trap. In 325 Building, it was collected by the use of a silica gel trap, whereas in 324 Building a molecular sieve trap was used. Tritium gas (HT) in the 324 Building stack was collected by oxidizing the gas with palladium and forming water, which was then absorbed as HTO. The results showed that during the week of 1-22-79 to 1-29-79, the tritium (HTO) level from the 325 Building stack exceeded the limit of 100 mCi/week by 30 mCi. The required corrective action reduced future releases below the 100 mCi/week limit.

At this time, tritium from the 324 Building stack is showing detectable levels from the storage of NWVP waste. The levels are considerably lower than the environmental

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specification limits, but plans are being considered to routinely sample and analyze the 324 Building stack effluent until the waste is removed from the building.

4.4.4 Krypton-85

Krypton-85 was detected by the use of a converted Triton®. The Triton was calibrated with a known amount of krypton-85. It was then placed in the 324 stack building with a representative amount of stack effluent flowing through it. A visual display of $\mu Ci/m^3$ on the Triton face, plus a chart recorder, provided krypton data. After the chart curve was integrated over a week, the total curies/week released could be determined. This data was consistently 70% of the amount expected by fuel ORIGEN-code calculations.

The Environmental Evaluations group samples routinely for krypton-85 in the Hanford area. Normal background is 30 to 35 pCi/m^3; during the month of January 1979, it rose to 170 pCi/m^3. No more data is available at this time from the environmental group.

4.5 INTERBUILDING PIPELINE

The performance of the 3/8-in. (dissolver solution) and the 3/4-in. (HLLW) pipelines is summarized in Tables 4.14 and 4.15. Included are the physical properties of the two solutions.

The entire volume of dissolver solution (7800 L) from dissolution of irradiated fuel was transferred without incident through the 3/8-in. line in 28 separate batches. Each transferred batch was followed by a 25-L flush of 2.5M nitric acid. The transfer rates ranged from 110 to 130 L/h, at a receiving tank vacuum of -46 to -51 cm of mercury.

Approximately 16,000 L of dilute HLLW in 22 batches was transferred without any problems through the 3/4-in. line from 325-A Building to the 324 Building. The flow rate ranged from 590 to 690 L/h.

The normal volumetric flow of process off gas through the interbuilding pipeline was 0.71 m^3/min (25 scfm). Vacuum available in the 325-A Building, provided by the air jet in 324 Building, was at all times in excess of 76 cm of water. There were no incidents of

TABLE 4.14. Summary 3/8-Inch Dissolver Solution Pipeline Performance

Transfer Rate (L/h)	110 to 130
Uranium Concentration (g/L)	310 to 325
Acid Concentration (M)	2.2 to 2.5
Freezing Point Temperature (°C)	-10 to -1
Volume per Transfer (L)	275 to 285
Receiving Tank Vacuum (cm Hg)	-46 to -51
Specific Gravity (g/cc at 28°C)	1.45 to 1.50
Uranium Transferred in Dissolver Solution (kg)	2350
Volume of Dissolver Solution Transferred (L)	7800

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TABLE 4.15. Summary 3/4-Inch HLLW Pipeline Performance

HLLW Specific Gravity	1.06 to 1.10
Total Volume HLLW Transferred (L)	16,000
Average Flow Rate HLLW (L/hr)	590 to 690
Jet Dilution (volume %)	10 to 12

high radiation in monitoring the off-gas line. Condensation of vapor in the vent gas line did not affect the performance of the line.

During cold testing of the 3/8-in. pipeline in December and January with uranyl nitrate solutions used to simulate dissolver solution, the line was plugged twice with frozen UNH. Each time the line was freed after $0.5\underline{M}$ HNO_3 was pumped through the line from the 325-A Building. Concentrations of uranium and nitric acid were adjusted to lower the freezing point to -10°C, and no further plugging was encountered.

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