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AEC Research and Development Report

A FACILITY FOR CHEMICAL SEPARATION OF PU^{238}

by

A. L. Coogler and B. F. Fowler
Laboratory Services Division

August 1962

[redacted]

E. I. du Pont de Nemours & Co.
Savannah River Laboratory
Aiken, South Carolina

[redacted]

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Printed in USA. Price $0.90

Available from the U. S. Atomic Energy Commission
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DP-719

NUCLEAR TECHNOLOGY - CHEMISTRY
AND CHEMICAL ENGINEERING
(M-3679, 26th Ed.)

[redacted] Number 31 [redacted]

A FACILITY FOR CHEMICAL SEPARATION OF Pu^{238}

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E. I. du Pont de Nemours & Co.
Explosives Department - Atomic Energy Division
Technical Division - Savannah River Laboratory
Aiken, South Carolina

Contract AT(07-2)-1 with the
United States Atomic Energy Commission

Approved by
D. E. Waters, Research Manager
Laboratory Services Division

[redacted]

ABSTRACT

A facility for separation of Pu^{238} and Np^{237} from irradiated neptunium dioxide target slugs was designed, constructed, and operated at the Savannah River Laboratory. This report describes the facility and includes design details of equipment.

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A FACILITY FOR CHEMICAL SEPARATION OF Pu^{238}

SUMMARY

A small-scale facility to separate and purify Pu^{238} and to recover Np^{237} from irradiated target slugs was designed and installed in nine months. Existing shielded cells and gloved boxes in the main laboratory building were used to house the facility. Final products were plutonium nitrate solution and neptunium dioxide powder.

The main problems encountered during the design of the facility were: (1) to provide adequate containment and shielding of the radioactive materials, (2) to select chemical- and radiation-resistant materials, and (3) to provide for remote operation and maintenance of process equipment.

Equipment for dissolving the irradiated targets, removing fission products, and separating neptunium and plutonium required gamma shielding. This equipment, consisting of a dissolver, tanks, and resin columns for three ion exchange cycles[1], was contained within three stainless steel and "Homalite" boxes installed in two heavily shielded cells of the SRL High Level Caves. Precipitation, filtration, and calcination equipment for preparing neptunium dioxide and concentrated plutonium nitrate required alpha containment and was installed in a gloved box designed for plutonium chemistry and known as the "Dry Chemistry Facility".

This report contains a discussion of the major design problems and a detailed description of the facility and equipment. Equipment design details are based on information originally contained in a rough draft design manual that was used for preparing operating procedures and for training operating and maintenance personnel. Details of the process and operations are discussed in another report[2].

DISCUSSION

DESIGN CONSIDERATIONS

The chemical process for which the facility was designed required that:

(1) irradiated aluminum - neptunium dioxide slugs be dissolved in 10M HNO_3 with mercury and fluoride catalysts,

(2) fission products and undesirable cations be removed and Pu^{238} and Np^{237} be separated by ion exchange,

(3) plutonium nitrate be purified and concentrated by precipitating the peroxide and redissolving it in HNO_3, and

(4) neptunium nitrate be converted to the dioxide by precipitating the oxalate and calcining it to the oxide.

Several auxiliary operations were also required. These included:

(1) cask handling and unloading of target slugs

(2) slug storage

(3) preparation of chemical reagent solutions

(4) sampling of process solutions

(5) loading of transfer casks

(6) packaging of material for shipment

(7) waste disposal

The capacity of the facility was 40 to 80 g of total actinide per batch at a maximum rate of three batches per week. The equipment design required sufficient flexibility to permit minor changes in process chemistry and use of the facility for further process development experiments.

The materials to be processed presented unusual design problems because of their corrosive natures and high levels of radioactivity. In some cases, the alpha particles were energetic enough to be a potential heat problem. These factors and the need for a relatively long service life required that special attention be given to the selection of construction materials. The necessity for maintaining continuous production required the incorporation of features for remote maintenance and replacement of equipment and components.

Stainless steel and radiation-resistant plastics were selected as construction materials for the containment boxes and for all process equipment. The use of low-melting plastics in contact with process solutions was prohibited unless the plastic component could be flushed after use and could be easily and remotely replaced. Pneumatically operated valves were used wherever possible. This type of valve minimized the use of remote manipulators and thereby minimized the number of manipulator failures and reduced the hazards associated with changing and repairing contaminated manipulators. All equipment pieces except the dissolver and the in-cell tanks were designed for remote replacement. Wherever possible, in-cell tanks were piped to provide alternative routings and usage in case of failure of individual units. To facilitate remote replacement of equipment and piping, all connections inside the containment boxes were made with ferruled tubing fittings. In addition, all pipe lines were fabricated on jigs, and the jigs were retained for the fabrication of replacement items. Standardized locations were established for tanks and valve racks to minimize the number of jigs required.

DESCRIPTION OF FACILITIES AND EQUIPMENT

The interim production facility consists of the following four main sections:

(1) Cold feed preparation facility

The cold feed preparation facility (see Figure 1) is located outside of the High Level Caves wing and is protected from the weather by a canvas tent. The facility consists of an elevated platform supporting tanks for chemical storage and for the make-up and addition of process reagents. Pumps and air pressure tanks are provided for transfer of solutions into the process system.

(2) High Level Caves processing facility

The High Level Caves processing facility (see Figure 1) contains equipment for dissolving, feed preparation, and ion exchange separation. The installation consists of three in-cell containment boxes, process equipment, instrument panels, and operating consoles. The dissolution and decontamination box (Box 1) is located in Cell 1. The separation or partition box (Box 2) and the Pu^{238} concentration box (Box 3) are located in Cell 2 of the High Level Caves.

Slug casks may be introduced into Cell 1 adjacent to the transfer port of the dissolution box. The casks can be opened and the slugs transferred to the dissolver or to a shielded storage drawer by means of General Mills and master-slave manipulators.

Shielded lines leaving the back face of Cells 1 and 2 are connected to the sampling station, resin removal station, plutonium bottling station, and neptunium transfer cask station.

(3) High Level Caves auxiliary facilities

The auxiliary facilities provide equipment for handling and sampling both product and waste. Operating and instrument controls and vacuum, exhaust, and alarm systems are included.

(4) Plutonium finishing facility

The plutonium finishing facility (see Figure 2) is located in the existing plutonium process chemistry laboratory known as the "Dry Chemistry Facility". This laboratory, consisting of a large compartmented gloved box with appropriate operating and service areas, equipment, and utilities, was specially designed for the handling of relatively large amounts of Pu^{239} in solutions or as dry powder or metal. Facilities are provided for transferring solutions into or out of the system, precipitating, filtering, and calcining. Materials may be introduced into the boxes through transfer ports and removed through bag rings; liquid wastes are piped to a high level drain line. Figure 2 shows a side view of the finishing facility gloved box.

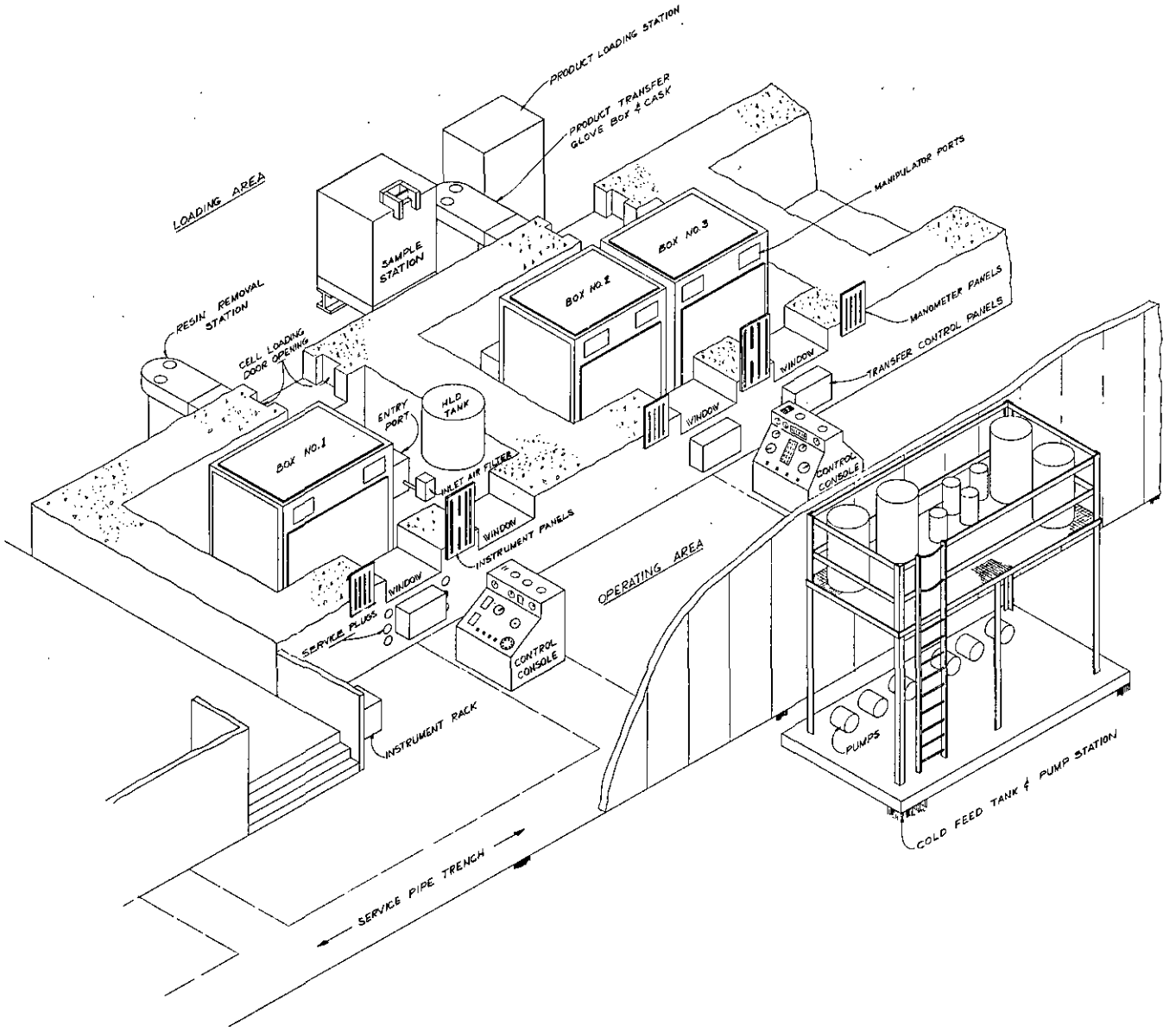


FIG. 1 HIGH LEVEL CAVES ARRANGEMENT

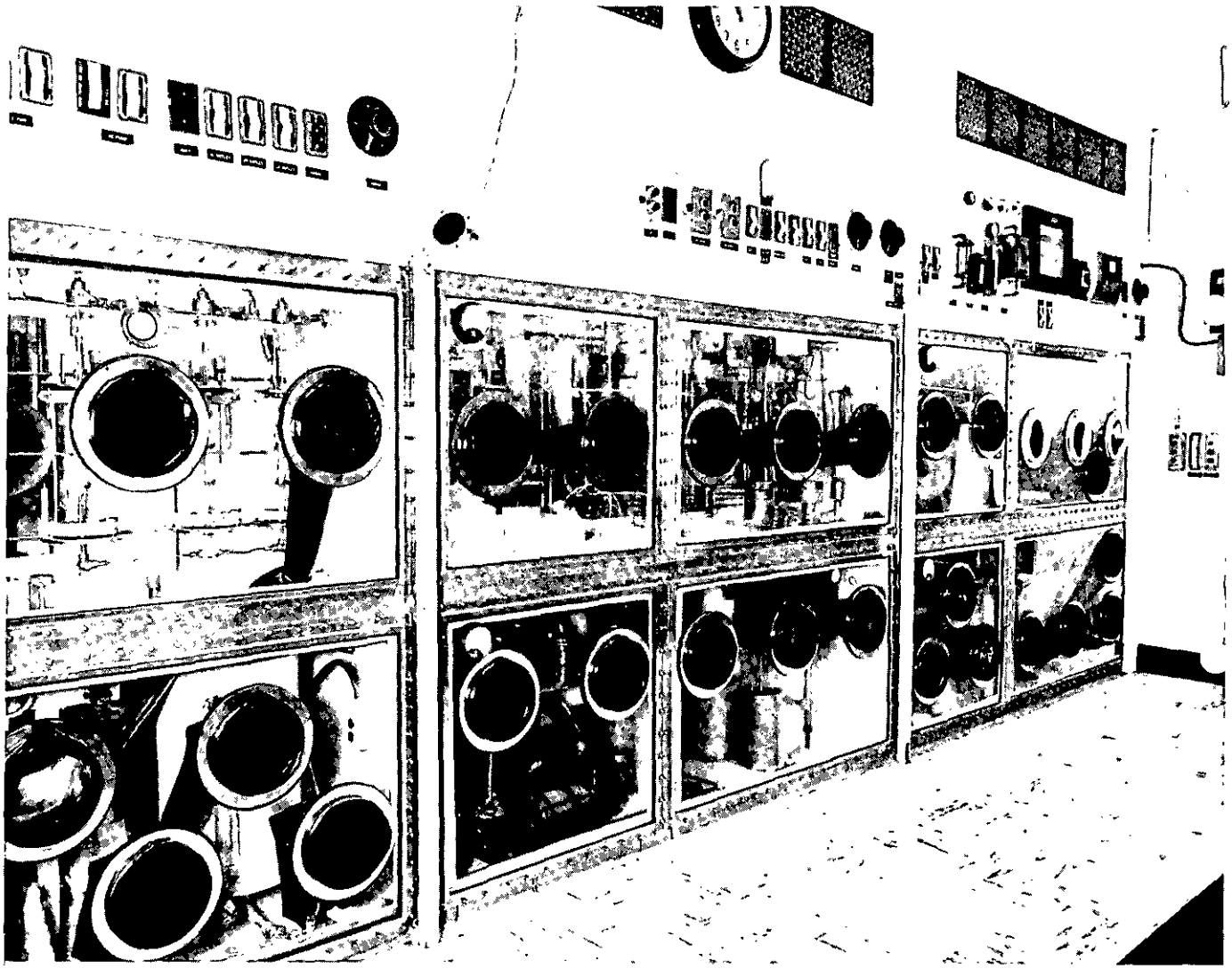


FIG. 2 DRY CHEMISTRY FACILITY ARRANGEMENT

COLD FEED PREPARATION FACILITY

General Description

The cold feed area consists of storage and mixing tanks for preparing process solutions and pumps to transfer the solutions. The area is outside the High Level Caves and is enclosed with a tarpaulin. The area is heated by electrical space heaters to prevent freezing of solutions and damage to equipment.

There are nine chemical feed tanks located on a 7-ft-high, 5-ft by 11-ft steel platform. The platform is constructed of checkered plate covered with acid-resistant paint. A secondary tray to catch acid spills is located below the platform.

A concrete pad below the platform supports nine pumps, one for each chemical feed tank. Additional space in the area is used for mixing and for storage of acid drums and chemicals. Scales and a weigh tank are provided for weighing and mixing the batches of solution to be transferred to the feed tanks.

The asphalt floor of the area is sloped to a vitreous pipe drain filled with limestone. A safety shower is located by the platform.

There is a two-way intercom installed between the outside area and the operating area of Cells 1 and 2.

Equipment

Feed Tanks

Installed feed tanks consist of the following nine type 304L stainless steel vessels:

Tank No.	Capacity	Use
E-1	20 liters	Elution solution for Column RC-1
E-2	20 liters	Elution solution for Column RC-2
E-3	20 liters	Elution solution for Column RC-3
W-1	55 gallons	Decontamination wash solution for Column RC-1
W-2A	55 gallons	Partitioning wash solution for Column RC-2
W-2B	55 gallons	Decontamination wash solution for Column RC-2
W-3	55 gallons	Decontamination wash solution for Column RC-3
SC-A	55 gallons	Scrub solution for dissolver off-gas scrubber SC-1
DS-A	30 liters	Feed solution for dissolver DS-1

Each tank is equipped with a guarded "Pyrex" sight glass and a plugged pipe coupling is located at the top for addition of chemicals to the tank. The 55-gal tanks are equipped with air-operated stirrers. All tanks are vented to a header running to a limestone drain.

Three 20-liter chemical addition tanks, FJA-1, FJA-2, and FJA-3, are located in the cold feed area. These tanks are equipped with filling funnels and are piped to the feed adjust tanks in Boxes 1, 2, and 3. Chemicals are transferred to the respective feed adjust tanks by air pressure. The chemical addition tanks are constructed of type 304L stainless steel, and each is equipped with a sight glass, pressure gauge, drain valve, vent valve, and 20-psig compressed air service.

Pumps and Piping

A separate pump is piped independently to each of the nine chemical feed tanks. The pumps for the transfer of W-1, W-2B, W-3, and SC-A tanks are Eco "Gearchem" pumps, 700 series with 3/8-inch ports. The pumps for E-1, E-2, E-3, W-2A, and DS-A tanks are Eastern D-11 centrifugal pumps. An acid drain below the pumps slopes to an earth drain.

The Eco "Gearchem" pumps have 200-mesh stainless steel strainers in the inlet lines to protect the pump gears from foreign material. All of the pumps discharge through 1/4-inch-OD stainless steel tubing headers to the cell. The discharge line has a pressure gauge, a "Kel-F" 5-μ filter, and a recycle line that returns to the top of the tank. The recycle line is equipped with a valve to regulate the discharge pressure or to recirculate the tank contents for mixing. The "Gearchem" pumps are protected with a 60-psig safety valve.

The W-2A recycle line is piped through a heat exchanger to cool the partitioning wash solution. A 45°F chilled water line passes through an insulated chamber, where a cooling coil contacts the recycle line.

Solution Make-Up

Scales and a weigh tank are provided for weighing and mixing solutions. The scale is a Fairbanks-Morse scale with a 0- to 500-lb range dial with 1/2-lb graduations. It is equipped with balance beams of 100-lb and 200-lb capacity.

The weigh tank is a 55-gal stainless steel drum equipped with an air-operated stirrer. Transfer of solutions from the weigh tank or from the storage tanks is performed with an Eco "Gearchem" pump equipped with portable polyethylene and stainless steel suction and discharge lines.

Building process cold water and distilled water services are available for solution make-up and general use.

Auxiliary Tanks

Three auxiliary tanks are installed in the operating area on the outside cell wall.

Auxiliary Dissolver Feed Tank, DS-B

A 750-ml tank is provided for adding reagents to the dissolver, DS-1. The tank is constructed from stainless steel and equipped with an entry funnel, a polyethylene sight gauge, and a pressure gauge. Liquid is transferred into the cell from the DS-B tank by 20-psig air pressure. The transfer line is valved at the instrument panel.

Resin Feed Tank, RF

A 7-liter tank for transferring resin into the cells is provided. The resin slurry is moved through 3/8-inch stainless steel lines into the boxes to the ion exchange columns by means of 20-psig air pressure controlled from Console 1. A three-way valve below the tank allows the flow to be directed to the proper resin column.

The tank is constructed of stainless steel and is equipped with an entry funnel, a polyethylene sight gauge, a sparge head, a pressure gauge, and a vent to Cell 1.

Auxiliary Elution Feed Tank, E-3 Aux

A 4-liter graduated cylinder, guarded by an expanded metal frame, is provided for feeding an accurately measured volume of elution solution through the resin column in Box 3.

HIGH LEVEL CAVES PROCESSING FACILITY

The chemical processing equipment in the High Level Caves is housed in three stainless steel containment boxes located in two general purpose cells. Aluminum-jacketed slugs are received in a shielded cask, which is introduced into Cell 1 and opened remotely. The irradiated slugs are then transferred with a General Mills manipulator to the dissolution and decontamination box (Box 1) or to a storage drawer.

Box 1 contains a dissolver with off-gas condenser and caustic scrubber, a process filter, a resin column, and associated tanks and piping. The process solution is transferred from Box 1 to the partitioning box (Box 2), located in Cell 2 of the High Level Caves. Box 2 contains a resin column and associated equipment. Neptunium product is removed from Box 2 to a cask loading station located outside the rear wall of Cell 2. The concentrated neptunium solution is transferred to the finishing facility for conversion to neptunium dioxide. The remaining process solution is transferred to the plutonium concentration box (Box 3) located adjacent to Box 2 in Cell 2. This box contains a resin column for decontaminating and concentrating the plutonium solution. The concentrated plutonium solution is bottled for shipment or trans-

ferred to the finishing facility for further processing.

Single line piping diagrams of this equipment are included in the Appendix (Figures A-1, A-2, and A-3).

High Level Caves Cells

Two of the general purpose cells in the High Level Caves are used. Each cell is approximately 6 ft by 12 ft in floor area and 17 ft high. The walls are 3-ft-thick high density concrete with two lead-glass windows in the operating face of each cell. A General Mills heavy duty electric rectilinear manipulator and a one-ton bridge crane are mounted on continuous rails that run the length of the two cells. The manipulator and crane can be moved from cell to cell by raising steel barriers in the intercell wall. There is an access door at the rear of each cell and the cell roof covers are removable. A shielded transfer and storage drawer is installed in the intercell wall.

Each cell is equipped with a sump pit to catch liquids spilled in the cell. The liquid can be steam jetted to a high level drain. Service plugs and transfer blocks are provided for piping and electric services into the cell. Cell air is exhausted to the building exhaust system. Each cell is lined with a disposable plastic liner to prevent gross contamination of the cell. "Plexiglas" panels with glove ports and bag rings are installed in each cell doorway.

Process Containment Boxes

The primary containment boxes, located in Cells 1 and 2 of the High Level Caves, are constructed of stainless steel angle framing and have stainless steel and/or "Homalite" side, top, and bottom panels. Each box is 8-1/2 ft high and 4 ft deep. Box 1 is 6 ft wide, and Boxes 2 and 3 are each 5 ft wide.

Each box is equipped with an air lock entry port. The air locks have top opening lids and a sliding tray. A sliding door separates the air lock from the inside of the box. Figures 3 and 4 are pictures taken of Box 2 during construction.

A 50-lb-capacity bridge crane is provided in each box. The hoist is powered by a 1/30-hp reversible capacitor electric motor driving a rope drum through a 200-to-1 worm gear reducer. A replaceable polyethylene rope with a stainless steel hook is attached to the rope drum. The electrical controls for the hoists are located in the operating area.

A light rack is located over the top "Homalite" panel of each box. The racks support groups of 4-ft fluorescent lamps mounted in a "Unistrut" frame. The Box 1 rack contains 22 lamps and the Box 2 and 3 racks contain 18 lamps each. The lamps in each light rack are divided into four subcircuits separately switched at the operating area consoles. The light racks can be removed via the cell roof slab opening for relamping and maintenance.

The containment boxes are fabricated in two sections. The bottom section is completely enclosed with 16-gauge stainless steel; the bottom and sides are continuously welded to angle framework. The bottom section is approximately 32 inches high and separated from the top section by a 3/8-inch reinforced plate. The bottom section contains the process tanks and dissolver.

The top box sections have "Homalite" or stainless steel side and top panels, and contain process piping, valves, resin columns, filters, liquid traps, and other process equipment. Piping from the top section extends through the 3/8-inch deck plate into the process tanks and dissolver. The bridge cranes and air locks serve the upper box sections. Connections for process piping and electrical and instrument services are made at bulkhead fitting and Amphenol plug panels in the upper section.

The upper section of each box is provided with two master-slave manipulator ports. The manipulators are provided with gauntlets which prevent gross contamination of the manipulators. Each box is equipped with a fire detector and a water spray nozzle. The fire detector is connected to the building fire alarm system.

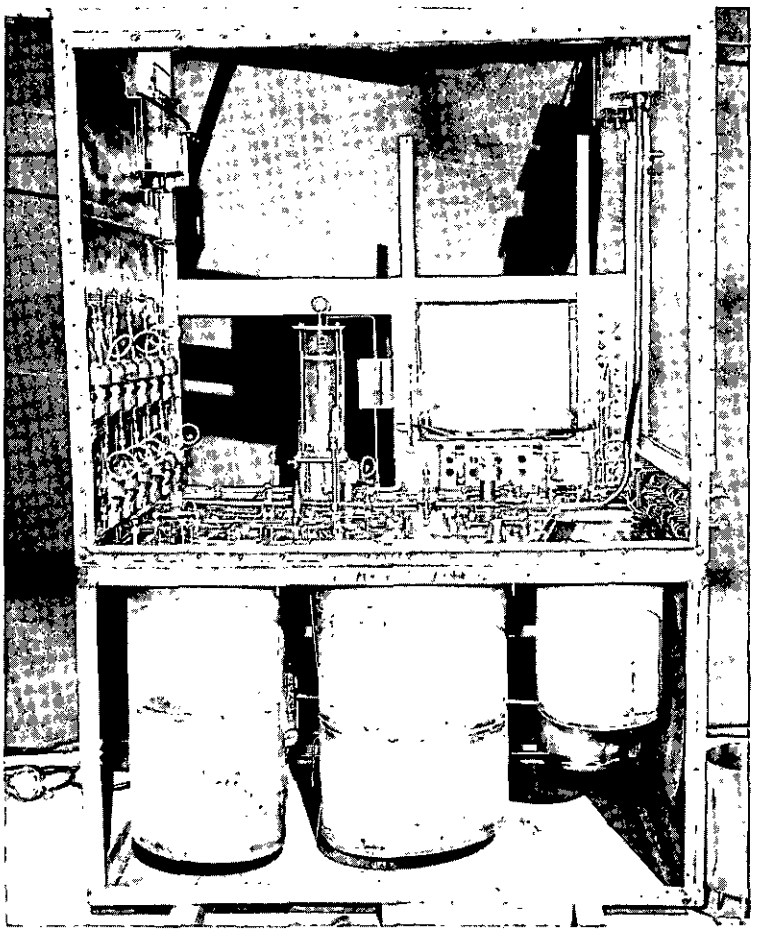


FIG. 3 BOX 2 FRONT VIEW

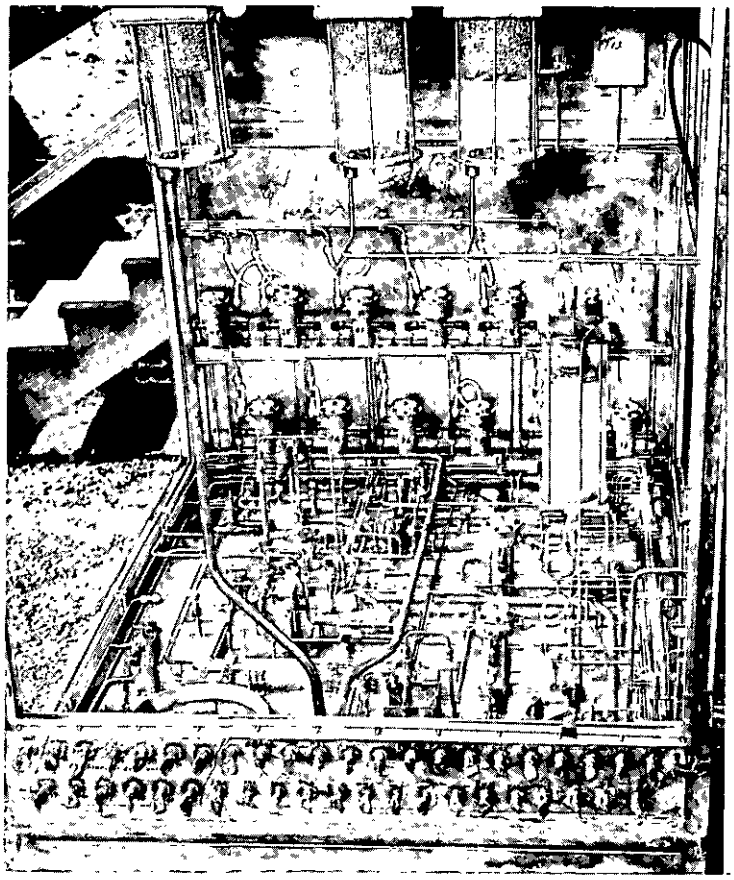


FIG. 4 BOX 2 SIDE VIEW

Process Equipment

Process Tanks

The process tanks are fabricated from type 304L stainless steel and plate. Type 308L stainless steel welding rod was used; 100% X-ray and dye tests were specified. The tanks include:

Tank Description	In Box 1		In Box 2		In Box 3	
	Tank No.	Volume, liters	Tank No.	Volume, liters	Tank No.	Volume, liters
Transfer	T-1	38	T-2	38	T-3	89
Feed adjust	FJ-1	53	FJ-2	53	FJ-3	89
Feed	F-1	38	F-2	53	F-3	89
Feed waste	FW-1	38	FW-2	89	FW-3	89
Wash waste	WW-1	138	WW-2	138	WW-3	138
Product tank 1	P-11	13	P-21	13	P-31	12
Product tank 2	P-12	13	P-22	13	P-32	12
Dry waste	DW-1	29	DW-2	29	DW-3	29

The tanks for each containment box are welded underneath a common top plate which also serves as a working level deck. The tank bottoms are sloped 5 degrees.

Each tank is equipped with a dip line which extends to the low point of the tank bottom. Liquid transfers into and out of the tanks are normally made through the dip lines. The waste and product tanks are equipped with inlet tubes connected to the resin column piping.

All tanks except the transfer and feed tanks are permanently vented and equipped with three tube bubbler packs. Two bubbler tubes per tank are used for liquid level determinations. The third tube is designed for use in specific gravity determinations. The bubbler lines are 1/8-inch tubing and are supplied with 0.1-cfh compressed air through purgerator meters in front of the cells.

The transfer and feed tanks are connected with pressure-vacuum vent lines and are pressurized or evacuated to transfer process solutions.

The feed adjust tanks are equipped with 1/30-hp variable-speed stirrers, which are controlled remotely. The waste and product tanks are equipped with $2\frac{1}{2}$-inch 10μ-fritted stainless steel air sparge heads to be used for mixing.

All tanks except the feed and transfer tanks are equipped with sample dip lines, which are piped to the sample station in the cell loading area.

The feed adjust and feed tanks are equipped with electric heaters, cooling water jackets, and thermocouple wells. Each tank has three 1000-watt ring heaters mounted on the bottom of the tank. The cooling water outlets from the top of the cooling water jackets are connected to the low level drain line in the service trench behind the cells. The thermocouple wells are equipped with stainless steel-jacketed thermocouples with polyethylene-sheathed lead lines, which extend to Amphenol connectors at the box wall and from there to temperature recorders in the operating area.

Each tank is provided with a spare fitting, which can be used as a tank connection for installation of any new lines or liquid level alarm probes. The spare fitting is a "Swagelok" female connector, which has been drilled through for a 1/4-inch tube.

A dry waste tank is provided in each box. They are open top vessels with liners for storage of solid waste matter (for example, old valves and gauntlets). The box deck plates slope to the top of each dry waste tank. Liquid leaks and deck plate flushes drain into the annular space between the tank and waste tank liner. The waste liquid can be sampled and recycled or transferred to the high level drain.

Dissolver-Condenser

The dissolver unit, located in Box 1, consists of a 50-liter boiling pot and attached condenser. The pot is equipped with three ring-type electrical heaters (4500 watts total), a cooling water jacket, and four thermocouple wells. A slug entry hatch, designed to withstand a dissolver pressure of 2 psig, is provided in the dissolver top. The dissolver bottom is sloped and a process dip tube is installed through the dissolver top to the low point in the pot. The pot contains four bubbler lines designed for use in determining liquid level, specific gravity, and presence of foam.

The condenser consists of a 3-ft-long section of 4-inch pipe containing nine bubble caps and an 8-inch demister wire section. The condenser has three separate cooling water jackets and three thermocouple wells. It is designed for off-gas rates of 20 cfm and is exhausted to the off-gas system through a caustic scrubber.

Scrubber

A caustic scrubber, installed in Box 1, removes iodine from the dissolver off-gas stream. It is constructed of a 2-ft-long section of 4-inch pipe. A 0.2-gpm caustic spray nozzle is located in the scrubber pipe between two 8-inch demister wire sections.

The scrubber is designed for gas flow rates up to 20 cfm. Gases exhausted from the scrubber pass through a demister filter unit in Box 1, a liquid trap in Cell 1, and CWS (Chemical Warfare Service) filters in the loading area before going into the building off-gas exhaust system.

Caustic solution from the scrubber flows by gravity through a weir to the high level drain system. A conductivity-type flow detector activates indicating lights on the Cell 1 console.

Resin Columns

The ion exchange columns are constructed of heavy-wall, precision-bore "Pyrex" tubing. Each resin column in Boxes 1 and 2 is 4.95 inches in ID and 15 inches long and contains 4.1 liters of resin. The resin column in Box 3 is 2.5 inches in ID and 15 inches long and contains 1.1 liters of resin. The resin is retained between 150-mesh stainless steel screens. The top retaining screen is mounted in a spring-loaded piston with polyethylene wipers to maintain the resin in a compacted bed. A 3/8-inch tube is located between the retaining screens to permit resin charging and removal. The columns are installed with "Swagelok" fittings and polyethylene ferrules. A transparent shield is provided around the "Pyrex" column to prevent loss of product or equipment damage if the column should shatter. Figure 5 shows a typical resin column.

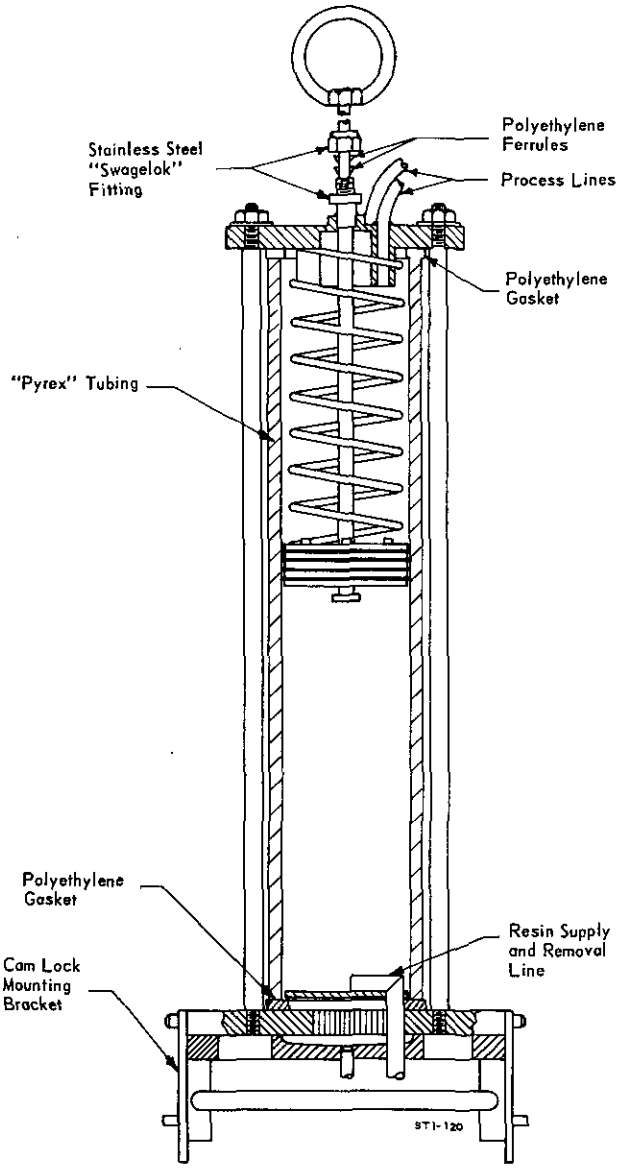


FIG. 5 TYPICAL RESIN COLUMN

Process Filter

The process filter (FT-1), provided in Box 1 for filtering dissolver solution, is a Micro Metallic Company 5μ-fritted stainless steel filter unit with a 4-ft^2 filter area. The filter housing is welded closed and equipped with "Swagelok" fittings. A standard jig is provided for fabricating replacement filters.

High Level Waste Hold Tank

A 125-liter stainless steel high level waste hold tank is installed in an upper corner of Cell 1. High level liquid waste from Boxes 1, 2, and 3 can be transferred to this tank by vacuum and then drained by gravity into the building high level drain system. Waste liquid from the resin waste station is also discharged to the waste hold tank.

The high level waste hold tank is equipped with three process waste inlet lines, a vacuum supply line, a drain valve, a spray nozzle, and high and low level conductivity probes. A process header from each containment box enters at the top of tank. Valves are provided for these lines on one wall inside Cell 1. The vacuum supply line is valved at the sample station and a vacuum gauge and a vent valve are located in the operating area. The tank drain valve handle extends into the operating area through a service plug. The spray nozzle is connected to a process cold water line for tank flushing and decontamination. The high and low level conductivity probes are connected to the liquid level alarm panel.

Waste Jet

A high level drain jet is installed in each box for emergency use in case the high level waste hold tank system should fail. The jet is a $\frac{1}{2}$-inch Schutte and Koerting stainless steel jet. Water at 75 to 100 psig is used as the jetting fluid.

Valves and Piping

Two types of valves are used in the containment box piping. For unidirectional flow Hoke AOTY-440 bellows seal valves with modified pneumatic operators are used. The modified operators have stainless steel rather than brass parts and "Hypalon" diaphragms. Where multidirectional flow is desired, Republic plug valves are used. The valve plugs are polyethylene in Box 1 and "Teflon" in Boxes 2 and 3.

The valves are equipped with "Swagelok" fittings that are spaced to match vertical tube connections in the process piping. All valves of like kind are interchangeable and valve jigs are provided for fabricating replacement valves.

The basic piping material is 1/4-inch type 304L stainless steel tubing. Bubbler packs have 1/8-inch type 304L tubing, and the resin and waste systems have some 3/8- and 1/2-inch type 304L tubing.

Single line piping diagrams for Boxes 1, 2, and 3 are included in the Appendix (Figures A-1, A-2, and A-3).

HIGH LEVEL CAVES AUXILIARY FACILITIES

Auxiliary Equipment

Sampler Station

A 14-stage sampler is installed in a modified junior cave located in the cell loading area. The 3-inch-thick steel junior cave shell is provided with a "Plexiglas" front containing glove ports, a bag ring, and an air lock. The junior cave is equipped with a high level drain for liquid waste and is exhausted by the building off-gas exhaust system.

The 14-stage sampler consists of two racks. The top rack can be moved vertically with pneumatic cylinders. The rack contains a pair of stainless steel needles for each of the 14 sampling points; each pair consists of one long and one short needle. The needles are positioned to provide a sample of predetermined volume. The bottom rack holds 14 standard 10-cc pyramid sample bottles with diaphragm tops and can be moved horizontally on centering guides.

Resin Waste Station and Disposal Cask

A resin waste removal station is provided outside the Cell 1 wall in the loading area. The disposal cask can be attached to the station.

The resin waste station consists of a 20-inch by 14-inch by 8-inch stainless steel enclosure with a "Plexiglas" top panel and a sliding door. The station is permanently piped to the resin waste header in Cells 1 and 2 and to the high level waste hold tank with 3/8-inch stainless steel tubing.

The resin waste cask consists of a disposable, 85-liter, stainless steel tank contained within a reuseable lead liner. A reuseable stainless steel and "Plexiglas" gloved box is mounted on top of the resin tank and shield assembly.

The gloved box is equipped with a sliding door transfer port that matches the port on the resin removal station. The waste tank assembly is attached to the removal station by toggle clamps. A neoprene gasket seals the joint between the sliding door faces of the gloved box and waste station.

The disposal cask is equipped with a resin slurry inlet which is connected to the resin waste header. A liquid waste outlet from the waste tank is equipped with a 200-mesh stainless steel screen and is connected to the high level waste hold tank line. The connections from the station to the waste tank are made with 3/8-inch polyethylene tubing in the gloved box. These lines are equipped with valves at the tank connections.

Resin transfers are made by vacuum supplied from the high level waste hold tank. The spent resin in the process resin columns is slurried with dilute nitric acid from the cold feed area. The resin slurry flows from the column through the resin waste piping into the waste tank. The resin is retained in the waste tank by the 200-mesh screen, and the liquid is drawn into the high level waste hold tank.

The resin waste station is exhausted by the off-gas exhaust system at approximately 20 cfm. Air inlet is through a 4-inch by 4-inch by 6-inch CWS filter mounted on the waste tank gloved box; a negative pressure of approximately one inch of water is maintained.

Product Transfer Station and Transport Casks

A product transfer station is provided outside the Cell 2 wall in the loading area. Shielded casks which can be attached to the transfer station are provided to transport product solution from the transfer station to the finishing facility.

The transfer station consists of a 20-inch by 14-inch by 8-inch stainless steel enclosure with a "Plexiglas" top panel and a sliding door. The station is permanently piped to the product hold tanks and vacuum header in Cell 2 with 1/4-inch stainless steel tubing.

Each cask assembly consists of a cylindrical shielded tank with a gloved box mounted on the top. A dolly is provided for each cask. Two casks are provided for neptunium product and two for plutonium product.

Each plutonium cask contains a 2.1-liter stainless steel tank equipped with a cooling water jacket. The tank is shielded with one inch of paraffin and one inch of lead around the sides and bottom. Two pipes enter the top of the tank. One line is a dip line and extends to within 1/8 inch of the bottom of the tank. The other line is a short line extending 1/4 inch below the tank top. Each line is provided with a valve. A bypass line and valve connects the two tank lines. The valves are installed above the tank in a shielded cavity. One inch of polyethylene shielding is provided for the tank top and the cavity is filled with lead shot to a level two inches above the valves.

A stainless steel gloved box is welded to the top of the shielded tank and valve section. It is equipped with two glove ports, a 4-inch by 4-inch by 6-inch CWS air inlet filter, and a sliding door panel, which matches the product transfer station sliding door panel. Extension handles from the shielded valves extend into the gloved box section. "Swagelok" connectors are provided for connecting the tank dip tube and short tube to the product transfer and vacuum lines in the product transfer station. Cooling water and drain connections are provided outside the gloved box section for the tank cooling water jacket.

The neptunium product transfer casks are similar to the plutonium casks except there is no cooling water jacket or paraffin and polyethylene shielding. The neptunium transfer casks have a capacity of 12.7 liters.

Product Loading Station

A plutonium product loading station is provided in the cell loading area adjacent to the product transfer loading station. It consists of a two-section "Micarta"-faced plywood and "Plexiglas" gloved box. Product solution measuring and sampling is performed in the upper section. The solution is then put into special stainless steel shipping containers in the lower section.

The upper gloved box section contains a 4.5-liter stainless steel product transfer tank, a 1-liter graduated cylinder, and associated valves and piping. 1/4-inch tubing connections to the product transfer station are made through an 8-inch plastic tube connected to glove ports.

The lower gloved box section contains a pneumatically operated rack which positions containers beneath a loading port in the upper section.

The loading station gloved box is exhausted through a CWS filter to the off-gas exhaust. Air flow into the box is provided through double duststop filters in the gloved box entry air lock.

Decontamination Station

The stainless steel shipping containers are decontaminated in a modified laboratory hood. The hood face is replaced with a sliding front with glove ports. Loaded containers are triple bagged and placed in a stainless steel container on a dolly for transport to the hood. The bagged containers are put into the hood on a holder. With the hood front in the "down" position, the bags are removed, and the containers are decontaminated.

Operating Controls and Instrumentation

Most in-cell equipment is operated remotely from consoles located near the front face of the cell. For convenience, process instrumentation for each containment box is grouped near the appropriate control console. In addition, each box is equipped with a pair of master-slave manipulators; a General Mills manipulator serves the area within the cells but outside the containment boxes. The manipulators are used for material handling, maintenance, and limited process operation.

Process Transfer Control Panels

The process transfer control panels are completely enclosed steel and "Plexiglas" boxes housing valves and piping for applying vacuum or pressure to the containment box process transfer and feed tanks. There are three transfer control panels, one for each process containment box. The Box 1 panel is located beneath the Cell 1 window; the Box 2 and 3 panels are located under the Cell 2 windows.

The Box 1 transfer control panel controls vacuum and pressure supply for transfers into and out of the T-1 (transfer) and F-1 (feed) tanks. The Box 2 panel controls the T-2 and F-2 tanks, and the Box 3 panel controls the T-3 and F-3 tanks.

A typical transfer or feed tank is equipped with a pressure-vacuum vent line which is piped through a transparent liquid trap and a filter trap in the containment box to a transfer control panel. The liquid trap contains a liquid level alarm probe. Inside the transfer control panel the line is connected through a float check valve to the common port of

a three-way plug valve. The through-side ports of the valve are connected to a 20-psig pressure supply, a vacuum supply, and a vent header. Gauges are provided for the tank pressure-vacuum vent lines and for the vacuum supply line. A valve is provided for venting the vacuum header to the vent header which exhausts into the cell containment box.

Instrument and Control Consoles

Instrument and Control Console 1 is located in the cave operating area in front of Cell 1. It houses some of the control valves, electrical gear, and instrumentation for the Box 1 process. Console 2 is located in the operating area in front of Cell 2. It contains controls similar to Console 1 and serves Boxes 2 and 3.

The consoles are fabricated from steel angle and sheet. Valves, electrical switches and controls, and instruments are mounted on the side, top, and end panels. Doors and removable panels provide access to piping and wiring inside the consoles.

The consoles provide mounting space for the following valves and electrical and instrument controls:

(1) plug valves connected to compressed air and vent headers to operate the Hoke pneumatic valves in the "hot" process piping and to pressurize or vent the cold feed adjust tanks (FJA), the dissolver addition tank (DSB), and the resin feed tank (RF)

(2) pressure regulator and air valves to supply sparge mixing heads in the waste and product tanks

(3) water supply valves for the cooling jackets of the feed adjust tanks, feed tanks, dissolver and condenser, and containment box and high level waste hold tank spray lines

(4) box light switches, tank and dissolver heater switches and powerstats, stirrer switches and rheostats, and containment box bridge crane controls

(5) purgerator flow meter and Magnehelic gauges for indication of containment box negative pressure, dry waste tank liquid level, dissolver liquid and foam indication, and pressure drop across the dissolver, condenser, and scrubber

Console 2 also contains a thermocouple temperature recorder which records the feed adjust and feed tank temperatures in Boxes 2 and 3.

Instrument and Control Panels

Instrument and Control Panels 1 and 2 are mounted on the outside front walls of Cells 1 and 2, respectively. These panels provide mounting space for control valves and rotameters for column wash and elution lines and the off-gas exhaust scrubber solution line, control valves from the feed adjust and dissolver addition tanks, and purge rate meters for bubbler lines into the "hot" process tanks.

Manometer Racks

The manometer racks are mounted on the outside front wall of Cells 1 and 2. One rack is supplied for each of the three containment boxes. Each rack contains five 30-inch U-loop water manometers for determining liquid level in the feed adjust, feed waste, wash waste, and two product tanks in each box. The manometers are connected parallel with the purge lines from the purgerators on the instrument and control panels.

Recorder and Instrument Panel

A panel is provided in the operating area for an eight-point temperature recorder and a six-position battery-powered radiation alarm unit.

Box 1 process temperatures are relayed from thermocouples located in the dissolver, condenser, feed adjust, and feed tanks to the eight-point temperature recorder.

The radiation alarm unit consists of six radiation meters, which energize an alarm buzzer when any one of six radiation probes detects a preset radiation level. Radiation detection probes are located at each of the three transfer control panels below the cell windows, at the vacuum pumps on the Cell 1 roof, and at each of the two primary off-gas exhaust filter banks behind Cells 1 and 2.

Liquid Level Alarm Panel

The liquid level alarm panel is located in the operating area. It houses an alarm bell, indicating lights, and four liquid level relays. The relays are connected to conductivity probes in the Box 1, 2 and 3 transfer and feed tank liquid traps and the high level waste hold tank.

Conductivity probes in the liquid traps from the Box 1 transfer and feed tanks are tied in parallel to one relay. Liquid in either trap will cause the alarm bell to ring and the Box 1 overfill indicator light to turn on.

Conductivity probes in the liquid traps from the Box 2 and 3 transfer and feed tanks are tied in parallel to another relay. Liquid in any one of the four traps will sound the alarm and light the Box 2 and 3 overfill indicator light.

The other two relays are connected, one each, to the high level and low level conductivity probes in the high level waste hold tank. A green light on the panel indicates when the tank is empty. When the tank is approximately 10% full the lower probe will contact the liquid and cause the green light to switch off and an amber light to switch on. If the tank is filled to 90% of capacity the alarm bell will sound and a red light will switch on.

An acknowledge button is provided to silence the alarm bell in all cases; however, the appropriate alarm light or lights will remain on until the cause for the alarm has been relieved. Relay and light test buttons are provided for each of the four relays.

Jet Pump

A pump is provided adjacent to Console 1 to boost the building process water pressure for use with the box waste jets. The pump is an Eco "Gearchem" pump, 700 series, with 3/8-inch ports.

The process water line is equipped with a strainer upstream of the pump and a bypass and relief valve around the pump. The line branches to each of the three boxes to service jets J-1, J-2, and J-3. Operating pressure is approximately 75 psig. Each branch line is equipped with a gauge and a valve for pressure regulation.

Box Microphones

Microphones are installed through service plugs to each of the three containment boxes. The amplifier, speaker, and controls for the microphones are located on the wall in front of Cell 2.

Control and Service Piping

All control and service piping to Boxes 1, 2, and 3 is routed through the consoles and panels located in the operating area.

Lines from the cold feed area are run through the building wall into a service trench below the floor of the operating area to the consoles and panels. Process cold water, 20-psig air, and 90-psig air are supplied from building service headers existing in the service trench.

All lines into the cells from the consoles and panels enter through service plugs. The tubing is spiraled in small bundles through the plugs which are then filled with poured lead or lead shot. "Swagelok" unions are provided at each end of the tubing in the service plugs for ease of assembly.

Ball check valves are installed in the cell entry lines where practical to prevent the possibility of liquid back-up and possible contamination spread in the operating area. The valves are located inside the cells between the service plugs and the containment boxes.

Piping from the cold feed area and operating area into the cells is 1/4-inch stainless steel and copper tubing except for purge lines, which are 1/8-inch copper tubes, and cooling water lines, which are 3/8-inch copper tubes.

Auxiliary Services

The exhaust, electrical, and vacuum service systems are described in this section. Process cold water, distilled water, 20-psig compressed air, 90-psig compressed air, high level drain, and low level drain services are supplied from existing building facilities and are not discussed.

Exhaust Systems

The processing facility utilizes two existing exhaust systems. The cell exhaust system is retained for exhausting Cells 1 and 2. An extension of the off-gas exhaust system is provided for containment box, dissolver, and vacuum pump exhaust.

Normal cell exhaust is 500 cfm per cell. Air inlet is through fiberglass duststop filters located in air slots on the cell roof. A 20-inch by 20-inch by 2-inch thick fiberglass exhaust filter is located near the floor of each cell. Each cell exhaust is equipped with a duststop prefilter and a 24-inch by 24-inch by 12-inch-thick CWS absolute filter and exhaust fan. A second filter unit and fan are installed as spares. The fans are discharged into exhaust stacks.

An off-gas exhaust system serves the three cell containment boxes, the dissolver exhaust, the vacuum pump discharge and pump housing, the product and waste transfer stations, the sample station, and the product loading station. A single line diagram of the off-gas exhaust system is provided in the Appendix (Figure A-4).

The processing facility off-gas exhaust header is separated from the main exhaust header by two completely enclosed Flanders Model 6C22-CN2N2, Size C, CWS filters. The Flanders filters are piped in parallel and one is in service at all times. Service runouts from the facility header are equipped with 4-inch by 4-inch by 6-inch CWS cartridge filters prior to the Flanders filters. The cartridge filters are housed in gloved boxes. The sample station and product loading station filters are installed in individual stainless steel housings.

Power Supply

Lighting panels A and E, instrument panel 11-RL, and emergency panel ES supply 110 v normal, instrument, and emergency power, respectively. These panels are located in the High Level Caves operating area on the wall opposite Cell 1. Instrument power is supplied to the intercom system between the cold feed area and the operating area. Emergency power is supplied to the liquid level alarm system and the cold feed pumps. All other equipment and instruments are supplied normal 110 v electrical power.

The fire alarm detectors in Boxes 1, 2, and 3 are connected in series to the building fire and evacuation alarm panel.

Vacuum System

A separate vacuum system is provided for the processing facility. The system consists of three Model 1400B Welch Duo-Seal vacuum pumps, stainless steel piping, and plastic and stainless steel valves. Vacuum service is supplied to the Box 1, 2, and 3 process transfer control panels, the high level waste hold tank, and the sample station. A diagram of the vacuum system is included in Figure A-4 in the Appendix.

The vacuum pumps are installed on the Cell 1 roof. Each pump is piped to a specific use point. Individual switches with pilot lights are located in the operating area at convenient locations. Valved cross connections are provided between the pumps so that each pump can be used as a spare for either or both of the other pumps.

Each pump suction line is equipped with a dry ice liquid trap. The trap has a valved drain line to the building high level drain system. Each pump is contained in a stainless steel and "Plexiglas" enclosure to permit removal of the entire pump and enclosure for decontamination and maintenance. The pump enclosures are exhausted to the off-gas exhaust system.

The vacuum pump discharge lines are connected into a common header equipped with an oil trap. The trap discharge line is exhausted to the off-gas exhaust system.

FINISHING FACILITY

General Description

The finishing facility is provided to concentrate plutonium nitrate solution and convert neptunium nitrate solution to a dioxide powder. Plutonium is precipitated as the peroxide, collected on a filter, and dissolved to the desired concentration. Neptunium is precipitated as the oxalate, collected on a filter, and calcined to the dioxide.

The finishing facility is installed in an 8-ft section of a "Dry Chemistry Facility" gloved box, which is 24 ft by 4 ft by 6-1/2 ft high and is located in the center of a 24-ft by 24-ft radiochemical laboratory. The gloved box has gloved panels on both sides and is separated by vertical partitions and air locks into three 8-ft-long sections. Each 8-ft section is divided into an upper and lower box by a horizontal partition which serves as a working surface for the upper box.

The gloved box is provided with an off-gas exhaust system, which exhausts approximately 10 cfm from each gloved box section. Air entry into the gloved boxes is through 4-inch by 4-inch by 6-inch CWS filters; a negative pressure of 1/2 to 1 inch of water is maintained. The box exhaust is filtered through two series CWS filters in the off-gas exhaust system before being expelled into the building exhaust stack.

The middle 8-ft dry chemistry gloved box section houses the plutonium and neptunium finishing equipment. A precipitator, filter, and filtrate catch tank are provided for plutonium concentration. An anion exchange column and associated tanks is provided for product recovery from the filtrate solution. A precipitator, filter, filtrate catch tank, and calcining furnace are provided for producing neptunium dioxide. Tanks and precipitators are located in the lower gloved box section; valves, piping, filters, and the calcining furnace are located in the upper section.

A cask unloading station and a plutonium product loading station are installed in an adjacent 8-ft gloved box section. The cask unloading station is identical to the product loading station in the High Level Caves processing facility.

The plutonium product loading station is a 24-inch by 16-inch by 12-inch deep stainless steel box equipped with a "Plexiglas" front. Process piping connections for product transfer are provided. A shipping container gloved box can be attached to the station via matching 12-inch bag rings.

Process Equipment

Tanks

The finishing facility process tanks are fabricated from type 304L stainless steel pipe and plate. These tanks include:

Tank No.	Volume, liters	Description
T-1	2.5	Plutonium filtrate catch tank
T-2	50	Waste hold and column feed adjust tank
T-3	50	Column feed tank
T-4	5.5	Column elution displacement and tails tank
T-5	5.5	Column elution product tank
T-6	5.5	Neptunium filtrate catch tank
F1H	0.6	Plutonium product tank
FWT	175	Neptunium waste hold tank

Dip tubes are installed through the top of each tank. The T-1, T-2, and T-6 tanks are equipped with process inlets. Each tank is equipped with a vacuum-vent line, and vacuum is used for process liquid transfers. The T-3 tank is equipped with a 15-psig air line to provide pressure for transferring feed solution through the resin column.

Each tank except the T-3 tank is equipped with a $2\frac{1}{2}$-inch-diameter, 10μ-fritted stainless steel sparge agitator. The sparge heads are piped to a vent valve in the upper gloved box section and are operated by applying vacuum to the tank and opening the sparge vent valve.

Each tank, except the T-3 tank, can be sampled. A sample needle holder for each tank is located in the upper box section. Each sample needle holder is equipped with a hypodermic needle connected to a 1/16-inch polyethylene tube which extends through a 1/4-inch stainless steel tube into the tank. An evacuated pyramid sample vial with a self-sealing diaphragm cap is filled by inserting it over the hypodermic needle. Another needle holder, connected to a vacuum supply, is used for evacuating sample vials. An O-ring sealed cap is provided for each sample needle holder.

All tanks, except the T-3 tank, are equipped with liquid level sight gauges. The sight gauges are 6-mm "Pyrex" glass tubes connected at the bottom and top of the tanks with 1/4-inch "Swagelok" elbows. Each sight gauge is provided with a "Plexiglas" guard and a calibrated meter stick.

The FWT and T-2 tanks are equipped with drain lines connected to the building high level drain. The T-2 tank is equipped with two 500-watt emersion heaters, a cooling water jacket, and a thermometer well. A small water-cooled condenser is provided on the T-2 tank at the vacuum-vent connection.

The F1H tank is equipped with a bottom inlet-outlet line, a vacuum-vent line, a sample line, and a cooling water jacket. A porous stainless steel frit welded over the inlet-outlet connection is provided for vacuum sparge mixing.

Precipitators

Precipitators P-1 and P-2 are fabricated from type 304L stainless steel. The precipitators are fully baffled vessels agitated with simple four-blade paddles driven through reduction gears by Gast air motors. Each precipitator is equipped with process dip lines, process inlet lines, and sample lines.

The plutonium precipitator (P-1) has a capacity of 3 liters. It is shielded with 1 inch of lead and is equipped with a thermometer well and a cooling jacket. A refrigerating unit circulates chilled glycol solution through the cooling jacket.

The neptunium precipitator (P-2) has a capacity of 5.5 liters and is shielded with 1/2 inch of lead. The precipitator is equipped with a cooling jacket, two 250-watt emersion heaters, and a thermometer well.

Filters

The plutonium filter (F-1) is constructed from 3-inch type 304L stainless steel pipe and a 10μ stainless steel frit. The filter is permanently installed and is equipped with a scraper to break up the filter cake and a platinum gauze catalyst to aid in dissolving the cake. The neptunium filter (F-2) is constructed of type 347 stainless steel pipe and frit material. The type 347 stainless steel is used to permit calcining in the filter without contaminating the product with iron. The neptunium filter is installed in a secondary housing and is removable. The capacity of each filter is approximately 350 cc.

Resin Column

The resin column is constructed from "Pyrex" pipe, 2 inches in diameter and 12 inches long, with stainless steel end fittings. The column has a resin capacity of 500 cc. The resin is held between "Teflon" felt retainers. The top felt retainer is mounted in a spring-loaded piston, which keeps the resin in a packed bed.

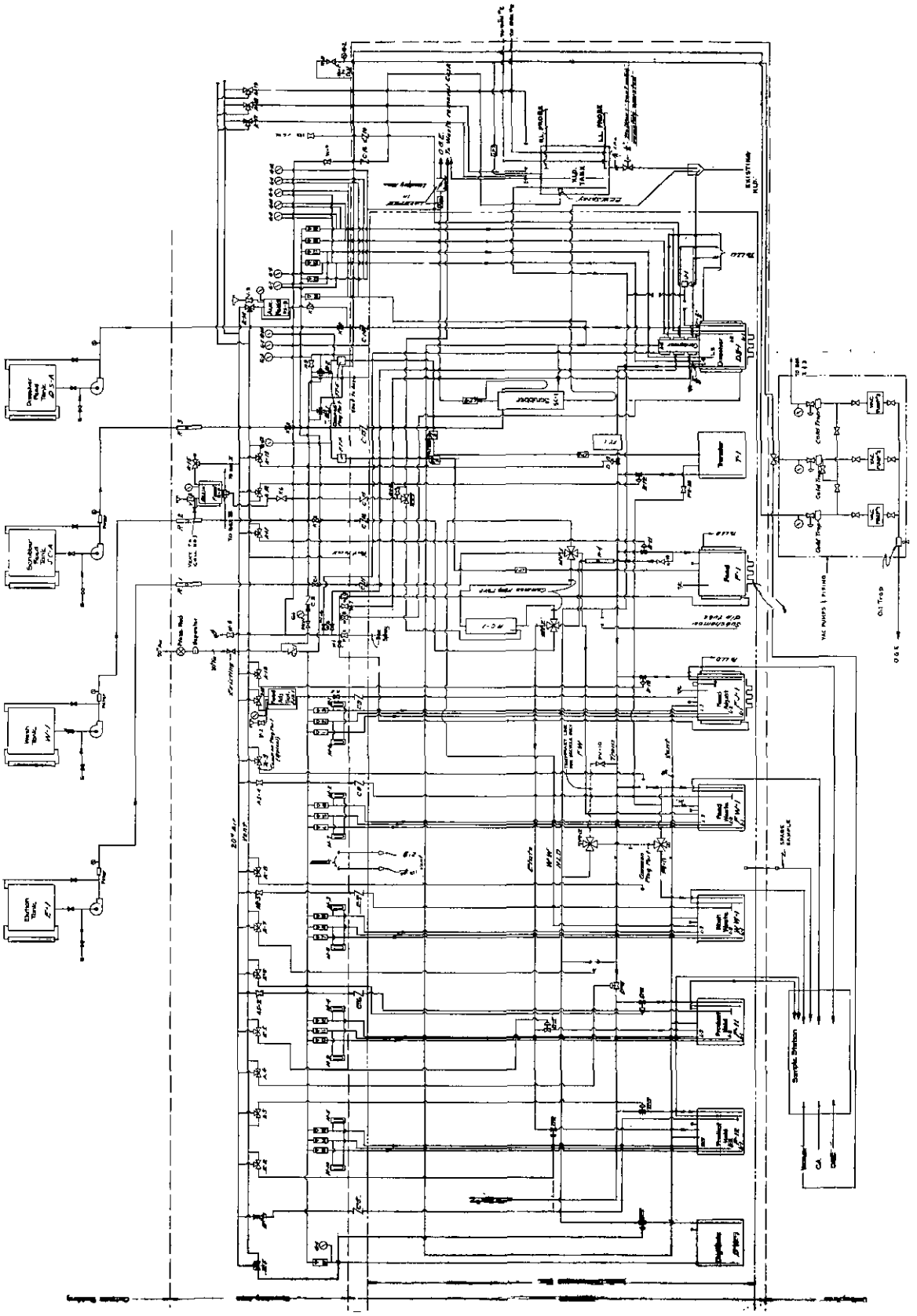
Valves and Piping

Figure A-5 in the Appendix shows the finishing facility piping diagram and a table describing the valves and rotameters. The principal valves are stainless steel Republic plug valves with "Teflon" plugs and Hoke and Republic needle valves.

Process piping is fabricated from 1/4-inch type 304L stainless steel tubing and "Swagelok" fittings. Cooling water and refrigerant lines are 1/2-inch tubing.

Separate process, vent, and vacuum headers are provided for the neptunium and plutonium processing equipment. The vent headers discharge to the gloved box atmosphere through liquid traps and CWS filters. The two vacuum lines are connected to a common header through individual liquid traps and CWS filters. A Welch Duo-Seal vacuum pump, located in the lower box section, provides vacuum to the system through a vacuum regulator. A solenoid-operated vent valve is installed in the common vacuum header. The vacuum pump power supply and the vent valve solenoid are interlocked with conductivity probes in the liquid traps. If process liquid is drawn into either trap, the pump will automatically become de-energized and the solenoid valve will vent the header.

The P-1 precipitator and the T-1 and T-3 tanks are equipped with pressure relief valves, which are piped to the off-gas exhaust system through a surge tank and CWS filter.



A-1 PIPING SCHEMATIC FOR BOX 1 OF HIGH LEVEL CAVES

Calcining Furnace

A 2000-watt calcining furnace is provided for calcining neptunium oxalate to the dioxide. It is designed to hold the F-2 filter unit and is equipped with gas sweep pipe connections, a cooling jacket, and a thermocouple well. The furnace temperature can be controlled manually with a powerstat or automatically with a temperature controller-recorder.

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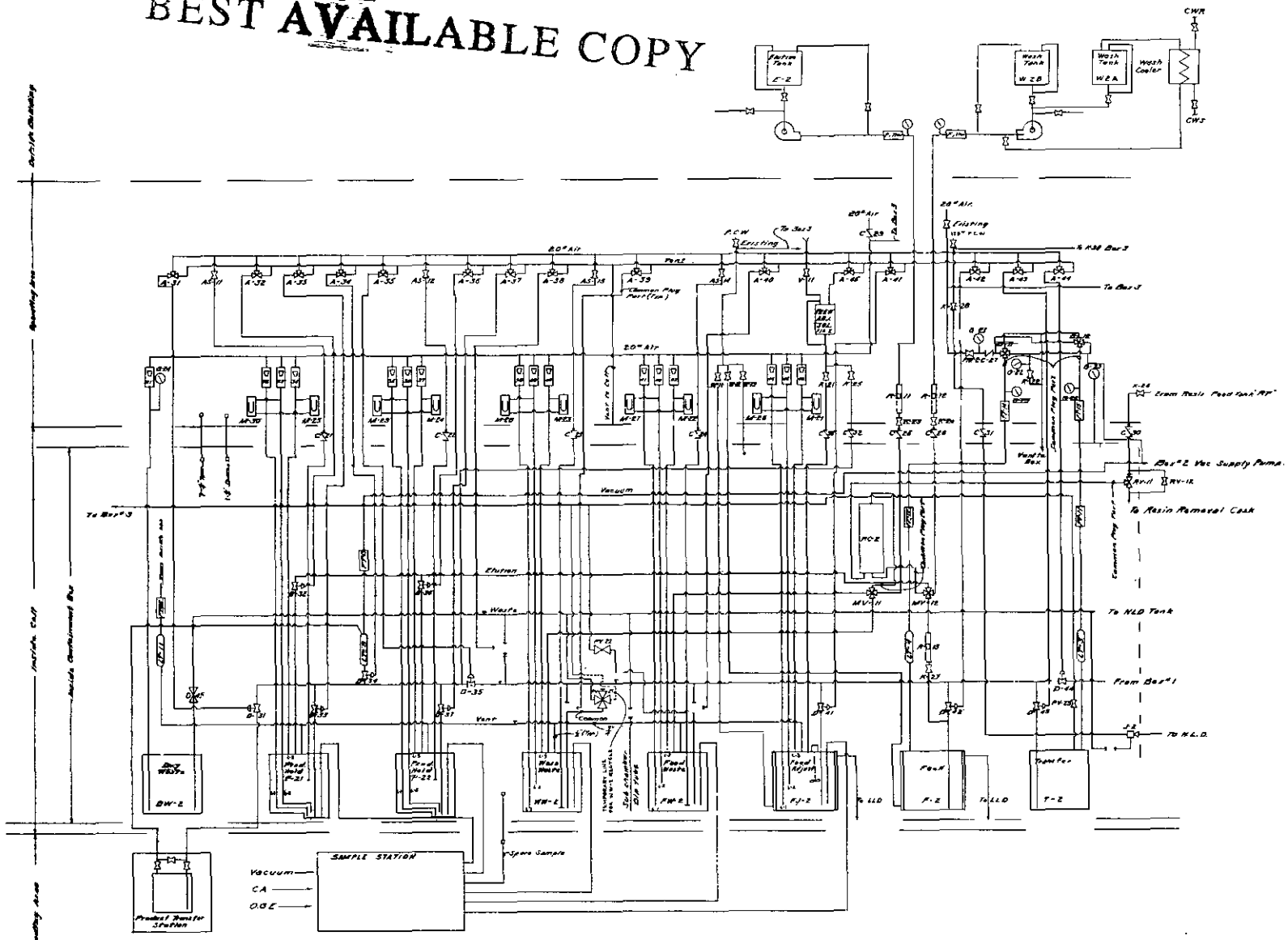
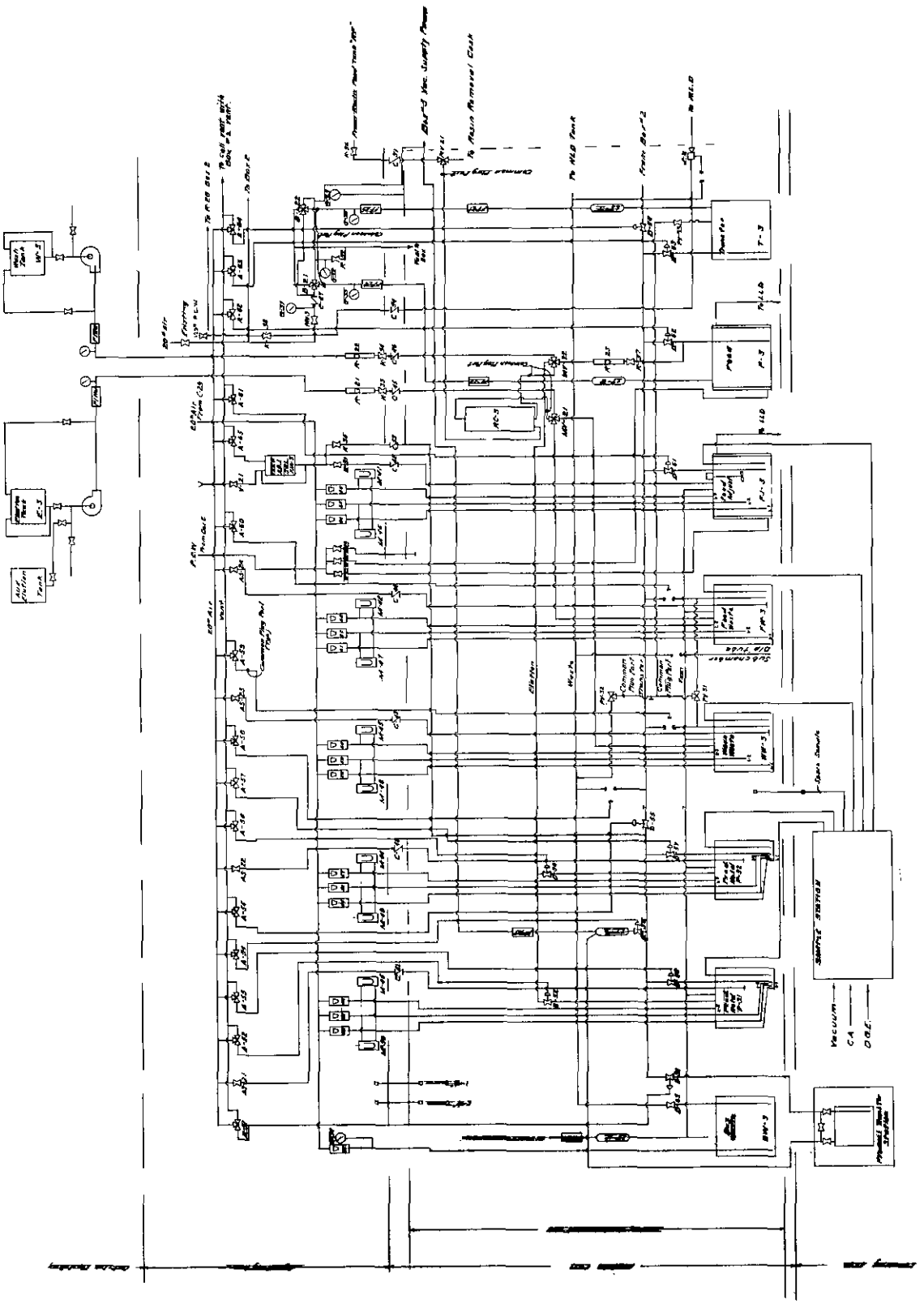
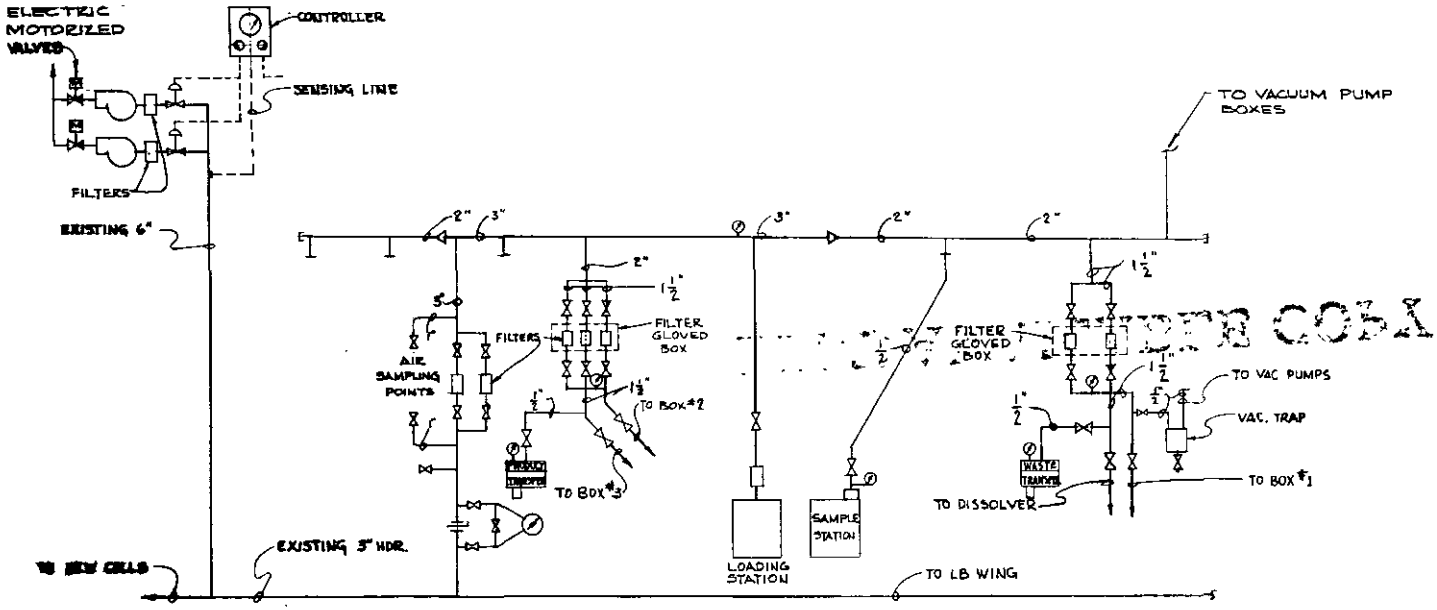


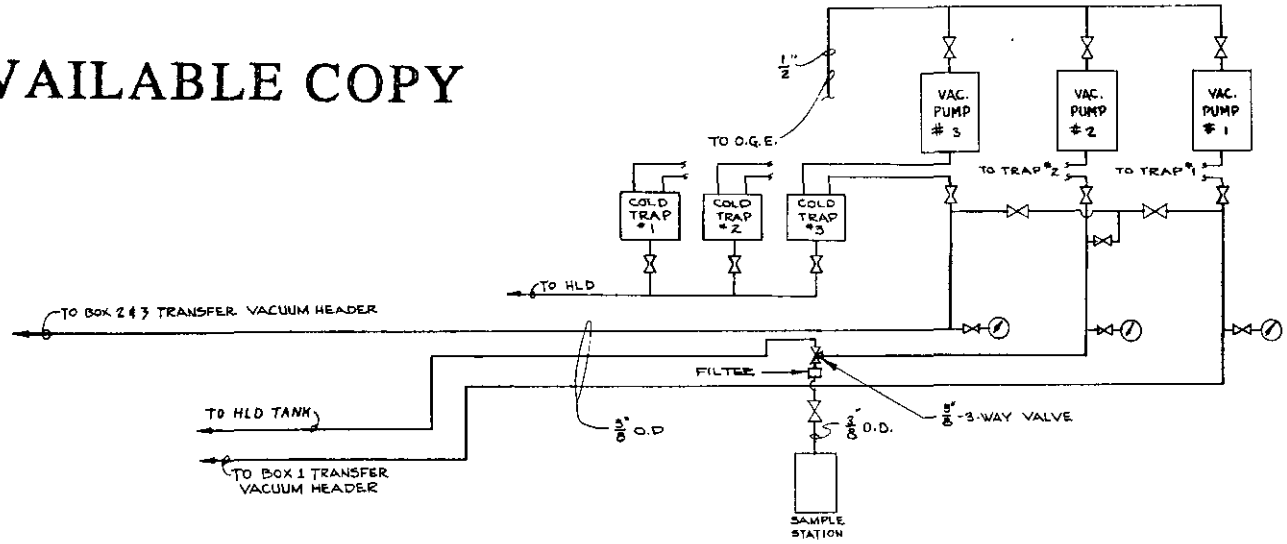
A-2 PIPING SCHEMATIC FOR BOX 2 OF HIGH LEVEL CAVES



A-3 PIPING SCHEMATIC FOR BOX 3 OF HIGH LEVEL CAVES

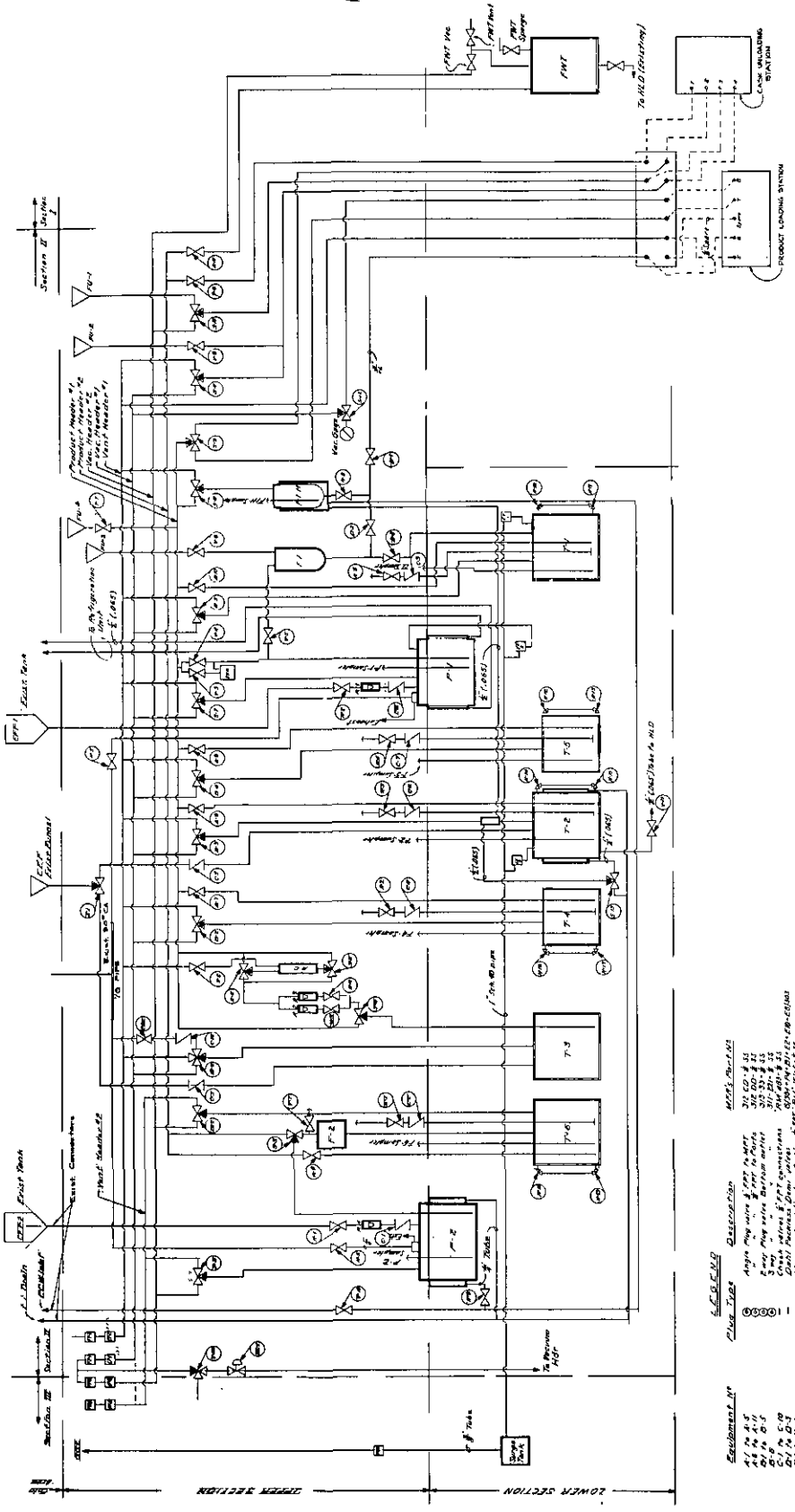


O.G.E. PIPING SCHEMATIC



VACUUM PIPING SCHEMATIC

A-4 PIPING SCHEMATIC FOR EXHAUST AND VACUUM



A-5 PIPING SCHEMATIC FOR DRY CHEMISTRY FACILITY