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ENGINEERING EVALUATION OF
VOLATILITY PILOT PLANT EQUIPMENT

F. W. Miles
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CHEMICAL TECHNOLOGY DIVISION

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ABSTRACT

The ORNL Volatility Pilot Plant engineering experience and operating techniques are compiled for reference. Different systems within the plant are presented separately. For each system, equipment and operating details, evaluations, and recommendations are given. The report covers all operations up to August, 1958.

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1.0 SUMMARY

The Oak Ridge National Laboratory Volatility Pilot Plant was operated from 1956 to 1958 studying the decontamination and recovery of uranium from molten salt reactor fuels. Engineering experience and operating techniques developed during this period of operations are compiled to serve as a reference source.

The salt charging system (a charge melt vessel, heated by an electric furnace, and associated equipment) was used to transfer fuel from small shipping cans into the fluorinator. The principal difficulty encountered was plugging due to inadequate heating.

Molten salt in the fluorinator was sampled with ladles. Even though many modifications were made, the sampling system was never entirely satisfactory: sampling itself was difficult; removal of the sample from the ladle was a problem; dissolution of the sample was difficult; and the samples were not always truly representative of the entire melt.

In the fluorinating system (an L-nickel vessel, electric furnace, and auxiliaries such as instrumentation), uranium was fluorinated to UF_6 and separated from non-volatile fission products and the salt mixture which constituted the bulk of the fuel. Numerous operational difficulties (such as plugging) were encountered, but most of these were, or could be, alleviated by relatively minor design changes and/or revised operating methods. Even though there were shortcomings in the detailed design of the system, the basic design was satisfactory.

The Complexible Radioactive Products (CRP) trap was designed to remove some of the contaminants (such as zirconium, niobium, and chromium fluorides) from the UF_6 stream leaving the fluorinator. The last design used (a bed of NaF pellets at $400^\circ C$) worked well except that the necessary uniform temperature was hard to obtain and the problem of removal and disposition of the contaminated pellets had not been solved.

All UF_6 piping was enclosed in a heated duct to prevent UF_6 condensation and to contain escaping UF_6 in the event of a leak. In spite of nonuniform temperature distribution, operation of the system was satisfactory.

An absorption system utilizing NaF pellets (absorption of UF_6 and fission products at $\sim 100^\circ C$ followed by desorption of UF_6 at $\geq 390^\circ C$) was used for final decontamination of the UF_6 stream. The system was generally satisfactory for the program in which it was used, but the long heating and cooling times required and the difficulty of handling contaminated NaF would necessitate redesign of the equipment for more efficient scheduling and/or processing of higher activity fuels.

Decontaminated UF_6 was condensed in the cold trapping system. This system included two cold traps in series (-40° and $-55^\circ C$) and a chemical trap (NaF at room temperature) to ensure against loss of the UF_6 . The cold traps had built-in heaters for product transfer. In spite of the inability to reach the design temperature of $-62^\circ C$ in the second cold trap and of occasional Freon vapor locks caused by heating the traps, operation of the system was satisfactory.

Product UF_6 was collected by evacuating inerts from the cold traps, heating the cold traps to liquefy the UF_6 , and transferring the product to a 5-in. shipping cylinder. The operation required about twelve hours, and the portion of product which had to be recondensed in the cold traps and held until the next run was extremely erratic; other than this, the system was satisfactory.

In the scrubbing system, excess fluorine in the off-gas stream was neutralized with KOH solution in a spray tower before the inert gases were discharged through the building off-gas header to the stack. The only significant difficulty encountered was occasional plugging of the gas inlet line, believed to have been caused by evaporation of KOH-KF solution adhering to the gas inlet pipe.

Product UF_6 was sampled by melting the contents of a shipping cylinder and draining a portion into a sample tube (or tubes) mounted on a special apparatus. The necessary samples were obtained, but the operational difficulties in obtaining a sample and the air activity resulting from the sampling made the system barely adequate rather than truly satisfactory.

After completion of fluorination, the salt remaining in the fluorinator was disposed of through the waste salt system. Waste salt was transferred by pressure through a transfer line into a disposable waste can, where it froze. This can, in its shielded carrier, was transported to the burial ground for permanent burial. Difficulties were encountered with plugs in the transfer line (primarily at the fluorinator, in the freeze valve vents, and at the exit nozzle) and with salt splatter from the space between the exit nozzle and the waste can, but operations were improved by modifications. The system was satisfactory for the activity levels involved, but would be inadequate for high activity levels.

Nitrogen was used as the inert gas, and fluorine was the fluorinating agent. The supply systems for these two gases incorporated several special features, such as a dryer in the N_2 system, and HF trap in the F_2 system, and extensive instrumentation. In prior work with fused salt fuels, helium had been specified as an inert gas; however, no difficulties were encountered with nitrogen. With proper materials, equipment, and operating techniques, both gas systems could be operated with no major difficulties.

The ARE system was designed specifically for removing the Aircraft Reactor Experiment fuel from its dump tank, transferring it in a single batch to a hold tank, and then transferring the fuel in six batches to the fluorinator. It was necessary to maintain fuel in the hold tank in a molten state for about half a year. There were no major difficulties with this system.

Valves used in gas lines were commercial units; those used in molten salt lines were line plugs formed by freezing a reservoir of salt. An extensive development program was necessary to improve some valve designs to the point where they met operating requirements. For future programs, the air operated on-off valves should be completely redesigned; all other types of valves (both commercial and "freeze" valves) were satisfactory.

A flange leak-detecting system, basically a pressure-monitored nitrogen buffer at the ring joint, was installed on some of the flanges. This system identified leaking flanges and ensured that any leakage was nitrogen rather than direct leakage between the process and atmosphere. The system worked very well and, in the future, will be extended to all other critical flanges.

All joints (except the above flanges) were tested by fluorine pressure with KI-starch to indicate leaking fluorine. After repair of all leaks discovered by this method, the over-all system was tested by pressure drop. This test showed that the leakage was no greater than 0.035%/min at 20 psig, and was probably much less.

All heating in VPP was electrical. Autoresistance heating was used for molten salt transfer lines. For this, low-voltage (~7 v) high-amperage (~435 amp) power is put directly on the pipe itself; the electrical resistance of the pipe results in the pipe serving as a heating element. The only major difficulty was in attaining uniform temperature distribution. All heating requirements other than for molten salt lines were met by conventional resistance heaters either as commercial heaters or furnaces or as commercial heating elements locally formed into heaters. All electrical heating was quite dependable and satisfactory.

Radiation safety required continuous vigilance. The most serious problem encountered was air borne α contamination, which periodically necessitated the use of masks. The highest average exposure reported for any week was less than 50 mrem/man, and there were no over exposures.

2.0 INTRODUCTION

The Oak Ridge National Laboratory Volatility Pilot Plant was constructed in 1955 and 1956 (1) to aid in developing the Fluoride Volatility Process (2) and to decontaminate and recover the uranium in the Aircraft Reactor Experiment (ARE) (3) fuel. The pilot plant was operated intermittently from 1956 to 1958. The results obtained during this period of operation have been reported from a process standpoint (4, 5, 6). Corrosion experience is the subject of a separate report (7). These reports summarize all data which are pertinent from either a process or a corrosion standpoint, and thus present some equipment performance and engineering data. However, there is no previous publication reporting the engineering experience in any complete form.

This report is a compilation of all available engineering experience gained in operating the ORNL Volatility Pilot Plant. Data have been extracted from topical reports, status and progress reports, log books, unrecorded observations of operating personnel, etc. It is intended primarily as an equipment reference source.

In format, the equipment is divided into twenty-one logical systems. The eleven sections from the salt charging system (Section 3) through the waste system (Section 13) parallel the flow of material through the plant. Subsequent sections cover service and special equipment such as the Nitrogen System (Section 14) and Radiation Safety Including Criticality (Section 23). For each system a table of contents is presented, details of the equipment pieces are recorded in a table,^a and the operating procedure is given along with critical operating steps. Next, each component is thoroughly evaluated with pertinent cross-referencing in the equipment table. Then the equipment evaluation is summarized, conclusions are drawn, and recommendations are recorded. And finally, essential appendices are included.

It is expected that this report will be the initial reference in any search for engineering experience on all operations to date in the Volatility Pilot Plant, that at least ninety per cent of such searches can be completed with no additional reference needed, and that references will be given for all others.

Basically, the ORNL Fluoride Volatility Process involves contacting a uranium-bearing molten salt fuel with elemental fluorine, further decontaminating the evolved UF_6 by an absorption-desorption cycle in sodium fluoride, and cold-trapping and recovering the UF_6 . Through all runs, the usual batch weighed ~150 kg, had a volume of ~52 liters, and contained ~10 kg of uranium. A flow-sheet of the process is shown in Fig. 2.1,

The ORNL Volatility Pilot Plant is located in Cells 1 and 2 of Building 3019 and in adjacent areas. The physical layout is shown in Figs. 2.2 and 2.3. Building 3019 was originally constructed for the bismuth phosphate process for

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^aFor simplicity, trade names are used for equipment pieces where these are better known than the generic terms. Examples are: (a) Variac or Powerstat instead of autotransformer and (b) Pyrovane or Wheelco rather than temperature controller. The equipment numbering system is delineated elsewhere. (8, 9).

Fig. 2.1. Flowsheet for ORNL Fluoride Volatility Pilot Plant.

Fig. 2.3. Typical Section, 3019 Bldg.

plutonium recovery. The building has since been used as a pilot plant for several fuel processing programs. During the period of volatility operations in the east end of the building, the Thorex Pilot Plant was operated in the remainder of the cells.

Cells 1 and 2 are 27 ft high by 19 ft north-south. Cell 1 is 11 ft east-west, Cell 2 20 ft east-west. Shielding is provided by concrete. North walls are 5 ft thick, south walls 4 ft thick, east and west walls 5 ft thick, and the roof of each cell 4⁺ ft thick. Each cell has a roof hatch 9 ft by 9 ft and a stairway and labyrinth entrance from ground level down to cell floor level. In addition, Cell 1 has a 1⁺ ft thick wall running east-west, forming what is called Cell 1-A (3-1/2 ft by 11 ft) in the south portion of Cell 1. The south wall of the cells forms most of the south wall of the building while the north face is adjacent to the "Operating Gallery". The area over the cells is enclosed, forming the "Penthouse", and is used for some nonradioactive operations as well as for access to the cells through the cell hatch covers (or roof plugs). The Operating Gallery is at a level approximately that of the upper third of the cells; directly beneath the operating level is the "Pipe Tunnel" used for equipment which must be close to the cells, is nonradioactive, and needs little attention.

Initial operations of the pilot plant were in September, 1956. From this date until November, 1957, operations consisted of mechanical check-out of equipment ("M" runs) and "cold" operations ("C" runs) in portions or all of the plant with non-irradiated material. The ARE fuel was processed ("E" runs) from November, 1957, until March, 1958. Following the "E" runs, another reactor charge was processed ("L" runs); some of these "L" runs were "spiked" with plutonium (10, 6) and/or fuel material from in-pile loops in order to make additional decontamination studies. In August, 1958, the pilot plant was shut down for modifications which would permit the processing of heterogeneous fuels with higher levels of radioactivity. This report covers all operations up to August, 1958.

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3.0 SALT CHARGING SYSTEM

3.1 Introduction

The salt charging system was used to feed the molten fluoride salt into the fluorinator. This system handled salt batches varying in size from two to ten gallons and was different from the ARE charging system. The principal steps in operating this system were:

- a. Placing the solid fluoride salt in the charge melt vessel.
- b. Melting the salt in a nitrogen atmosphere and allowing it to drain to the fluorinator.

Radiation exposure was reduced by using a shielded charge carrier and by performing the operation remotely.

3.2 Equipment

Fig. 3.1 gives the equipment arrangement in the salt charging system. To eliminate difficulties, several changes in components were made during the "C" runs. The components of this system and major changes made in them are listed and described in Table 3.1.

3.3 Operation

3.3.1 Operating Procedure^b

Major steps in operating the salt charging system were:

- a. Preparing the salt charge which included putting the salt into the charge cans or crushing the material and bagging.
- b. Loading the salt charge into charge melt vessel and closing the furnace lid.
- c. Purging charge melt vessel and setting other nitrogen purges.
- d. Setting the necessary valves.
- e. Putting the required instruments (including temperature controllers, autoresistance transformers, PC-45, PR-45, LR-2, PR-33, TR-1A, TR-1B, and TI-4) in service and synchronizing the charts.
- f. Heating: (a) the charge melt vessel (to be held at 600°C); (b) the salt transfer line and freeze valve (to be kept $\geq 570^{\circ}\text{C}$); and (c) the fluorinator (to be maintained at 600°C).

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^a Sec. 16.4.1 through 16.4.14.

^b Charging a normal batch (~ 52 l) required ~ 12 hr from "heat-on" FV-102 until the entire charge was in FV-100.

Fig. 3.1. Equipment Arrangement for Salt Charging System

Table 3.1
List of Salt Charging System Components*

Components	Drawings and Sketches		Miscellaneous Information	References
	Drawings	Sketches		
Charge Line (including FV-108 Freeze Valve), MS-102-1 to MS-108-1	E-22767 D-27768 D-22777 D-22922 C-23198 D-23224 D-23225 D-31350	Cell I Equipment Layout Cell I Piping Wiring Diagram (sheet 2, item "F") Furnace Retort - FV-102 Map II Freeze Valve Cell I Piping Detail, (sheet No. II) Freeze Valve and Furnace Installation Cell I Piping	Control Equipment - Pyrovanne (thermocouple on pipeline) Varic and orometer. Period of Use - All runs. Heater Data - A power supply of 7-8 volts and 450 amp. was applied (Secs. 21.2; 21.3; 21.3.1; 21.3.2; and Fig. 21.1). The stub (item 3) shown on Dwg. No. C-23198 was removed.	This Report - Secs. 3.4.1; 3.4.2; 3.4.4; 3.5; 3.6; 5.4.1, item a; 5.5; 5.6; 13.4.2; 13.4.3; 13.5; 13.6; 17.4.4; 17.5.4; 17.6; 21.2; 21.3; 21.3.1; 21.3.2; 21.4.1; 21.4.6; 21.5; 21.6. Figs. 3.1; 21.1. Table 21.1 Engineering File Folder No. F-22. Other Literature - 11, 22 (p. 47), and 12.
Charge Melt Vessel, FV-102	D-22922 D-22923 D-31350 Fig. 3.2	Furnace Retort - FV-102 Retort FV-102 Basket Cell I Engineering Piping "Cold" Salt Charge Can.	Control Equipment - Cf. FV-502; FE-45; FV-45 and LI-9. Period of Use - The type 347 stainless steel vessel was used through Run C-7 and the "L" nickel vessel is the remaining runs.	This Report - Secs. 3.4.1; 3.4.2; 3.4.3; 3.5; 3.6; 5.4.1, item a; 21.4.1; 21.5; 21.6; Fig. 3.2. Engineering File Folder No. F-22. Other Literature - 11, 22 (pp. 2, 3, 5 and 10).
Charge Melt Furnace, FV-502	D-22777 E-25207 D-25208 Vendor Dwg. No. A-862	Wiring Diagram (sheet 3, item "A") Mount for Furnace, FV-502 General Arrangement FV-502 Mount Section Assembly	Control Equipment - Two Pyrovanes interlocked (thermo- couple for one is annulus between FV-502 and FV-102 and that for other on surface of FV-102. Period of Use - All runs Furnace Data - American Electric Furnace Co., Inc., Boston 22, Mass.; Serial No. 9601; Purchase Order, W25X-20420-2; Property No. K-63519; 36 kw; 230 V., 3 phase delta, 60 cycle, one zone; heating elements, No. 5 ea. Chromel "A" 8.3 MSI.	This Report - Secs. 3.4.2; 3.4.3; 3.5; 3.6; 22.4.1; 22.4.2; 22.5 and 22.6. Table 22.1. Engineering File Folder No. F-26. Other Literature - 22 (pp. 3, 11, and 47).
Outlet Pipe Heater, FV-502A	D-22777 D-31350 Vendor Dwg. No. A-862	Wiring Diagram (sheet 3, item "B") Cell I Engineering Piping Section Assembly	Control Equipment - Pyrovanne (thermocouple on pipeline at heater) and Varic. Period of Use - All runs Heater Data - American Electric Furnace Co., Inc., Boston 22, Mass.; 1.25 kw., 35 V., single phase; heating elements No. 7 ea. Chromel "A", 7.3 MSI; Transfer supplied by Vendor.	This Report - Secs. 3.4.1; 3.4.4; 3.5; 3.6; 22.4.1; 22.4.2; 22.5; 22.6. Table 22.1. Engineering File Folder No. F-26.

* Salt charging system was not used during the "E" runs. See Sec. 16.0.

Table 3.1 (Continued)

Components	No.	Drawings and Sketches Titles	Miscellaneous Information	References
Vent Lines Lower Furnace, FV-508	D-22778 D-31350	Wiring Diagram (sheet A, item "C") Cell I Engineering Flow Sheet	<u>Control Equipment</u> - Pyrovens (thermocouple was welded to pipeline about midway of heater) and Varior. <u>Period of Use</u> - All Runs. <u>Heater Data</u> - Envi-Duty Electric Co., Milwaukee, Wis.; Type No. M-5012-8; Serial No. 91370; Property No. X-63588; 2.5 KW, 115/230 V., 1 phase.	<u>This Report</u> - Secs. 3.4.5; 3.4.1; 3.6; 3.5; 13.4.2; 13.4.3; 22.4.1; 22.4.2. Table 22.1. <u>Engineering File Folder No.</u> F-22. <u>Other Literature</u> - 32 (p. 47).
Vent Lines Upper Furnace, FV-509	D-22778 D-31350	Wiring Diagram (sheet A, item "B") Cell I Engineering Flow Sheet	<u>Control Equipment</u> - Controlled separately by equipment similar to that for FV-508. <u>Period of Use</u> - All Runs. <u>Heater Data</u> - Envi-Duty Electric Co., Milwaukee, Wis.; Type No. M-3012-8; Serial No. 91373; Property No. X-63593; 1.65 KW., 115/230 V., single phase.	Same as for FV-508 above.
Manipulator, FV-908	E-24500 through E-24523 Inclusive	Manipulator Assembly Main - FV-908 Manipulator Parts List, (sheet 2 of 2)	<u>Period of Use</u> - All Runs.	<u>This Report</u> - Secs. 3.4.6; 3.5; 3.6. <u>Engineering File Folder No.</u> F-23. <u>Other Literature</u> - 32 (p. 9).
Fuel Carrier, FV-950	D-18643 through D-18647 Inclusive	Fuel Removal Carrier Fuel Removal Carrier Details (sheet A)	<u>Period of Use</u> - All Runs.	<u>This Report</u> - Secs. 3.4.6; 3.4.7; 3.5; 3.6. <u>Engineering File Folder No.</u> F-24.

- g. Watching LR-2 for indication of feed salt flowing into the fluorinator and maintaining the PR-45 reading at 1 in. of H_2O pressure during the transfer.
- h. After all the salt had transferred, keeping the vent lines furnaces at $650^{\circ}C$ to melt any salt freezing in these lines during transfer.
- i. Cutting off the power to the charge melt vessel, the molten salt line, and freeze valve.
- j. During step i., maintaining PR-45 at 1 in. of H_2O pressure while the equipment cooled.

NOTE: When the salt charge was bulky such as crushed salt, it was necessary to perform more than one salt charging operation to obtain a normal batch of ~150 kg (52 liters).

System modifications necessitated additional minor steps in the procedure. For the complete salt charging procedure, see Secs. 3.7.1 and 3.7.2.

3.3.2 Critical Operating Steps

- a. Avoiding blowing sand seal when placing the charge melt furnace lid to prevent losing sand seal and/or sucking sand into FV-102.
- b. Purging all the air out of FV-102 initially and then maintaining a nitrogen atmosphere in the vessel during the rest of the charging operation by keeping PR-45 at one in. of H_2O pressure to avoid losing the sand seal and/or sucking air into the system. (To remove all air the vessel was purged for 16 hr before starting to heat the equipment.)
- c. Having the vessels and entire charge line hot enough (FV-102 at $600^{\circ}C$, FV-100 at $600^{\circ}C$, and charge line including freeze valve FV-108 at $\geq 570^{\circ}C$) to melt salt in FV-102 or to maintain the salt fluid in the charge line and FV-100.
- d. Sealing FV-108 before fluorination of the batch.

3.4 Equipment Performance^a

3.4.1 Charge Line, MS-102-1 to MS-108-1^b

The charge line included the molten salt line between FV-102 and FV-100 and also the Mark II freeze valve, FV-108.^c This line was similar structurally to the Mark I waste line of Sec. 13.4.2. Operationally, however, there was a difference, that is, gravity transfers were used in the

^a After the "I" runs, the charging line was dismantled and moved to Burial Ground No. 3 (Sec. 23.4.16b). The remaining equipment was retained for future processing.

^b Sec. 21.4.1 and Table 21.1.

^c The Mark I (pot-type) freeze valve design tested in the UNOP Section was never used in the VPP (Sec. 17.4.4).

charge line and either pressure or siphon transfers in the waste line. The gravity transfer usually occurred at one of three rates. These rates with accompanying items of equipment behavior were:

- a. Moderate (most common) - transfer required about one hour; little extra effort involved in maintaining the PR-45 reading at one inch of water pressure; no tendency to blow the sand seal.
- b. Slow transfer required several hours probably because of a partial plug somewhere in the line; no trouble met with keeping the PR-45 reading at one inch of water pressure; no tendency to blow the sand seal.
- c. Fast transfer required less than one-half hour probably because nearly all of the charge was molten in FV-102 before transfer started; much trouble encountered with keeping the PR-45 reading at one inch of water pressure; marked tendency to blow sand seal. There was no "sure-fire" way of obtaining a moderate transfer rate although this was approached by having all of the charge line $\geq 570^{\circ}\text{C}$ when the charge started melting. In some cases, a very slow or a "no-flow" transfer rate was increased by blowing nitrogen through FV-108 and by pressurizing FV-100 with nitrogen. Although this practice was successfully used at times, it cannot be recommended for two reasons: (a) danger of blowing sand seal and (b) possibility of plugging the waste line or instrument dip lines of FV-100. The preferred alternate to this method was having the entire charge line $\geq 570^{\circ}\text{C}$ (Sec. 21.3.2).

Charge line plugs most commonly formed in these places:

- a. Adjacent to FV-502A. Plugs near this heater resulted from cold spots in the line caused by openings in the insulation. Such plugs were eliminated by stuffing Fiberfrax^b in crevices in the insulation. Sometimes such openings in the insulation formed while heating the line to temperature. Consequently, inspection and some patching with the line at temperature were required at times.
- b. Freeze-valve-to-vent lines joints. These plugs were similar to those discussed in Secs. 13.4.2 and 13.4.3.

Early in the pilot plant work the autoresistance ground lug on the FV-102 end of the charge line was welded to the charge line a few inches below FV-502. In such an arrangement, the length of pipe between FV-502A and the autoresistance

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^a Line temperatures were measured with thermocouples placed as described in Sec. 21.4.6.

^b Aluminum silicate vitreous ceramic fiber - ORNL Stores Cat. No. 15-002-3000.

ground lug was too cold to keep the salt fluid. The situation was similar to that created at the fluorinator by putting the lug on the charge line near the vessel. The first remedy in which a tubular heater was added was unsatisfactory. Finally, after other ineffective efforts were made, the nickel autoresistance ground lug was welded to the FV-102 flange as for FV-100.^a This solution was effective and relatively simple to perform.

- c. FV-100 wall. These plugs disappeared after the nickel autoresistance electrode was welded to the FV-100 flange (Sec. 5.4.1a and item b. above).

Although most of the plugs were caused by inadequate heating resulting from misplacing the ground electrode, the plug in Run C-6 evidently was a mixture of composition 108 (56 mole per cent NaF - 37.5 ZrF₄ - 6.5 UF₄) and Zircon ore. Visual observation work performed on such mixtures indicated that higher-than-normal melting points (m.p. of C-108 = 550°C.)⁽¹¹⁾ were possible.⁽¹²⁾ In addition, gas formation (probably SiF₄) and higher-than-normal viscosities were noticed. It was presumed that other fused salt mixtures would be affected similarly by Zircon ore. Finally, higher-than-normal melting points and viscosities were observed with C-108 and corrosion scale mixtures. This corrosion scale was that formed in the type 347 stainless steel FV-102 in early runs (Sec. 3.4.2).

Although a 3/8-in. NPS charge line was never tried, experience with a 3/8-in. NPS charge line should parallel that with the Mark E waste line (Sec. 13.4.2).

The freeze valve loop did not tend to "blow" or lose its seal despite the "swallowing" phenomenon (Sec. 3.4.2). The nitrogen purge lines were apparently effective siphon breakers. The vent line on the FV-100 side of the loop was replaced with a nitrogen purge line to achieve better control of the inert atmosphere in the system. This change also allowed a more positive UF₆ shut-off by a manual valve (V-74C) in the Gallery which replaced HCV-3 in the freeze valve vent.

Prior to the "C" runs, a hole was burned in FV-108 by attempting to weld an autoresistance lug to the pipe containing salt. The salt evidently melted because of local heating and expanded until it ruptured the pipe. Attempts to repair the hole only increased the damage thereby necessitating the fabrication of a new freeze valve.

3.4.2 Charge Melt Vessel, FV-102

The vessel fitted snugly into the charge melt furnace and was its liner (retort). The original vessel made of type 347 stainless steel suffered severe scaling (oxidation) in the presence of heat, air and fluoride fumes from melting salt because of poor sealing of the furnace

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^a The lug was moved to the FV-102 flange after Run C-2. Also cf. item c. below.

lid to the retort (See Vendor's drawing on asbestos strip seal.). Although the scaling was eliminated when a tight closure was achieved with the sand seal, the vessel was replaced after Run C-7 by one made of "L" nickel having a trough for the sand and an N_2 purge inlet near the vessel bottom. The nickel vessel was entirely satisfactory.

The sand seal was satisfactory but had several disadvantages. First, it was rather delicate, capable only of withstanding gas pressure (or vacuum) of <4 in. water. After chrome ore, Zircon ore, and ordinary sand were tried as sealing materials, a mixture of half Zircon ore and half clean sand was found to be the most satisfactory seal. It afforded fair resistance to rupture and fair "self healing" after rupture.

To avoid rupturing the sand seal by pressure surges during operation, a 1-1/2 in. NPS vent with a pressure control valve (PCV-45) actuated by PE-45 was installed between FV-102 and the cell off-gas duct.^a A bypass around the control valve provided emergency venting in case the valve action was too slow. Two small pots were installed in the bypass. The one farther away from FV-102 contained water in which the vent bypass pipe was submerged about three inches to preserve the seal with LE-9 indicating the water level. The pot nearer FV-102 served to catch this water in the event of a negative pressure surge in FV-102. The arrangement was reasonably satisfactory, since the sand seal did not "blow" because of pressure surges during operation and no water got into FV-102. On several occasions vacuum surges in FV-102 were indicated on PR-45 when the salt started to melt and drain by gravity from the vessel. These surges were especially pronounced when the salt began to drain suddenly after the transfer had been delayed by a cold spot in the FV-102 charge line to the fluorinator. This "swallowing" effect could usually be anticipated and counteracted by increasing the N_2 purge rate to FV-102 when the salt transfer was expected to start.

The main difficulty with the sand seal occurred during lifting and lowering of the furnace lid. The lid acted as a large piston, moving the FV-102 atmosphere in the direction the lid was moved. Because of this, the lid had to be set very carefully into the sand seal to avoid blowing sand outward from FV-102 as the lid came to rest. Conversely, the lid had to be lifted very carefully to prevent sucking sand into the vessel. The small capacity of valve PCV-45 to pass gas was responsible for much of this difficulty. Zircon ore was sucked into the vessel in Run C-6 resulting in a high melting plug in the FV-102 drain. The drain pipe had to be sawed open and the plug drilled through from below (Sec. 3.4.1). Although only one such incident occurred during the entire VPP operation, a repetition during an extremely "hot" run would present a very difficult repair problem. Despite the delicate nature of the sand seal, it effectively prevented entry of air into FV-102 since no difficulty from O_2 or water vapor was experienced after it was installed.

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^a Cf. Ref. No. 13 for details of instruments (Table 17.7 and Secs. 14.4.5, 16.4.3, 16.4.4, and 17.4.3).

The basket and charge cans for FV-102 were satisfactory for "cold" runs. Several modifications of the charge cans were made before the final design shown in Fig. 3.2 was developed to suit Y-12 salt filling requirements. The lids were used to retain an inert atmosphere in the cans during filling. At the VPP, the lids were removed, and 4 cans were loaded upside down in the basket for lowering into FV-102. Scale on the charge cans indicated that the N_2 purging in FV-102 did not remove all the air from the inverted cans, although no serious difficulties occurred because of this. During "hot" runs, the basket became highly contaminated from salt that accumulated on it.

3.4.3 Charge Melt Furnace, FV-502^a

The furnace performed satisfactorily throughout the pilot plant runs, heating to the desired temperatures (600°C. normal, 700°C. maximum) without a failure. Time to reach 600°C. from room temperature was 3-1/2 hr; time to cool from 600°C. to room temperature was 20 hr. The original asbestos tape seal for the lid was not satisfactory, allowing air to enter the re-tort as the furnace cooled. Vent and purge connections of 1/2-in. NPS were provided to the lid originally; these were later replaced with 1-1/2-in. NPS fittings (Sec. 3.4.2).

The mount for FV-502, supported on a pivot for swinging the furnace under the hatch, worked well the only time it was needed, that is, when the stainless steel FV-102 was replaced by the nickel vessel.

3.4.4 Outlet Pipe Heater, FV-502A^a

The outlet heater performed satisfactorily without a failure throughout the pilot plant runs. The time-to-600°C. of the pipeline was 5 hr. At times, the pipeline was heated higher than 600°C. with 700°C. being the maximum.

Care was required to maintain thermal insulation around the base of FV-502A because the joint where the outlet pipe insulation ended at the furnace base tended to open when the pipe insulation shrank or slid downward from the furnace. As delineated in Sec. 3.4.1, Fiberfrax was packed in such cracks.

3.4.5 Vent Lines Heaters, FV-508 and FV-509^a

The two vent lines heaters performed satisfactorily without failure. Cold spots tended to occur at the points where the N_2 purge lines joined the freeze valve because furnace FV-508 was originally installed too high above the freeze valve. The situation here roughly paralleled that for FV-506 and FV-507 in Secs. 13.4.2 and 13.4.3 (Cf. also Sec. 3.4.1).

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^a Table 22.1 and Secs. 22.4.1, 22.4.2, 22.5, and 22.6.

Fig. 3.2. "Cold" Salt Charge Can

3.4.6 Manipulator, FV-908

The manipulator in conjunction with the fuel carrier FV-950 was intended to provide remote charging of small cans of radioactive salt. Because of a misunderstanding in the design phase, the manipulator was built to handle a smaller weight than a single charge can filled with salt (~75 lb). Thus, it could not invert the cans and lower them into the melt vessel as intended. Consequently, this operation had to be performed by hand. The manipulator was used to lift and lower the lid of the charge melt furnace and it performed this operation satisfactorily, although the arrangement of the manipulator crank handles were awkward and experience was required to operate them.

A closed-circuit television system was originally purchased to assist the remote charging but was not installed because the activity levels were never high enough to require its use, and because the manipulator could not perform the main operations which were to be observed by TV. The TV would have been helpful in maneuvering the furnace lid, since the manipulator had no index to indicate where its hook engaged the lid handle and since this was not directly visible to the operator when the lid was swung away from the furnace.

3.4.7. Fuel Carrier, FV-950

The carrier was originally designed to handle the aluminum cans into which the ARE fuel was to be poured, although it was never used for that purpose. In the VPP, it was used to transport radioactive loop salt and fragments of ARE components (pump parts and piping) in nickel cans from the Gamma Gardens storage area (southeast corner of Building 3550) to Building 3019 Penthouse, where the cans were lowered into the charge melt vessel. Although it proved awkward to invert the cans before placing them in the fuel carrier (This had to be done manually and required personnel exposure to radiation.), no parts or crumbs of salt fell through the retaining screen in the inverted cans. This operation was intended to be performed by the manipulator over the open charge melt vessel so that crumbs of "hot" salt would fall into FV-102. The carrier handled the cans satisfactorily despite the lack of a convenient means to handle the wires by which the inverted cans were lowered into FV-102 from the carrier.

3.5 Summary and Conclusions

In general, the charge salt system operated successfully and fulfilled its designed function. Some changes were required to correct original flaws, but the over-all design and construction were satisfactory. Time required was 12 hr.

The gravity transfer ordinarily required about an hour although sometimes it was much slower or faster. Having the entire charge line $\geq 570^{\circ}\text{C}$. was usually reasonable assurance of transferring at the one-hour rate.

Charge line plugs usually resulted from inadequate heating at: (a) the freeze-valve-to-vent-line joints, (b) the outlet pipe heater, or (c) the fluorinator wall. The remedy for each of these plugs is discussed. Plugs in b. and c. were largely eliminated by welding electrodes to the FV-100 and -102 flanges.

Although most charge line plugs were caused by inadequate heating, the plug in Run C-6 apparently was a composition 108:Zircon ore mixture having a higher melting point than C-108.

The freeze valve loop did not "blow" or lose its seal despite the "swallowing" phenomenon during transfers presumably because the nitrogen purge lines were effective siphon breakers.

An attempt to weld an autoresistance lug to a pipe containing salt resulted in burning a hole in the pipe.

The original type 347 stainless steel charge melt vessel corroded and was replaced after Run C-7 with one made of "L" nickel. The nickel vessel was satisfactory.

The sand seal and PCV-45 combination was superior to the asbestos gasket originally used to seal the charge melt vessel. The main trouble with the sand seal occurred while lifting and lowering the furnace lid. But a "swallowing" effect during the transfer was potentially troublesome. This effect could usually be anticipated and counteracted by increasing the nitrogen purge rate when the salt transfer started. The sand seal apparently caused one plug when Zircon ore was pulled into the vessel as discussed.

The basket and charge cans were less satisfactory for "hot" than for "cold" runs. This occurred because, through a misunderstanding in the design phase, the manipulator was built to handle a smaller weight than the 75-lb charge can. Consequently, the manipulator was used only to lift and lower the furnace lid. It required experience to operate the manipulator because of the awkward arrangement of the handles.

The fuel carrier handled cans satisfactorily in spite of the two difficulties discussed.

The furnace and heaters were satisfactory and required the following times to heat their respective components to temperature: (a) furnace = 3-1/2 hr, (b) outlet pipe = 5 hr, and (c) vent lines = 3 hr. After flowing the charge to the fluorinator, it required 20 hr to cool the charge vessel to room temperature.

3.6 Recommendations

It is recommended that the salt charging system be used "as-is" unless the activity of the charge salt approaches that for the "hottest" "L" runs in which case an improved means for remotely putting the salt into the charge melt vessel would be needed. Further, the following are recommended:

- a. That the operating procedure should: (a) emphasize having the entire charge line $\geq 570^{\circ}\text{C}$ by the time the charge starts to melt and (b) mention the two most likely cold spots - at the outlet pipe heater and at the freeze-valve-to-vent-lines joints.
- b. That nickel autoresistance ground lugs such as on the fluorinator and the charge melt vessel continue to be put on the vessels.

- c. That welding not be done on pipelines containing salt.
- d. That any future charge melt vessel be made of "L" nickel or a material of equal or better corrosion resistance in fused fluoride salts.

3.7 Appendix

3.7.1 Operating Procedure: Loading Melt Vessel

LMV _____
Date _____
Time _____
Oper. _____

PART I: PRELIMINARY CHECK

- ___ 1. Open or adjust PV-4 to give 4.5 psig on PI-32 on control panel.
- ___ 2. Record FI-3. _____ scfm.
- ___ 3. Check that TR-1A and TR-1B are "on" and operating properly, and that charts are synchronized.
- ___ 4. Record the following temperatures:

TR-1A-1 _____	TR-1B-3B _____
TR-1A-2 _____	TR-1B-5B _____
TR-1B-1 _____	TR-1B-9 _____

(If any of the above temperatures are above 100°C., check with supervisor before proceeding to next step.)

- ___ 5. Check that TIC-1B-9 _____ and TIC-1A-1 _____ are "off".
- ___ 6. Set PC-45 on manual _____ and open PCV-45 to 15 psig _____.
- ___ 7. Open V-83B to give 60% on PR-45.
- ___ 8. Remove cover by raising it slowly straight up with the manipulator.
- ___ 9. Set cover to one side and check to see that the vessel is empty and ready to receive feed.
- ___ 10. Close V-83B.

Checked by _____
Time _____
Date _____

NOTE: Get supervisor's approval before proceeding to Part II, "Loading Salt."

Approved _____
Time _____
Date _____

PART II: LOADING SALT

- ___ 11. If loose material is to be charged, place it in the charge melt vessel first, and record the weight in step 14 below.
- ___ 12. Turn each of the cans to be loaded upside down and lower it carefully into the charge melt vessel.

13. Record the following data:

Time Loaded	Can No.	* True Gross Wt.	*True Drained Wt.	*True Net Wt.
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____

*Corrected for zero reading on scales.

14. Set PC-45 on manual _____ and adjust PCV-45 to 15 psig _____.

15. Read the following instruments: PR-45 _____%, FI-49 _____ in H₂O, LI-9 _____ in.

16. Add or remove water from seal pot until LI-9 reads 3 in.

17. Replace FV-102 cover carefully, using the following procedure:

- Make sure plenty of Zircon sand is uniformly distributed around the periphery of the seal.
- Raise the cover off the rack with the manipulator, swing the cover around and center it over the charge melt vessel, and then slowly lower the cover down into the seal.
- Tamp the cover to make a good seal, making certain the cover is level.
- Clean off (and save) the excess sand on the top of the charge melt vessel.

18. Open V-73A _____, V-80 _____, and V-83A _____.

During the following operations record the requested numbers on data sheet.

19. Open V-83C until FI-35 reads approximately 6%. Time open _____.

20. Open V-83B until FI-35 increases to approximately 12%. Time done _____.

21. Open V-73B until FI-35 increases to approximately 20%. Time done _____.

22. Approximately 1 hr later close V-73B _____ and V-83B _____. Time done _____.

23. Set FI-13 _____ and FI-25 _____ to 1 cfh each.

24. Set PC-45 on "auto" and adjust set point to 60% (+ 1 in. H₂O pressure).

25. Slowly close V-83C until PC-45 is controlling at setpoint.

26. Record FI-3 _____ slm, FI-32 _____ psig, PR-45 _____. Time completed _____.

By _____.

NOTE: Check with supervisor before proceeding to "Charge Salt Transfer." Upon completion of this, return to Part III of "Loading Melt Vessel," "Retrieving Charge Cans."

PART III: RETRIEVING CHARGE CANS

27. When TR-1A-2 falls below 200°C, open V-83B until FI-35 reads 10% _____.

28. Set PC-45 on manual _____, and open PCV-45 to 15 psig _____.

29. Remove cover by raising it slowly straight up with the manipulator. Time _____.

30. Swing cover away from operator and set it on platform provided.

31. Close V-83B.

32. Pull out cans using crane and place them on blotter paper on the Penthouse floor.

33. Reweigh cans and record weights under "Drained Wt" in step 13 above.

Time completed _____.

By _____.

3.7.2 Operating Procedure: Charge Salt Transfer

Approved by: _____
Shift Supervisor

CST _____
Date _____
Time _____
Oper. _____

1. The following valves on the panelboard should be closed:

HX-8 _____, HX-12 _____, HX-32 _____, and HX-34 _____.

2. The following valves on the panelboard should be open:

HX-6 _____, HX-11 _____, HX-35 _____, and HX-36 _____.

3. The following valves in the gallery should be open:

V-78 _____, V-79 _____, V-80 _____, and V-81 _____.

4. Adjust PV-44 to read 4.5 psig _____ and PV-4 to 4.5 psig _____.

5. The following valves in the gallery should be closed:

V-90A _____, V-90B _____, V-92 _____.

6. The following valves in the Penthouse should be open:

V-27 _____, V-72 _____, and V-112 (or 113) _____.

7. Set the following purges:

Valve No.	FI-No.	Location	Flow Rate	Purge Point
_____	----	Penthouse	1 cfh (ea. side)	LE-2
_____	----	Penthouse	1 cfh	DE-2
-87	27	Penthouse	150 cc/min	S-100-1
-73	13	Gallery	1 cfh	LN-108-1
-74A	14	Gallery	1 cfh	LN-1
-75A	15	Gallery	1 cfh	LN-104-1
-83A	25	Gallery	1 cfh	LN-102-1
-86A	26	Gallery	150 cc/min	LN-106-1
-91A	31	Gallery	1 cfh	LN-103-1

8. Set PC-45 on "auto" and adjust setpoint to +1 in. H₂O (60%).

9. Read PR-45 _____%, LI-9 _____ (In gallery.).

10. Record FI-3 _____ scfm, PI-32 _____ psig.

11. The following instruments should be in service and charts synchronized:

DR-2 _____, LR-2 _____, PR-33 _____, TR-1A _____, TR-1B _____, and
TI 4 _____.

12. Switch TX-1B, EX-1B-4, and TX-1B-7 to position "B."

13. Turn the following controllers on, and adjust the set points as indicated:

Controller	Setpoint, °C.	Time Set
TIC-1A-5	650	_____
TIC-1A-6	610	_____
TIC-1A-8	650	_____
TIC-1B-3B	500	_____
TIC-1B-4B	650*	_____
TIC-1B-11	700	_____

*NOTE: If necessary, raise the set point of TIC-1B-4B until TR-1B-1 is equal to or greater than 550 °C.

14. Adjust the following Variacs:

Variac	Setting	Time Set
TC-1B-4	(140) 145* volts	
TC-1B-11	(190) _____ volts	

*Adjust until EI-1B-4 reads ~435 amps.

15. When above controllers are controlling at the set point, proceed to step 16.
16. Turn the following controllers on, and adjust the set points as indicated:

Controller	Set Point, °C	Time Set
TIC-1A-1	660	
TIC-1A-2	640	
TIC-1B-9	670	

17. Adjust the following Variac:

Variac	Setting	Time Set
TC-1B-9	(140) _____ volts	

18. Time when TR-1A-2 reaches 500°C _____. When TR-1A-2 reaches 500°C, observe closely TI-4-43. If an abrupt rise in temperature occurs, notify supervisor immediately. Also observe LR-2 for buildup of salt in the fluorinator. Record initial reading of LR-2 ____%.
19. Record the time of the following events, if noted:

- Salt begins to melt _____.
- LR-2 shows a buildup _____.
- LR-2 stops rising _____. Final level ____%.
- TR-1A-2 stops rising _____. Final Temperature _____°C.

20. Check FI-13 and FI-14 for flow at 1 cfh each. If either line is plugged, proceed to step 22 _____. Otherwise, proceed to step 25 _____.

21. Turn the following controllers on and/or adjust the set points as indicated:

Controller	Set Point, °C	Time Set
TIC-1B-3B	650	
TIC-1B-7B	650	
TIC-1B-7B	650	

22. Time when TR-1B-3B and TR-1B-7B levels off _____.
23. After 15 min record LR-2 reading _____%, and proceed to step 25 _____.

24. Shut off TIC-1A-1 _____, TIC-1B-3B _____, TIC-1B-4B _____, TIC-1B-7B _____, and TIC-1B-9 _____. Time off _____.

25. Lower Variacs TC-1B-4 _____ and TC-1B-9 _____ to zero volts.

26. Check PR-45 while FV-102 is cooling to see that 1 in. H₂O pressure is maintained.

27. When TR-1B-1 falls below 450°C, and the freeze valve is sealed, proceed to "Feed Salt Fluorination," subject to approval by supervisor.
- Time reached _____.

28. When TR-1A-2 falls below 200°C return to Part III of "Loading Melt Vessel" to retrieve the charge cans.

Time reached _____.

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4.0 MOLTEN SALT SAMPLING SYSTEM

4.1 Introduction

Both the feed and waste salt mixtures were sampled while molten in the fluorinator using the molten salt sampling system. The principal steps in this sampling operation were:

- a. Lowering a weighted ladle into the melt and allowing it to remain long enough to fill with salt.
- b. Withdrawing the filled ladle and processing the sample for analyses.

Radiation exposure was reduced by lead shielding and venting the fluorinator vapors.

4.2 Equipment

The general arrangement of the molten salt sampling equipment was as shown in Fig. 4.1 for all pilot plant work. This figure also shows the latest model of each individual component. The various component models are listed and described in Table 4.1.

4.3 Operation

4.3.1 Operating Procedure^a

Steps in operating the molten salt sampling system were:

- a. Sparging FV-100 with 20 slm of N_2 for 45 min (feed salt) or 30 min (waste salt) through line X-100-1.
- b. Using liquid level (LR-2) and density (DR-2) data to compute the depth to lower the sample ladle.
- c. Placing empty ladle on end of reel wire.
- d. Making certain that the sampler hood was securely in place, and that both air-lock doors were closed.
- e. Setting N_2 purge for the ladle guide pipe to FV-100 and maintaining the pressure in the hood (PI-51) at one inch of water while taking the sample.
- f. Opening V-88; lowering ladle to the predetermined depth; letting ladle stay at this position for ~20 seconds; then lifting ladle to the sampler hood.
- g. Closing V-88 and opening V-129 to a vacuum reading of 2 to 3 inches of water on PI-51.

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^aSampling required ~2 hr including 45-min melt sparge time.

FV-500 lift is not shown.

Fig. 4.1. Equipment Arrangement of Molten Salt Sampling System

Table 4.1
List of Molten Salt Sampling System Components

Components	No.	Drawings and Sketches Title	Miscellaneous Information	References
Sampler	E-26107	Sampler Arrangement and Wiring Diagram	<u>Period of Use</u> - All runs	<u>This Report</u> -
Reel and	D-26114	Sampler Reel Assembly		Secs. 4.4.1, item a; 4.4.1, item f; 4.5; 4.6.
Pedestal	D-26115	Sampler Reel Details		<u>Engineering File Folder No. F-21.</u>
	D-26116	Sampler Reel Details		<u>Other Literature</u> - 52 (pp. 53, 54).
	D-26117	Sampler Reel Details		
	E-26119	Pedestal Frame		
	E-26120	Pedestal Cover Plate		
	D-26121	Details		
	E-26122	Pedestal Assembly		
Ladle Guide Pipe	See Dwg. for the Sampler Reel and Pedestal		<u>Mark IA</u> <u>Period of Use</u> - Through the "C" runs. This model had sharp edges or shoulders within the guide tube as described in Sec. 4.4.1, item b.	<u>This Report</u> - Secs. 4.4.1, item b; 4.4.1, item f; 4.5; 4.6; 5.4.1, item a. <u>Engineering File Folder No. F-21.</u>
	See Dwg. for the Sampler Reel and Pedestal		<u>Mark IB</u> <u>Period of Use</u> - "E" and "I" runs. In this model, the edges and shoulders inside the guide pipe were eliminated as described in Sec. 4.4.1.7.	
Ladle to Wire Connector	D-26113	Sample Ladle and Connector	<u>Period of Use</u> - All runs. The length of the connector varied from 2 1/2 to 4 1/2 inches.	<u>This Report</u> - Secs. 4.4.1, item c; 4.5. <u>Engineering File Folder No. F-21.</u>
Ladle	D-26113 MIT File No. SPS-X-278 Sketches MS-7,6,5,4,3, and 2.	Sampler Ladle and Connector (Mk. IA) Sampler Ladle (Mk. IB) Mark IC, II, III, V, VI, VIII and IX respectively	<u>Period of Use</u> - Graphite ladle through the "C" runs; copper ladle in the "E" and "I" runs. More than ten designs have been tried. The Marks IV and VII designs are unavailable.	<u>This Report</u> - Secs. 4.4.1, item d; 4.4.1, item f; 4.5; 4.6. <u>Engineering File Folder No. F-21.</u> <u>Other Literature</u> - 15, 16.
Sample Carrier, PV-950	D-26107 D-26108 D-26109 D-26110 D-26111 D-26112	Sample Arrangement and Wiring Diagram Sample Carrier Details Sample Carrier Assembly Sample Carrier Details Sample Carrier Details Sample Carrier Details	<u>Period of Use</u> - Not Used (cf. Sec. 4.4.1, item e).	<u>This Report</u> - Secs. 4.4.1, item e; 4.5; 4.6. <u>Engineering File Folder No. F-21.</u> <u>Other Literature</u> - 52 (pp. 9, 53, 59).

Table 3.1 (Continued)

Components	Drawings and Sketches		Miscellaneous Information	References
	No.	Title		
Sampler Hood, PV-955	E-26107	Sampler Arrangements and Wiring Diagram	<u>Mark I</u>	<u>This Report -</u>
	D-26118	Sampler Hood	<u>Period of Use - "C" and "E" Runs.</u>	<u>Secs. 4.4.2; 4.5; 4.6</u> <u>Engineering File Folder No. P-23.</u>
	E-26107	Sampler Arrangements and Wiring Diagram	<u>Mark II</u>	
	D-26118	Sampler Hood	<u>Period of Use - "L" runs. This model had two glove ports, and airtight, an H_2 supply and a vent whereas Mark I did not have these features.</u>	

- h. Disconnecting ladle from reel wire; removing sample from ladle; submitting sample for analyses.

Equipment modifications necessitated minor changes in the procedure. For the complete procedure, see Sec. 4.7.

4.3.2 Critical Operating Steps

- a. Determining accurately the depth to lower the ladle.
- b. Leaving the ladle in the molten salt long enough (~20 seconds) to collect the sample.
- c. Being careful not to kink the reel wire.^a
- d. Being certain that the combination of excessive FV-100 vapor space pressure and HCV-8 open would not cause a plug to form in the FV-100 F₂ inlet line. (Sec. 5.4.1c).
- e. Agitating the melt adequately to ensure collecting a representative molten salt sample.^b

4.4 Equipment Evaluation

4.4.1 Sampler Assembly, FV-900^c

a. Sampler Reel and Pedestal

The sampler reel and pedestal were satisfactory as originally designed, and no change in them was made.

The reel wire was made of nickel. It was very susceptible to kinking unless great care was exercised in winding it on the reel. When kinked, even to a small extent, it was necessary to replace the entire length of wire to operate the sampler.

b. Ladle Guide Pipe

Model differences were:

Mark IA - Threaded joints in guide pipe and untapered shoulder at entrance to guide pipe from the fluorinator vapor space.

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^a Sec. 4.4.1a.

^b Sec. 4.4.1f.

^c After the "L" runs, the sampling equipment was moved to Burial Ground No. 3 (Sec. 23.4.16b).

Mark IB - Butt-welded joints with grinding of welds where possible to eliminate internal shoulders (especially at V-88 and the adjacent tee) and tapered shoulder at guide pipe entrance from the fluorinator vapor space.

These refinements reduced hang-ups of the ladle in the guide tube. Previously hang-ups consumed considerable time, especially at times when attempting to pull the filled ladle from the fluorinator vapor space into the guide tube (Sec. 5.4.1a).

c. Ladle-to-Wire Connector

The connector caused the ladle to sink in the melt. While immersed, the ladle filled with salt. The over-all length of the connector was varied between 2-1/2 in. and 4-1/2 in. Generally, the longer heavier lengths were preferred because these caused the ladle to sink more quickly and presumably more completely.

d. Ladle

More than ten ladle designs described in Table 4.1 were tried. The first few designs were made of high-density graphite and the later ones of thin-wall copper tubing. The graphite ladles were very expensive because of the great amount of machining required. In addition, it was difficult to remove molten salt samples from graphite ladles without contaminating the sample with graphite and spreading radioactivity. The copper ladles were relatively inexpensive, and it was easier to remove samples from the copper ladles as well as to prevent sample contamination and/or the spreading of radioactivity. The ladle in use when the plant was shut down after the "L" runs was Mark IX (6, Fig. 9.1, p. 40). Although this ladle was the most satisfactory of all designs to date, removing the molten salt required careful handling and resulted in some personnel exposure to radiation as described in Sec. 4.4.1f.

e. Sample Carrier

The sample carrier was not used because: (a) the radioactivity of the samples handled could be successfully attenuated using lead bricks inside the hood; (b) the Mark II hood design would not accommodate the sample carrier (This occurred because of the limited time available for designing the Mark II hood.); and (c) the carrier did not contain a wire-cutting mechanism.

f. Discussion

The MIT practice school at Oak Ridge conducted a sampling study using a graphite ladle which was helpful, but the sample contained graphite from the ladle. (14).

The changes made in the sampler assembly improved its operability. However, these shortcomings were retained:

- (1) Two men were required to operate the sampler because of the necessity for carefully threading the ladle into the guide tube from point of entry until the ladle passed through V-88. Whereas it was physically possible for one man to operate the molten salt sampler, this constituted a difficult and time-consuming task because of the position of the reel toggle switch relative to the gloved entry into the sampler hood. A minor redesign could have remedied this situation.
- (2) One-half hour was consumed per sample, principally because of the slow speed of the reel. Providing a two-speed reel could reduce the time per sample considerably. The fast speed would be used while the ladle was in the guide tube except near V-88. However, using the fast speed would accentuate the wire-kinking problem at the reel.

In a technique used to reduce the sampling time, more than one ladle was attached to the reel wire. Although this scheme has been used successfully, it was subject to these troubles: (a) jamming of the ladles in the guide tube and (b) incompletely filling the ladles with salt. Therefore, this technique was never widely practiced.

- (3) Some trouble was still experienced with the ladle hanging in V-88 despite using a gate valve and providing as much open area as possible in the ladle guide tube. Most of the hang-ups were apparently within the valve body which probably could have been reduced by using a larger valve.
- (4) Removing the salt sample from the ladle was not only time consuming but also a radiation hazard to personnel. In the best method found, the ladle was hammered while contained within a piece of Tygon tubing closed at the ends. The powdered salt formed was poured as accumulated into the plastic bottle in which the sample was submitted for analysis. The advantage of the simplicity of this method was reduced by the radiation exposure received by the personnel. In one of the other methods tried, the entire copper ladle and sample were dissolved. Although this method was the simplest for pilot plant personnel and could be used with little radiation exposure, the entire sample was difficult to dissolve.

- (5) The nickel reel wire tended to kink unless handled very carefully.

Whether representative samples were obtained with the molten salt sampler was questionable. In a waste salt sampling study, differences in analyses among three kinds of samples [(a) sampler, (b) flowing stream, and (c) waste can half-section] indicated that the sampler results were low in uranium (15). This finding initiated fluorinator mixing study which showed that a conical bottom with center-spaced draft tube should produce adequate agitation (16). No comparative sample work has been performed on feed salt samples.

Duplicate feed salt samples varied $\sim \pm 1\%$ ($\sim \pm 0.05$ mg/g) and waste salt samples $\sim \pm 20\%$ ($\sim \pm 0.001$ mg/g). The high percentage deviation for the waste samples was attributed to the very low U concentration and to the method of analysis (15).

4.4.2 Sampler Hood, FV-995

Model differences were:

Mark I - No N_2 supply, vent, air-lock, or glove ports.

Mark II - N_2 supply, vent, air-lock, and glove ports.

These refinements to the sampler hood reduced not only the time required to take samples in the "E" and "L" runs but also the radioactivity received by personnel and the release of α contamination in the Penthouse. The sources of air-borne α were: (a) Pu added to runs L-3 and -4, L-6, and L-8 and (b) residual UF_6 in the ladle guide tube.

4.5 Summary and Conclusions

The assemblage of the following components produced a molten salt sampler which was suitable for taking feed salt and waste salt samples in the "L" runs: (a) pedestal and reel, (b) Mark IB ladle guide pipe, (c) 4-1/2 in. ladle-to-wire connector, (d) Mark IX ladle, and (e) Mark II sampler hood. Its chief disadvantages were: (a) it required two men to operate and (b) one-half hour was consumed per sample. This high unit sample time could be reduced by filling more than one ladle at a time, a technique which was not entirely satisfactory. The time required for sampling was ~ 2 hr.

Eliminating shoulders in the guide pipe at the valve (V-88) and at the fluorinator end of the guide pipe reduced hang-ups of the ladle.

The sample carrier was not used because it contained no wire-cutting device and because there was insufficient time to fit it into the Mark II hood design. Actually, the carrier was not needed to reduce personnel exposure to radiation since this was done with lead bricks.

Analyses of waste salt samples taken in different ways left doubt as to the validity of ladle-drawn samples. No such work was done with feed salt samples.

The variability of duplicate feed salt samples was $\sim \pm 1\%$ ($\sim \pm 0.05 \text{ mg/g}$) and of duplicate waste salt samples $\sim \pm 20\%$ ($\sim \pm 0.001 \text{ mg/g}$).

4.6 Recommendations

It is recommended that:

- Any new design using the ladle sampling method require only one operator and include: (a) a larger valve in the ladle guide pipe, (b) a means for remote removal of the sample from the ladle, (c) a two-speed ladle travel as described to reduce the unit sample time, and (d) shielding other than by lead bricks to enable handling samples of higher activity.
- Further study be done on the validity of waste salt samples taken by the ladle method and study also be done on the validity of feed salt samples taken by this method.
- A study be performed to see whether the method of molten salt sampling should be changed.

4.7 Operating Procedure: Molten Salt Sampling

FSP _____
Date _____
Time _____
Operator _____

- Record LR-2 reading _____%, DR-2 reading _____%.
- Compute density of liquid: First multiply DR-2 reading (in %) by 0.005.
$$(\text{DR-2 reading}) (0.005) = (\quad) (.005)$$
- Add 3.0 to answer in (2) to give actual density.
$$3.0 + \quad = \quad \text{(Should be between 3.0 and 3.5).}$$
- To correct LR-2 liquid level for this density, divide the % reading on LR-2 by the answer in (3).
$$\text{Corrected level} = \frac{(\text{LR-2}\%)}{\text{Density from (3)}} = \frac{(\quad)}{(\quad)} = \quad$$
- To obtain cm of salt multiply answer in (4) by 2.03.
$$(\quad) 2.03 = \quad \text{cm of salt.}$$
- Obtain sampler counter correction by subtracting "cm of salt" from 708.
$$708 - (\text{cm of salt}) = 708 - (\quad) = \quad \text{is counter correction}$$
- Raise toggle switch of fluorinator sampler pedestal until connector hits the stop.
Counter reading = _____ cm.
- To calculate counter reading when ladle is immersed, subtract counter correction calculated in step (6) from counter reading in step (7).
$$(\quad) - (\quad) = \quad \text{cm.}$$

9. Set PV-50 on 2 psig. Close V-129 _____.
10. Check that hood is securely in place and both doors of air-lock are closed.
11. Open V-87C _____, and partially open V-87B _____ until PI-52 reads 1-1.5 inches of water.
12. Open V-119 until PI-51 reads about 1 inch of water.
13. Open V-88 _____. Regulate V-87B to maintain 1 inch of water on PI-51.
14. Depress toggle switch to lower sample ladle.
15. When counter reads the number in (8), release the toggle switch.
16. Wait 20 seconds, then raise toggle switch to retrieve sample ladle.
17. Close V-88 _____, V-87B _____, and V-119 _____.
18. Partially open V-129 _____ until PI-51 indicates 2-3 inches of water vacuum.
19. Disconnect wire and place ladle and wire in air-lock. Close inner door _____.
20. Open outer door and retrieve sample.
21. Attach 14-1/2 in. of 20-gauge nickel wire to ladle and place in air lock _____.
Close outer door _____, open inner door and insert free end of wire into connector and tighten set screws _____.
22. Repeat sampling as per runsheet - record data below:

Date	Time
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

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5.0 FLUORINATING SYSTEM

5.1 Introduction

In the fluorinating system, the uranium was separated from the nonvolatile fission products and salt mixture constituents by volatilization as UF_6 . Some of the volatile fluorides of fission products and melt ingredients - ZrF_4 , NbF_5 , RuF_6 , and CrF_3 - left the fluorinator with the UF_6 . Subsequently, the UF_6 was collected in cold traps while these impurity fluorides were either complexed on NaF beds or retained in the tail gas stream. The principal step in the operation of the fluorinating system was bubbling F_2 into the molten salt to effect the reaction, $UF_4 + F_2 \rightarrow UF_6$, until the uranium content of the salt was < 20 ppm. The operation was performed remotely.

5.2 Equipment

Figure 5.1 is the equipment arrangement of the fluorinating system with the Mark I fluorinator. Two other vessel designs (Marks IIA and IIB) have also been used. The principal differences among the three models are recorded in Secs. 5.4.1a and b. Details relative to the fluorinator and its auxiliary components are listed and described in Table 5.1.

5.3 Operation

5.3.1 Operating Procedure^a

Steps in operating the fluorinating system were:

- Maintaining the temperatures of the feed salt (~ 150 kg of ~ 50 M % NaF-46 ZrF_4 -4 UF_4) in the fluorinator at $\sim 600^\circ C$, the CRF trap at $\sim 400^\circ C$, the heated duct at slightly $> 65^\circ C$, and both absorbers at $65-75^\circ C$.
- Putting the necessary instruments and charts in service and establishing all N_2 purges using 4-1/2 psig N_2 .
- Sampling the feed salt (Sec. 4).
- Setting valves as required and seeing that the cold traps were at operating temperatures.
- Ascertaining that FV-124 and FV-158 were filled with NaF pellets and that FV-650 was running.
- Making certain that the scrubber was operating normally, and that there was adequate scrubbing capacity for a normal fluorination (Sec. 11.3.1).

^aFluorination required ~ 8 hr from first admitting F_2 until the U content of the melt was < 20 ppm.

^bThat is, $> 57^\circ C$, the sublimation point of UF_6 at 14.7 psia (17, p. 4).

Notes: 1. Position of
LA-5 shown on Fig. 6.1.
2. Upper part of FV-100,
FV-501, and FV-503 in-
sulated with 2 in.
Superex.

Fig. 5.1. Equipment Arrangement in the Fluorinating System (Mark I FV-100)

Table 5.1
LIST OF FLOURINATING SYSTEM COMPONENTS

Components	Drawings and Sketches		Miscellaneous Information	References
	No.	Title		
Flourinator, FV-100 (Freeboard ~ 150", Melt Level ~ 24 in. (150 kg salt containing ~10 kg of U ₃ O ₈))	E-21381, Rev. 2	Flourination Vessel Assembly and Details	<u>Mark I</u>	<u>This Report</u> -
	D-23197	FV-100 Flourinator Alterations	<u>Control Equipment</u> - Cf. FV-500. <u>Period of Use</u> - Through the "C" Run.	Secs. 5.4.1; 5.4.1, items a, b, c, and d; 5.4.7; 5.4.5, item b; 5.5; 5.6; 3.4.1; 3.5; 3.6; 6.4.1; 6.4.1, items a, b, c, and d; 6.4.4; 13.4.2, 13.5; 13.6; 15.4.4; 15.5; 16.4.14; 16.5; 18.3; 21.4.4; 21.4.5; 21.4.6. Figs. 3.1 and 4.1.
	D-30361, Rev. 1	Shell Design for Vessel FV-100	<u>Mark IIA</u>	<u>Engineering File Folder No. F-27.</u>
	E-30362	Top Flange Design for Vessel FV-100	<u>Control Equipment</u> - Cf. FV-500. <u>Period of Use</u> - "E" Run; Run L-1 through -4. The record of the Mark IIA design is kept on prints of original tracings in VFT Engineering File Folder No. F-27. These original tracings have now been altered to conform to the Mark IIB design.	<u>Other Literature</u> - 7, 10, 17, 18, 19, and 22 (pp. 2, 14).
	D-31350	Cell I Engineering Flowsheet		
	D-30361, Rev. 2	Shell Design for Vessel FV-100	<u>Mark IIB</u>	
	E-30362, Rev. 1	Top Flange Design for Vessel FV-100	<u>Control Equipment</u> - Cf. FV-500. <u>Period of Use</u> - Run L-5 through -9 and M-62 through -64	
Flourinator Furnace Liner, FV-101	D-19703	Liner for FV-500	<u>Period of Use</u> - All Runs.	<u>This Report</u> - Secs. 5.4.2; 5.5; and 5.6. <u>Engineering File Folder No. F-27.</u> <u>Other Literature</u> - 20 and 22 (p. 14).
Furnace, FV-500	C-24498 D-34089	Flourinator Furnace FV-500 Modifications Wiring Diagram (sheet 3, item "C")	<u>Control Equipment</u> - Two interlocking pyrometers (thermocouple for one in annulus between FV-500 and FV-101 and that for other in the molten salt. <u>Period of Use</u> - All Runs. <u>Furnace Data</u> - American Electric Furnace Co., Inc.; Boston 22, Mass.; Serial No. 9132, Cat. No. F-16243, P. O. No. W25X-88449; Req. No. C-4444; 30 kw., 230 v., 131 amp, single phase, MFR's. Dega. No. Y-2378, X-2337 and Y-737-1.	<u>This Report</u> - Secs. 5.4.3; 5.5; 22.4.1; 22.4.2; 22.5; 22.6; Fig. 3.1; 4.1. Table 22.1. <u>Engineering File Folder No. F-27.</u> <u>Other Literature</u> - 22 (pp. 2, 15, and 47).
Furnace Lift, FV-910	D-25200	Furnace Lift - FV-910 Assembly	<u>Mark I</u>	<u>This Report</u> -
	D-25201	Furnace Lift FV-910 Details	<u>Period of Use</u> - Through Run K-2. This model was manually operated.	Secs. 5.4.4; 5.4.2; 5.5.
	D-25202	Furnace Lift FV-910 Details		<u>Engineering File Folder No. F-29.</u>
	See Mark I		<u>Mark II</u>	<u>Other Literature</u> - 40 (p. 15).
	Drawings Sketch F-1-16	Wiring Diagram for FV-500 Furnace Lift	<u>Period of Use</u> - Run E-3 through -6, L-1 through -9, and M-62 through -64. <u>Furnace Lift Data</u> - Reversing switch, DE Motor, Model No. 526220118, 1/4 H.P., 1725 RPM; 115/230 v., 5.6/2.8A; Boston Reliometer, Cat. No. M21-50-88, MTG A, output 80W +35, output torque ~10.	

Table 5.1 (Continued)

Components	No.	Drawings and Sketches Title	Miscellaneous Information	References
Lower Mantle Heater, FV-501	D-22777 D-22785, Rev. 1	Wiring Diagram (sheet 3, item "D") Details of Fluorinator Upper Heater (Note: This heater was termed "upper" before adding FV-501A. After this time, it was called "lower".)	<u>Mark I</u> <u>Control Equipment</u> - Two interlocking Pyrometers (thermocouples for one welded to vessel wall between heating elements and for other welded to one of the heating elements) and a Variac. <u>Period of Use</u> - All runs through Run L-4. <u>Heater Data</u> - Nine 1000-w., 230-v., G.E. No. 4422 Calrods were attached to FV-100 mantle, 15.5 kw, three-phase delta.	<u>This Report</u> - Secs. 5.4.3; 5.4.6; 5.4.5, items c and d; 5.5; 5.6, 22.4.1, 22.4.2; 22.5; and 22.6. Fig. 4.1. Table 22.2. <u>Engineering File Folder No.</u> P-27. <u>Other Literature</u> - 32 (p. 47).
	D-34889	Wiring Diagram (sheet 3, item "D")	<u>Mark II</u> <u>Control Equipment</u> - Same as for Mark I. <u>Period of Use</u> - Runs L-5 through -9; M-62 through -64. <u>Heater Data</u> - Six 1500-w., 230-v., G.E. No. 4422 Calrods were attached to FV-100 mantle, 15.5 kw, three-phase delta.	
Upper Mantle Heater, FV-501A	D-34889	Wiring Diagram (sheet 3, item "DA")	<u>Control Equipment</u> - Pyrometers (thermocouple welded to the vessel surface between heating elements) and a Variac. <u>Period of Use</u> - Runs L-5 through -9; M-62 through -64. <u>Heater Data</u> - Six 1500-w., 230-v., G.E. No. 4422 Calrods were attached to FV-100 mantle, 15.5 kw, three-phase delta.	<u>This Report</u> - Secs. 5.4.6; 5.4.5; 5.4.5, item a; 5.4.5, item b; 5.5; 5.6; 22.4.1; 22.4.2; 22.5; 22.6. Fig. 4.1 Table 22.2. <u>Engineering File Folder No.</u> P-27.
Fluorinator Instrumentation	D-31350	Cell No. I Engineering Flowchart	<u>Functions of Instruments and Periods of Use:</u> LR-2 Liquid Level All Runs PR-33 Vapor Space Pressure All Runs DR-2 Liquid Density All Runs PA-33 Pressure Alarm All Runs PK-33A Pressure Switch All Runs PA-57 Pressure Alarm All L-Runs; Runs M-62 thru -64 PK-33B Pressure Switch All L-Runs; Runs M-62 thru -64 Thermocouples were put in melt and on surfaces and lines to maintain needed temperatures.	<u>This Report</u> - Secs. 5.4.7; 5.4.2, 5.5; 5.6; 5.3.1; 5.3.1; 13.3.1. Figs. 15.4; 7.2; Secs. 15.4.4a, b, c, and d; 16.4.2; 17.4.2a; 21.4.6. <u>Engineering File Folder No.</u> P-27. <u>Other Literature</u> - 13.

- g. Setting the F_2 flow rate to bubble 20 slm of F_2 into the feed salt.
- h. Cooling the first absorber with compressed air when the temperature in that vessel increased. (A temperature increase indicated that UF_6 was being sorbed by the NaF. Usually cooling the second absorber was unnecessary because essentially no UF_6 passed through the first absorber.)
- i. Continuing to fluorinate until the salt level recorder (LR-2) indicated a decrease of 6.5 chart divisions. (This decrease in liquid level occurred while UF_6 was being volatilized from the salt, reflecting a change in density rather than in melt level.)
- j. Sampling the waste salt. (Sec. 4).
- k. Refluorinating at 20 slm for one hour.
- l. Sampling the waste salt.
- m. Repeating steps j. and k. until the U content of the waste salt was approximately 20 ppm.

System modifications necessitated only minor changes in the procedure. For the complete procedure, see Sec. 5.7.1.

5.3.2 Critical Operating Steps

- a. Keeping the fluorinator pressure below ~3 psig to reduce the tendency to form plugs in the F_2 inlet line.
- b. Maintaining the heated duct temperatures $\geq 65^\circ C.$ to avoid condensing UF_6 in line H-103-1.
- c. Holding the OH^- and the F^- concentrations in the scrubber at ≥ 1.0 molar and ≤ 1.0 molar, respectively, to avoid passing F_2 through the scrubber (Sec. 11.3.2a).
- d. Making certain that the U content of the waste salt was < 20 ppm before finishing the fluorination step.
- e. Cooling the first absorber with air to keep its temperature below 120° to $150^\circ C.$ and, therefore, minimize the tendency to decompose the $UF_6 \cdot 3NaF$ complex.
- f. Holding the CRF trap temperature at $\sim 400^\circ C.$ to reduce UF_6 absorption and/or UF_5 formation.
- g. Keeping cold traps at operating temperatures.

5.4 Equipment Evaluation

5.4.1 Fluorinator, FV-100^a

a. Model Differences - Mark I and II

1. Positions of off-gas line and CRP trap.

Mark I: Off-gas line was on the northwestern side of the vessel near the top of the vessel.

Mark II: In the northwestern quadrant of the top flange.

In both cases, the CRP trap was located in the off-gas line within a few inches of the vessel. In Mark I, the side position of the CRP trap offered no possibility of dumping its contents into the molten salt without physically handling the trap packing material. In Mark II, CRP trap dumping, although physically possible, failed because of two factors: (a) the NaF pellets agglomerated, and (b) the dumping and/or bed vibrating mechanism (Syntron) was inadequate.

2. Vessel wall thicknesses at the supports.

Mark I: 1/4 in.

Mark II: 1/2 in..

During the "C" runs, the metal cross-section of the Mark I vessel warped, becoming slightly elliptical. This condition was most severe at the support ears where internally the major diameter (on east-west line) was 13.513 in. and the minor diameter was 13.217 in. In Mark II, increasing the wall thickness at the support ears to 1/2-in. evidently eliminated the vessel warpage since the metal cross-section remained circular.

3. Locations of the autoresistance ground lugs for salt transfer lines.

Mark I: Individual ground lugs were placed near the vessel wall on each salt line.

Mark II: Common ground lug was placed on the lower vessel flange.

Placing the autoresistance ground lugs on the molten salt lines adjacent to the vessel wall effected cold spots

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^aAfter use, all equipment except the furnace lift was moved to Burial Ground No. 3 (Sec. 23.4.16b). The furnace lift was retained for future processing.

at these locations and, therefore, interfered with salt transfers in the feed and waste lines. In Mark II, a common ground electrode for all transfer lines welded to the lower vessel flange apparently corrected this trouble.^a

4. Nickel pipe stub lengths in waste salt transfer line at the fluorinator wall.

Mark I: 2-in. long.

Mark II: 1/2-in. long.

For both Mark I and II, the nickel pipe stub in the waste salt transfer line at the FV-100 wall created a cold spot which was heated to the desired temperature with FV-500A as described in Sec. 13.4.2.

5. Splash plate and draft tube.^b

In Mark IIA, the splash plate was raised 5 in. and the draft tube shortened 4 in. with the total distance between the bottom of the splash plate and the top of the draft tube being 9 in. greater than in Mark I. Changing the relative locations of the splash plate and draft tube produced no known effect on fluorine consumption, fluorination time, CRP trap plugging, or vessel wall encrustations.

6. Vessel accesses.

Mark I: Access was obtained only by removing the top flange.

Mark II: Limited access was possible through the hand-hole incorporated in the top flange.

7. Lower end of molten salt sampler guide pipe (at bottom of FV-100 top flange, Sec. 4.4.1b).

Mark I: Square end caused ladle to hang on ascent.

Mark II: Tapered end eliminated hanging of the ladle at this point.

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^a Secs. 3.4.1, 13.4.2, 21.4.1, 21.4.3, 21.4.4, 21.4.5, and 21.4.6.

^b The Mark I dispersion nozzle shown on Dwg. E-21381 was a UNOP model and was never used at the Pilot Plant. The draft tube of Dwg. D-23197 was the Mark II design of the gas dispersion device.

b. Model Differences - Mark IIA and IIB

1. Splash plate and draft tube.

Mark IIA: Splash plate; 4-in. NPS draft tube.

Mark IIB: No splash plate; 3-in. NPS draft tube.

This change produced no known effect on F_2 consumption, fluorination time, CRP trap plugging, or vessel wall deposits.

2. Instrument purge line tips.

Mark IIA: Line cut 15° to the horizontal per Dwg. E-30362.

Mark IIB: Internally tapered end per Dwg. E-30362, Rev. 1.

The tapered tips of Mark IIB apparently reduced salt plugs in instrument purge lines.

3. Corrosion specimen nipple lengths were:

Mark IIA: 2-1/2-in. long.

Mark IIB: 7-1/2-to 9-1/2-in. long.

Increasing the distance of the Swagelok thread from the top flange virtually eliminated thread galling.

4. Top flange and hand-hole cover flange leak detector connections.

Mark IIA: None

Mark IIB: Connections were added.

Incorporating the FV-100 top flange and the hand-hole cover flange in the leak detector system enabled continuously monitoring the top flange for leaks. See Sec. 18.3, Fig. 18.1, and Table 18.1 for further details.

c. Plug Formation.

1. The fluorine inlet line.

Most of the inlet line plugs apparently stemmed from maloperation and from the faulty design of other system components instead of from inadequate fluorinator design.^a Therefore, attempted remedies involved improving operating techniques and altering the designs of other systems rather than that of the fluorinating system. Examples of this were:

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^a For the Mark II vessel, a several in. bed of loose asbestos cement was placed on the top flange primarily to reduce salt plugs in the F_2 inlet line. Although the benefit of this measure was not assessed, it was apparently much less than that attainable by eliminating maloperation.

(a) the HCV-7 and -8 interlock switch-over at ~8 psig as discussed in Sec. 5.4.7 and (b) the N_2 - F_2 interlock described in Sec. 15.4.4. The inlet line plugs resulting from the NiF_2 precipitation during Run L-4 were exceptions to the above comment about inlet line plugs. At that time, plugs also occurred in the fluorinator instrument probes, the CRP trap, and the waste line as mentioned in Sec. 13.4.2. This material was difficult to remove from the fluorinator. In fact, removal required transferring through an instrument probe as much as possible;^a freezing the salt; removing the top flange after cutting the dip tubes; and chipping out the hard mass with a hand chisel and a jack hammer. While thus removing the salt, dished head scars ~125 mils deep with external bulges of ~1/16 in. maximum were made. This damage did not affect the utility of the vessel during the five remaining "L" runs and Runs M-62, -63 and -64.

2. The fluorinator instrument probes.

Instrument probe plugs were believed to be significantly reduced by using the tapered tips referred to in Sec. 5.4.1a.

3. The CRP trap.

The inlet and outlet tubes used in the Mark II CRP trap were erroneously thought to have eliminated CRP trap plugging in the last four "E" runs because CRP trap plugging again became chronic during the first four "L" runs (Sec. 6.4.1c). In addition to the inlet and outlet tubes, a CRP trap bypass line (with heater FV-500B) was installed between Runs E-2 and -3 (Sec. 6.4.4).

Gas entrainment studies (Runs M-62 through -64) in the Mark IIB fluorinator indicated that: (a) the upper section should be at a temperature of 400°C or lower to minimize entrainment (18), (b) similar studies should be made in a new-design vessel (18), and (c) freezing and remelting a full salt charge (~150 kg) produced no known vessel damage.

Attempts at fluorinating through the waste line have not been entirely satisfactory. In Run L-8, for example, the entire fluorination was performed through the waste line with little difficulty and with no apparent change in fluorine consumption. In Run L-9, however, fluorinating through the

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^a Only about 1/4 of the salt was removed in this way because a hole in the purge line several inches below the original salt level stopped the transfer.

waste line was unsuccessful because the desired fluorine flow rate could not be maintained. Although no known differences in the condition of the waste line for the two runs existed, a plug was believed to have formed in the unheated section of the waste line above the melt level as mentioned in Sec. 13.4.2. In both the Mark I and II vessels, a rim of salt solidified internally a few inches above the melt level. This solidified salt probably contributed to the plugs in the waste line above the melt level which were mentioned above in this section and in Sec. 13.4.2. Such a situation could present a serious problem during decontamination after processing highly radioactive salt mixtures. In the Mark I design, this was not deemed of major concern because of the low activity of foreseeable feed salts. Consequently, no effort was expended to eliminate this rim. In the Mark II design, however, the mantle heater was redesigned, this measure being partially effective as delineated in Sec. 5.4.5b.

d. Corrosion data.

Fluorinator corrosion data were (7):

1. Mark I Vessel.

Service conditions (all work through "C" runs): Total time, ~1160 hr including ~61 hr with F_2 ; temperature range, 600 to 725°C; salt, NaF-ZrF₄-UF₄.

Findings:

- (a) Maximum attack was in the vapor phase.
- (b) Maximum thickness affected was 73 mils of which 47 mils was metal loss, 21 mils was intergranular penetration of the interior surface, and 5 mils was intergranular oxidation of the exterior surface.

2. Mark II Vessel and Internal Pipes and Tubes.

Rather severe corrosion occurred on the internal walls of the Mark II fluorinator and on its internal components. The attack was mostly intergranular in nature and was considerably more severe where the metal was contacted by the molten salt. It was thought that sulfur, occurring as a contaminant in the molten salt, was responsible for the intergranular penetration. In the three phases of experiments

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^a A record of decontaminating practices is in Secs. 23.4.15b and 23.4.15c.

conducted with the Mark II vessel, the two most significant factors corrosionwise seemed to be: (a) the time of exposure at temperature to molten salts, and (b) the amount of fluorine sparged through the salt. Further detail is available elsewhere (19).

The stainless steel sheathed thermocouples used during the gas entrainment studies (Runs M-62 through -64) corroded badly both in the salt and vapor space. Although severe corrosion was anticipated, stainless steel sheathed thermocouples were used because those with Inconel sheaths were unavailable.

3. Corrosion Specimens in Mark I and II Vessels.

A wide divergence in process conditions occurred. In general, corrosion was greatest at the salt-vapor interface. INOR-2, INOR-8, HyMu-80, Waspalloy, Hastelloy W, and 90 Ni-10 Co have shown promise in individual tests and will be studied further.

5.4.2 Fluorinator Furnace Liner, FV-101

The fluorinator furnace liner was never tested for its primary purpose, that is, to contain the fluorinator charge in case of vessel failure. Such a failure did not occur.

At times the liner shorted out LA-5 thereby falsely indicating a fluorinator failure. Usually this happened when the liquid temperature was greater than the normal temperature of 600°C. Consequently, lowering the fluorinator temperature generally stopped the alarm.

Misalignment of the liner with respect to the furnace caused the liner to contact the Mark II vessel in the southeastern quadrant. This fact probably caused the vessel to be hottest in that region, thereby explaining why the greatest corrosive attack on the Mark IIB vessel was in that vicinity (20,7).

5.4.3 Fluorinator Furnace, FV-500^a

This furnace was satisfactory. The time-to-600°C of the vessel was either ~3 hr in normal operation, that is, with the vessel empty, or ~8 hr starting with a 150 kg charge of equimolar NaF-ZrF₄ at room temperature.

In cooling the vessel, the furnace was lowered in five or more steps to hasten cooling. The rapidity of furnace lowering was governed, however, by the appearance of the vessel. That is, lowering in each step was done only as long as the visible part of the surface of the vessel was at black

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^a Table 22.1 and Secs. 22.4.1 and 22.4.2.

heat. This limit on furnace lowering was necessary to avoid undue thermal shock. In this way, the vessel temperature was reduced to essentially room temperature in ~30 hr.

5.4.4 Fluorinator Furnace Lift, FV-910

Both the Mark I (hand-operated) and II (power-operated) designs of the furnace lift were satisfactory for raising and lowering FV-500. Although the Mark II design eliminated the hard manual work of Mark I, operating Mark II also necessitated entering the cell.

Cell entrance requirement in the Mark II design was deemed necessary to adequately follow the ascent of FV-500 whereas remote operation might have resulted in equipment damage had the vessel contacted the liner. In the Mark IIB fluorinator, misalignment occurred as delineated in Sec. 5.4.2. Later, however, when processing materials of higher activity, remote operation will be mandatory.

5.4.5 Fluorinator Lower Mantle Heater, FV-501^a

a. Model Differences - Mark I and II

The Mark I design is shown on Dwg. D-22785. In the Mark II design the heated portion of the vessel was effectively lowered a few inches from that achieved in the Mark I design, by bending the Calrods symmetrically as compared with the unsymmetrically bent Calrods in Mark I and then placing the bend against the FV-100 insulation support as compared with the "up" position of bends in Mark I. This arrangement lowered the hot portions of the Calrods to the vessel insulation support whereas in Mark I the necessity for bringing the Calrod terminals away from the vessel for electrical connections left several inches of the vessel surface above the insulation support untouched by the hot Calrod surfaces. A compromise was made in Calrod positioning for the Mark II design. That is, the bottoms of the bends were put at the insulation support instead of a few inches lower as desired. This compromise was made because of the difficulty in removing or slotting the support and the need for an insulation support.

b. Discussion

Both Mark I and II were capable of heating the fluorinator wall to 600°C. The Mark II design heated a greater portion of the vessel surface to 600°C, thereby reducing the size of the rim of salt above the melt level. The time required to heat the vessel wall to 600°C was ~3 hr. This time could be reduced by

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^a Table 22.2 and Secs. 22.4.1 and 22.4.2.

setting TIC-1A-11 somewhat higher than the customary 650°C. set point because of the interlocking arrangement of this Pyrovane with TIC-1A-8. Two thermocouples were used: one welded to the vessel surface at the Calrods and the other welded to one of the Calrods (Secs. 5.4.7 and 22.4.2).

5.4.6 Fluorinator Upper Mantle Heater, FV-501A^a

This heater was installed after Run L-4 to keep the upper 15 inches of the fluorinator vessel at 450°C. to reduce the amount of ZrF_4 entering the CRP trap and prevent UF_6 sorption by salt splatterings on this section of the fluorinator. This heater was not needed, however, since the upper 15 inches of FV-100 could be kept at 450°C. without using FV-501A. In addition, no heavy salt or ZrF_4 deposits collected on the upper portion of FV-100 nor on the top flange. Whether the amount of ZrF_4 entering the CRP trap decreased was not ascertained.

5.4.7 Fluorinator Instrumentation^b

LR-2, a pneumatic-type instrument, monitored the liquid level in the fluorinator. The liquid level data were used for: (a) charging the fluorinator,^c (b) calculating the ladle depth for molten salt sampling,^d (c) terminating the first fluorination of a batch,^e and (d) stopping the waste salt transfer.^f LR-2 was almost indispensable for both operations and data work.

LR-2 was relatively satisfactory although it was out-of-service about one-tenth of the time. These operational difficulties occurred:

- a. The high-pressure purge line plugged. The tapered tips added to the submerged purge lines in the Mark IIB vessel apparently reduced the number of instrument purge line plugs.

^a Table 22.2 and Secs. 22.4.1 and 22.4.2.

^b Ref. 13 for details of instrument components (Sec. 14.4.5).

^c Sec. 3.3.1.

^d Secs. 3.3.1 and 4.3.1.

^e The first fluorination for each batch was stopped when the LR-2 reading decreased 6.5 chart divisions (Sec. 5.3.11).

^f Sec. 13.3.1.

- b. The calibration was changed by both vessel and purge line corrosion. An example of such a change in calibration occurred with Mark I vessel during the "C" runs.
- c. At times during maloperation, F_2 and/or UF_6 flowed through the low-pressure purge line to the Main Transmitter Rack. This occurred when the pressure in the vapor space was greater than that of the purge nitrogen.
- d. The reading was spurious with vapor space pressures greater than $\sim 4\frac{1}{2}$ psig because the nitrogen purge flow was interrupted.

PR-33, a pneumatic-type instrument, recorded the pressure in the FV-100 vapor space and was necessary both for operations and data work. Little trouble with it occurred although it was possible for F_2 and/or UF_6 to flow through the purge line to the Main Transmitter Rack as for LR-2. Its "down-time" was practically nil.

DR-2, a penumatic-type instrument, recorded the density of the molten salt. It was used for: (a) calculating the ladle depth for molten salt sampling (Secs. 3.3.1 and 4.3.1) and (b) determining the termination of the first fluorination for each batch (Sec. 5.3.1). DR-2 was more troublesome than LR-2, having been out-of-service about half-time. The operational troubles with DR-2 were similar, however to those with LR-2. In addition, melt solidification could effect the rupture of the DR-2 bellows unless the instrument equalizing valve was open (Sec. 16.4.8). The importance of removing a density instrument from service when the high-pressure probe was thought to be plugged, and also when salt was frozen in that vessel is mentioned in Secs. 14.4.5 and 16.4.16. Generally, instrument purge line plugging with LR-2 was more frequent than with DR-2 since DR-2 had two submerged purge lines to one for LR-2. The high "down-time" of DR-2 necessitated learning how to operate without it. Even though the fluorination could be performed without it, density data were helpful in data-work.

5.4.8 Interlocks

Interlock data are given in Table 5.2.^a Prior to interlocking HCV-7 and -8, these valves were operated independently and plugging of line X-100-1 was more frequent. PA-33 and PX-33A were helpful safety features on the fluorinator during the pilot plant work to date. In the future, however, when the CRP trap plugging problem is solved, these instruments may not be needed.

As for PA-33 and PX-33A, PA-57 and PX-33B may not be needed when CRP trap plugging is eliminated. In one or two runs, PX-33B was disconnected

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^a Cf. also Sec. 15.4.4, items b and c.

Table 5.2

INTERLOCKS AFFECTING FLUORINATION AND/OR FLUORINE FLOW

Valve or Instrument	Action	Purpose	Operational Phase Used
HCV-7 and -8 ^a (See Fig. 5.2)	HCV-7 was closed when -8 was open and vice versa; operated either manually at Main Panelboard or by PX-33A.	To enable fluorinating and to reduce line X-100-1 salt plugs stemming from high FV-100 vapor space pressure.	Waste salt transfer Fluorination
PA-33	Sounded when FV-100 vapor space pressure rose to 8 psig; reset when pressure again fell to 8 psig.	To warn of high pressure in vapor space.	Waste salt transfer
PX-33A	Overrode panelboard control of HCV-7 and -8 by closing -8 and opening -7 when vapor space pressure reached 8-1/2 psig; when pressure again fell to 8 psig, switch reset.	To reduce line X-100-1 salt plugs stemming from high FV-100 vapor space pressure.	Waste salt transfer
PX-33B	Cutoff F ₂ flow when the FV-100 vapor space pressure rose to 4-1/2 psig; when this pressure again fell to 4 psig, reset.	To reduce line X-100-1 salt plugs stemming from high FV-100 vapor space pressure.	Fluorination
PA-57 ^b	Sounded when PX-33B, PX-57, PX-58, PX-59, or PX-60 cutoff F ₂ flow; reset when F ₂ flow started again.	To warn of F ₂ flow cutoff,	Fluorination or any other operation while F ₂ was flowing.
PX-57 ^b	Cutoff F ₂ flow when ΔP_1 (PE-6 reading minus PE-10 reading) decreased to 0.5 psi; when ΔP_1 exceeded 0.5 psi, reset.	To keep F ₂ out of the N ₂ system.	Same as for PA-57

^aSee Sec. 17.4.2, item b for a description of HCV's.^bThese instruments were connected as shown in Fig. 15.5; also cf. Sec. 15.4.4.

Table 5.2 (Continued)

Valve or Instrument	Action	Purpose	Operational Phase Used
PX-58 ^b	Cutoff F_2 flow when ΔP_2 (N_2 pressure in line LN-112-1 minus F_2 pressure in line LN-121-1) decreased to 0.5 psi; when ΔP_2 exceeded 0.5 psi, reset.	Same as for PX-57	Same as for PA-57
PX-59 ^b	Cutoff F_2 flow when ΔP_3 (N_2 pressure in line LN-124 minus F_2 pressure in line LN-121-1) decreased to 0.5 psi; when ΔP_3 exceeded 0.5 psi, reset.	Same as for PX-57	Same as for PA-57
PX-60 ^b	Cutoff F_2 flow when ΔP_4 (N_2 pressure in line LN-MTR minus F_2 pressure in line LN-121-1) decreased to 0.5 psi; when ΔP_4 exceeded 0.5 psi, reset.	To keep F_2 out of the N_2 system.	Fluorination or any other operation while F_2 was flowing

NOTE: See also Secs. 15.4.1, item b and 15.4.2, item b.

^bThese instruments were connected as shown in Fig. 15.5; also cf. Sec. 15.4.4.

Actions of HCV-7 and -8:

1. HCV-7 opened when -8 closed to equalize pressure in the dip tube of line X-100-1 and in the FV-100 vapor space; HCV-7 closed when -8 opened to allow gas to flow through the dip tube.
2. Pressure switch (PX-33A) closed HCV-8 and opened HCV-7 when pressure in the vapor space rose to 8-1/2 psig to reduce dip tube plugs resulting from high pressures in the vapor space.
3. The actions of PX-33B, PA-57, and PX-57 had no effect on HCV-7 and -8. See Section 5.4.7 for these actions.

Fig. 5.2. Arrangement of HCV-7, HCV-8, and Related Fluorinator and Fluorine Instrumentation

to enable completing the fluorination since its removal permitted sufficient F_2 flow at higher than 4-1/2 psig. Generally, however, if F_2 would not flow below 4-1/2 psig, increasing the F_2 pressure was far less beneficial than eliminating the plug causing the high FV-100 pressure.

Whether the PA-57 and PX-33B combination as connected served a useful purpose is debatable. PA-57 probably did since it made operating personnel cognizant of a 4 psig vapor space pressure. PX-33B, however, contributed little by shutting off the F_2 flow. Instead of being helpful, it probably was detrimental. For example, when PX-33B shut off the F_2 flow, it left HCV-8 open and HCV-7 closed thereby not allowing the pressure to equalize between line X-100-1 and the vapor space. Then, unless HCV-8 was closed and -7 opened manually, an increase in vapor space pressure by the instrument purges without a corresponding increase in line X-100-1 pressure would force salt into this line with a plug as a likely result. Because of this possibility of plug formation, it would have been better to use PX-33B as a differential pressure switch operating as recommended.

Several combinations of HCV-7, -8, PA-33, PX-33A, PA-57, and PX-33B have been used. The one discussed above was the most satisfactory despite the fact that when PX-33B shut off the F_2 flow it was essential to manually switch HCV-7 and -8 to avoid F_2 inlet line plugs.

Thermocouples were used for temperature measurement and control in suitable thermowells, on vessel surfaces, and on pipelines (Secs. 21.4.6 and 22.4.2).

5.5 Summary and Conclusions

The fluorination equipment operated relatively satisfactorily. Heating-up and cooling-down times are given in previous sections for the furnace and heaters, and the equipment troubles encountered are highlighted. Time required was 8 hr.

All three FV-100 designs were operable. Increasing the wall thickness at the vessel supports eliminated vessel warpage occurring with a thinner-wall fluorinator.

Locating the autoresistance ground lug on the vessel flange apparently eliminated cold spots on the salt transfer lines near the FV-100 vessel wall.

Removing the splash plate had no known effect on fluorination time, fluorine consumption, CRP trap plugging, or wall deposits on the upper FV-100 wall.

Galling of Swagelok threads on corrosion tubes was decreased by lengthening these tubes from 2-1/2 in. to ~7-1/2 to 9-1/2 in.

Fluorine inlet line plugs resulted primarily from maloperation.

Whether fluorinating through the waste line is feasible was not determined from the limited work done.

Results of gas entrainment studies indicated the desirability of keeping the upper section of the vessel at a temperature of $\leq 400^{\circ}\text{C}$. to minimize entrainment. In addition, similar studies on a new vessel would be beneficial.

Redesigning the lower mantle heater reduced the rim of salt in FV-100 above the melt level whereas the upper mantle heater was not needed to keep the upper vessel wall at 450°C .

Severe corrosion occurred on both vessels. In both cases, the attack was mostly intergranular on the internal walls. In Mark I, the metal in the vapor phase was more severely attacked. In Mark II, corrosion was most severe where the metal was contacted by the molten salt. Apparent factors were: (a) presence of sulfur, (b) the time of exposure at temperature to molten salts, and (c) the amount of fluorine sparged through the salt.

The furnace liner, the furnace, and the furnace lift were satisfactory.

LR-2, PR-33, and DR-2 were useful instruments. LR-2 and PR-33 gave nearly full-time service, but DR-2 was available about half-time because of dip-tube plugs. From an operational viewpoint, the need for LR-2 and PR-33 was clearly established whereas the need for DR-2 was questionable since several runs were made without it. For report information, however, data from all three instruments were beneficial. The tapered tips on instrument purge lines introduced in the "I" runs apparently reduced purge line plugs.

The fluorinator valve and pressure (HCV-7, HCV-8, PA-33, PX-33A, PA-57, and PX-33B) instrumentation discussed was the best arrangement which has been tried. But the PA-57 and PX-33B combination would have been more useful connected differently as recommended.

LA-5 was not evaluated since a fluorinator failure did not occur.

Thermocouples enabled maintaining the necessary temperature throughout the system.

5.6 Recommendations

It is recommended that:^a

- a. The splash plate be eliminated in future designs of fluorinators.
- b. The use of tapered tips on fluorinator instrument purge lines be continued.
- c. The use of the common vessel ground lug for autoresistance heated salt transfer lines on the fluorinator be continued.

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^a Cf. Sec. 13.6 relative to recommendations on eliminating cold spots: (a) in waste line inside of fluorinator vessel and (b) at nickel pipe stub between the fluorinator wall and the Inconel waste line.

- d. The corrosion specimen entry tubes be so designed that the tubing threads (Swagelok) on corrosion specimen assemblies be kept a sufficient distance from the adjacent high-temperature surface to avoid galling.
- e. The new fluorinator be so designed as to eliminate any cold section of the vessel wall on which a rim of salt might form.
- f. A feasibility study be made before deciding to develop a new alloy to replace nickel as the fluorinator material of construction.
- g. The new fluorinator design include an upper section operated at $\leq 400^{\circ}\text{C}$. to minimize entrainment.
- h. The need for a fluorinator furnace liner and LA-5 be re-evaluated.
- i. LR-2, PR-33, DR-2 be incorporated in the new fluorinator design.
- j. The instrumentation of a new-design fluorinator provide for interlocking HCV-7 and -8 such that the latter would close and the former open when the ΔP (line X-100-1 pressure minus vapor space pressure) decreases to 1/2 psi and then reverse when the ΔP increases to one psi (An alarm would sound at a ΔP of 1/2 psi and stop and reset at one psi.); further the $\text{N}_2\text{-F}_2$ interlock should be changed so that HCV-8 would close and -7 open when the F_2 flow was cutoff, and these valves would reverse when F_2 flow was resumed.
- k. The need for any additional safety instrumentation to that mentioned above in item j. should be thoroughly studied before installation.

5.7 Operating Procedure: Feed Salt Fluorination (Rev. April 1, 1958)

FSF _____
 Date _____
 Time _____
 By _____

PART I - PRELIMINARY OPERATIONS

1. Turn on EX-FV-522 _____ and EX-FV-650 _____. Time _____
2. Check that the following instruments are in service, their charts synchronized, and their purge rotameters set: DR-1 _____, FR-1 _____, DR-2 _____, FR-2 _____, LR-2 _____, FR-4 _____, LA-5 _____, LR-4 _____, FR-5 _____, FR-6 _____, FR-8 _____, LR-8 _____, FR-9 _____, PR-10 _____, PR-11 _____, PR-12 _____, PR-16 _____, PR-33 _____, PR-34 _____, PI-38 _____, TR-2A _____, TR-2B _____.
3. Open HX-11 _____, HX-35 _____, HX-36 _____, and HX-8 _____.
4. Adjust PI-6 to 4.5 psig.
5. Adjust FR-1 to 20 slm (80% of full scale). Time on _____.
6. Check that TIC-1A-5 is set on 660°C _____, 1A-6 on 610°C _____ and that TR-1A-6 reads at least 600°C _____. If not, adjust TIC-1A-5, 6.
7. Set controller TIC-1A-3 on 400°C _____ and adjust TC-1A-3 to give the desired heating _____.

8. When TIC-1A-3 attains 400°C. check TR-1C-11 _____. If TR-1C-11 is less than 340°C., increase setpoint on TIC-1A-3 _____.
 9. Record readings on proper data sheet.
 10. Approximately 45 minutes after step 5, close FCV-1 _____.
 Time _____
 11. Close HX-8.
 12. Sample fluorinator _____ times; use runsheet FSP-1:

Code Samples _____ FS 1a to Lamb
 _____ FS 1b to Lamb
 _____ FS 1c to Laing
 _____ FS 1d to Laing
 _____ FS 1e to Feldman
 _____ FS 1f to _____

- 12a. Supervisor sign for sampling _____
 13. When TR-1B-4B and TR-1B-5B drop to 150°C., proceed to step 14.
 TR-1B-4B reading _____ TR-1B-5B reading _____
 14. Close V-88 _____ and V-125 _____ on PV-100 sampler, V-126 _____ and V-127 _____ on cross-over between PE-33 and PE-34 lines.
 15. Adjust these regulators to read 4.5 psig. PV-4 _____, PV-8 _____, PV-44 _____, PV-50 _____, PV-53 _____ should read approximately 15 psig _____.
 16. Normal purge rate should continue to flow through FI-13 _____, FI-14 _____, FI-15 _____, FI-25 _____, FI-26 _____, FI-27 _____, FI-30 _____, FI-31 _____. (FI-27 is on FV-100 sampler.)

PART II - FLUORINATION

17. Open the following valves: HX-11 _____, HX-12 _____, HX-13 _____, HX-14 _____, HX-15 _____, HX-16 _____, HX-30 _____, and HX-34 _____.
 18. Close the following valves:
 HX-8 _____, HX-18 _____, HX-19 _____, HX-22 _____, HX-23 _____, HX-24 _____, HX-25 _____, HX-26 _____, HX-27 _____, HX-32 _____, HX-35 _____, HX-36 _____.
 19. The following controllers should be set:

TIC	Setpoint	Recorder Reading	Time Set
2A-5	80	75	_____
2A-8	75	75	_____
2A-11	130	120	_____
2A-12	145	120	_____
2B-9		120	_____
2B-10		120	_____

20. Adjust the following Variacs:

Variac-TC	Setting	Time Set
2A-5	60	_____
2A-8	60	_____
2A-9	60	_____
2A-10	60	_____
2A-11	60	_____
2A-12	60	_____

21. Wait for all duct temperatures on TI-4, 5 to attain 65°C. before continuing _____.

22. After TR-2A-6 _____ and TR-2A-7 _____ cool (or heat) to 65-75° C, start up scrubber _____.
23. Take scrubber inlet sample _____ SI _____ for OH⁻ and U⁺⁶.
24. Set PC-10 on "auto" and adjust setpoint to 4 psig (20% full scale).
25. Set FC-2 on manual _____ and HX-1 on high range _____.
26. Turn on fluorine supply cut-off switch. Depress energizer button on trailer flow device to initiate flow, then adjust FR-2 to 20 slm (80% of full scale).
The time fluorine was turned on _____.
27. When flow is steady, open HX-8 _____.
28. Close HX-12 _____. Time close _____.
29. Switch controller FR-2 to "auto."
30. When UF₆ begins to appear in the first absorber (temperatures rise on TR-2A-7), turn off TIC-2A-5 and TIC-2A-8 _____.
31. Turn on air sparger to FV-520. Time on _____.
32. Record pertinent data on sheets provided.
If this is the first fluorination on the feed charge, go to steps 33-40.
33. First Fluorination
- Continue fluorination until the liquid level decreased 6.5%.
Initial liquid level LR-2 _____
                                        - 6.5
Final liquid level should be _____
34. Time F₂ flow stopped _____. LR-2 reading _____.
35. Raise the output pressure to full scale on PC-10 (manual) _____.
36. Adjust FR-1 to 20 slm (80% of full scale) _____.
37. Thirty minutes after step 36, close FCV-1. Time _____.
38. Close HX-8.
39. Sample FV-152 _____. Code _____ SI _____.
40. Consult with W. H. Carr before proceeding with DOP or WST runsheets. ^a
41. As soon as DR-2 and LR-2 level off for 30 minutes, shut off fluorine flow _____. Time _____.
42. Turn off air flow to FV-520 when TR-2A-6 and -7 fall to 80°C. Time off _____.
43. Raise the output pressure to full scale on PC-10 (manual) _____.
Lower the output pressure to zero on FR-2 _____.
44. Adjust FR-1 to 20 slm (80% of full scale) _____.
45. Thirty minutes after step 36, close FCV-1. Time _____.
46. Close HX-8.
47. Sample fluorinator 2 times: Code samples _____ WS-1a, _____
WS-1b. If extra samples pulled: Code _____ WS-1c, _____ WS-1d _____.
48. Sample FV-152 _____. Code _____ SI _____.
49. Open HX-12 _____.
50. Set PC-10 on "auto" _____ and adjust setpoint to 4 psig (20% of full scale).
51. Set FC-2 on manual _____ and HX-1 on high range _____.
52. Turn on fluorine supply cut-off switch and depress energizer button on flow device at trailers _____. Adjust FR-2 to 20 slm (80% of full scale). The time fluorine was turned on _____.

^aIf this is a continuation fluorination on the feed charge, go to steps 41-65.

53. When flow is steady, open HX-8 _____.
54. Close HX-12 _____. Time closed _____.
55. Switch controller FR-2 to "auto."
56. When UF₆ reappears in the first absorber (temperature rise on TR-2A-7) turn off TIC-2A-5 _____ and TIC-2A-8 _____.
57. Turn on air sparger to FV-520. Time _____.
58. Sixty minutes after time in (54) shut off fluorine flow. Due off at _____. Actually shut off at _____.
59. Raise the output pressure to full scale on PC-10 (manual) _____. Lower the output pressure to zero on FR-2 _____.
60. Adjust FR-1 to 20 slm (80% of full scale).
61. Thirty minutes after step 60, close FV-1. Time _____.
62. Sample fluorinator 4 times.
Code _____ WS-2a
_____ WS-2b
_____ WS-2c
_____ WS-2d
- If more samples are pulled code _____ WS _____, _____ WS _____, _____ WS _____.
64. Wait for U analysis. Consult with W. H. Carr before proceeding with DOP or WST runsheets.
65. Sample FV-152. Close _____ SI _____. Time _____.

By _____

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6.0 CRP TRAP SYSTEM

6.1 Introduction

During fluorination, the Complexible Radioactive Products (CRP) trap sorbed some of the volatile fluorides (ZrF_4 , NbF_5 , and Cr_xF_y) from the fluorinator exit gases stream while others including UF_6 passed through and flowed to the absorbers. The sorbed volatile fluorides were impurities and were, therefore, subsequently discarded with the NaF sorbent. Operation of the trap was performed remotely. The major operational step was maintaining the NaF packing at $\sim 400^\circ C$ to prevent UF_6 sorption.

6.2 Equipment

All four models of FV-102 (Mark Nos. IA, IB, IIA, and IIB), provided the flow path for fluorinator exit gases to the heated duct. For Mark IIB, the equipment arrangement was as shown in Fig. 6.1. Differences among the various models are described in Sec. 6.4.1a, b and c.

The Mark IA vessel was termed "snow trap" because it was designed to remove ZrF_4 and NbF_5 "snow" from the exit fluorinator gases.^a In the early pilot plant work, chromium fluorides were also found in these gases. This fact initiated the attempt to remove chromium fluorides as well as ZrF_4 with the Mark IA vessel by altering the design. The altered trap (Mark IB) was proved inadequate in subsequent pilot plant runs. After this, the Mark IIA model was built and used starting with the "E" runs. Still later (after Run E-2), minor alterations produced the Mark IIB design. The Mark IIA and IIB vessels were termed Complexible Radioactive Products (CRP) traps because they were designed to remove chromium fluorides and ZrF_4 . Details of the components for the four FV-103 models are listed in Table 6.1.

6.3 Operation

6.3.1 Operating Procedure

Steps in operating the CRP trap system were:

- a. Having the CRP trap packed with 7.0 kg of 1/8-in. NaF pellets and assembled leak-tight.
- b. Holding the temperature of the NaF bed at $\sim 400^\circ C$ during fluorination to prevent sorption of UF_6 .

---

^a ZrF_4 and NbF_5 are subsequently referred to as ZrF_4 .

Fig. 6.1. Equipment Arrangement in the CRP Trap System

Table 6.1
LIST OF CRP TRAP SYSTEM COMPONENTS

Components	Drawings and Sketches		Miscellaneous Information	References
	Nos.	Titles		
Snow (CRP) Trap, FV-103	D-22760	Cell I Engineering Flowsheet	<u>Mark IA</u>	<u>This Report</u> -
	D-23195	FV-103 Fluorinator Snow Trap	<u>Control Equipment</u> - None.	<u>Secs. 6.4.1, 6.4.1, item a; 6.4.1, items b and d;</u>
	D-23223, Rev.1	Cell I Heated Pipe and Duct Detail	<u>Period of Use</u> - All runs through C-8. <u>Trap Packing</u> - Ni mesh.	<u>6.5; 6.6; 8.3.1; 8.4.1, item b; 10.4.12;</u> <u>15.4.3.</u> <u>Engineering File Folder No. F-27.</u> <u>Other Literature</u> - 17, 20 (p. 15).
Snow (CRP) Trap, FV-103			<u>Mark IB</u>	<u>Same as for Mark IA plus Sec. 6.4.2 and</u> <u>Figs. 4.1 and 5.1.</u>
			<u>Control Equipment</u> - FV-503 (Mark I).	
			<u>Period of Use</u> - Runs C-9 through C-15.	
			<u>Trap Packing</u> - 1/8-inch NaF pellets.	
			<u>Mark IIIA</u>	<u>This Report</u> -
	E-30362	Top Flange Design for Vessel FV-100	<u>Control Equipment</u> - FV-503 (Mark II).	<u>Secs. 6.4.1; 6.4.1, item b; 6.4.1, items c, d;</u>
Snow (CRP) Trap, FV-103	D-30363	Snow Trap Details for Vessel FV-100	<u>Period of Use</u> - Runs E-1 and E-2.	<u>6.4.2; 6.5; 6.6; 8.3.1.</u>
	D-30364	Miscellaneous Details for Vessel FV-100	<u>Trap Packing</u> - 1/8-inch NaF pellets.	<u>Engineering File Folder No. F-27.</u>
		Snow Trap		<u>Other Literature</u> - 19, 22, 23.
Snow (CRP) Trap, FV-103	D-31350	Cell I Engineering Flowsheet	<u>Mark IIB</u>	<u>This Report</u> -
		See all	<u>Control Equipment</u> - FV-503 (Mark II).	<u>Secs. 6.4.1; 6.4.1, item a; 6.4.1, item b;</u>
		Drawings for	<u>Period of Use</u> - Runs E-3 through E-9; "L" Runs; and	<u>6.4.2; 6.5; 6.6; 8.3.1; 17.4.1, item g;</u>
		Mark IIA	<u>Runs M-62 through M-64.</u>	<u>18.3.</u>
		ORNL-LB-DWG. (Photo) ARE Snow Trap	<u>Trap Packing</u> - 1/8-inch NaF pellets.	<u>Fig. 6.1; Fig. 6.3.</u>
Snow (CRP) Trap, FV-103	No. 29111	FV-103 Thermowells Assembly		<u>Engineering File Folder No. F-27.</u>
	Sketch No. B-1-9-15	Inlet and Outlet Tubes for FV-103		<u>Other Literature</u> - 19 and 21.
	Fig. 6.3			
Snow (CRP) Trap Heater, FV-503	D-22775	Wiring Diagram (sheet 1, item "J")	<u>Mark I</u>	<u>This Report</u> -
			<u>Control Equipment</u> - None.	<u>Secs. 6.4.2; 6.4.1; 6.4.1, items a, b, and d;</u>
Snow (CRP) Trap Heater, FV-503			<u>Period of Use</u> - Runs C-9 through C-15.	<u>6.5; 6.6; 22.4.1; 22.4.2; 22.5 and 22.6.</u>
			<u>Heater Data</u> - Four 1500W sheath, 1000-w., 230-v.	<u>Fig. 5.1.</u>
			Calrods were connected to single phase power: (a) three	<u>Table 22.2.</u>
			were bent in a "U" shape and equally placed on cylinder	<u>Engineering File Folder No. F-27.</u>
			elements around the trap. (The "U" ends were bent to fit	
Snow (CRP) Trap Heater, FV-503			the trap to heat its bottom surface.) and (b) the fourth	
			was bent around the short line between FV-100 and FV-103.	

(Continued)

Table 6.1 (Continued)

Components	Drawings and Sketches		Miscellaneous Information	References
	Doc.	Titles		
Snow (CRP) Trap Heater, PV-503 (Continued)	D-31358 Sketch No. E-10-18-15	Wiring Diagram (sheet 8, item "B") Arrangement of Calrod Heaters for PV-103	<u>Mark II</u> <u>Control Equipment</u> - Wheeler and Powerstat (thermocouple placed on vessel wall). <u>Period of Use</u> - "E" and "F" runs and Rums M-62 through M-64. <u>Heater Data</u> - Six 1500 ^W sheath, 1000-v., 230-v., Calrods were wrapped helically around the vessel; three-phase delta.	<u>This Report</u> - Secs. 6.4.2; 6.4.1; 6.4.1, items b, c, and d; 6.5; 6.6; 22.4.1; 22.4.2; 22.5 and 22.6 Table 22.2. <u>Engineering File Folder No.</u> P-27.
H-103-1 line Heater, PV-503A	D-31358	Wiring Diagram (sheet 8, item "C")	<u>Control Equipment</u> - Wheeler and Variac (Thermocouple was put on the first flange in line H-103-1.). <u>Period of Use</u> - "E" and "F" runs, Rums M-62 through M-64. <u>Heater Data</u> - ~400 w., 115 v. About 50 feet of industrial heating cable was wrapped helically around the pipeline and flange from the CRP trap to a point several inches inside the heated duct. The advance of the helix was ~1/4 inch per turn.	<u>This Report</u> - Secs. 6.4.3, 6.3.1; 6.4.1; 6.5; 6.6; 22.4.1; 22.4.2; 22.5 and 22.6. Fig. 6.2. Table 22.3. <u>Engineering File Folder No.</u> P-27. <u>Other Literature</u> - 11.
PV-103 By- pass or Line H-100-2 Heater, PV-500B	D-31357	Wiring Diagram (sheet 1, item "C")	<u>Marks I and II</u> These two designs differed only in two respects: (a) period of use and (b) line heated (Fig. 6.3). <u>Control Equipment</u> - Pyromax and Variac. <u>Period of Use</u> - Mark I: Rums E-3 through E-6 and L-1 through L-4. Mark II: Rums L-5 through L-9 and M-62 through M-64. <u>Heater Data</u> - ~400 w.; 115 v. About 50 feet of industrial heating cable was wrapped helically around the pipeline from the top of loose insulation and PV-100 top flange to a point several inches inside the heated duct. The advance of the helix was ~3/8 inch per turn.	<u>This Report</u> - Secs. 6.4.4; 6.3.1; 6.5; 6.6; 22.4.1; 22.4.2; 22.5 and 22.6. Fig. 6.3. Table 22.3. <u>Engineering File Folder No.</u> P-27. <u>Other Literature</u> - 11.

- c. Maintaining the nitrogen purge on the NaF fill line at one cfm to prevent UF_6 condensation in this line.
- d. Keeping the exit gases line H-103-1 at a temperature $>65^\circ C$ to prevent condensing UF_6 ; or keeping bypass line H-100-2 at a temperature $>65^\circ C$ when using the FV-103 bypass.
- e. Changing the NaF in the trap as required.^b

System changes necessitated alterations in the procedure from time to time. Since the CRP trap system operating procedure was part of that for the fluorination system, see Sec. 5.7.1 for the complete procedure.

6.3.2 Critical Operating Steps

- a. Maintaining the temperature of the NaF bed at $\sim 400^\circ C$.

Higher temperatures than $400^\circ C$. caused UF_5 to form which was permanently complexed by the NaF whereas lower temperatures required CRP trap desorption to prevent UF_6 loss.

- b. Keeping a clear flow-path through FV-103 upon which continuing the fluorination depended.

6.4 Equipment Evaluation

6.4.1 CRP (Snow) Trap, FV-103^c

- a. Model Differences - Mark IA and IB

Differences between Mark IA and IB were:

1. Packing

Mark IA - Nickel Mesh

Mark IB - 1/8-in. NaF pellets

The nickel mesh in the Mark IA model was supposed to remove ZrF_4 "snow" from the FV-100 exit gases. The study (Runs M-21 through -48) indicated that: (a) nickel mesh removed a small portion ($<1/10$) of ZrF_4 "snow" from the exit gases; (b) sintered nickel disks plugged and, therefore, could not be used; (c) chromium fluorides as well as ZrF_4 were present in the FV-100 exit gases; (d) NaF was a promising FV-103 packing since it sorbed chromium fluorides as well as ZrF_4 , and since it would not sorb UF_6 at $\sim 390^\circ C$.^d

^a The temperature of $\sim 65^\circ C$. was safely above the sublimation point of UF_6 [$\sim 57^\circ C$. at 14.7 psia (17, p. 4)] .

^b In pilot plant work through the "L" runs, the frequency of NaF bed changing was unpredictable because of operational troubles.

^c After use, all equipment was moved to Burial Ground No. 3 (Sec. 23.4.16, item b).

^d Desorption of UF_6 from the absorbers was performed at $\sim 390^\circ C$. as delineated in Sec. 8.3.1.

As a result of this study, 1/8-in. NaF pellets were used as the bed in the Mark IB and all subsequent FV-103 designs. In addition, the Mark IB vessel was heated with Calrods to reduce UF_6 sorption.

2. Heater

Mark IA - No heater.

Mark IB - Calrod heater, Mark I FV-503.

See Sec. 6.4.2 for details and under "Mark IB and IIA Differences" in this section.

b. Model Differences - Mark IB and IIA

1. Gas Flow Scheme

Mark IB - Downflow

Mark IIA - Upflow

The gas downflow through Mark IB resulted in plugs of NaF pellets in line H-103-1. The pellets were generally blown into this line by high fluorinator pressures accompanying F_2 inlet line plugs. Plugs evidently resulted because of (a) the packing of pellets and/or (b) the sorption of UF_6 by the NaF. With the upflow pattern in Mark IIA, the plugs formed by item (a) did not form but those resulting from item (b) did.

2. Position Relative to FV-100

Mark IB - On the side of FV-100

Mark IIA - Above FV-100

Placing the trap above the top flange made it physically possible to dump the FV-103 contents into the fluorinator. Despite this possibility of dumping, however, the scheme never worked either because of faulty equipment design and/or the agglomeration of the NaF pellets.

3. Vessel Heater

Mark IB - Calrods placed along cylindrical elements and bent under the vessel bottom heated neither cylindrical surface nor bottom of the vessel adequately (Mark I FV-503).

Mark IIA and IIB - Calrods wrapped helically around the vessel heated the cylindrical vessel surface to $400^\circ C$ as desired. Since the trap was welded to the FV-100 top flange, there was no trap bottom to heat (Mark II FV-503).

See Sec. 6.4.2 for further details.

4. Exit Gases Line Heater

Mark IB - Without an auxiliary heater, the temperature of the joint between FV-103 and line H-103-1 was believed to be $<65^{\circ}\text{C}$.

Mark IIA - With heater FV-503A, line H-103-1 and the flange outside of the heated duct were kept above 65°C with no UF_6 condensing.

c. Model Differences - Mark IIA and IIB

1. Flange Leak Detector

Mark IIA - No flange leak detector.

Mark IIB - The FV-103 top flange, the flange on line H-103-1, and the flange on the NaF fill line were added to the flange leak detector system. Having these flanges connected to the flange leak detector system was helpful in leak testing as discussed in Sec. 18.3, see also Fig. 18.1 and Table 18.1.

2. Thermowells

Mark IIA - Contained no internal thermowell for monitoring the NaF bed temperature because a thermowell would have conflicted with the NaF dumping mechanism.

Mark IIB - Two thermowells running the length of the trap were installed, one near the axis of the bed and the other $1/8$ in. from the trap inner surface. The thermowells made it possible to measure temperature profiles of the bed.

3. CRP Trap Bypass

Mark IIA - No bypass for fluorinator exit gases was provided.

Mark IIB - A bypass (line H-100-2) was installed so that the fluorination could be continued if the CRP trap plugged. Previously, a CRP trap plug stopped the fluorination until the plug could be eliminated.

The two CRP bypass designs installed are shown in Fig. 6.2. The Mark I model was unsatisfactory because of plugs developing in V-126 (Hoke No. 413) as delineated in Sec. 17.4.1, item g. The Mark II model was never used and, therefore, cannot be evaluated. Its design appeared to be superior, however, to the Mark I design because the $1/4$ -in. SMMD valve has a larger port than the Hoke No. 413 valve, and because the line size was larger throughout.

Fig. 6.2. FV-103 By-Pass Designs

4. Inlet and Outlet Tubes

Mark IIA - No inlet and outlet tubes. The depth of bed sintering at which no F_2 would flow was around 4 to 5 inches. It was felt that the addition of an inlet tube to disperse the gas vertically in the bed would reduce the shutdowns caused by sintering. In addition, an outlet tube would provide additional outlet area if the top of the NaF bed crusted over.

Mark IIB - Inlet and outlet tubes were provided to increase the effective flowpaths into and out of the trap as shown in Figs. 6.1 and 6.3. Operating experience while using these tubes indicated that the tendency to form plugs during maloperation outweighed the advantage offered by the tubes. For example, CRP trap plugs were numerous during Runs L-1 through -4 with the tubes in place. In addition to this experience, whether the tubes were needed in periods of smooth operation (Runs E-3 through -6 and L-5 through -9) was not ascertained.

d. Additional Remarks, FV-103

ZrF_4 "snow" removal by the Mark IA trap mesh was unsatisfactory. More "snow" deposited on the vessel surfaces upstream from the nickel mesh than on the mesh. In addition, relatively large amounts of material passed through the trap and deposited in the pipeline between the trap and the first absorber as well as probably in the first absorber. This pipe deposit apparently aggravated the tendency of HCV-11 to leak and, during decontaminating, necessitated water-washing this portion of the system. The apparent effects of this material on the first absorber are delineated in Sec. 8.4.1, item b.

The series of developmental runs (M-21 through -48) referred to under "Mark IA and IB Differences" initiated the heated trap packed with NaF to remove both ZrF_4 "snow" and chromium fluorides. Although the Mark IB trap was difficult to heat evenly and inadequately retained NaF, the additional data obtained with it were valuable in designing the Mark IIA trap.

Despite the superiority of Mark IIA to Mark IB, Mark IIA was not satisfactory because:

1. The vibrator and remote discharging device which utilized a remote valve operator to move the trap sieve plate did not work satisfactorily. The two principal reasons for its failure were (a) insufficient movement of the sieve plate and (b) agglomeration of the NaF bed.

Fig. 6.3. FV-103 Inlet and Outlet Tubes

2. The temperature profile of the NaF bed could not be determined without thermowells in the NaF bed.
3. The uranium retention was >100 grams. Inability to determine NaF bed temperatures probably contributed to the high uranium retention. The thermowells installed in Mark IIB helped to correct this situation.
4. The trap plugged frequently. Although the mechanisms causing CRP trap plugs are not understood, two observations were made: (a) a relationship seemed to exist between maloperation and FV-103 plugging. (Maloperation usually involved higher-than-normal FV-100 melt temperatures and pressures and excessive time-at-temperature.); (b) plugs formed by NaF particle agglomeration and/or plugging of the holes in the sieve plate. (Some NaF agglomerates were rather soft, i.e., could be easily crushed by hand, and resembled poly-fluorides.^a Others were yellowish possibly indicating the presence of U or Cr.^b Sieve plate plugs were very hard and had to be drilled out.)

Although the Mark IIB model showed the same tendency to plug as did Mark IIA, it did enable temperature profile determination in the NaF bed. The temperature gradient could be reduced from ~100°C. to ~80°C. by decreasing the upper fluorinator mantle temperature in Zone No. 1 from 550 to 450°C. In addition, uranium retention in Mark IIB was reduced from >100 g to <10 g by desorbing the NaF at a minimum temperature of 400°C. after fluorination.

The Mark II FV-103 showed significant corrosive attack, particularly nearest the fluorinator where intergranular penetration occurred (19, 7).

The highest bed activities occurred after Run L-7 and were gross β - 160 counts/min/mg U and gross γ - 3.4×10^3 counts/min/mg. U. Nb⁹⁵ was the chief γ emitter (21). Bed activities during the "E" runs were lower (22, 23).

Unloading FV-103 was done either with a portable vacuum cleaner or with FV-420 (Sec. 10.4.12).

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^a If the agglomerates contained polyfluorides, HF was probably present in the gases leaving the fluorinator. Before the "E" runs, the HF trap (FV-163) was installed to remove HF from the F₂ to the plant as described in Secs. 15.4.1a and 15.4.3. The effect of the HF trap addition on CRP trap plugging has not been assessed.

^b Sec. 8.4.1b.

A gas sampler for use between the fluorinator and absorber was recommended in the study made by the M.I.T. practice school in Oak Ridge (14). This sampler was never used because (a) the need for such a sample never arose and (b) the difficulty of taking an uncontaminated sample and analyzing it was so great.

6.4.2 CRP (Snow) Trap Heater, FV-503

Mark I heated the bottom of the NaF bed to $\sim 300^{\circ}\text{C}$. and the top (external cylindrical surface) of the trap to $\sim 200^{\circ}\text{C}$. in ~ 4 hours. Cooling data to room temperature are not available.^a This heater was inadequate because each of the three U-bent Calrods was intended to heat two perpendicular surfaces, that is, both the cylindrical and bottom surfaces of the trap. Instead of such an arrangement, it would have been better to use four Calrods, three equally placed circumferentially along cylinder elements and the other on the bottom surface of the trap.

Mark II heated the NaF bed to 400°C . in $2\frac{1}{2}$ hr with TC-1A-3 set on 35%. The temperature profile range from bottom to top of trap was $\sim 80^{\circ}\text{C}$.^c The 400°C . bed could be cooled to essentially room temperature in 12 hr. Although the sheath-to-electrode resistance of the Calrods was reduced to 5,000 ohms during decontamination after Run L-4,^d subsequent repeated slow "baking out" at successively higher temperatures increased the resistance to 20,000 ohms. Then the heater was used as before without further incident.

6.4.3 FV-103 Exit Line Heater, FV-503A

This heater was satisfactory for keeping the temperature of line H-103-1 and the $1\frac{1}{4}$ NPS flange above 65°C .^e The time-to-temperature for line H-103-1 was $\frac{3}{4}$ hr with Variac TC-1B-12 set at 60v. The cooling time to $\sim$ room temperature was $\frac{1}{4}$ hr.^f

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^a Schematically shown in Fig. 5.1, Fig. 22.2 and Secs. 22.4.1 and 22.4.2.

^b Sec. 8.4.1, item b. The Calrod around the line between FV-100 and FV-103 is not mentioned here, but it was used as indicated in Table 6.1.

^c Schematically shown in Fig. 6.1 (Fig. 22.3 and Secs. 22.4.1 and 22.4.2).

^d This decontamination was performed very carefully to avoid ruining the Calrods; that is, the FV-103 and -503 surfaces were wiped with the aqueous HNO_3 and Tide solution instead of being soaked therein. Normally the sheath-to-electrode resistance is > 1 megohm.

^e This line was kept at $\sim 115^{\circ}\text{C}$. during fluorination. Also cf. Sec. 6.3.1 and 6.4.1.

^f With a room temperature of 75°C at times in this vicinity.

6.4.4 FV-103 Bypass Heater, FV-500B^a

This heater kept line H-100-2 above 65°C.^b The time-to-temperature was 3/4 hr with TC-108 set at 100 v. The cooling time to room temperature was ~ 1/2 hr.

Plugs in the FV-103 bypass were mentioned in Sec. 6.4.1a.

6.5 Summary and Conclusions

Although the Mark IIB trap was the best of the "snow" and CRP traps, there were still improvements needed. Difficulties and changes in the models are highlighted in the foregoing sections, and heater data and times-to-temperature are given.

The Mark IA "snow" trap packed with nickel mesh removed a small percentage of the ZrF_4 and none of the chromium fluorides from the FV-100 exit gases. The Mark IB trap packed with NaF was inoperable because of inadequate heating and plug formation. Corresponding uranium retention was ~ 500 g. The Mark IIA trap was still unsatisfactory because of plug formation although the uranium retained (~ 100 g) was lower than for Mark IA. One-eight in. sodium fluoride pellets was the most satisfactory CRP trap packing used. Other materials tested were nickel mesh and sintered nickel disks.

The Mark IIB trap enabled reducing the uranium retention to < 10 g but was extremely susceptible to plugging during maloperation while fluorinating. With this trap, the desired 400°C. NaF bed temperature could be maintained during fluorination with a minimum temperature profile range of ~ 80°C. Although the temperature performance was better than for Mark IB, the 80°C. range was greater than desired.

The NaF dumping scheme incorporated in the Mark IIA trap design did not work.

The Mark I FV-103 bypass was inoperable because of a plugged Hoke No. 413 valve (V-126). The Mark II model was never tried.

The inlet and outlet tubes incorporated in the Mark IIB trap did not solve the plugging problem as was first supposed. The only generalization valid for plugs was that they were present during maloperation.

The effect of the HF trap (FV-163) addition on the CRP trap plugging has not been determined.

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^a Fig. 22.3; Secs. 22.4.1 and 22.4.2.

^b Sec. 6.3.1, item d.

The uranium retention was reduced from >100 g to <10 g with the Mark IIB trap by desorbing the NaF bed at ~ 400°C. following the fluorination.

Lines H-103-1 and H-100-2 were maintained as desired at ~ 100°C. with industrial heating cable. Despite the fact that the FV-503 Calrods were successfully used after decontamination, the decontamination procedure used did reduce the sheath-to-electrode resistance appreciably.

6.6 Recommendations

It is recommended that:

- a. A NaF-packed, Calrod-heated, trap continue to be used between the fluorinator and the absorber to reduce deposits in line H-103-1 and agglomeration in the first absorber.
- b. A higher heater watt density be used at the top of the trap than at the bottom of the trap to minimize the range of the temperature profile.
- c. The FV-103 top flange and any flange in line H-103-1 be incorporated into the flange leak detector system.
- d. Thermowells similar to those in the Mark IIB trap be included in any new design.
- e. Additional work be done on dumping the CRF trap contents directly into the fluorinator.
- f. The HF trap continue to be used, and an HF analyzer be incorporated in the F_2 line from the HF trap.
- g. Desorbing the CRF trap following fluorination and prior to absorber desorption be continued.
- h. Using industrial heating cable be continued on UF_6 lines such as H-103-1 and H-100-2.
- i. A UF_6 bypass around the new CRF trap similar to the Mark II design be provided.
- j. Extreme care be exercised in decontaminating vessel surfaces containing tubular heaters such as Calrods to avoid "shorting out" the elements.

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7.0 HEATED DUCT SYSTEM

7.1 Introduction

The process piping between the fluorinator and the main process chemical trap (FV-124) was maintained at a temperature of $> 65^{\circ}\text{C}.$ ^a by the heated duct system to prevent the deposition of UF_6 . In addition, the flow of air within the heated duct made it possible to recover uranium from UF_6 leaks in this process piping and to reduce cell contamination resulting from such leaks.

7.2 Equipment

Fig. 7.1 is a schematic arrangement of the equipment in the heated duct system. Details of the individual components are listed in Table 7.1.

7.3 Operation

7.3.1 Operating Procedure

a. Equipment Shakedown

Major steps in equipment shakedown were:

1. Drilling holes in the duct walls as indicated at "X" notations on Fig. 7.1 to roughly balance the air flow through the heated duct.
2. Adjusting the Mercoid Ductatherms and rearranging or adding Calrods to obtain a nearly constant temperature throughout the heated duct.

NOTE: See Sec. 7.4.1 for equipment shakedown details.

b. Operation

The steps involved in putting the heated duct in operation were incorporated in the operating procedures of other VPP systems. These steps were required:

1. Putting all the lid sections on the heated duct.
2. Having FV-158 filled with 11 kg of 1/8-in. NaF pellets.
3. Turning on both the blower switch (EX-FV-650) and the heated duct switch (EX-FV-522).

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^a The temperature of 65°C is above the sublimation temperature [$\sim 57^{\circ}\text{C}$. at 14.7 psia (17, p. 4)].

Table 7.1
List of Heated Duct System Components

Components	No.	Drawings and Sketches Title	Miscellaneous Information	References
Heated Duct	D-22758	Cell #2 Equipment Layout and Heated Duct Routing.	<u>Control Equipment</u> - Order FY-522. <u>Period of Use</u> - All Runs. The heated duct consisted of steel frame-work covered with Johns-Manville 1/2-inch thick Marinite - 35 - Type A. The joints were at first caulked with Glidden Caulking Compound No. 578 Natural. Later, Johns-Manville No. 450 insulating cement was tried and was preferred as delineated.	<u>This Report</u> - Secs. 7.4.2; 7.4.1; 7.4.1, item a; 7.4.1, item b; 7.4.1, item c; 7.4.1, item d; 7.4.4; 7.5; 7.6; 9.4.7 d; 10.4.2; 17.4.2, e2; 17.5.2. <u>Engineering File Folder</u> No. P-8, -9, -61. <u>Other Literature</u> - 19, 24, 32 (p. 29).
	D-22761	Cell #2 Engineering Flowchart		
	D-22765	Cell #2 Ductwork Plan and Elevation		
	D-22766	Cell #2 Heated Duct Sections		
	D-23211	Cell #2 Duct and Lead		
	D-23222	Cell #2 Lead Shield - Plan and Details		
	D-23223	Cell #1 Heated Duct and Pipe Details		
	D-25909	Heated Air Duct Electrical Layout - Isometric		
	Q-1679-37	Cell #1 Heated Duct Thermocouples		
	Q-1679-38	Cell #2 Heated Duct Thermocouples		
Heated Duct Heaters, FY-522	D-25909	Heated Air Duct Electrical Layout, Isometric	<u>Control Equipment</u> - A thermostat and transformer relay was used for each of thirteen tubular heaters shown in Fig. No. D-25909. Two interlocking switches (one for FY-522 and the other for FY-650) were provided. <u>Period of Use</u> - All Runs. <u>Heater Data</u> - Thirteen 1000-w., 230-v., tubular heaters were placed as shown in Fig. D-25909 and the other three as in Fig. 7.1. Power supply - three phase.	<u>This Report</u> - Secs. 7.4.3; 7.4.1, item c; 7.4.4; 7.5; 7.6; 8.4.7; 21.4.1. <u>Engineering File Folder</u> No. F-61. <u>Other Literature</u> - 32 (p. 29).
	D-34891	Wiring Diagram (sheet 5, item "B")		
Heated Duct Blower, FY-650	D-19712	Off Gas Scrubber Flowchart	<u>Control Equipment</u> - Electrical switch interlocked as described for FY-522 and ammeter on the motor. <u>Period of Use</u> - All Runs. <u>Motor Data</u> - U. S. Electrical Motors, Inc., Los Angeles, Calif. Type HRS, Serial No. 2293605; 1/2 H.P., 220/400 v., 2.5/1.3 A., three phase, 60 cycles, 1200 rpm. <u>Blower Data</u> - Sutorbilt One Pump, Size 21LB, Serial No. 91, Property No. 158855. <u>Drive Data</u> - Reeves Flexi-Speed, Size 203, 300 to 1800 rpm.	<u>This Report</u> - Secs. 7.4.4; 7.4.1, item b; 7.4.2; 7.5 and 7.6. <u>Engineering File Folder</u> No. F-59. <u>Other Literature</u> - 26, 32 (pp. 29, 47).
	D-34893	Wiring Diagram (sheet 7, item "F")		
Heated Duct Chemical Trap, FY-158	D-19712	Item FY-158 Chemical Trap for Duct Air Clean-up.	<u>Period of Use</u> - All Runs. This trap was packed with 11 kg. of 1/8-inch NaF pellets.	<u>This Report</u> - Secs. 7.4.3; 7.5; and 7.6. <u>Engineering File Folder</u> No. F-46. <u>Other Literature</u> - 23, 32 (p. 30).
	D-32782	Off-Gas Scrubber Flowchart		

7.3.2 Critical Operating Steps

- a. Having a nearly constant temperature throughout the duct of $\geq 65^{\circ}\text{C}$. to avoid depositing UF_6 .
- b. Making certain that all lid sections were placed on the heated duct.
- c. Turning on both EX-FV-650 and EX-FV-522. (The heaters could not be turned on with EX-FV-522 unless EX-FV-650 was "on.")

7.4 Equipment Evaluation

7.4.1 Shakedown Experiences

Shakedown items are discussed below. The effect of each independently was not assessed. After shakedown, however, heated duct operation was satisfactory. No run down-time was caused by UF_6 depositing in the heated duct piping.

a. Air-Flow Balance

In making the holes in the duct wall as mentioned in Sec. 7.3.1a, the hole sizes and number of holes were roughly determined depending on the distance away from the blower and the duct cross-section.^a Despite the fact that the determination was only approximate and that no distribution of air flows was later measured, the temperatures in the duct were adequate to prevent UF_6 from depositing after the shakedown changes were made. After equipment shakedown, however, it was questionable whether the air flow rate in the duct was great enough to transfer UF_6 leaks to FV-158 as discussed in Sec. 7.4.4.

b. Moving FV-158 and FV-650 to the Penthouse

The chimney effect which occurred in the heated duct at the cold traps with FV-158 and FV-650 in Cell 2 resulted in moving these components to the Penthouse. The cold lines in the vicinity of FV-222 caused by the chimney effect were warmer after the change. The Penthouse location chosen was adjacent to the cell offgas duct to make a short exit gases run (5 to 10 ft) from the blower to the duct. This design assured a better gas flow rate through the heated duct than did the Cell 2 location with a ~ 60-ft exit line to the duct, an arrangement which seriously reduced the amount of gas moved by the blower.

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^a The holes varied in size from ~ 3/8 to 3/4 inch.

c. Calrod Additions and Rearrangement

Three 1000-w Calrods were added to the original heated duct electrical system, increasing the number of tubular heaters to sixteen. One of the additional Calrods was added to each phase of the three-phase circuit.^a These heaters were placed adjacent to the piping as follows: (a) in the duct at the FV-120 extremity (line A-A in Fig. 7.1), (b) in the sleeve of Cell 1-2 wall along the side of the process piping (line B-B in Fig. 7.1), and (c) in the duct near the FV-121 lines (line C-C in Fig. 7.1). These additions increased the temperatures in these locations as desired.

The heating element nearest FV-124 was moved closer to line X-222-1, and this section of the duct was altered to contain the pipeline and heater and also to afford easier access to the inlet line flange of FV-124. These two changes along with adding heat lamps mentioned in item d. below afforded better heating of this line.

d. Heat Lamps

Two 250-w heat lamps were directed on the section of bare line between the end of the heated duct and FV-124 to avoid UF₆ deposition in this portion of line X-222-1 (cf. Fig. 9.1).

7.4.2 Heated Duct^b

The heated duct was satisfactory although the initial lids were not strong enough to support a man. Because the section of the heated duct near FE-26 was a convenient walk-way, steel supports were added to prevent damage to the duct lid in this vicinity.

For the first half of the VPP work, the walls and lids of the heated duct were made of Marinite,^c and the lid joints (Marinite-to-angle iron) were made with Glidden caulking compound No. 578 Natural. Short-time tests indicated that such joints would be satisfactory at expected temperatures of 110°C. (24). Subsequent use of these joints corroborated the result from the tests. In fact, the bond between the Marinite and angle iron was so strong that breaking the joint usually required destroying the Marinite. Since several occasions arose which necessitated opening sections of the duct and since each of these openings necessitated replacing a substantial portion of

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^a The three-phase delta heating arrangement shown as item "H" in Drawing No. D-22779, Rev. 1 did not include any of the three Calrods added. As revised, therefore, this drawing should have shown six 1000-w tubular heaters in one phase and five in each of the other two phases.

^b After use, equipment was moved to Burial Ground No. 3 (Sec. 23.4.16b) except for the heated duct blower and chemical trap.

^c Johns-Manville 1/2-in. Marinite-36-Type A. This was chosen for its relatively low thermal conductivity of 0.77 (BTU)(in.)/(ft)²(°F)(hr) at 200°F (1, p. 6).

the Marinite, asbestos cement^a was successfully substituted for the caulking compound. Using asbestos cement, the joint between Marinite and angle iron was adequate, and breaking this joint did not destroy the Marinite.

The high cost and special ordering of Marinite probably will lead in the future to the substitution of Flexiboard. Flexiboard is cheaper and is stocked in ORNL stores. Flexiboard was used successfully in the new lid for FV-526 (Sec. 10.4.2).

Prior to Run E-2, the HCV-22 bonnet leaked UF₆ into the heated duct (Sec. 17.4.2,e.2). Most of this UF₆ remained in the vicinity of HCV-22, some of it coloring the Marinite yellow. Other UF₆ leaks at PE-14 and -15 in the "C" runs left UF₆ in the heated duct near the top of FV-222 (25, p.21). Although the UF₆ from these leaks was evidently not swept to FV-158 as desired, little cell contamination resulted because most remained inside the duct (Secs. 7.4.4; 9.4.7, item d).

Corrosion in the Monel piping in Cell 2 appeared insignificant on samples from piping inside the heated duct (19).

Air temperatures were measured with bare thermocouples having hot junctions extending inside the duct. These thermocouples were purposely placed away from heating elements and pipelines to avoid being overly influenced by them (Sec. 21.4.2). Temperatures of resistance-heated equipment were determined with thermocouples as delineated in Sec. 22.4.2.

7.4.3 Heated Duct Heaters, FV-522

After the heater additions, heater rearrangement, and heated duct alteration near FV-124 discussed in Sec. 7.4.1c, the heated duct temperatures were satisfactory (range: ~ 60° to 135°C.).

The FV-522 system and FV-650 were interlocked so that the heaters could not be turned on unless the blower was on (Sec. 7.4.4), a safety measure to avoid high local temperatures in the duct with the blower not running.

A minor amount of trouble was met with cold lines caused by the chimney effect in the vertical section of the heated duct in Cell 1. Any opening in the horizontal section of the duct adjacent to the fluorinator accentuated this trouble.

It required ~ 2 hours for the heated duct to heat from room temperature to the normal operating temperature range of ~ 60 to 135°C. This wide temperature range was probably the chief disadvantage to tubular heaters because of the high wattage per unit length (40 watts per inch for the heaters used). The low temperature extreme was on line H-103-1 (in area denoted by item a. below).^b Evidently this thermocouple read slightly low; otherwise, more

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^a Johns- Manville No. 450 insulating cement.

^b Sec. 8.4.7.

trouble with UF_6 condensing in this line during desorption would have probably occurred. Other cold areas in the heated duct included:

- a. The area where the piping left the duct going to and from FV-120. This area was especially cold when the Cell 2 door was open during cold weather. The remedy included adding the heater mentioned in Sec. 7.4.1c as well as caulking the annular holes around lines H-103-1 and H-120-1 as these lines passed through the duct wall.
- b. The duct extremity at FV-124. This area was heated adequately after moving the heater closer to line X-222-1 and rebuilding this section of ductwork (Sec. 7.4.1, item b).

No trouble with tubular heaters has occurred. Both TNK's and Calrods have been used in the heated duct (Sec. 21.4.1).

7.4.4 Heated Duct Blower, FV-650

The operation of this blower had been satisfactory. As stated in Sec. 7.4.1b, the Cell 2 location seriously reduced the amount of air handled. Even in the Penthouse location, there was some doubt whether the pumping rate of the blower was adequate to pull UF_6 vapor from piping leaks into FV-158 for subsequent recovery. This doubt arose from the fact that very little UF_6 was collected in FV-158 when the two UF_6 leaks mentioned in Sec. 7.4.2 occurred. In each case, considerable yellowing of the Marinite in the vicinity of the leak indicated that UF_6 sorbed on the Marinite instead of being swept to FV-158.

The blower lubricating schedule formulated earlier was followed although radioactive operation was a deterrent to routine lubrication (26).

7.4.5 Heated Duct Chemical Trap, FV-158

This trap containing 11 kg of 1/8-in. NaF pellets operated satisfactorily. With the air inlet at the top, whether any UF_6 had been sorbed could be determined qualitatively by removing its lid and observing the color of the NaF bed.^a In this way, the presence of UF_6 could be detected without removing the trap contents. This arrangement saved operating time because little UF_6 ever reached this trap. The heaviest loading of this trap was 7 grams of U which was < one % of its capacity (23). Because so little of its capacity was used at the highest loading, no UF_6 ever passed through the trap. The trap bed was generally used for a number of runs without changing. The frequency of changing was sporadic. Unloading was done with a portable vacuum cleaner.

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^aA yellowish coloration indicated the presence of UF_6 (Sec. 10.4.9).

7.5 Summary and Conclusions

After equipment shakedown, heated duct operation was satisfactory. No run down-time was caused by UF_6 depositing in the heated duct piping.

Shakedown activities described included: (a) making an air-flow balance to evenly distribute heat and to provide movement of vapors to the chemical trap for sorbing any UF_6 in the heated duct; (b) moving the chemical trap and blower from Cell 2 to the Penthouse to eliminate cold areas near FV-222 caused by the chimney effect and to increase the blower throughput; (c) adding three additional heaters to increase duct temperature in certain areas; (d) moving the heater nearest FV-124 adjacent to the pipeline to better heat this line and altering this section of the duct to accommodate the relocated heater; and (e) adding two heat lamps to heat the bare section of pipeline between that extremity of the duct and FV-124.

Marinite was suitable for the walls and lids of the heated duct. However, Flexiboard would be a good substitute because it is cheaper and is stocked in ORNL Stores whereas Marinite must be specially requisitioned.

Glidden caulking compound No. 578 Natural has been successfully used on Marinite-to-angle iron joints. Its extremely strong bond, however, necessitated destroying the Marinite to open the duct. Therefore, asbestos cement which forms a weaker but adequate bond was substituted for the caulking compound.

UF_6 leaks in the heated duct have occurred: (a) near HCV-22 in the early "E" runs and (b) near the top of FV-222 in the "C" runs. These leaks were easily identified by the yellowish coloration of the Marinite. Since no uranium was found in FV-158 in either case and considerable yellowing of the Marinite occurred, these leaks raise the question of whether the air flow rate in the heated duct system was adequate to pull UF_6 vapor into FV-158 for subsequent recovery.

The extreme temperatures in the heated duct were ~ 60 and $135^\circ C$. under normal operating conditions. This wide temperature range was evidently caused by the high wattage per unit length (40 watts per inch) of the heaters. Some trouble with cold areas occurred. It required ~ 2 hours to heat the duct from room temperature to operating temperature. No trouble with the tubular heaters occurred.

No difficulty was experienced with the blower after it was moved to the Penthouse although its inability to transfer UF_6 vapor to FV-158 indicated low pumping rate. The lubricating schedule was followed although radioactive operation was a deterrent to following this schedule.

The operation of FV-158 was satisfactory. Its maximum uranium loading was $< 1\%$ of capacity; therefore, no UF_6 passed through it. The unloading schedule was sporadic. The top gas inlet facilitated detecting whether any UF_6 had been collected.

Corrosion of the Monel piping in Cell 2 appeared insignificant.

7.6 Recommendations

It is recommended that the heated duct be used in its latest state of development except that:

- a. If tubular heaters continue to be used in the heated duct, TNK's or GE Calrods be used without substituting other brands because of the success with these heaters in the past.
- b. The pumping capacity of FV-650 be determined and compared with its rated capacity.
- c. FV-650 and its motor be lubricated before subsequent operations.
- d. Studies be made on the advisability of using a heating element with a lower wattage per inch and of substituting Flexiboard for Marinite.

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8.0 ABSORBING SYSTEM

8.1 Introduction

In the absorbing system, UF_6 was separated from volatile fluorides of fission products and melt ingredients - ZrF_4 , NbF_5 , and CrF_3 .^a This was accomplished by sorbing the UF_6 and these other fluorides with NaF and then desorbing the UF_6 preferentially while the other fluorides remained on the sorbent. The chief steps in the operation of the absorbing system were: (a) sorbing the subject fluorides from the fluorinator exit gases on NaF and (b) desorbing the UF_6 by heating the NaF bed in a stream of F_2 which swept the UF_6 to the cold traps.

8.2 Equipment

Two absorbers alike in design and connected in series were used in the Volatility Pilot Plant. Figure 8.1 shows an assembled absorber. Since model differences in the absorber do not show in this sketch, these are delineated in Sec. 8.4.1a. Details relative to the vessel and its auxiliary components are listed in Table 8.1.^b

Some thermocouples were placed in the thermowells of the absorbers; others were welded to the external surfaces of the absorbers (Sec. 22.4.2).

8.3 Operation

8.3.1 Operating Procedure

Steps in operating the absorbing system were:

Sorption

- a. Maintaining the temperatures of both absorbers at $\sim 80^\circ C$ (Compressed air for cooling during sorption was removed.); the heated duct at $\geq 65^\circ C$ (Sec. 7.4.3); and the cold traps at $-40^\circ C$ and $-55^\circ C$ respectively.

^aThe reaction mechanism of UF_6 with NaF is unknown and is, therefore, termed "sorption" while the vessel is called "absorber" and the system "absorbing" (23).

^bFV-124 is in the Cold Trapping System (Sec. 9.4.8).

^cThe warm part of the system was kept at a temperature slightly above the sublimation point of UF_6 [$57^\circ C$ at 14.7 psia, (17, p. 4)], with the NaF beds held only slightly $> 65^\circ C$ to avoid a temperature rise to > 150 to $200^\circ C$ during sorption. The cold traps were kept cold to prevent UF_6 loss [vapor pressure of UF_6 at $-40^\circ C$ was 8.5×10^{-3} psia (17)]. Cf. Sec. 8.4.7.

Fig. 8.1. Equipment Arrangement in the Absorbing System

Table 8.1
List of Absorbing System Components

Components	No.	Drawings and Sketches Titles	Miscellaneous Information	References
Absorbers (Trep packing - 25 kg. of 1/8- inch haf pellets and 13 kg. of 1/4-inch #1 shot), FV-120 and -121	D-19714, Rev. 1 and 2 D-22761	FV-120 and FV-121 Absorber Cell II Engineering Flowchart	<u>Mark IA</u> <u>Control Equipment</u> - Under FV-520 and -521. <u>Period of Use</u> - Through the "C" runs. The diffuser ring per Dwg. D-19714 (unrevised) could not be readily built and, therefore, was never used. Design used was that on Dwg. D-19714, Rev. 2.	<u>This Report</u> - Secs. 8.4.1; 8.4.1, item a; 8.4.1, item b; 8.3.1; 8.4.5; 8.4.7; 8.5; 8.6; 9.4.6; 15.4.5, item b; 15.5; 18.4.1; and 18.4.2. <u>Engineering File Folder No. F-39</u> . <u>Other Literature</u> - I, 17, 21, 22, 23, 24, 25, 31, 32 (pp. 1, 17, 18, 58, 59).
Absorber Furnaces, FV-520 and FV-521	D-22761 D-22776 <u>Revi-Duty Dwg.</u> M-7420-A M-7420-1002 M-7420-1004 M-7420-2004 M-7420-2005 M-7420-2006 M-7420-2007	Cell II Engineering Flow Sheet Wiring Diagram (Sheet 4, Item "F" (FV-520) [Item "G" (FV-521)] General Assembly Furnace Wiring Furnace Wiring Top Section Base Assembly Shell Assembly Bottom Section Top Assembly Split Covers	<u>Control Equipment</u> - Pyrotherm (thermocouple on the external vessel surface) and Variac for each furnace. <u>Period of Use</u> - All runs. <u>FV-520 Data</u> - Revi-Duty Electric Co., Milwaukee, Wis., Serial No. 92750, P.O. No. D-4716 and D-55559; X-51482; 15 kv., 230 v., three phase. <u>FV-521 Data</u> - All data were same as for FV-520 except (a) Serial No. 91749 and (b) X-63585.	<u>This Report</u> - Secs. 8.4.2; 8.3.2; 8.5; 8.6; 22.4.1; 22.4.2; 22.5 and 22.6. <u>Engineering File Folder</u> - No. F-40.
Inlet Pipe Heaters, FV-520A and -521A	FV-520A D-22761 D-22780	FV-520A Cell II Engineering Flowchart Wiring Diagram (sheet 6, item "A")	<u>FV-520A</u> <u>Control Equipment</u> - Wheeler (thermocouple welded to the pipeline between adjacent turns of the heating cable) and Variac. <u>Period of Use</u> - All runs. <u>Heater Data</u> - About 50 ft. of industrial heating cable was wrapped helically around the FV-120 inlet line pipe flange; covered with stainless steel shingstock; and insulated per details Sec. 22.4.2. Power required was ~400 w. at 110 v.; advance of helix was ~1/2 inch.	<u>This Report</u> - Secs. 8.4.3; 8.5; 8.6; 22.4.1; 22.4.2; 22.5; and 22.6. Table 22.3. <u>Engineering File Folder No. F-40</u> .
Similar to that for FV-520A.			<u>FV-521A</u> <u>Control Equipment</u> - Similar to that for FV-520A. <u>Period of Use</u> - All runs. <u>Heater Data</u> - Similar to that for FV-520A.	Similar to that for FV-520A.

Table 8.1 (Continued)

Components	Drawings and Sketches		Miscellaneous Information	References
	No.	Title		
Outlet Pipe	FV-520B D-22761	Cell II Engineering Flowchart	<u>Control Equipment</u> - Similar to that for FV-520A.	<u>This Report</u> -
Heaters, FV-520B and FV-521B	D-22780	Wiring Diagram (sheet 6, item "B")	<u>Period of Use</u> - All Runs.	Secs. 8.4.4; 8.5; 8.6; 22.4.1; 22.4.2; 22.5; and 22.6.
	FV-521B D-22761 D-22780	Cell II Engineering Flowchart Wiring Diagram (sheet 6, item "D")	<u>Heater Data</u> - Similar to that for FV-520A.	Table 22.3. <u>Engineering File Folder No.</u> 7-40.
Gas Preheaters, FV-520C and FV-521C	D-22761	Cell II Engineering Flowchart	<u>FV-520C and -521C</u> <u>Control Equipment</u> - Pyrowox (thermocouple welded to the pipeline about midway of heater) and Varisc. <u>Period of Use</u> - During "C" Runs. <u>Heater Data</u> - A 1000-w., 110-v., clampbell heater 12-in. long was fixed around the inlet pipe of reasel as closely as possible to the furnace lid and insulated with 2-in. Superex the outside surface of which was then covered with Thermatex "B".	<u>This Report</u> - Secs. 8.4.5; 8.5; 8.6; Table 22.3 <u>Engineering File Folder No.</u> No record of either heater is kept in the Engineering File or on any engineering drawing.
Heat Lamps, FV-120 and -121	D-22761 D-22773	Cell II Engineering Flowchart	<u>Control Equipment</u> - None. <u>Period of Use</u> - "E" and "L" Runs. <u>Heater Data</u> - Two 250-w., infra-red heat lamps were shown on the inlet and outlet pipeline flanges (one on each flange) for each chamber; power supplies were from 1P-8, for FV-120 circuit 14 and the FV-121 circuit 16.	<u>This Report</u> - Secs. 8.4.6, 8.5 and 8.6. <u>Engineering File Folder No.</u> 8-40. <u>Other Literature</u> - 17.
Absorber Instrumentation				<u>This Report</u> - Secs. 8.4.7; 8.3.1; 8.4.1, item b; 8.5; 8.6; 7.4.3; 9.4.3; 9.4.7, item c; 21.4.6. <u>Engineering File Folder No.</u> 7-40. <u>Other Literature</u> - 13.

- b. Putting the necessary instruments and charts in service and establishing all N_2 purges using 4-1/2 psig N_2 .
- c. Setting valves as required.
- d. Controlling at indicated set points heaters on FV-220 inlet pipe, absorber inlet and outlet pipes, and absorber inlet and outlet flanges (heat lamps).
- e. Making certain that the scrubber was operating normally, and that there was adequate scrubbing capacity for a normal sorption and desorption.
- f. Having FV-124 and -158 packed with NaF and FV-650 running.

Desorption

- g. Adjusting the F_2 flow rate through the absorbers to 8 slm and continuing steps a - f as needed.
- h. Turning on absorber furnaces and setting their controllers at 400°C.
- i. Continuing to heat the absorbers until the upper and lower temperatures in both absorbers exceeded 390°C.
- j. Turning off the absorber furnaces and the heaters referred to in item d.
- k. Shutting off the F_2 flow 15 minutes after doing item j.
- l. Purging absorbers with N_2 for 40 minutes.
- m. Sampling the absorber beds in segments as required to obtain pertinent analytical data (27, p. 37). (Such sampling was done once at the end of Run E-6 and once at the end of Run L-9. In prior runs, sampling was done at odd intervals because of the developmental nature of those runs.)

System changes necessitated only minor alterations in the procedure. The sorption procedure was part of the fluorination procedure given in Sec. 5.7. For the complete desorption procedure, see Sec. 8.7.1.

8.3.2 Critical Operating Steps

- a. Maintaining the heated duct temperatures and the FV-220 inlet (heated by FV-527) $\geq 65^\circ\text{C}$ to prevent depositing UF_6 in the piping.
- b. Holding the OH^- concentration in the scrubber solution at $\geq 1.0\text{ M}$ to avoid passing F_2 through the scrubber.
- c. Having the absorbers at $65^\circ - 75^\circ\text{C}$, before absorption to avoid operating time loss (Sec. 8.4.2).

- d. Having both absorbers at $\sim 80^{\circ}\text{C}$ before desorption to avoid depositing UF_6 on the cold surfaces of the second absorber and to reduce the time required to heat the NaF to 80°C . (About four hours could be consumed in heating the cold absorbers to 80°C following excessive cooling during sorption.) See Sec. 8.4.2.
- e. Holding cold traps at operating temperatures to minimize the amount of UF_6 passing through them.
- f. Having FV-124 and -158 in service to sorb any UF_6 in gases passing through the cold traps or UF_6 leaking out of the system piping, respectively.
- g. Maintaining F_2 flow and not exceeding a temperature of 400°C in the absorbers to reduce the formation of UF_5 . (Nonvolatile UF_5 represented a uranium loss.)

8.4 Equipment Evaluation

8.4.1 Absorbers, FV-120 and -121^a

a. Model Differences - Mark IA and IB

Mark IA contained thermowells as shown on ORNL Dwg. D-19714, Rev. 1 and Mark IB as shown on ORNL Dwg. D-19714, Rev. 2.

The thermowells in Mark IB were superior to those in Mark IA in these ways: (a) enabled measuring inlet and outlet gas temperatures as well as the bed temperature at a point higher in the bed than beforehand, (b) used structurally stronger thermowells in the NaF bed (1/4-in. NPS Sch. 40 Inconel vs. 1/8-in. NPS Sch. 40 nickel), and (c) facilitated the installation of thermocouples because of the larger i.d. of the 1/4-in. NPS.

The Mark IB design was also superior because flanges were incorporated in flange leak-detector system (cf. Secs. 18.3, 18.4.1, 19.4.2; Fig. 18.1; Table 18.1).

b. Absorber Evaluation

Charge for an absorber was 25 kg of 1/8-in. NaF pellets,^{b,c} with 13 kg of 1/4-in. nickel shot placed as shown in Fig. 8.1.

Absorber capacity was nominally 10 kg of U as UF_6 . Theoretical capacity (0.9 g U/g NaF) was 22.5 kg (28, p. 33).

^aAfter use, the absorbers and associated equipment were moved to Burial Ground No. 3 (Sec. 23.4.16, item b).

^bAttempts to reduce processing costs by using less expensive NaHF_2 instead of NaF failed because of polyfluoride formation in downstream NaF beds and condensation of HF in unheated sections of the system piping (29). The economic justification was shown (30, 31, p. 9). Also cf. Sec. 9.4.8.

^cUsing minus 20-mesh NaF instead of the more expensive 1/8-in. pellets was not feasible because of difficulties encountered with fines.

Absorber losses on the average were:

1. One absorber passed ~ 25 to 30 g of U in the exit gases.
2. Two absorbers and cold traps in series as in most runs passed ~ 11 g of U out in tail gases.
3. After desorption at 400°C., 14 g of U remained equally distributed vertically on the absorber bed.^{b,c}

Experiences with the absorbers were:

1. Difficult to unload because of the small neck. Discharging was done by vacuum in a manner described elsewhere to determine the distribution of uranium and/or impurities in the bed (21). It was difficult to remove the absorber bed without spreading NaF dust.
2. Uneven gas distribution in gas diffuser ring. A rough qualitative test performed by bubbling air through the diffuser ring while the ring was under water showed that about two-thirds of the air was discharged adjacent to the gas inlet pipe. Consequently to prevent UF₆ from heavily loading the NaF above this region the absorber was filled to the bottom of the diffuser ring with 13 kg of nickel shot. Then, the NaF pellets were charged on top of the nickel shot. Although the efficacy of this scheme was not fully determined, less caking of NaF in the bottom of the absorber occurred after this change than beforehand.
3. Long time (e.g., 12 hrs) required to heat the center of the bed to 390°C. Two means of shortening the heat-up time of the center of the absorber bed have been tried. First, the gas preheater mentioned in Sec. 8.4.5 was added. Second, the distance from the outer circumference to the center of the bed was reduced from 5" to 4" by axial placement of a piece of 2-in. NPS (31, p. 26). This reduced by one inch the distance through which heat from the absorber furnace flowed to reach the most-distant NaF pellets. Some reduction in heat-up time was realized, but the improvement was slight. In addition, exact axial placement of the center pipe was extremely difficult because of the small neck and geometry of the absorber design. Consequently, using this axial pipe technique for reducing heat-up time was discontinued.

^a The U passing the absorbers and cold traps was collected on NaF in FV-124. The U collected in FV-124 and that remaining in the absorber bed after desorption was reclaimed by dissolution (Sec. 9.4.8; 32).

^b In the "E" and "L" runs, the desorption in the final run of each series was performed at 600°C. with no marked decrease in the U retention of the bed. Cf. footnote above.

^c Sec. 8.3.1.

4. Pressure build-up before FV-120^a. In the pre-CRP trap period, considerable trouble with pressure buildup before FV-120 was experienced. This apparently did not occur in post-CRP trap work. Generally, three observations were made during the period of time that FV-120 plugging was prevalent:

- (a) Sintering and/or agglomerating of NaF pellets occurred. The first of the two series-connected NaF beds was very susceptible to plugging. Plugging did not occur, however, in the last four "E" runs or the last five "L" runs. And the real reason(s) for these exceptions cannot yet be formulated. One possibility is that maloperation during fluorination was associated with plugging because maloperation was absent in these above mentioned "E" runs and present in almost every other run. "Maloperation" here is defined as embracing the following out-of-the-ordinary fluorination conditions: (1) pressure rises in the fluorinator, (2) excessive fluorination time, and (3) higher-than-normal fluorination temperatures.

The second possibility is that the HF in the F₂ reacted with the NaF to form polyfluorides (Sec. 15.4.1a). An HF trap (Sec. 15.4.3) was installed prior to the "E" runs to remove the HF from the F₂.

- (b) Coloring of some NaF pellets took place, the colors being yellow, brown and pink (31, p.28). Analyses of the colored pellets disproved the belief that all yellowish NaF pellets are high in uranium content. For instance, many of the yellowish pellets contained substantial percentages of chromium and little uranium. Along with these findings, the brown and pink pellets were also found to contain high percentages of chromium. Apparently the differently colored Cr-rich pellets represented chromium in different valence states.
- (c) More than three-fourths of the holes in the diffuser rings of both absorbers were plugged. The holes in the FV-120 diffuser ring plugged with this vessel in the FV-120 position (first absorber in series). After the FV-120 diffuser ring disintegration (item 5 following), the vessels were exchanged. Then, after the FV-121 vessel was used for several runs in the FV-120 position, examination of its diffuser ring revealed similar plugging. FV-121 was restored with a circumferential weld as was FV-120, after drilling out the plugs.

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^a Pressure build-up was noted on PE-12 and PE-38 (Sec. 8.4.7).

5. Disintegration of FV-120 diffuser ring.^a The cause of this incident was not definitely known. But three out-of-the-ordinary practices might have contributed to it either individually or collectively:
- (a) No F_2 conditioning of FV-120 prior to the sodium fluoride preparation. F_2 conditioning was not indicated in the sodium fluoride preparation procedure because the conditioning with HF was thought to be adequate although not as effective as that with F_2 .
  - (b) Pure F_2 being fed to the vessel with the bed at $\sim 600^\circ C$. instead of at $375^\circ C$. as indicated by the sodium fluoride preparation procedure. Heating the bed to this higher temperature before adding F_2 was intentional rather than accidental. Reasons for this were: (a) to minimize the HF released from the NaF during desorption by finishing the $NaHF_2$ to NaF conversion at $\sim 200^\circ C$. above the normal desorption temperature, and (b) to further reduce the sodium fluosilicate content of the NaF since sodium fluosilicate reacts irreversibly with UF_6 .
  - (c) The accelerated cooling technique used on FV-120 after desorption. In this technique, FV-120 was isolated from the system by valving after desorption. Then, the inlet and outlet flanges were uncoupled and blanked off. Next, the vessel was lifted out of the furnace to hasten cooling. The furnace was left open with a small flow of air to it to cool more rapidly. After the vessel and furnace had cooled to the desired temperatures, the vessel was lowered into the furnace and recoupled to the system (33). Whether the vessel was then conditioned with F_2 before being used again for the absorption-desorption cycle is unknown. Although the mechanism causing the diffuser ring failure is unknown, sufficient heat was released to partially melt the Inconel ring. It is rather probable that the heat came from a localized highly exothermic reaction like that of fluorine with organic material(s) such as dirt, grease, or a rag which might have been accidentally left in the absorber after charging with $NaHF_2$. Repair of the diffuser ring was accomplished by: (1) cutting the vessel circumferentially in a plane normal to its axis and 12 to 15 in. above the bottom of the vessel; (2) dressing up the melted edges of the diffuser ring and welding on plates to seal the ring, this operation reducing the ring to about two-thirds its original size; (3) removing the plugs from about three-fourths of the diffuser ring holes; and (4) restoring the vessel with a

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^a Sec. 15.4.5b (7).

circumferential weld. After FV-120 was so repaired, the original FV-120 was henceforth used in the FV-121 position and vice versa. This exchange was made because of the reduced size of the FV-120 gas diffuser ring.

6. Shielding. The lead shielding around the FV-120 inlet line H-103-1 and the concrete-block wall around the FV-120 and -520 assembly were adequate shields for the radiation levels encountered in Cell 2. The highest bed activities which occurred during the "L" runs were: gross β - 51 counts/min/mg U and gross γ - 220 counts/min/mg U. Cs^{137} was the principal γ emitter (21). Activities during the "E" runs were lower (22,23).
7. Flange leak-detector connections. The top flanges and the inlet and outlet lines flanges of both absorbers were incorporated into the flange leak-detector system before Run E-3. During subsequent runs, leaks were detected by this system in the top flanges to both vessels whereas no leak was found at the inlet and outlet flanges. In each case, the leak was greatest with the vessel at $\sim 100^\circ\text{C}$ and least at $\sim 400^\circ\text{C}$ (during desorption). Since the system pressure was about atmospheric, applying 30 psig N_2 dynamically to the leak-detector connections at the absorber top flanges eliminated any air-leakage into the vessels through the flange leaks. Attempts to stop the leak by drawing the flange bolts tighter with the vessel at $\sim 100^\circ\text{C}$ failed. The bolt nuts would not move at the 175 ft-lb. limit of the torque wrench used. Trying to tighten the bolts with the vessel at 400°C was not feasible.

The NaF sampler for the absorbers recommended in the study made by M.I.T. practice school at Oak Ridge was never used because larger samples than could be taken with it were desired (14). Usually the bed was sampled in segments as mentioned in Sec. 8.3.1.

8.4.2 Absorber Furnaces, FV-520 and -521^b

To secure proper furnace lid closure, it was necessary to "hot-rod" both lids to accommodate the Mark IB thermocouple wells and the top flange leak-detector plumbing.

Significant time-lags were encountered while heating and cooling in the $< 100^\circ\text{C}$ range. For example, assuming the absorbers were at 90°C while preparing for fluorination, it required 3 to 4 hours to reach the desired $65\text{--}75^\circ\text{C}$ range. In the same way, if the absorbers were at 50°C while preparing for fluorination,³ or

^aSecs. 18.3, 18.4.1, 18.4.2; Fig. 18.1; Table 18.1.

^bTable 22.1 and Secs. 22.4.1, 22.4.2, 22.5, and 22.6.

4 hours were required to heat to the required temperature range. The full impact^a of this time-lag was felt when the fluorination was delayed by operating error.

Absorber cooling was hastened by applying a compressed air stream to the vessels; otherwise, the cooling time-lag mentioned above would have been much greater.

It required about 14 hours to cool the absorbers to $< 100^{\circ}\text{C}$. following the desorption step. In this case also, compressed air was blown on the vessel after it had cooled to $\sim 300^{\circ}\text{C}$. This cooling step was not usually critical time-wise, however, because subsequent parts of the run could be carried on while the absorbers were cooling.

The absorber heat-up time during desorption was ~ 12 hours. The UF_6 -bearing vessel (FV-120) required about two hours longer than did the second absorber because of the heat required to decompose the $\text{UF}_6 \cdot 3\text{NaF}$ complex (23).

8.4.3 Inlet Pipe Heaters, FV-520A and -521A

These heaters were satisfactory. The heat-up time to 100°C . was ~ 2 hours. See Table 22.3, and Secs. 22.4.1, 22.4.2, 22.5 and 22.6.

8.4.4 Outlet Pipe Heaters, FV-520B and -521B

These heaters were satisfactory. The heat -up time to 100°C . was ~ 2 hours. See Table 22.3, and Secs. 22.4.1, 22.4.2, 22.5, and 22.6.

8.4.5 Gas Preheaters, FV-520C and -521C

These heaters were removed during the "C" runs after demonstrating^b their ineffectiveness in reducing the time-to-temperature of the absorbers. Their failure resulted from: (a) the small surface areas of the heated surfaces, (b) the low contact times between the gas and the heated surfaces, and (c) the presumably low over-all heat transfer coefficient.

8.4.6 Heat Lamps for Flanges

These heat lamps were effectively used to keep the absorber inlet and outlet pipe flanges (shown in Fig. 8.1) at $> 65^{\circ}\text{C}$. Without them the flanges were cold enough (i.e., $< 57^{\circ}\text{C}$.) to condense UF_6 (17, p. 4). The heat-up time to $> 65^{\circ}\text{C}$ was about one hour.

8.4.7 Absorber Instrumentation (Sec. 14.4.5; 13)

PE-38 indicated the pressure of the gases leaving FV-103 in line H-103-1. This instrument satisfactorily indicated plugs in the system piping to the scrubber, especially in FV-120 (Sec. 8.4.1b). But, in the effort to reduce the probability of UF_6 leaks in the system during Run E-3, the PE-38 connection was capped off (Sec. 9.4.7c).

^a This is the reason for listing items c. and d. as critical operating steps in Sec. 8.3.2.

^b Table 22.2.

Experience with instruments FE-5 and FE-12 paralleled that with FE-6 and FE-13 (Sec. 9.4.7c). Prior to capping off these connections as for FE-38 and others during Run E-3, however, FE-12 also indicated plug formation in FV-120.

FE-34 indicated the pressure of the gases in line H-120-1 between FV-120 and -121. This instrument enabled detecting plugs in FV-121 and downstream of this vessel throughout the VPP work. The plug in the FV-220 inlet during Run L-7, for example, was found with FE-34 (Sec. 9.4.3).

The various thermocouples used in this system were indispensable for monitoring these temperatures: (a) absorber beds and gas inlets and outlets (as shown on Fig. 8.1), (b) external surfaces of the vessels, and (c) heated duct (Sec. 7.4.3).

8.5 Summary and Conclusions

Each absorber was capable of sorbing 10 kg of U as UF_6 (< 50% theoretical capacity) when packed with 13 kg of nickel shot and 25 kg of 1/8-in. NaF pellets. About 25 to 30 g of U passed through one such absorber while only ~ 11 g of U passed through four vessels in series (FV-120, FV-121, FV-220, and FV-222 in that order) as normally used in the Volatility Pilot Plant. After a normal desorption at ~ 400°C, about 14 g of U remained equally distributed on the absorber bed.

UF_6 in the tail gas stream did not represent a process loss. This material was sorbed on NaF in FV-124 and was later reclaimed by aqueous processing.

Attempts to use either crushed NaF or $NaHF_2$ instead of 1/8-in. NaF pellets met with difficulties as outlined earlier.

Operational experiences with the absorber vessels included:

- a. Mark IB thermowells were superior to those in Mark IA.
- b. The small necks made unloading the NaF from the absorbers difficult.
- c. The uneven gas distribution of the diffuser rings was probably improved by using nickel shot.
- d. The 10-in. diameter of the NaF bed made it difficult either to heat or cool the center of the bed.
- e. Diffuser ring hole plugs and bed agglomeration were significantly lower in post-CRP trap runs.
- f. Coloration of NaF pellets qualitatively showed the presence of both U and Cr.
- g. The mechanism causing the disintegration of the FV-120 diffuser ring is unknown although three out-of-the-ordinary practices were performed during the associated runs.

- h. Absorber shielding was adequate for the activity of the material handled.
- i. Leaks in both vessel top flanges were found with the flange leak detector system.

The absorber furnaces were adequate although 12 to 14 hours were required for desorption. Cooling the absorbers after desorption by blowing compressed air on the vessels required ~ 14 hours.

Prior to sorption, it required ~ 4 hours either to heat a cold absorber or cool a warm absorber to the desired 65-75°C. temperature range. This resulted in an operating time loss unless done simultaneously with other preparations for fluorination. Prolonged cooling after sorption also caused time loss in desorption.

The performances of auxiliary heaters were:

- a. Inlet and outlet line heaters were satisfactory and required ~ 2 hours to reach the operating temperature of 100°C.
- b. Heat lamps requiring a one-hour heat-up time were adequate for maintaining the inlet and outlet pipe flanges at > 65°C.
- c. Gas preheaters were unsatisfactory.

The instrumentation was partially satisfactory. All pressure instruments (PE-12, -34, and -38) successfully indicated downstream plug formation. But PE-12 and -38 were removed from service in Run E-3 to reduce the probability of having UF₆ leaks. FE-5 was never used because of the improper range. Thermocouples were used to measure temperatures.

8.6 Recommendations

It is recommended that:

- a. The absorber be redesigned with at least these features: (a) shorter heating and cooling times, easier unloading, and more uniform gas distribution than in Mark I; (b) adequate provision for unit shielding based on the material to be processed; (c) incorporation into a flange leak detection system; and (d) provision for inlet and outlet line heaters and flange heat lamps as before if these are needed.
- b. Two absorbers be used packed with 1/8-in. NaF pellets in series as before with some form of CRP trap upstream of the first absorber to reduce plugging in the first absorber.
- c. None of the three extraordinary practices associated with the FV-120 diffuser ring failure be used.
- d. Operating procedures be altered to ensure having the absorbers in the designated temperature range (65-75°C.) when ready for fluorination and also when ready for desorption to avoid a loss of operating time.

- e. The use of gas preheaters similar to those (FV-520 and -521C) used during early absorber work be discontinued.
- f. Pressure instrument(s) continue to be used to indicate down-stream plug formation.

8.7 Operating Procedure: Description of Product (Revised November 1, 1957)

DOP _____
 Date _____
 Time _____
 Oper. _____

1. Close the following valves on the panel:
 HX-8 _____, HX-18 _____, HX-19 _____, HX-22 _____, HX-23 _____, HX-24 _____,
 HX-25 _____, HX-26 _____, HX-32 _____, HX-35 _____, HX-36 _____.
  2. Open the following valves on the panel:
 HX-12 _____, HX-13 _____, HX-14 _____, HX-15 _____, HX-16 _____, HX-30 _____,
 and HX-34 _____. Keep HX-11 open when fluorinator requires venting.
  3. Open V-27 _____ and V-72 _____ on roof. Close V-126 _____ (Cell one)
 Close V-127 _____ (Cell two)
  4. The following instruments should be in service and charts synchronized:
 LR-4 _____, FR-8 _____, PR-34 _____, PR-26 _____, TR-2A _____,
 TR-2B _____, TR-2C _____, TR-2D _____, TR-2E _____.
  5. Set the following controllers:
Controller - TIC	TIC Set Point*	Recorder Point **	Time Set
2A-11	130	120	_____
2A-12	145	120	_____
2B-2	58	80	_____
2B-9	135	105	_____
2B-10	135	105	_____
FV-527	100	80	_____
6. Adjust the settings on the following Variacs:
Variac - TC	Setting	Time Set
2A-11	60	_____
2A-12	60	_____
2B-2	90	_____
2B-9	60	_____
2B-10	60	_____
TC-527	80	_____
7. Record periodic data on sheets provided.

* Set point is setting on controller.

** Control point is reading on recorder.

8. Turn on brine switches in pipe tunnel _____. Set pressure on each brine reservoir at 10-15 psig.
- FV-830 _____, FV-832 _____. Start compressors on FV-830 _____, FV-832 _____. Time _____.
9. Turn on FV-352 _____ agitator in FV-152 and start FV-450 _____. Recirculate KOH through valves 4, 8, 10 into tower. Use (top, side) fluorine inlet line _____. Time _____.
10. Sample FV-152. Coded "SI".
11. Set PC-10 on "auto" and adjust set point to 4 psig (20% full scale).
12. Set PC-2 on manual _____.
13. Turn on fluorine supply and adjust FR-2 to 8 SIM (18% full scale). Time fluorine on _____.
14. When flows have levelled out, switch controllers to "auto".
15. Set these controllers:
Controller - TIC	Set Point	Control Point	Time Set
2A-5	a	400	_____
2A-8	b	400	_____
- a) Adjust so TR-2E-3 is maintained at 420°C. (Maximum).
- b) Adjust so TR-2E-9 is maintained at 420°C. (Maximum).
16. Adjust the settings on these Variacs:
Variac	Original Setting	Reduce (When Controlling)	Time Set
TC-2A-5	220		
TC-2A-8	220		
17. Record data on sheets provided.
18. Continue operation until TR-2A-6, -7, -9, and -10 and TR-2E-5, TR-2E-11, each exceeds 390°C.
Point	Time Reached 390°C.
TR-2A-6	
TR-2A-7	
TR-2A-9	
TR-2A-10	
19. When all above points reach 390°C, shut off these controllers: TIC-2A-5 _____, TIC-2A-8 _____, TIC-2A-11 _____, TIC-2A-12 _____, TIC-2B-9 _____, TIC-2B-10 _____. Time _____.
20. Fifteen minutes after step 19, shut off F₂ flow _____. Reset FR-2 and PC-10 to zero output pressure _____. Time _____.
21. Set FR-1 at 50%.
22. Set PI-6 on 4.5 psig _____. Time _____.
23. Forty minutes after step 21, close FV-1 _____.
24. Close: HX-12 _____, HX-13 _____, HX-14 _____, HX-16 _____, HX-30 _____, HX-34 * _____, HX-35 * _____, and HX-36 * _____. Time _____.
25. Remove FV-120 _____, FV-121 _____, and FV-124 _____ for sampling.
26. Sample FV-152. Code SI _____.
27. As soon as FV-124 is reloaded and replaced, open HX-34 _____, HX-35 _____, HX-36 _____. Time _____.
- By _____

* Omit unless otherwise instructed.

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9.0 COLD TRAPPING SYSTEM

9.1 Introduction

In the cold trapping system, the UF_6 desorbed from the absorbers was collected from the F_2 -bearing sweep gas by freezing it out on two series-connected cold traps. To avoid UF_6 loss, the exit sweep gas from the cold traps passed through a chemical trap before being scrubbed in the caustic scrubbing tower. Major steps in operating this system were:

- a. Keeping the two cold traps at $-40^\circ C$. and $-55^\circ C$. respectively, and having the chemical trap and scrubber operating normally.
- b. Directing the appropriate UF_6 -bearing gas stream through the two cold traps, the chemical trap, and caustic scrubber in that order.

9.2 Equipment

The general arrangement of the equipment in the cold traps system is shown in Fig. 9.1. The measured volumes of the cold traps are given. Details of individual components are recorded in Table 9.1.

9.3 Operation

9.3.1 Operating Procedure

Steps in the operating procedure for the cold traps system were:

a. Cooling Procedure

1. Having the F-11, -13, and -22 circuits filled with their respective Freons, the cooling water turned on, the thermostat set at the desired operating temperature for each unit ($-40^\circ F$ for FV-830 and $-60^\circ F$ for FV-832), and the brine cooler switch at each unit turned on.
2. Making certain that the pressure in each cold trap vacuum jacket was < 3 in. of Hg. [PA-36 and -37 sounded when the vacuum jacket pressures were > 3 in. of Hg. When this happened, the pressure switches (PK-36 and -37) could be reset by pumping down the jackets to an absolute pressure below one in. of Hg.
3. Turning on the refrigerating units switches at the Main Panel-board.
4. Setting the F-11 flow rate in each circuit at about $4\frac{1}{2}$ gpm by adjusting valves V-60 and -61.
5. Performing the desired operational step, i.e., the feed salt fluorination (Sec. 5.3.1), the desorption of product (Sec. 8.3.1), or the collection of residual UF_6 vapor in product collection

Volumes of Cold Traps as Installed in VPP (measured by R. G. Nicol)
 FV-220 (Bounded by HCV's 13, 15, and 19)---12.9 liters. FV-222 (Bounded by HCV's 14, 15, 18, and 22)---50.0 liters.

Fig. 9.1. Equipment Arrangement in the Cold Trapping System

Table 9.1
List of Cold Trapping System Components

Components	No.	Drawings and Sketches Titles	Miscellaneous Information	References
9" Cold Trap and Heaters, FV-220	D-34891 22757	Wiring Diagram (sheet 5, items "A", "B", "C") Assembly and Details of Vacuum Jacket for FV-220	Control Equipment - Wheelco (thermocouple) and Varian for each of three heating units; inlet, middle and outlet. Period of Use - All Runs. Heater Data - See Vendor's Drawings. Vessel Data - Manufactured by Vulcan Manufacturing Div. of the Vulcan Copper and Supply Co., Cincinnati, Ohio; expansion joints fabricated by Flexonics Corp.; Req. No. WJX-27688	This Report - Secs. 9.4.1; 9.3.2; 9.4.2; 9.4.7, item b; 9.5; 9.6 Table 22.3 Engineering File Folder No. F-41. Other Literature - 37, 21, 22 (pp. 3, 19, 20-22, 39, 45, 47).
Refrigerating Unit for FV-220, FV-830	D-23193, Rev. 5 D-34893 Vendor Dwg. E-36-28 R-512-3A E-14-3C 2883-35 R-512-3A	Refrigeration and Scrubber Make-up Flowchart Wiring Diagram (sheet 7, item "A") Brine System Electrical Schematic Diagram Brine and Refrigeration Schematic Brine System Wiring Diagram General Layout Brine and Refrigeration Schematic	Control Equipment - Freon 11 flow rate, FE-11; Freon 11 temperature controller was built into the unit. Period of Use - All Runs. Refrigerating Unit - Tenny Engineering Co., Inc., Baltimore, Md.; Serial No. 2347, Model No. Kolpak; Refrigerants: 8 lb. of F-22 and 3 lbs. of F-13; Coolant (brine): F-11; flow rate: 5 gpm at a 20-ft. head; K-61967, P. O. No. WJX-2348-1.	This Report - Secs. 9.4.2; 9.3.2; 9.4.7, item a; 9.5, 9.6 Engineering File Folder No. F-5a. Other Literature - 35, 22 (pp. 4, 25-27, 47).
Inlet and Brine Heater, FV-227 (FV-220)	D-22761 D-34895	Cell II Engineering Flowchart Wiring Diagram (sheet 9, item "D")	Control Equipment - Pyrovarc (thermocouple welded to the FV-220 inlet line between adjacent turns of the heating cable) and Varian. Period of Use - All Runs. Heater Data - ~90 ft. of industrial heating cable was wrapped helically around the FV-220 inlet and product drain lines; advance on helix was ~3/8" ; power required : ~400 watts, 110 volts.	This Report - Secs. 8.4.7; 9.4.3; 9.4.1; 9.5; 9.6; 22.4.1; 22.4.2; 22.5; 22.6 Table 22.3 Engineering File Folder No. F-41. Other Literature - 21.
6" Cold Trap and Heaters, FV-222	D-22761 D-34891 E-21358 E-22756	Cell II Engineering Flowchart Wiring Diagram (sheet 5, items "D", "E", and "F") 6" Cold Trap (FV-222) Assembly and Details of Vacuum Jacket for FV-222.	Control Equipment - Wheelco (thermocouple) and Varian for each of three heating units, inlet, middle and outlet. Period of Use - All Runs. Vessel and Heater Data - Fabricated at E-25, cf. dwgs. expansion joint fabricated by Flexonics Corp., P. O. No. WJX-27688.	This Report - Secs. 9.4.4; 9.4.1; 9.4.7, item b; 9.5; 9.6 Table 22.3 Other Literature - 37, 22 (pp. 3, 19, 22-24, 39, 45, 47).

¹The thermocouple for each Wheelco was welded to the vessel shell with its leads being brought through the vacuum jacket in 1/4-in. MP6. This pipe was welded to the jacket at one end and sealed at its other extremity outside of the vessel insulation where the thermocouple leads emerged from it.

Table 9.1 (Continued)

Components	No.	Drawings and Sketches Title	Miscellaneous Information	References
Refrigerating Unit for FV-222, FV-832	D-34893 (See Dugs, for FV-830)	Wiring Diagram (sheet 7, item "B")	Control Equipment - Freon 11 flowrate, PB-12; Freon 11 temperature controller was built into the unit. Period of Use - All runs. Refrigerating Unit - Tenney Engineering Co., Inc., Baltimore, Md., Serial No. 2348, Model No. Kolpak; Refrigerants: 6 lb. of F-22 and 4 lb. of F-13; coolant (brine): F-11; flow rate: 5 gpm at a 30°F. heat; I-61968, P. O. No. W29K-4348-1.	This Report - Secs. 9.4.5; 9.4.6; 9.3.2; 9.4.4; 9.4.7, item a; 9.4.7, item b; 9.5 and 9.6. Table 22.3. Engineering File Folder No. F-34 Other Literature - 35, 36 (pp. 4, 25-27, 47).
Cold Traps Instrumentation	D-22761 - D-23193, Rev. 5	Cell II Engineering Flowchart Refrigeration and Scrubber Make-Up Flowchart	Functions of Instruments: PB-6 Gas Flowrate into FV-220. PB-7 Gas Flow rate between FV-220 and FV-222. PB-8 Gas Flow rate into FV-150. PB-11 Freon 11 Flow rate out of FV-830. PB-12 Freon 11 Flow rate out of FV-832. PB-13 Gas Pressure into FV-220. PB-14 Gas Pressure between FV-220 and FV-222. PB-15 Gas Pressure Out of FV-222. PB-16 Gas Pressure Into FV-150. PA-36 Pressure Alarm on FV-220 Vacuum Jacket. PK-36 Pressure Switch on FV-220 Vacuum Jacket. PI-36 Pressure Indicator on FV-220 Vacuum Jacket. PA-37 Pressure Alarm on FV-222 Vacuum Jacket. PI-37 Pressure Alarm on FV-222 Vacuum Jacket. PI-37 Pressure Indicator on FV-222 Vacuum Jacket.	This Report - Secs. 9.4.7; 9.4.7, item a; 9.4.7, item b; 9.4.7, item c; 9.4.7, item d; 9.4.7, item e; 9.4.7, item f; 9.4.7; 9.3.2; 9.4.1; 9.4.2; 9.4.4; 9.4.5; 9.5; 9.6; 7.4.2; 10.4.1; item b; 10.4.13; 21.4.2. Engineering File Folder No. F-42. Other Literature - 13.
Main Process Chemical Trap, FV-124	D-19702	FV-124 Chemical Trap	Period of Use - All Runs Trap Packing - 11 kg. of 1/8-in. NaF pellets.	This Report - Secs. 9.4.6; 9.5.5; 9.6; 8.4.1, item b; 10.4.12; 18.D. Engineering File Folder No. F-47. Other Literature - 52 (p. 29).
Batch Type UF ₆ Detector, FV-123	Sketch No. W-8-20-15 (Fig. 9.2)	FV-123 UF ₆ Detector	Period of Use - Runs M-50 through M-56.	This Report - Secs. 9.4.9; 9.5.6; 9.6. Fig. 9.2. Engineering File Folder No. F-47.

(Sec. 10.3.1). (Making certain that the main chemical trap was filled with NaF, and that the scrubber was operating properly were parts of these operational steps.)

6. After step (5) was finished, shutting off the refrigerating units by cutting off both the brine cooler switches at the units and the refrigerating units switches on the Main Panel-board. (Either one of the two switches for a given unit would cut off the unit whereas both had to be "on" to start it. But both switches should be turned off after use to prevent starting the unit by inadvertently throwing either switch.)

b. Heating Procedure

1. After step (6) in Sec. 9.3.1a, the inlet, middle, and outlet heaters of both cold traps were used to heat the cold traps to $\sim 80^{\circ}\text{C}$. (This was done only during product collection as described in Sec. 10.3.1.)
2. Periodically bleeding both F-11 reservoirs as necessary to keep the pressure at 10 to 15 psig.

9.3.2 Critical Operating Steps

- a. Maintaining the level of F-11 in each reservoir at about mid-way in the sight glass. (Because F-11 was vented during intermittently bleeding vapor from the brine circuit, F-11 additions were necessary at times.)
- b. Maintaining the F-13 and F-22 circuits of each refrigerating unit filled. (The frequency of replenishing these circuits varied. The inability of the units to attain the normal operating temperatures usually indicated the need for replenishing the F-13 and/or F-22.)
- c. Keeping 10 psig of dry instrument air pressure above the liquid in each F-11 reservoir. (This was specified in the Tenney operating instructions. The low head developed by the F-11 pump apparently necessitated this surge tank over-pressure.)
- d. Having cooling water flowing to each unit. (The cooling water was needed in the F-13 circuit. Since water was drawn to each unit automatically, the water supply was left on continuously. No cooling water flowed when the unit was "off".)
- e. Reading temperatures of the cold traps from recorders instead of from temperature controllers. (Since the temperature controllers had been calibrated in a different temperature range from that reached during product collection, the controller reading was misleading.)

- f. Having both the brine cooler switch at the unit and the switch on the panelboard "on" was required before either refrigerating unit would start.
- g. Bleeding off F-11 vapor was essential at times before F-11 could be pumped.

Starting F-11 to pumping was always difficult after having had the cold traps hot during product collection. Since the boiling point of F-11 was 74.7°F, the vapor pressure of F-11 with the cold traps hot was considerably above atmospheric pressure. F-11 vapor vapor-locked the brine pump. Until F-11 flow could be established, actions taken were: (1) to start the refrigerating unit, (2) to bleed off vapor from the system through the surge tank valve or the F-11 bleed valve (V-55 and/or -57 for FV-830 and V-56 for FV-832), and (3) to increase the dry instrument air pressure in the surge tank to ~ 20 psig. Since it was not ascertained which of these actions was most beneficial, the three were usually tried simultaneously. The time required to start F-11 flow in FV-830 with FV-220 hot was about one and one-half hours. The time required in FV-832 was usually about one-half hour.

At times this same trouble was encountered, although to a lesser degree, when starting the units in hot weather.

Detailed operating procedures are given in Sec. 9.7.1, appropriate parts of Secs. 5.7.1, 8.7.1, and 10.7.2, and the Tenney bulletin (34).

9.4 Equipment Evaluation^a

9.4.1 5-in. Cold Trap, FV-220

The operation of FV-220 was satisfactory with no maintenance being required. But two changes in the original design were needed:

- a. An additional heater was required to heat simultaneously the trap inlet line and product drain line adjacent to the trap. (For more detail, see Sec. 9.4.3).
- b. A means should have been provided to reduce the time required to start the F-11 flowing in the brine system. This change was never made. See Secs. 9.3.2, 9.4.2, and 9.6.

See Sec. 9.4.7b relative to evacuating the FV-220 vacuum jacket.

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^a After the "L" runs, most of the equipment was dismantled and moved to Burial Ground No. 3 (Sec. 23.4.16b). However, the cold traps were left in place for future processing.

9.4.2 Refrigeration Unit for FV-220, FV-830

The design operating temperature of -40°C . in FV-220 was attained. The FV-220 inlet and outlet heaters had little effect on the -40°C . operating temperature (35). Only one part replacement and minor miscellaneous maintenance were needed. After approximately 2500 hours of service, the thermal overload on the F-13 compressor burned out and was replaced. The three Freon systems needed replenishing intermittently. The F-11 circuit was refilled most frequently because starting the F-11 flow necessitated bleeding off F-11 vapor to eliminate vapor locks in the pump as delineated in Sec. 9.3.2.

Characteristic operating data of this unit for cooling FV-220 to -40°C . with the F-11 flow set at $\sim 4\text{-}1/2$ gpm were:

- With FV-220 initially at room temperature - $\sim 2\text{-}1/4$ hours.
- With FV-220 initially at $\sim 80^{\circ}\text{C}$. during product collection - $\sim 3\text{-}1/2$ hours. (This did not include the $1\text{-}1/2$ hours normally required to start the brine flow; Sec. 9.3.2.)

See Sec. 9.4.7a for information on FE-11.

9.4.3 FV-220 Heaters^a

The inlet, middle, and outlet heaters were satisfactory. Heating characteristics were:

- Time required to heat FV-220 from -40°C . to $\sim 80^{\circ}\text{C}$. was $3\text{-}1/2$ hours.
- Controller settings at operating temperature ($\sim 80^{\circ}\text{C}$.):

	Pyrovens, $^{\circ}\text{C}$.	Variac, volts
Inlet	58	*
Middle	70	*
Outlet	190	80

* Not available

In the early "M" runs, insufficient heating of both FV-220 and -222 inlet lines to keep these lines above 65°C . was discovered (35)^b. Neither the heated duct nor the cold traps inlet heaters adequately heated these lines. The satisfactory remedies were: (a) FV-527 for the FV-220 inlet and (b) insulation for the FV-222 inlet. FV-527 was wrapped around both the FV-220 inlet line and the product drain line adjacent to the cold trap because this drain line was also too cold.

^a Table 22.3.

^b That is, above the triple point of 64°C at 22 psia (17, p. 4).

The need for FV-527 was demonstrated later in Run L-7 (21, pp. 33,34).^a During the desorption of product in this run, this heater was left off accidentally. Melting the UF₆ plug which had formed delayed the desorption step several hours. The heating characteristics were: (a) Time from room temperature to operating temperature (~ 80°C.) was approximately one hour. (b) Pyrovane and Variac settings to maintain operating temperature were 120°C. and 50 v, respectively.

9.4.4 6-In. Cold Trap, FV-222

The operation of this trap was successful. In the "C" runs, normally < 5 g of U as UF₆ passed through FV-222 and was collected on FV-124 (36, 31, 29). A cast brass coupling failure resulted in a UF₆ leak in Run C-7 causing a low recovery in that run (37, p. 31). This coupling formed the closure for the TE-2C-6 thermocouple well. The alpha radioactivity released caused more concern than the loss of product because the product contained normal uranium. In the subsequent repair, an Inconel adapter with welded joints was used. At the same time, a similar change was made in the TE-2C-4 thermocouple well. No further trouble has been experienced with either thermocouple well.

The comment in Sec. 9.4.1 regarding reducing the time required to start F-11 flowing in the FV-830 brine system also applies here.

See Sec. 9.4.7b relative to evacuating the FV-222 vacuum jacket.

9.4.5 Refrigeration unit for FV-222, FV-832

The operation of this refrigerating unit was satisfactory although the -62°C. design temperature in FV-222 could not be reached. Consequently, FV-222 was operated with no known deleterious effect at a temperature of -55°C. The FV-222 inlet and outlet heaters had a pronounced effect on the trap temperature (35). Only minor miscellaneous maintenance involving the intermittent replenishing of Freons-11, 13 and 22 was required. As for FV-830, F-11 replenishing was most frequent.

Characteristic operating data of this unit for cooling FV-222 to -55°C. with the F-11 flow rate set at ~ 4-1/2 gpm were:

- a. With FV-222 initially at room temperature - ~ 3-1/2 hours.
- b. With FV-222 initially at ~ 80°C. during product collection - ~ 6-1/2 hours.

This time did not include the half-hour usually needed to start the brine flow (Sec. 9.3.2). The reason why the brine flow in FV-832 was started more quickly than in FV-830 was not ascertained.

See Sec. 9.4.7a for information on FE-12.

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^a Table 22.3 and Secs. 9.4.1, 9.4.7, 22.4.1, 22.4.2, 22.5 and 22.6.

^b Alpha radioactivity was monitored with a portable disk sampler (Sec. 23.4.3); γ-β smears (Sec. 23.4.8) and urine samples (Sec. 23.4.13) were taken; and masks and other protective devices were worn as needed (Sec. 23.4.14).

9.4.6 FV-222 Heaters^a

The inlet, middle, and outlet heaters were satisfactory.^b Heating characteristics were:

- a. Time required to heat FV-222 from -55°C. to ~ 80°C. was 2-3/4 hour.
- b. Controller settings at operating temperature (~ 80°C.):

	<u>Pyrovane, °C.</u>	<u>Variac, volts</u>
Inlet	75	*
Middle	85	*
Outlet	95	170

* Not available

9.4.7 Cold Traps System Instrumentation (13)

- a. FE-11 (FV-830) and FE-12 (FV-832)^c

These orifices measured the Freon -11 flow rates in the brine circuits. After the initial leaks were eliminated, these flow meters were satisfactory. Generally, the flow rates were set at ~ 4-1/2 gpm by adjusting V-61 for FE-11 and V-60 for FE-12. Since the flow rates tended to increase slightly with time, intermittent adjustments were required to maintain the desired values.

- b. PA-36, PX-36, and PI-36 (FV-220); PA-37, PX-37 and PI-37 (FV-222)

The inner liner of each cold trap was surrounded by a vacuum jacket serving two purposes: (a) primarily to detect a leak in the inner liner and (b) secondarily to aid in insulating the inner liner.^d A pressure alarm, a pressure switch, and a mercury manometer monitored the pressure in each vacuum jacket. The pressure alarm and pressure switch worked as follows:

1. Contacts on the pressure switch closed when the absolute pressure fell to 1 in. of Hg. This set the pressure switch for actuation described in item 2.

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^a Table 22.3.

^b Comment on heating FV-222 inlet line in Sec. 9.4.4.

^c Secs. 9.4.2 and 9.4.5.

^d Secs. 9.4.1 and 9.4.4.

2. Contacts on the pressure switch opened when the absolute pressure rose to three in. of Hg. thereby setting off the pressure alarm on the Main Panelboard.

NOTE: After the alarm sounded following the rise of the pressure to 3 in. of Hg (abs.), it was necessary then to evacuate the jacket to a pressure of 1 in. of Hg (abs.) to set the pressure switch for another actuation (item 1).

The mercury manometer could be used as desired to determine the pressure in the vacuum jacket.

This instrumentation was satisfactory. No known leak to the vacuum jacket of either cold trap occurred. The pressure alarms did sound a few times during the two and one-half year period in which the "C", "E", and "L" runs were made. These alarm actuations were attributed to very slow leaks in the vacuum jackets and showed that the instrumentation worked properly. In each case, the vacuum jacket was evacuated to an absolute pressure below one in. of Hg to reset the pressure switch by opening the appropriate valve, V-51 for FV-220 and V-52 for FV-222. The frequency of occurrence was approximately semiannually or annually.

Although the mercury manometers were adequate for intermittently checking the pressure in the jackets, they had these disadvantages:

- (a) Required too much space in the cell.
- (b) Could be blown free of mercury by pressurizing.
- (c) Had glass covers which were easily broken.

Another type of pressure instrument would have been more suitable.

c. FE-6 and PE-13^b

These pneumatic-type instruments monitored flow rate and pressure at the inlet to FV-220. In early work, neither of these instruments was useful because: (1) the range of FE-6 was too great to measure VFP flow rates and (2) the range of PR-13 (0-1 psig)

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^a The vacuum pump used was FV-420 (Sec. 10.4.12).

^b Sec. 14.4.5.

was too small for the line pressures encountered about half the time. Changing the ranges to match VPP conditions approximately coincided with the decision made in Run E-3 to reduce the probability of leaks by capping off some instrument connections.^a Consequently, little subsequent work was done with FE-6 and PE-13. Since the remaining "E" and the nine "I" runs were made successfully without them, these instruments were not needed to operate the plant. But flow rate and pressure data at this point would have been helpful to operating as well as to data processing personnel.

d. FE-7, PE-14 and -15^b

The experiences with these instruments paralleled that with FE-6 and PE-13. One difference was that PE-14 and -15 were used in the high-pressure portion of VPP whereas PE-13 was not. Because the pressure in this portion of VPP was 50 psig during product collection an N_2 purge of $\sim 4\frac{1}{2}$ to 10 psig could not be used. In addition, bleeding N_2 into the cold traps while draining product would have increased the quantity of inert gases therein, and UF_6 could have collected in the cold purge lines between the cold traps and the Main Transmitter Rack. Because of these three objections to using the customary pneumatic-type instrument with an N_2 purge, two other schemes were tried:

- 1.) Valving off the purge lines during product collection and not using these instruments in that operational step.

Scheme 1. was beset with UF_6 leaks: (a) at tubing and pipe joints and (b) at the three-way solenoid used in one design at PE-15 because of a mistake in connecting lines to its ports (Sec. 7.4.2).

2. Putting the pressure transmitter in the heated duct with no N_2 purge as for PR-26 (Sec. 10.4.13).

Scheme 2. was preferable to scheme 1. But this was never tried because these instruments were put out of service along with others after Run E-3 (Sec. 9.4.7c).

e. FE-8 and PE-16^b

As for FE-6 and PE-13, the original-design instruments had inadequate ranges. The subsequent instruments (FE-8 with a range of 0-35 liters/min. and PE-16 with a range of -0.5 to + 4.5 psig) were satisfactory. In addition to monitoring the gas flow

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^a Besides FE-6 and PE-13, other instruments taken out of service at that time were FE-5, -7; PE-12, -14, -15, and -38. (Sec. 8.4.7).

^b Sec. 14.4.5

to the caustic scrubber, FE-8 warned operating personnel of any unusually high gas flow which might indicate that a valve in the system had been inadvertently left open. One of the most helpful uses of FE-16 was its pressure rise occurring when the scrubber inlet line plugged. One disadvantage was that leaks in this vicinity were hard to find because of the large number of joints.

f. Thermocouples

The thermocouples used to measure temperatures were satisfactory and gave indispensable operating data. As stated in Sec. 9.3.2, relying on temperature controllers for temperatures instead of temperature recorders was poor practice. Thermocouples were placed as described in Sec. 22.4.2.

9.4.8 Main Process Chemical Trap, FV-124

The operation of this trap was relatively satisfactory.^a Uranium retentions have varied from zero to 5,334 g with essentially no uranium passing through the trap (27, 25, 36, 31, 29, 22, 21, and 24). Retained uranium did not represent a loss because it was recoverable (32). An upper NaF retainer screen was added after pressure surges from 50 psig to atmospheric pressure forced NaF pellets up to V-72 in the Penthouse. Two other changes as yet not made are desirable:

- a. Incorporating the top flange in the flange leak-detector system (Sec. 18).
- b. Altering the trap design to facilitate loading and unloading.

The normal FV-124 charge was 11 kg of 1/8-in. NaF pellets. NaF pellets were preferable to the minus 20-mesh NaF particles tried because of the absence of fines. Unloading FV-124 was done either with a portable vacuum cleaner or with FV-420 (Sec. 10.4.12).

In the early VFP work when NaHF_2 was converted to the NaF in the absorbers, FV-124 was left empty to prevent the formation of NaHF_2 therein (Sec. 8.4.1b).

9.4.9 Batch-Type UF_6 Detector, FV-123

During the "C" runs, a batch-type UF_6 detector was built and used in the inlet line to FV-124. The operating principle of this detector was that the increase in temperature occurring when a small quantity of NaF sorbed UF_6 could be detected with a thermocouple. The design of the device is shown in Fig. 9.2. It worked satisfactorily but was not used in enriched-uranium runs because of the frequent changing required.

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^a Sec. 8.4.1b.

Fig. 9.2. Batch-Type UF_6 Detector, FV-123

9.5 Summary and Conclusions

9.5.1 Entire Cold Traps System

The operation of the cold traps system was satisfactory. Some minor design changes were made while a few needed design changes have not yet been done. Heating characteristics of the various heaters are recorded.

9.5.2 FV-220 (5-in. Cold Trap) and FV-830

The design operating temperature of -40°C . was attained. Since the temperatures of the inlet and product drain lines were $< 65^{\circ}\text{C}$. while the trap was cold, FV-527 was added to prevent UF_6 from solidifying in these lines. One UF_6 plug in the inlet line during Run L-7 when FV-527 was inadvertently left off confirmed the need for this heater. The inlet and outlet heaters had little effect on trap cooling.

Maintenance on FV-830 included replacing a thermal overload after about 2500 hours of service and intermittently replenishing the three Freon circuits.

At a $4\text{-}1/2$ gpm flow rate of F-11, the time required to cool FV-220 from room temperature to -40°C . was $2\text{-}1/4$ hours. With the trap initially at $\sim 80^{\circ}\text{C}$. after product draining, the cooling time under similar conditions was $3\text{-}1/2$ hours, this time excluding the $1\text{-}1/2$ hours required to start F-11 flowing. A way to decrease the time to start F-11 flowing was needed but never received any design attention.

9.5.3 FV-222 (6-in. Cold Trap) and FV-832

The 6-in. cold trap attained only -55°C . instead of the design value of -62°C . However, this fact had no known deleterious effect on plant operation. Since the FV-222 inlet line could not initially be heated to $> 65^{\circ}\text{C}$. while the trap was cold, this line was insulated. The inlet and outlet heaters had a pronounced effect on trap cooling.

A cast brass coupling on a thermowell failed in Run C-7 releasing a quantity of normal UF_6 in Cell 2. The α radioactivity released was of more concern than the loss of the normal uranium. In the repair, an Inconel adapter with welded joints was used. Another thermowell of similar design was changed in the same way.

In the "C" runs, normally < 5 g of U as UF_6 passed through both FV-220 and -222 in series.

The only maintenance work on FV-832 was intermittently replenishing the Freons in its three circuits.

At the $4\text{-}1/2$ gpm flow rate of F-11, the time required to cool FV-222 from room temperature to -55°C . was $3\text{-}1/2$ hours. With the cold trap initially at $\sim 80^{\circ}\text{C}$. after product draining, the cooling time was $6\text{-}1/2$ hours, this

time being exclusive of the half-hour needed to start the F-11 flow. Why it took three times as long to start the F-11 flow in FV-830 as in FV-832 was not ascertained. At any rate, the need for a means of reducing such time losses was evident.

9.5.4 Instrumentation

FE-11 and -12 were satisfactory as installed. Usually the F-11 flow rates on both FV-830 and -832 were set at about 4-1/2 gpm. To maintain these flow rates, intermittent valve adjustments were required.

PA-36, PX-36, and PI-36; PA-37, PX-37, and PI-37 performed satisfactorily, although no UF_6 leak apparently occurred to detect with them. PA-36 and PX-36; PA-37 and PX-37 actuated at semiannual or annual frequencies. Such actuations were attributed to slow leaks in the vacuum jackets. When this occurred, normal operating conditions were restored by again evacuating the vacuum jacket to <1 in. of Hg (abs.). The mercury manometers were space consumers and subject to mercury blowing and glass breakage. Another type of pressure instrument would have been more appropriate.

FE-6 and -7; PE-13, -14, and -15 were initially installed with improper ranges for VPP. Obtaining instruments with suitable ranges and the decision to eliminate these instruments to reduce the leak probability in VPP occurred almost simultaneously. Consequently, these instruments were no longer used, and the purge lines were capped in Cell 2. The experiences encountered with PE-14 and -15 are recounted as a matter of record because these instruments were in the high-pressure portion of VPP during product collection. Pressures could have been satisfactorily measured at these points with arrangements similar to that for PE-26. (Sec. 10.4.13).

FE-8 and PE-16 initially also having had unsuitable ranges were satisfactory after the ranges were changed. These instruments were useful in normal operations, in trouble-shooting, and in data work.

Thermocouples were successfully used and found to be indispensable.

9.5.5 FV-124

This trap was relatively satisfactory, passing little or no UF_6 at loadings up to ~5,000 g of U.

An NaF retaining screen at the top of the trap bed was added to prevent the blowing around of NaF pellets in the system.

Two needed items not yet done are: (a) adding the top flange to the flange leak-detector system and (b) altering the trap design to facilitate loading and unloading.

The normal trap charge was 11 kg of 1/8-in. NaF pellets.

9.5.6 FV-123

This UF₆ detector worked satisfactorily but was not needed in the "E" and "L" runs.

9.6 Recommendations

It is recommended that the cold traps system be operated in the future with the equipment as described herein except for the following changes:

- a. A means of reducing the time required to start P-11 flowing in the brine circuits after product draining be provided, especially for the FV-220 and -830 combination.
- b. A new design FV-124 be built to facilitate loading and unloading.
- c. The mercury manometers (PI-36 and -37) be replaced with more suitable pressure instruments.
- d. Any future PE placed in the high-pressure portion of VPP be installed similarly to PE-26.
- e. No more cast brass fittings be used in the VPP (Sec. 15.6).

9.7 Operating Procedure: Operation of Refrigeration Systems

Preliminary to startup, the three circuits of each system will have been filled with Freon-22 (high stage), 13 (low stage) and 11 (brine circuit). The power circuit to the compressors will be "on", and the brine cooler switch will be closed.

NOTE: Vent valves 55, 56, and 57 should not be opened as this would bleed off Freon gas.)

- ___ 1. Open these valves to provide cooling water to the first stage:
63 ____, 64 ____, 65 ____, and 66 ____.
- ___ 2. Open these valves on refrigeration lines near FV-830 and FV-832:
58 ____, 60 ____, 61 ____, and 62 ____.
- ___ 3. Turn on FV-830 and FV-832 at the Main Panelboard.
- ___ 4. A temperature controller is provided at FV-830 and another at FV-832 which can be set to give a desired temperature. The temperature is indicated on TE-2D-2 and TE-2D-3, respectively.

To Shutdown:

- ___ 5. Close V-60 ____ and V-61 ____ on outlets from refrigeration system. Turn off FV-830 ____ and FV-832 ____ control circuits on Main Panelboard.

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10.0 PRODUCT COLLECTING SYSTEM

10.1 Introduction

The UF_6 product having been previously frozen out in the cold traps after desorbing was heated and then collected in the product cylinder. The product was transferred in both the liquid and gaseous states, finally being frozen out in the product cylinder. The principal steps in the operation of this system were:

- a. Draining liquid UF_6 to the product cylinder.
- b. Thermally transferring UF_6 vapor to the chilled product receiver.
- c. Purging residual UF_6 out of the lines.

10.2 Equipment

The general arrangement of the product collecting system is shown in Fig.

10.1. Descriptions of the components are given in Table 10.1.

10.3 Operation

10.3.1 Operating Procedure^a

Steps in the operation of the product collecting system were:

- a. Having ~10 kg of U as UF_6 frozen out on the cold traps; 11 kg of 1/8-in. NaF pellets in FV-124; 3 kg of NaF pellets in FV-222; FV-420 operating; and joints at FV-122 and -124 leak-tight (as determined by the KI-starch leak-test described in Sec. 17.4.2, e2.)
- b. Taring and connecting the product cylinder to the pigtails; putting WE-1B in service.
- c. Conditioning the product cylinder and leak-testing (KI-starch method) the pigtail connections and valves. [Conditioning was completed at ~ 5 to 10 psig F_2 pressure (Sec. 15.4.5a) and then the KI-starch leak-testing was done with the system filled with F_2 at this pressure (Sec. 17.4.2, e2)].
- d. Heating the product cylinder to ~80°C.^b

^a Product collecting required 10 to 12 hr from system evacuation to closing the cylinder valves.

^b While draining the liquid UF_6 , the system was kept at a temperature of ~80°C, i.e., safely above the triple point (64°C. at 22 psia) but not high enough to vaporize all of the UF_6 (17, p. 4).

Fig. 10.1. Equipment Arrangement in the Product Collecting System

Table 10.1
List of Product Collecting System Components

Components	Drawings and Sketches		Miscellaneous Information	References
	No.	Title		
Product Cylinder, PV-126	K-25 Dugs.		<u>Control Equipment</u> - PV-326.	<u>This Report</u> -
	D-22-1954-5A	Withdrawal Cylinder Details	<u>Period of Use</u> - All Runs.	Secs. 10.4.1; 10.3.1; 10.4.2; 10.4.3; 10.5; 10.6;
	Ref. No. 17.	A Brief Guide to UF ₆ Handling	The cylinders were color-coded and numbered. In color-	17.4.1, item f.
	Figs. 1 and 2		coding, a yellow band was painted around the top. Then,	<u>Engineering File Folder</u> No. F-44.
	<u>Sketches</u>		with black letters on the band, "O.S.M.L." was lettered	<u>Other Literature</u> - 17, 22 (pp. 24, 25, 56).
	B-7-19-15	UF ₆ Product Cylinder Valve Orientation	on one side and "V.P.F." on the other side.	
	B-3-11-15	F.V.P.F. Product Cylinder Holder		
Product Cylinder Heater, PV-526	D-22763	Cell II Engineering Flowsheet	<u>Control Equipment</u> - Pyrovane (thermocouple was put	<u>This Report</u> -
	C-22797	Adjustable Base for Product Furnace	into the well in PV-126) and Varisc.	Secs. 10.4.2; 10.3.1; 10.4.1; 10.5; 10.6.
		PV-526 (The print is in R. P. Milford's	<u>Period of Use</u> - All Runs.	Table 22.3.
		File).	<u>Heater Data</u> - No. 840-381 manufactured by Trent, Inc.,	<u>Engineering File Folder</u> No. F-44.
	<u>Sketch No.</u>	Half of new Product Receiver	Philadelphia, Pa.; P. O. No. 425K-16634, Reg. No.	<u>Other Literature</u> - 22 (pp. 25, 47).
	M-4-9-15	Furnace Lid	D-55650; 1.6 kw., 220 v., 60 cycles, single phase, max.	
	D-34891	Wiring Diagram (sheet 3, item "D")	temp. - 200°F. After receipt at ORNL, heater was	
			modified to incorporate inlet and outlet air ports for	
			cooling PV-126.	
Product Cylinder Inlet and Outlet Lines Heater, PV-526A	D-22761	Cell II Engineering Flowsheet	<u>Control Equipment</u> - Pyrovane (thermocouple welded to	<u>This Report</u> -
	D-22780	Wiring Diagram (sheet 6, item "E")	one of the pipelines) and Varisc.	Secs. 10.4.3; 10.3.1; 10.5; 10.6; 21.4.1; 21.4.2;
			<u>Period of Use</u> - All Runs	21.5; 21.6.
			<u>Heater Data</u> - About 50 ft. of industrial heating cable	Table 22.3.
			was wrapped helically around the inlet and outlet lines	<u>Engineering File Folder</u> No. F-19.
			starting on one line and ending on the other; Advance	
			of helix was 3/8 in. per turn. Power: ~400 w. (110 v.);	
			uninsulated.	
Product Pigtail Heater, PV-526B	D-34895	Wiring Diagram (sheet 9, item "C")	<u>Control Equipment</u> - Pyrovane (thermocouple welded to	<u>This Report</u> -
			one of the pipelines) and Varisc.	Secs. 10.4.4; 10.3.1; 10.5; 10.6; 21.4.1; 21.4.2;
			<u>Period of Use</u> - Runs 1-2 through -9.	21.5 and 21.6.
			<u>Heater Data</u> - About 50 ft. of industrial heating cable	Table 22.3.
			was wrapped helically around the two pigtails starting	<u>Engineering File Folder</u> No. F-44.
			on one pigtail and ending on the other; advance of helix	<u>Other Literature</u> - 19.
			was ~3/8 in. per turn. Power: ~400 w. (110 v.);	
			uninsulated. Two 250-w. heat lamps were used to heat	
			the product cylinder valves. Prior to installing the	
			heating cable, heat lamps were used on pigtails and	
			valves.	

Table 10.1 (Continued)

Components	Drawings and Sketches		Miscellaneous Information	References
	No.	Title		
Product Weigh Station, FV-922	Q-1679-W-12-E	Weigh Element Details	<u>Period of Use</u> - All Runs.	<u>This Report</u> -
	D-22787	Product Weigh Station Assembly	Weigh element: Strain gauge. Manufactured by Baldwin - Lima - Hamilton; model U-1, range: 0-100 lb., P. O. No. W39K-18359, Req. No. D-35984.	Secs. 10.4.5; 10.4.8; 10.4.4; 10.5; 10.6
	D-22788	Product Weigh Station Details		<u>Engineering File Folder No. F-43</u> <u>Other Literature</u> - 52 (D. 53).
Product Cooling Coil, FV-226	D-21442	Cooling Equipment for Product Vessel, FV-126	<u>Control Equipment</u> - FV-820.	<u>This Report</u> -
	D-22788	Product Weigh Station Details	<u>Period of Use</u> - All Runs.	Secs. 10.4.6; 10.5; 10.6.
	D-34887	Wiring Diagram (sheet 1, item "A")	<u>Cooling Coil Data</u> - A packaged unit purchased from the Trane Co., LaCrosse, Wis.; P. O. No. W39K-50568.	<u>Engineering File Folder No. F-44</u> .
Product Cooler Blower, FV-622	D-21442	Cooling Equipment for Product Vessel, FV-126	<u>Period of Use</u> - All Runs.	<u>This Report</u> -
	D-22788	Product Weigh Station Details	<u>Blower and Motor Data</u> - X-49230, IAG Electric Co., No. 181-012EA, Style E-3, No. B-12 Fan, 450 cfm, 1/6 H.P. motor, 1750 rpm, 115 v.	Secs. 10.4.7; 10.5; 10.6.
	D-34887	Wiring Diagram (sheet 1, item "A")		<u>Engineering File Folder No. F-44</u> .
Refrigeration Unit for Product Cooler, FV-820	D-21442	Cooling Equipment for Product Vessel, FV-126	<u>Control Equipment</u> - Temperature controls were built into the units.	<u>This Report</u> -
	D-22788	Product Weigh Station Details	<u>Period of Use</u> - All Runs	Secs. 10.4.8; 10.5; 10.6.
	D-34887	Wiring Diagram (sheet 1, item "A")	<u>Refrigerating Unit</u> (two stages) Copeland, Sidney, Ohio. <u>Upper Component (Cool Stage):</u> X-47690, model 55 WFL, Serial No. 1006081; Charge-Freon-12. <u>Lower Component (Cold Stage):</u> X-47689, model 55 WFL, Serial No. 1007265; Charge-Freon-12. Motors: two A.C. Wagner Electric Co., 1/2 H.P., 1725 rpm, 1 phase, 60 cycles, 115 v., 7.6 a.	<u>Engineering File Folder No. F-44</u> .
Coolant Container for FV-224, FV-924	None	None	<u>Period of Use</u> - Preliminary runs only. Mark I - Shown on Dwg. D-19741. Mark II - Glass Dewar flasks available on the area. Mark III - Stainless steel Dewar flask; Hofmann Laboratories, Inc., Newark, N. J., Patent No. 2447607, X-67437.	<u>This Report</u> - Secs. 10.4.11; 10.5; 10.6 - <u>Engineering File Folder No. F-48</u> . <u>Other Literature</u> - 52 (p. 28).

Table 10.1 (Continued)

Components	Drawings and Sketches		Miscellaneous Information	References
	Fig.	Titles		
Vacuum Pump, PV-420	D-22761	Cell II Engineering Flowsheet	<u>Control Equipment</u> - Temperature of oil in pump type 2C, 4-in., 115-v., Titan thermostat set at 140°F., pressure at pump inlet PI-27, automatic vacuum release when pump stopped running of. Dag Q-1679-48-BU. <u>Period of Use</u> - All Runs. <u>Pump Data</u> - Kinney Compound, Kinney Manufacturing Co., Boston, Mass. CTD-B-6-10 (KC-46), Serial No. 07098, Property No. C400C 159063, 46 CFM, 500 rpm. A spare pump and motor of smaller capacity was on hand. <u>Motor Data</u> - C.E., 3 H.P., 1725 rpm, 220 v., 4 a., 60 cycles, 3 phase. <u>Heater Data</u> - Two 115-v., 250 - w., Chromalox strip heaters were strapped midway on the pump body.	<u>This Report</u> - Secs. 10.4.12, 10.3.1, 10.4.9, 10.5, 10.6, 6.4.1, item b, 9.4.7, item b, 9.4.8, 12.3.1, item a, 12.3.1, item b. <u>Engineering File Folder No.</u> F-49. <u>Other Literature</u> - 13, 22 (pp. 28, 47).
	D-22763	Cell II Piping Plan		
	D-22764	Cell II Piping Elevation		
	D-34687	Wiring Diagram (sheet 1, item A)		
	D-34693	Wiring Diagram (sheet 7, item D)		
	Q-1679-48-BU	VPP Vacuum Pump & Pump Timer System		
Chemical Trap for Product Purge, FV-122	D-19709-B2	FV-122, Chemical Trap	<u>Period of Use</u> - All runs. Packed with 3 kg of 1/8-in. NaF pellets. Changes made in the design are discussed in Sec. 10.4.9.	<u>This Report</u> - Secs. 10.4.9, 10.3.1, 10.4.12, 10.5, 10.6. <u>Engineering File Folder No.</u> F-45. <u>Other Literature</u> - 21, 22, 23, 22 (p. 28).
Cold Trap for Product Purge, FV-224	D-19710	FV-224 Cold Trap	<u>Period of Use</u> - Preliminary runs only.	<u>This Report</u> - Secs. 10.4.10, 10.5, 10.6. <u>Engineering File Folder No.</u> F-48. <u>Other Literature</u> - 22 (p. 28).
Instrumentation (Thermocouples and PE-26)	D-22761	Cell No. 2 Engineering Flowsheet	Locations of thermocouples and PE-26 are shown on drawing D-22761. <u>Period of Use</u> - All Runs.	<u>This Report</u> - Secs. 10.4.11, 10.5, 10.6, 9.4.7, item f, 21.4.2. <u>Engineering File Folder No.</u> F-44. <u>Other Literature</u> - 13, 17.

- e. Setting valves as needed to evacuate the cold traps and product receiver to 0.3 mm of Hg as determined by PI-27. This was the reasonable pressure limit in cold trap evacuation. A higher pressure left inert gases in the system; a lower pressure resulted in excessive UF_6 loss to FV-122 via the exit gases (36, p. 28).
- f. Isolating the cold traps and product cylinder from the rest of the VFP.
- g. Heating to and keeping at $\sim 80^\circ\text{C}$ the cold traps and piping of the evacuated portion of VFP using heaters FV-527, FV-526A, and FV-526B, the inlet, middle, and outlet heaters on both cold traps; and the heat lamps for the product cylinder valves.
- h. Draining the liquid UF_6 into the product cylinder by opening HCV-18 and HCV-19.
- i. After the liquid UF_6 had drained or when PR-26 reading exceeded 130% of that before starting the liquid to drain,^b starting FV-820 and -622, and bleeding dry instrument air into the FV-126 cooling system to prevent moisture from collecting on the cylinder.
- j. One hour after the product receiver temperature (TR-2C-6) reached 0°C and leveled off and the WR-1B reading leveled off, closing the product receiver valves and shutting off FV-820 and -622.
- k. Shutting off the heat to the cold traps and cooling them to -40°C (FV-220) and -55°C (FV-222) using FV-830 and -832, respectively.
- l. Evacuating the two cold traps by pumping for 5 minutes and then filling with N_2 to atmospheric pressure and repeating this step two more times.
- m. Sampling FV-152 and submitting for U^{+6} and OH^- analyses.
- n. Turning off FV-526A, FV-526B, and the heat lamps on the product receiver.

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^a While draining the liquid UF_6 , the system was kept at a temperature of $\sim 80^\circ\text{C}$, i.e., safely above the triple point (64°C at 22 psia) but not high enough to vaporize all of the UF_6 (17, p. 4).

^b FV-526 was shut off during liquid draining period.

^c Starting FV-830 and -832 was usually a time-consuming operation as delineated in Sec. 9.4.2.

- c. While chilling the pigtails with dry ice, disconnecting the product receiver; plugging the ends of the pigtails; and capping the ends of the product cylinder valves.
- p. Weighing the product cylinder and storing it safely in a criticality drum.
- q. Emptying and refilling FV-122 and -124 as needed.

System modifications necessitated additional minor steps in the procedure. For the complete procedure, see Secs. 10.7.1 and 10.7.2.

10.3.2 Critical Operating Steps

- a. Making certain that all product cylinder weighing was done in the prescribed fashion using the designated scales and standard weights.
- b. Assuring that the system prior to product removal contained no leak detectable by the KI-starch method.
- c. Making certain that the 0.3 mm of Hg pressure was attained on evacuation without extended pumping. (An extended pumping period indicated a leak which should be eliminated and an undue loss of UF_6 to FV-122 via the exit gases.)
- d. Having all of the pipelines and valves at $\sim 80^\circ C$ which eliminated the deposition of UF_6 in cold places.
- e. Assuring optimum UF_6 collection by the one-hour waiting period after TR-2C-6 reached $0^\circ C$ and WR-1B leveled off.
- f. Cooling the cold traps to operating temperatures and then evacuating the residual gases through FV-122 and chilling and capping the pigtails to minimize UF_6 losses.
- g. Continuously watching in Cell 2 for UF_6 leaks ("white smoke") while PR-26 read above atmospheric pressure.

10.4 Equipment Performance^a

10.4.1 Product Cylinder, FV-126

Three alterations to the standard 5-in. product cylinder used by K-25 produced a satisfactory product receiver for VPP: first, numbering and identifying for VPP; second, adding the central thermowell for monitoring the

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^aAfter the "L" runs, most of the equipment was dismantled and moved to Burial Ground No. 3 (Sec. 23.4.16b). However, the product cylinders, the cylinder heating and cooling equipment, the chemical trap, and vacuum pump were retained for future processing.

UF₆ temperature, and finally, changing the valves and valve orientation.

Two valve troubles were experienced:

- a. Solid UF₆ condensing in valves and preventing closure. This situation was remedied by: (1) wrapping pigtails all along and close to the valves with Mark II FV-526B instead of heating pig-tails with Mark I FV-526B (heat lamps) and retaining one heat lamp from Mark I FV-526B for heating each valve, (2) redesigning the FV-526 lid (Mark II) as described in Sec. 10.4.2, and (3) altering the FV-922 cylinder supporting device as delineated in Sec. 10.4.5.
- b. Valve leaking through the packing. This valve was repacked without further trouble as described in Sec. 17.4.1f.

The criticality containers for storing UF₆-bearing cylinders were satisfactory.

The product cylinder was kept at ~80°C during the liquid UF₆ transfer (Sec. 10.3.1).

In the "C" run product transfers, about 25 g of U as UF₆ was normally purged to FV-124 (25, 27, 36, 31, 29). This uranium did not represent a loss because it was recoverable (32). No such data were obtained in the "E" and "L" runs.

10.4.2 Product Cylinder Heater, FV-526^a

This heater operated successfully. The only necessary change was an altered lid made of Flexiboard which served two purposes: first, it minimized the drag of the cylinder on the lid thereby greatly reducing the effect of this drag on the cylinder weighing monitor, and second, it raised the cylinder valves out of the furnace so that UF₆ plugs could be eliminated by external heating (heat lamps).

The heating characteristics of FV-526 were:

- a. Time to heat empty cylinder from room temperature to ~80°C:^b one hour.

^a Table 22.3 and Secs. 21.4.1, 21.4.2.

^b See footnote for items d. and g. in Sec. 10.3.1.

- b. To keep cylinder at $\sim 80^{\circ}\text{C}$ ^a, Pyrovane and Variac settings were 85°C and 120 v, respectively (Sec. 10.4.1).
- c. Time to heat a product cylinder containing ~ 10 kg of U as UF_6 from room temperature to slightly $> 100^{\circ}\text{C}$ (for sampling): 6 - 8 hours.

10.4.3 Product Cylinder Inlet and Outlet Lines Heater, FV-526A^b

This heater worked satisfactorily. Performance data were:

- a. Time to heat lines from room temperature to $\sim 80^{\circ}\text{C}$ - $\sim 1/2$ hour.
- b. Pyrovane and Variac settings to maintain $\sim 80^{\circ}\text{C}$ were 120°C and 50 v, respectively.

10.4.4 Product Pigtaills Heater, FV-526B^b

This heater was satisfactory. The only difficulty was that heat lamps were also necessary to keep the product cylinder valves hot enough to prevent UF_6 deposition while collecting product. Instead of being a fault in the heater, this technique was an expedient. Using the industrial heating cable for pigtaills and auxiliary heat lamps for valves was less complicated than using industrial heating cable for both pigtaills and valves. The reason was that the heating cable would have required re-wrapping every time a cylinder was connected.

Prior to installing the heating cable, the pigtaills and valves were heated entirely with heat lamps. In this arrangement, the pigtaills were not heated as uniformly and considerable time was spent adjusting heat lamps.

Heater performance data were:

- a. Time to temperature ($\sim 80^{\circ}\text{C}$) - $\sim 2-1/2$ hours.
- b. Pyrovane and Variac settings to maintain $\sim 80^{\circ}\text{C}$ were 120°C and 50 v, respectively.^c

Corrosion of the pigtaills and Monel piping in the product collecting system was insignificant (19).

10.4.5 Product Weigh Station, FV-922

The product weigh station gave a rough record of the cylinder weight while collecting UF_6 . This record was very valuable in charting the progress

^aSee footnote for items d. and g. in Sec. 10.3.1.

^bTable 22.3 and Secs. 21.4.1, 21.4.2. Three valves, one in a temporary line connecting the pigtaills and the other two in a temporary UF_6 supply line to the pigtaills, plugged in early work (Sec. 17.4.1g).

^cController thermocouples were silver brazed to the pigtaills.

of this step in the process and, in addition, was normally within a kilogram of the actual cylinder weight as determined by Toledo scales checked by SS Material Accountability.

The operation of the product weigh station was beset with three difficulties:

- a. The drag occurring when the product cylinder and/or valves rubbed against the furnace lid. This drag which caused the weight record to be low was greatly reduced by the new-design lid mentioned in Sec. 10.4.2 and by suspending the cylinder from its thermowell. Previously, the cylinder was slung from the weigh element by an awkward circumferential clamp which accentuated the drag against the furnace lid.
- b. The drag resulting from the flexing of the pigtails. This drag could not be eliminated, but its effect on the weight record was compensated for while zeroing the recorder.
- c. The weigh element absorbing some of the heat from the heat lamps. This situation caused concern because black material dripped from the element at times. Actually, however, no specific damage to the element was apparent. This situation was largely corrected when most of the lamps were dispensed with in favor of industrial heating cable (Sec. 10.4.4).

10.4.6 Product Cooling Coil, FV-226

This cooling coil performed well throughout all runs.

10.4.7 Product Cooler Blower, FV-622

This blower was satisfactory.

10.4.8 Product Cooler Refrigerating Unit, FV-820

The operation of this refrigerating unit was satisfactory.^a Three comments are pertinent:

- a. The unit required maintenance about every 6 months.
- b. It was necessary to run both units to obtain cooling because of the cascade hook-up.
- c. Trouble was experienced with the overload on the lower unit which presumably was caused by starting the lower unit less than ~15 minutes after the upper unit was started.

The combination of FV-226, -622, and -820 was capable of cooling the product cylinder containing ~10 kg of U as UF₆ from ~80°C to 0°C in ~3-1/2 hours.

^aThis unit appears in Fig. 12.3.

10.4.9 Product Purge Chemical Trap, FV-122

The operation of this trap (shown on Dwg. No. D-19709-R2) was unsatisfactory. Consequently, the following changes were necessary:

- a. The flow scheme was reversed. Two disadvantages were met in the original flow scheme: (a) the qualitative test, i.e., yellowish coloration of NaF, for uranium could not be used to tell whether U had been picked up by merely observing the top of the NaF (Sec. 7.4.5) and (b) the arrangement made it possible to pull NaF in- to the vacuum pump.
- b. A spring-loaded sintered nickel disk was placed on top of the NaF bed to eliminate blowing NaF around in the system.
- c. The vessel was realigned and made more accessible by welding to a nearby steel column.

The original placement resulted in improper alignment of the vessel relative to its pipelines as well as the inaccessibility of the inlet and outlet flange bolts. Both of these factors made it difficult to change the NaF in the trap as well as to make the flanged joints leak-tight. After this change, the situation was improved although the flange bolts were still somewhat inaccessible. Whether the difficulty of rendering the flanged joints leak-tight resulted from the trouble in tightening the difficult-to-get-at bolts or the use of a flat-gasketed joint instead of a ring joint was never ascertained. At any rate, the result was either incomplete evacuation of the cold traps in steps e. and l. of the Operating Procedure (Sec. 10.3.1) or prolonged pumping with an accompanying high pick-up of UF_6 in FV-122.

It was felt that the proper solution to this situation was to replace the flat-gasketed top flange of FV-122 with a ring-joint flange and to make some of the bolts even more accessible. This change was not made while operating and should, therefore, be made before another processing period begins.

Uranium normally sorbed in FV-122 was reduced by better operating technique from ~25 g per run in the "C" runs to a total of 15 g in the last five "L" runs (25, 27, 36, 31, 29, 22, 21, and 23). Since FV-122 also sorbed residual UF_6 from product sampling during the "C" and "E" runs, however, only the uranium trapped during the "L" runs came from the product transfer step (Secs. 12.3.1a, 12.3.1b and 12.4.2c). The uranium did not represent a loss because it was recoverable (32).

This trap was packed with 3 kg of 1/8-in. NaF pellets. Unloading FV-122 was done either with a portable vacuum cleaner or with FV-420 (Sec. 10.4.12).

10.4.10 Product Purge Cold Trap, FV-224

This cold trap used in Runs C-3 through -8 was unnecessary because the product purge chemical trap was capable of collecting the UF_6 evacuated during product collection (25, 27, 36, 31). In addition, this trap was awkward to handle.

10.4.11 Coolant Container of FV-224, FV-924

The most suitable coolant container (Mark III) was the stainless steel Dewar flask. The original container (Mark I) was unsuitable because of its size and shape. The glass Dewar flask (Mark II) available at ORNL was too fragile.

In the early VPP work, both FV-224 and FV-924 were found to be non-essential equipment.

10.4.12 Vacuum Pump, FV-420

The vacuum pump was used as follows:

- a. To evacuate cold traps during product collection. The pump was adequate for this service. Difficulties were met in achieving the desired pressure (0.3 mm of Hg, cf. Sec. 10.3.1) only when leaks were present in the system. Leaks giving the most trouble were those in the FV-122 top flange and valve V-114.
- b. To evacuate the UF_6 sampler (Sec. 12.3.1a and 12.3.1b). The vacuum pump was satisfactorily used in UF_6 sampling.
- c. To evacuate the cold trap jackets (Sec. 9.4.7b). The vacuum pump was satisfactory for this service. No difficulty was experienced.
- d. To empty chemical traps FV-103, -122, and -124 (Secs. 6.4.1b, 9.4.8, 10.4.9). Using the vacuum pump to empty chemical traps was preferred over using a portable vacuum cleaner because it was more convenient and because it presented no exhaust gas problem. But in this service fines were bled to the vacuum pump which apparently increased the frequency of overhauling.

Specific troubles met were:

- a. It was necessary to use MFL (Miller's Fluorinated Lubricant) oil which was very viscous at room temperature and, therefore, required heating to $\sim 140^\circ F$ either to fill the pump or to start it. This heating preparatory to starting the pump was done both with heat lamps and with two strip heaters fastened to the pump body. A thermostat set at $140^\circ F$ and immersed in the oil was incorporated in the strip heaters circuit. The strip heaters method was preferred.

- b. A gluey mass which clogged the pump lubrication ports formed in the pump. Formation of this material was presumed to stem from: (a) heating the oil, (b) passing F_2 through the oil, and (c) the accumulation of NaF fines. Its formation necessitated this approximate maintenance schedule: (a) changing oil quarterly and (b) overhauling pump semi-annually or annually.^a The accumulation of NaF fines was believed to be the most damaging. This gluey mass affected the pumping capacity, although to an unknown degree.
- c. NaF pellets from FV-122 were sucked into the pump. This trouble was encountered only in early VFP work and resulted both from suddenly opening V-99 and from inadequately retaining the NaF in FV-122 (29, p.27). Later both of these difficulties were eliminated.
- d. A vacuum-break on the upstream side of the pump was necessary when shutting down the vacuum pump. The consequence of not breaking the vacuum was the possibility of sucking oil into the system. Since there was a great likelihood of this, an automatic vacuum-breaking device was installed. This device was actuated when the pump motor power supply shut off.^b This actuation would occur from a power shut-off occurring manually or by the circuit breaker but not from a power failure.
- e. The vacuum pump originally discharged exit gases to the cell. In early VFP work, the pump exhaust was piped to the cell off-gas duct.

Since the vacuum pump was indispensable during product collection, a spare pump of smaller capacity and suitable nipples for connecting it to the system were provided in the event of Kinney pump break-down. This spare pump was used when NaF pellets were accidentally pulled into the Kinney pump during Run C-10 (29, p.27). Its pumping capacity was inadequate, however.

10.4.13 Instrumentation

FE-26 (13) and thermocouples were indispensable in following the product collection operation. FE-26 monitoring the system pressure indicated the movement of the UF_6 from the cold traps to the product cylinder. For example, at the maximum pressure of ~ 50 psia, the UF_6 was a mixture of liquid and vapor. The subsequent decrease in pressure reading indicated the collection of UF_6 in the product receiver. Finally, when product collection was over, the FE-26 reading was nearly zero psia as it had been previously after system evacuation.

^aOverhauling was done at the K-25 Vacuum Pump Shop in Building 1401 under R. L. Leinart's supervision.

^bThis system contained a solenoid and a timer so that N_2 flowed for one minute only. Complete operational information is given in Engineering File Folder No. 49.

After PT-26 was placed inside the heated duct to keep it above 65°C^a, little trouble with it arose. Previously, it was essential to replace the Bourdon spring on two occasions because of UF₆ condensation.

Some trouble was experienced with PI-27, the pressure gage used to determine the extent of evacuation (0.3 mm Hg as in step d., Sec. 10.3.1) of the product collecting system. Although the reasons for the trouble were not fully ascertained, exposure to UF₆, F₂, and NaF fines was evidently a contributing factor.

10.5 Summary and Conclusions

Most of the components of the product collecting system gave little trouble. Principal difficulties were met with the product cylinder valves, the product purge chemical trap, and the vacuum pump. The characteristics of the heating equipment are recorded. The product collecting operation required 10 to 12 hours.

The standard 5-in. UF₆ cylinder was altered to identify with VPP and to adapt to the operation. Adaptation constituted installing a central thermowell and changing and reorienting the valves. Plugs occurring in cylinder valves were eliminated by redesigning the FV-526 lid and by heating each valve with a heat lamp. The packing in one valve on a cylinder containing UF₆ was replaced without incident as described. Criticality containers for the cylinders were successfully used.

The cylinder furnace lid was changed to minimize drag and to raise the valves out of the furnace for proper heating.

Pigtails heated adequately with industrial heating cable were unevenly heated with heat lamps. In addition, considerable time was consumed by adjusting the heat lamps. Corrosion of the product pigtails and Monel piping in the product collecting system appeared insignificant.

The product weigh station worked better when the cylinder was suspended by the thermowell than when suspended by a circumferential clamp. The reason was that less drag occurred from the thermowell suspension. The weight element exuded a black material as a result of heat absorption, but this caused no known damage to the element. The weighing device was suitable for monitoring the change in cylinder weight while collecting UF₆. Usually the final weight indicated by the device was within about one kilogram of the true UF₆ weight determined with Toledo scales checked by SS Material Accountability.

The product cylinder cooling system cooled a product cylinder with ~10 kg of U as UF₆ from 80°C to 0°C in 3-1/2 hours. Cooling was obtained only when both refrigerating units were running. Maintenance on the refrigerating units was required about semi-annually.

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^aThat is, above the triple point of UF₆ (See Step d, Sec. 10.3.1).

The product purge chemical trap required these changes:

- a. Reversed flow scheme to enable detecting U pick-up without emptying the trap.
- b. Redesigned for adequate retention of NaF pellets without the possibility of pellet movement.
- c. Realigned vessel to alter its position relative to its pipelines and to improve accessibility to the flanges bolts.

Because of the inaccessibility of the flanges bolts and presumably because of the flat-gasketed flanged joints, leaks were difficult to eliminate. Such leaks either resulted in incomplete evacuation in product collection or a prolonged "pumping-down" time. Incomplete evacuation increased the partial pressure of inert gases during product collection. Prolonged pumping augmented the pick-up of UF_6 in FV-122. Improvements in operating technique enabled reducing the uranium normally sorbed by this trap to a total of 15 g in the last five "L" runs. Uranium so collected was recoverable.

The product purge cold trap and its coolant container were used only through Run C-8.

The vacuum pump was used to: (a) evacuate the cold traps, cold trap jackets, and UF_6 sampler and (b) empty the chemical traps. At times, attaining a pressure of 0.3 mm of Hg during product collection was difficult, this difficulty usually stemming from leaks. The other evacuations presented no trouble. Emptying the chemical traps with the pump resulted in getting NaF fines in the vacuum pump oil. Specific troubles with the pump were: (a) the necessity for using MFL oil requiring heating, (b) the formation of a gluey mass in the pump lubrication ports, (c) NaF pellets inadvertently being sucked into the pump, (d) the necessity to break the vacuum when pump was shut off, and (e) release of exit gases in Cell 2 in the early design. The spare pump was inadequate for product collection. Reasons for the trouble with PI-27 were never fully ascertained. PE-26 and thermocouples were indispensable in following the product collection operation.

10.6 Recommendations

It is recommended that:

- a. The components of the product collecting system not mentioned below be used in the latest stage of development in future processing.
- b. The 15-minute waiting period between starting the first and second product cylinder cooling system refrigerating units be adhered to more closely; these units should be checked semi-annually.
- c. The product purge chemical trap be rebuilt with the following design features: (a) gas inlet at the top for ease in qualitatively detecting UF_6 pick-up, (b) adequate NaF pellet retention without pellet movement, and (c) flanged joints which are easy to make leak-free as determined by the KI-starch method. (This change includes making the bolts for flanges accessible.)

- d. NaF fines be kept out of the vacuum pump by: (a) screening NaF for use in the product purge chemical trap and (b) emptying chemical traps some way other than with the vacuum pump.
- e. An adequate spare for the vacuum pump be obtained and kept ready for service.
- f. The following maintenance schedule for the vacuum pump be adhered to until proven unnecessary: (a) change oil quarterly and (b) overhaul semi-annually or annually.
- g. A reliable, accurate low-pressure gage be installed instead of PI-27.

10.7 Appendix

10.7.1 Operating Procedure: Product Cylinder Handling (Revised Feb. 7, 1958)

PART A

PRH _____
 Date _____
 Time _____
 Oper. _____

1. Select a conditioned 5" UF₆ cylinder for use as a product receiver.
 Cylinder Number _____
2. Carefully weigh the cylinder (with the valve caps on, but with all other attachments removed) on Toledo Scale No. X-26515. Wt = _____ kg.
3. Zero reading of scale _____ kg.
4. Weigh the 25 kg check-weight. Wt = _____ kg.
5. If this number is not between 24.95 and 25.05 kg, have the scales checked, then repeat steps 2, 3, and 4.
6. Place the product receiver in the furnace (FV-526).
7. Attach the lifting lug to the cylinder.
8. Connect the pigtails to the cylinder using new aluminum gaskets. Be sure to use a back-up wrench.
9. Attach the lifting lug to the weighing assembly, making sure the cylinder is centered in the furnace.
10. Install lids on furnace, then make final check that the cylinder hangs freely from the weighing element. Time done _____.
11. Close V-53 _____, V-99 _____, V-114 _____, on suction side of FV-420, and V-115 on discharge side, and V-111 _____ on product sampler.
12. Open V-71A _____.
13. Open HX-23 _____, and HX-26 _____.
14. Close HX-22 _____, HX-24 _____, and HX-25 _____.
15. Turn on FV-420.
16. Open V-99 slowly.
17. Pump system down for 5 minutes.
18. Close HX-26.
19. Open HX-25 until N₂ flow stops. (Watch PI-3.)
20. Close HX-25.
21. Open HX-26 for 5 minutes.
22. Close HX-26.
23. Open HX-25 as in step 19.
24. Close HX-25.

25. Open HX-26 for 5 minutes.
26. Close HX-26.
27. Open HX-25. When flow levels out record FI-3 _____, FI-32 _____.
28. Close HX-25. When flow levels out record FI-3 _____, FI-32 _____.
29. Proceed to LPS _____ runsheet.

PART B

30. After product has been collected in the cylinder, and the pigtaills purged, disconnect pigtaills from the cylinder.
31. Replace valve caps on cylinder _____ and plugs in the pigtaills _____.
32. Remove the lifting lug, and take the cylinder out of the furnace.
33. When cylinder has warmed to room temperature, weigh on Toledo Scale X-26515. Wt = _____.
34. Zero reading of scale _____ kg.
35. Weigh the 25- and the 10-kg check-weights together. Wt = _____ kg.
36. If this number does not lie between 34.95 and 35.05, have the scales checked, then repeat steps 33 and 34.
37. Return the cylinder to Cell 2 for sampling.
38. After product sampling operation, reweigh cylinder as above.
Cylinder weight _____ kg.
Check weights _____ kg.
Zero reading of scale _____ kg.
39. Store cylinder in VPP storage cage.

10.7.2 Operating Procedure: Product Removal Operation (Revised May 1, 1958)

PRO _____
Date _____
Time _____
Oper. _____

1. Begin this procedure after the installation of the cylinder according to Part A, PCH runsheet. Cylinder Number _____.
2. Close V-53 _____, -99 _____, -100 _____.
3. Open cylinder valves.
4. Condition cylinder and pigtaills with F₂. (See FCP runsheet).
5. Switch WX-1 to point B. WR-1B reading _____ %.
6. Close HX-13 _____, 14 _____, 16 _____, 25 _____, 30 _____.
7. Open HX-15 _____, 18 _____, 19 _____, 22 _____, 23 _____, 24 _____, 26 _____. Time _____.
8. Set the following controllers. Record information on appropriate data sheet.

Controller	Set Point	Time Set	Time Reached
TIC-2B-1*	*	_____	_____
TIC-2B-11	120	_____	_____

* Adjust until TR-2D-6 = 70°C

- ___ 9. Adjust heat lamps on pigtails until 66° Tempilstik melts along entire length of pigtails.
- ___ 10. Adjust TC-2B-11 to 50 v after reaching temperature in step 8.
- ___ 11. Check that FV-420 is running.
- ___ 12. Open V-99 slowly. Time open _____.
- ___ 13. When PI-27 falls to 0.3 mm Hg, close HX-26 _____ and V-99 _____. Time closed _____. (If minimum vacuum is greater than 0.3 mm Hg after pumping for 5 minutes, close HX-26 _____ and V-99 _____ and look for source of inleakage.)
- ___ 14. Close HX-18 _____, 19 _____, 23 _____.
- ___ 15. Promptly after step 14, turn off EX-FV-830 _____, and -832 _____. Bleed air from Freon reservoirs (if necessary) to keep pressure at 10-15 psig _____.
- ___ 16. Set the following controllers:

Controller	Set Point	Control Point	Time Set	Time Reached
TIC-2B-2	58	80	_____	_____
TIC-2B-3	70	80	_____	_____
TIC-2B-4	130	80	_____	_____
TIC-2B-5	75	80	_____	_____
TIC-2B-6	85	80	_____	_____
TIC-2B-7	95	80	_____	_____

- ___ 17. The Variacs should be adjusted to give, as nearly as possible, identical temperature records on TR-2C-1, 2, 4, 5, 6. Then,
  - a) Set TC-2B-2, 3, 4, 5, 6, 7 on 50% of full scale _____.
  - b) Make adjustments on individual Variacs to keep temperature records close together _____.
  - c) Then increase each Variac as the slope of the temperature curves begin to decrease (Curves flatten.) _____.
  - d) Finally, maintain TR-2C-1, 2, 4, 5, 6, at 80°C ± 5°C.
- ___ 18. Observe PR-26. When PR-26 reads equivalent of atmospheric pressure, station a man at entrance to Cell 2 to observe any leakage from the system. Reading on PR-26 _____ %.
- ___ 19. When PR-26 increased 15% above the reading in step 18, open HX-18 _____. Shut off TIC-2B-1 _____.
- ___ 20. Watch WR-1B. When reading is increasing less than 1 division in five minutes, open HX-19 _____.
- ___ 21. When WR-1B stops increasing 1 division in 5 minutes (or any time the pressure on PR-26 exceeds 30% above reading in step 18) start the product coolers.
- ___ 22. To start coolers: Turn on dry air supply to blower _____. (enough air to produce a slight hissing sound).
- ___ 23. Turn on blower.
- ___ 24. Allow 5 minutes to dry out system, then turn on cooler #1.
- ___ 25. Approximately 10 to 20 minutes later, turn on cooler #2.
- ___ 26. Maintain these conditions until TR-2D-6 and WR-1B both level off.

Instrument	Time Levelled Off	Reading
TR-2D-6	_____	_____
WR-1B	_____	_____

27. Wait 60 minutes then close the product cylinder valves. Time closed _____.
28. Turn off TIC-2B-2 _____, 3 _____, 4 _____, 5 _____, 6 _____, 7 _____.
29. Turn on brine pumps in pipe tunnel _____. Reduce pressure on each brine reservoir to 10-15 psig _____. Start compressors FV-830 _____, and 832 _____. Time _____.
30. After TR-2D-1 _____, 4 _____ and 5 _____ cool to -40°C , close HX-22 _____ and -24 _____.
NOTE: Between steps 30 and 37, while HX-14 and -16 are closed, close HX-35 _____ and -36 _____, then pull and refill FV-124*. (Yes _____, No _____) Time pulled _____. Fill in card file on FV-124 _____.
31. Open HX-25 until pigtaills are pressurized _____. (Observe FI-3 as indication of pressurizing.)
32. Close HX-25 _____ and open HX-24 _____.
33. After 15 seconds, close HX-24 _____.
34. Repeat steps 31, 32, and 33 _____.
35. Repeat steps 31, 32, and 33 again _____.
36. Close HX-18 _____ and -19 _____.
37. Check that FV-124 is buttoned up _____. Open HX-14 _____ and -16 _____.
38. Open V-99 _____. Read PI-27, or -28, _____ mm Hg.
39. Check that FV-120 is running.
40. Open HX-23 _____ and HX-26 _____.
41. Pump down for 5 minutes.
42. Close HX-26.
43. Open HX-25 _____, V-71A _____, and V-71B _____ until N_2 stops flowing. (Watch FI-3.)
44. Close HX-25.
45. Open HX-26, pump down for 5 minutes.
46. Close HX-26.
47. Open HX-25 until N_2 stops flowing.
48. Close HX-25.
49. Open HX-26, pump down for 5 minutes.
50. Close HX-26.
51. Open HX-25, until N_2 stops flowing.
52. Close HX-25.
53. Turn off TIC-2B-11 _____, and pigtail heat lamps _____.
54. Sample FV-152. Code SI _____, Submit for U^{+5} and OH^{-} .
55. Return to PCH runsheet Part B, for removal of product cylinder. Record cylinder weight here as well as in PCH runsheet.
Cylinder weight _____ kg.
Check-weights _____ kg.
Zero reading _____ kg.
56. Empty and refill FV-122 and FV-158 only if requested. FV-122 Yes _____; No _____; FV-158 Yes _____; No _____.

Time Completed _____
Operator _____

* Only if requested to refill.

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11.0 SCRUBBING SYSTEM

11.1 Introduction

In the scrubbing system, fluorine and a small amount of HF in the VPP tail gases were reacted with an aqueous KOH solution. The inert gases were subsequently exhausted to either the vessel or cell off-gas system in Building 3019. Principal steps in the operation of this system were:

- a. Maintaining adequate scrubbing capacity.
- b. Keeping the scrubber pump running and the inlet line to the scrubber unplugged.

11.2 Equipment

The general arrangement of the equipment in the scrubbing system is shown in Fig. 11.1. Details of the individual components are recorded in Table 11.1.^a

11.3 Operation

11.3.1 Operating Procedure

Steps in operating the scrubbing system were:

- a. Determining whether the scrubbing capacity in the surge tank was adequate for the operation to be done. (This was done by comparing the estimated moles of scrubbing capacity needed with the moles of OH^- ion in excess of 0.5 molar in the surge tank.)
- b. Providing the necessary KOH solution by the scrubber make-up procedure shown in Sec. 11.7.1.
- c. Setting the necessary valves (Sec. 11.7.2, Part B.), and starting the agitator, FV-352. (The top three sprays only were used.)
- d. Putting the PCV-39, FE-9, and LE-4 instrumentation in service.
- e. Turning on the scrubber pump, FV-450.

NOTE: The scrubber was shut down by turning off the scrubber pump and setting valves as necessary.

Altering the design of the VPP from time to time required minor changes in the scrubbing operating procedure. Details of the operating procedures for the scrubbing system are given in Secs. 11.7.1 and 11.7.2.

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^aThe scrubbing tower, surge tank, and circulating pump are shown in Fig. 14.2.

Fig. 11.1. Equipment Arrangement in the Scrubbing System

Table 11.1
List of Scrubbing System Components

Components	Fig.	Drawings and Sketches Title	Miscellaneous Information	References
Scrubbing Tower, FV-150	D-21408 D-22762 E-22789 D-23912 Q-1679-F-39-C Fig. 11.2 Fig. 11.3	Gas Disposal Unit Item No. FV-150 Off Gas Scrubber Flowchart Scrubber Piping Plan and Elevations Gas Disposal Unit Support Detail Off Gas Scrubber Thermocouples Jack Legs Added Between Vent Lines and Off-Gas Buttwelder Dual Gas Inlet for Scrubbing Tower FV-150	<u>Mark IA</u> <u>Instrumentation</u> - FE-9: orifice-type flowmeter purged with water. LE-4: pneumatic-type level instrument (FV-150) purged with H ₂ . <u>Period of Use</u> - All runs through the "G" runs. <u>Mark IB</u> <u>Instrumentation</u> - Same as in Mark IA. <u>Period of Use</u> - "B" and "I" runs. The following additions were made to Mark IA: (a) auxiliary scrubber inlet (cf. Fig. 11.3); (b) a surge drum (FV-151) and jack legs to the off gas lines (cf. Dwg. D-23218, Rev. 3 and D-23219, Rev. 3 and (c) an improved FV-39 system (cf. Fig. 11.4). In b. above, also cf. Fig. 11.2.	<u>This Report</u> - Secs. 11.4.1; 11.4.1, item a, 11.4.1, item b, 11.5; 11.6; 17.4.3, 17.6.3. Figs. 11.2; 11.3; 11.4; 17.7 (Table). <u>Engineering File Folder No. F-50.</u> <u>Other Literature</u> - 1, 12, 21, 22, 25, 26, 27, 28, 29, 30, 31, 32 (pp. 4, 30, 31).
Surge Tank, FV-152	D-19701 D-22789	Surge Tank Scrubber Piping Plan and Elevations	<u>Instrumentation</u> - See LE-4 for Mark IA, FV-150. <u>Period of Use</u> - All runs.	<u>This Report</u> - Secs. 11.4.2; 11.5; 11.6. <u>Engineering File Folder No. F-51.</u> <u>Other Literature</u> - 21, 22 (pp. 30, 31).
Surge Tank Agitator, FV-352	D-22762 D-22775	Off Gas Scrubber Flowchart Wiring Diagram (sheet 1, item "B")	<u>Period of Use</u> - all runs Lightnin directly coupled mixer-motor set manufactured by Mixing Equipment Co., Inc., 138 Mt. Road Blvd., Rochester, 11, New York; Property No. X-32044, Reg. No. D-2445-X; <u>Mixer</u> -model No. 52, serial no. 435749; <u>Motor</u> -style 73250, type PA, serial No. Q713433, Frame No. 17420, 220/440 v., 1.6/0.8 A., 1/2 H.P., 3 phase, 1725 rpm.	<u>This Report</u> - Secs. 11.4.3; 11.5; 11.6. <u>Engineering File Folder No. F-51.</u>
Circulating Pump, FV-450	D-22762 D-22781 Vendor Dwg. PSC 1526	Off Gas Scrubber Flowchart Wiring Diagram (sheet 7, item "C") 116 Weyco Pump w/M.S.Mari-Drive	<u>Control Equipment</u> - Ammeter. <u>Period of Use</u> - All runs. Weyco pump manufactured by Robbins and Mayers, Inc., 1934 Clark Blvd., Springfield, Ohio; X-63596, P.O. No. W10K-17221-1; pump type CD4, form 6H, serial No. A-95633-A, Frame 116, PM-F Flex. No. 120A Packing No. 12, Lovejoy coupling, Mamel Rotor No. 27, <u>Motor-Speed Reducer Set</u> (directly coupled) U. S. Electrical Motors, Inc., 204 Walker St. N.W., Atlanta, Ga., X-56292, P. O. No. W10K-22728, U. S. Variable Motor type VEM-REV-GB, design B, serial No. 2331244, 220/440 v., 7.6/3.8 A., 2 H.P., 1 phase, ratio 2.75, frame: 23-224-A-21, 80-560 rpm.	<u>This Report</u> - Secs. 11.4.4; 11.5; 11.6. Figs. 11.5; 11.6. <u>Engineering File Folder No. P-57.</u> <u>Other Literature</u> - 22 (pp. 30, 47).

Table 11.1 (Continued)

Components	Drawings and Sketches		Miscellaneous Information	References
	Fig.	Title		
Caustic Make-Up Tank, PV-140	E-22789 D-23193	Scrubber Piping Plan and Elevations Refrigeration and Scrubber Make-Up Flowchart	Period of Use - All Runs. O.H.S.L. Fabricated from a 50. gal. stainless steel drum.	This Report - Secs. 11.4.5; 11.5; 11.6. Engineering File Folder No. P-50. Other Literature - 22 (p. 30).
Caustic Make-Up Agitator, PV-340	D-22782 D-23193	Wiring Diagram (sheet 7, item "G") Refrigeration and Scrubber Make-Up Flowchart	Period of Use - All Runs. Agitator - Motor Mtr (directly coupled) K-62046, P. O. No. W2X-5693-1; Agitator - Chemineer, Inc., 1042 East First St., Dayton 2, Ohio, Model FMA-2, Serial No. D-416-2, rotation, cw, 30-in. shaft; Motor-Master Electric Co., 136 Davis Ave., Dayton 1, Ohio, Style 208302, Type - BA, Serial No. C014618, 115/230 v, 1/2 amp., 1 phase, 1725 rpm	This Report - Secs. 11.4.6; 11.5; 11.6. Engineering File Folder No. P-50. Other Literature - 22 (p. 30).
Caustic Transfer Pump, PV-440	D-22780 D-23193	Wiring Diagram (sheet 7, item "H") Refrigeration and Scrubber Make-Up Flowchart	Period of Use - All Runs Pump-Motor Set: K-50830, P. O. No. W2X-52414; Pump - Goulds Pumps, Inc., 75 Fall St., Seneca Falls, N. Y., Fig. 3750, No. 2914482, 30-ft. head at 1750 rpm; Motor: O.R., Model 567308000, 440 v, 1.3/1.2 a., 1425/1725 rpm, 3/4 H.P., 3 phase.	This Report - Secs. 11.4.7; 11.5; 11.6. Engineering File Folder No. P-50. Other Literature - 22 (p. 30).
Caustic Drum Tilt, PV-540	D-23193 Sketch No. 0-11-11-15	Refrigeration and Scrubber Make-Up Flowchart Drum Lift	Period of Use - All Runs. Palmer-Schile Co., 16000 Pullerton Ave., Detroit 27, Mich., No. B-731, P. O. No. W1X-62372.	This Report - Secs. 11.4.8; 11.5; 11.6. Engineering File Folder No. P-50. Other Literature - 22 (p. 30).

11.3.2 Critical Operating Steps

- a. Keeping the scrubber solution within (OH^-) limits of 0.5 and 2 M (10 wt %). The lower limit was used to avoid exhausting F_2 in the exit gases. At times in the "E" and "L" runs, however, the (OH^-) was run as low as 0.2 M. Whether appreciable F_2 passed out in the exit gases during such operation was not ascertained. Although the upper limit of 2. M (OH^-) in the starting solution was exceeded at times, this represented a convenient concentration based on the 45% KOH purchased and on the size of the scrubber make-up solution equipment. Because of the high solubility (107 g/100 ml of water or 18.4 M) of KF in water at 30°C, the (F^-) was of small relative importance unless the same solution was used and strengthened repeatedly.
- b. Keeping the scrubber gas inlet line unplugged. (A gas inlet line plug necessitated shutting off the F_2 .)
- c. Continuing to operate the scrubber as long as F_2 was flowing to avoid passing F_2 into the off-gas systems.

11.4 Equipment Evaluation^b

11.4.1 Scrubbing Tower, FV-150

a. General Information

Scrubber performance tests were run in October and November of 1956 (38). When a total of 2.74 cu ft of F_2 was contacted with 6 gpm of ~2 M KOH (two Schutte and Koerting Fig. 622C-30 coarse spray nozzles) at a solution temperature >30 to 35°C, the F_2 concentration in the off-gas was <100 ppm. Temperature was proposed as an important factor in the reaction:

In VPP operations, the scrubber performance data were:

1. Adequately handled F_2 flow rates up to 30 liters/min with ~10 gpm of KOH flowing through only the top three spray nozzles.
2. The uranium normally collected in the KOH solution was insignificant (27, 25, 36, 31, 29, 22, 23, 21).
3. Fission product activity in the KOH solution was low (21, 23).
KOH solution concentration limits established are given in Sec. 11.3.2.

^aThis solubility figure was interpolated from data in (39). The minimum temperature in the scrubbing solution was 30°C in the "L" runs (21).

^bAfter the "L" runs, the scrubbing equipment was retained for future processing.

The only serious corrosion observed occurred on the gas inlet liner. During early work, the corrosion noted on the Mark IA gas inlet liner was very severe as described elsewhere (40). In later work, corrosion on the Mark IA liner was less severe presumably because the KOH temperature at the top of the scrubbing tower was lowered from ~ 100 to 50°C (7)^a. However, two localized high-attack regions were found. Corrosion of the small gas inlet liner (Mark IB, Sec. 11.4.1b) produced only surface roughening (7). Vidigage data revealed only slight corrosion on the scrubbing tower shell (41).

Once while cleaning out the gas inlet liner, the flange of that assembly slipped off the pipe. Whether this was a weld failure or the separation of a poorly welded joint could not be determined. At any rate, to prevent a recurrence possibly followed by dropping the pipe inside the scrubber, the joint was remade using a heavy weld as shown on ORNL Dwg. D-21408, Rev. 2. Since that time, no similar trouble has occurred.

FE-9 and LE-4 monitored the flow rate of the KOH solution and the liquid level in the surge tank (Sec. 14.4.5). Both of these instruments were satisfactory. LE-4 was essentially trouble-free. However, the water purges of FE-9 were difficult to maintain because of: (a) rust in the water clogging up the purge lines and (b) the fact that the purge pressure needed to be high enough to overcome the 65 psig pressure in the KOH line.

b. Model Differences

Differences in Mark IA and IB were:

1. Vent line piping.

- (a) Mark IA piping design did not prevent KOH from flowing into the vent line piping. Although the gas exit line of the scrubber was ~ 10 in. above the liquor exit line, the KOH solution in the Mark IA design flowed into the exhaust gases header and thence into lines V-112-1, V-161-1, and V-162-1. This was first discovered in Run M-9 (between Runs C-2 and C-3) when solution dripped from an opening of line V-112-1 in Cell 1. A survey of the vent-line piping revealed the presence of KOH solution also in lines V-161-1 and V-162-1 at the Fluorine Transmitter Rack. No reaction of F_2 with the solution apparently occurred although the fact that such a happening was possible accelerated making the design change discussed under Mark IB.

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^a "Less severe" here means that the frequency of replacement might be once every year or two.

- (b) Mark IB - (1) eight foot jack-legs shown in Fig. 11.2 were added to prevent any liquid in the off-gas subheader from entering lines V-112-1, FV-128,^a and V-163-2; (2) the constant-level drum FV-151 shown in Fig. 11.1 toward which the off-gas subheader sloped was also added to allow liquid in the off-gas subheader to drain and to automatically shut off the circulating pump if the KOH level in FV-151 became too great. As far as is known, no occasion has arisen in which the jack-legs and FV-151 were needed.^b If so, however, the system worked since no further trouble with liquid in the vent piping has occurred.

2. Plugs in gas inlet line.

- (a) Mark IA - The existence of only one gas inlet necessitated shutting off the F_2 when this line plugged.
- (b) Mark IB - A second scrubber inlet line was installed as shown in Fig. 11.3.

The additional scrubber inlet was useful in that its presence avoided having to shut down when the gas inlet line plugged. But the new inlet line, being of smaller diameter, plugged more rapidly than the original inlet line. Consequently, the new gas inlet line could not be used for more than a few hours because of plugging.

3. PCV-39 design.

- (a) Mark IA - PCV-39 design^c apparently allowed pressure-vacuum surges in the scrubber.
- (b) Mark IB - PCV-39 design shown in Fig. 11.4 maintained the pressure differential across PCV-39 between slightly less than 1/2 in. and 2 in. of water.^d Maintaining the pressure differential between these limits assured that the exit gases would continuously flow in a direction away from the scrubber. This was a safety measure to prevent vapors in the off-gas line from entering the scrubber. At times, the off-gas line contained organic materials from the solvent extraction portion of the pilot plant. These, if present in the scrubber along with F_2 , might explode. The 2-in.

^aThat is, the line to FV-128 prior to changing FV-128 to FV-158.

^bIt is definitely known that FV-450 was never shut off automatically because of too great a liquid level in FV-151.

^cORNL Dwg. Q-1679-P-39-CV.

^dPressure differential = pressure on scrubber side of valve - pressure on off-gas side of valve (13).

Fig. 11.2. Jack-Legs Added Between Vent Lines and Off Gas Subheader

Fig. 11.3. Dual Gas Inlet for Scrubbing Tower, FV-150

Inst. No.	Make - Model	Range - Type	Set to -
PE-39A	Taylor 339 RF-5	2-8-in. H ₂ O	0 - 2 in. H ₂ O
PX-39A	M. H. Press. Sw.	0 - 15 PSIG	Closes At 4.5 psig
PE-39B	Taylor 339 RF-5	2-8 in. H ₂ O	0-2 in. H ₂ O
PC-39	Foxboro Model 58 PA	---	Reverse Action
PI-39	Moore MP Station	---	---
PCV-39	Existing Valve	---	---
PCV-39 Sol.Valve	Skinner 1/8", 3 Way	---	---

Fig. 11.4. Schematic Diagram of Mark II PCV-39

of water maximum pressure differential was adequate for exhausting the inert gases from the VPP operations.

11.4.2 Surge Tank, FV-152

This 275-gallon tank was satisfactory. The amount of corrosion was nil (41).

11.4.3 Surge Tank Agitator, FV-352

This agitator was suitable. As initially installed, the temporary arrangement with the dangling switch-box was unsafe. Later a safe installation was made.

11.4.4 Circulating Pump, FV-450

The Moyno pump was adequate for pumping the KOH solution over the scrubber. Two troubles were experienced:

- a. The packing gland overheated in early work, apparently from the corrosive action of aqueous KOH on the rubber connecting rod washers as well as the backward installation of a grease seal. The remedy consisted of using Teflon washers, correctly placing the grease seal, and replacing the chevron-type packing with 3/8-in. square graphite-impregnated asbestos which required no lubrication.

Within a year after this packing gland trouble was resolved, a slow drip from the packing started which could not be eliminated by tightening the packing gland. Actually, however, a slow drip from the packing was preferable to packing failure with no drip because the solution would provide some lubrication and reduce scoring of the shaft by the packing. Therefore, the best solution was to operate with the leak and collect it in a drain pan.

- b. The knuckle joint on the rotor-end of the connecting rod was worn severely. Others at Oak Ridge who have operated Moyno pumps thought that severe wear on the rotor-end knuckle joint of the connecting rod may be inherent in Moyno pumps.^a

To prolong the life of the knuckle joint, the consensus favored lubricating the joint as follows: (1) coating the contact surfaces with molybdenum disulfide^b and (2) then applying

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^aH. T. Kite and D. T. Martin of Y-12; R. J. DeBakker and S. M. DeCamp of ORNL.

^bApplied as Lubri-Bond "A" (manufactured by Electrofilm, Inc., 7116 Laurel Canyon Blvd., North Hollywood, California; stocked at Y-12 under Catalog No. 11-435-9606).

a caustic-resistant Dow-Corning stopcock grease. These lubricants were applied to both knuckle joints although no way was found for determining how much this treatment increased the life of either joint. It was presumed, however, that the life of the rotor-end knuckle joint is ~7000 hours, the service time at inspection (Fig. 11.5). Pertinent data were: (a) symptoms - intermittent squeak occurred in the pump with no apparent change in its performance; (b) condition of parts - the knuckle-joint pin was sheared; the pin severely enlarged the hole in the rotor-end of the connecting rod; and the packing scored the drive shaft slightly; and (c) repair - replaced the connecting rod and pins lubricating as described ^aabove; replaced the drive shaft, packing, washers and grease seals.

Although a routine lubrication schedule for the pump and motor was drawn up (26), the high radioactive background from other nearby equipment was a deterrent to following this schedule. No known damage occurred, however, to the pump or motor because of this lack of routine lubrication.

Before the start of cold runs in the fuel element dissolution program, the Moyno pump was disassembled for observation and repair. The operating time between this work and the prior work mentioned above was ~1200 hr. The observations made were: (a) the rotor-end knuckle joint had again worn severely (Fig. 11.6);^b (b) wear on the other parts was slight with the drive shaft being slightly scored.

Repair included:

1. Changing the stator (The stator had been used ~8,200 hr) and the connecting rod and its two pins.
2. Reusing the other parts including the packing.
3. Not lubricating the rotor-end knuckle joint because the worst problem is evidently not lack of lubrication but rather stress concentration along a line tending to shear the pin on both ends. (Aid in arriving at this conclusion came from T. S. Mackey of ORNL.)

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^aThe scoring was so slight that the shaft could have been metallized and machined to size had a spare shaft not been available.

^bThe five other photographs (Nos. 48456 through 48460) made are in Engineering File Folder No. F-57.

^cAlthough this work was scheduled, it had not been done as of June 13, 1960.

Drive-Shaft End

Rotor End

Fig. II.5. Ends of Connecting Rod for Moyno Pump after ~7000-hr Service Time.

ORNL
Photo No. 48455 A
UNCLASSIFIED

New

Used (~1200-hr Service Time)

Fig. 11.6. Comparisons of New and Used Moyno Pump Parts:
Rotor-End and Corresponding Pin of Connecting Rods.

11.4.5 Caustic Make-up Tank, FV-140; Caustic Make-up Agitator, FV-340; Caustic Transfer Pump, FV-440; and Caustic Drum Lift, FV-940

This caustic make-up equipment was adequate.

11.5 Summary and Conclusions

Most equipment in the scrubbing system performed satisfactorily. Major problems requiring attention were: (a) scrubbing solution entering the vent lines, (b) apparent pressure-vacuum surges in the scrubber, (c) plug formation in the gas inlet line to the scrubber, and (d) near-failure of a connecting-rod joint in the Moyno pump. Actions taken in solving these problems along with other pertinent data were discussed earlier.

11.5.1 Scrubbing Tower Performance

The scrubbing tower performance data were:

- a. Handled F_2 flow rates up to 30 liters/min with 10 gpm of KOH flowing through only the top three sprays.
- b. The uranium appearing in the scrubber solution during operations was insignificant.
- c. Fission product activity in the KOH solution was low. KOH solution concentration limits were: (a) 0.5 (minimum) to (b) 2.0 M OH^- (maximum).

A series of developmental runs considered beyond the scope of this report is referred to.

Corrosion of the scrubbing tower was apparently nil. The only corrosion noticed was the very severe attack on the gas inlet liner in early work which was less severe later. A weld "failure" on the gas inlet line apparently resulted from the poor design of that weld.

11.5.2 Scrubbing Tower Modifications

The Mark IB scrubbing tower was superior to Mark IA in that it contained remedies for earlier troubles:

- a. Eight-foot jack-legs and surge tank FV-151 to keep KOH out of the vent line piping.
- b. A second gas inlet line to avoid having to shut-down when the original tower inlet line plugged.
- c. Altered PCV-39 design to prevent pressure-vacuum surges in the scrubber.

FE-9 and LE-4 adequately monitored the KOH flow rate and surge tank liquid level. Trouble with the water purges at FE-4 was experienced.

The surge tank was satisfactory and suffered insignificant corrosion.

The surge tank agitator was suitable. This initially unsafe installation was later corrected.

11.5.3 Moyno Pump Performance

Although the Moyno pump was suitable for pumping the KOH solution, the troubles which occurred and remedies tried were:

- a. The packing gland overheating was corrected by using Teflon washers, correctly placing the grease seal, and replacing the chevron-type packing with 3/8-in. square graphite-impregnated asbestos. A slow drip occurring later was not corrected because it might reduce shaft scoring, compared with worn dry packing.
- b. Severe wear occurred on the rotor-end knuckle joint of the connecting rod. This wear may be inherent in Moyno pumps. Lubrication evidently does not reduce this wear because of the stress concentration along a line which tends to shear the pin. Details are given for the inspection and repair of the pump after the first ~7,000 hours of service, and after an additional ~1,200 hours of service. A near failure occurred after the first ~7,000 hours of service. Although radioactive operation was a deterrent to following the established lubrication schedule, no trouble with the pump or motor occurred because of the lack of scheduled lubrication.

The caustic make-up equipment operated satisfactorily.

11.6 Recommendations

It is recommended that the components of the scrubbing system continue to be used as described herein and further that:

- a. A drained drip pan be provided under the Moyno packing gland.
- b. The knuckle joints of the Moyno pump be inspected after every 2500 hours of operation, this inspection schedule be followed until suitable data are obtained for formulating a better one.
- c. More work be done on eliminating gas inlet line plugs in the scrubbing tower with moving the discharge end of the line out of the KOH spray to be considered.
- d. The rust and water pressure problems in FE-9 purges be solved.
- e. The F_2 concentration in exit scrubber gases be monitored during future operation.

11.7 Appendix

11.7.1 Operating Procedure: Scrubber Make-Up Procedure
(250 Gallons)

Batch SMP _____

Condition: Assume FV-152 hold tank is empty.

- _____ 1. Close these valves near FV-140 in the gallery: Valves 19 _____, 24 _____, and 21 _____.
- _____ 2. Close these valves near FV-152 on the roof: Valves 1 _____, and 15 _____.
- _____ 3. Add water to FV-140 up to the upper mark on the recirculation line (4" from the top of FV-140).
- _____ 4. Open valve 19 _____ and 20 _____.
- _____ 5. Turn on FV-440 pump.
- _____ 6. Turn off FV-440 after solution has been pumped (Some heel will remain.).
- _____ 7. After line to roof has drained back to FV-140, close valve 19.
- _____ 8. Record LR-4 liquid level _____ inches.
- _____ 9. Add water to FV-140 up to the upper mark, open valve 19, turn on FV-440 pump.
- _____ 10. Turn off FV-440 pump after solution has been pumped. Close valve 19 _____.
- _____ 11. Add water to FV-140 up to the middle of the bottom ring (11" from bottom).
- _____ 12. Hoist a drum of 45% KOH (caustic potash) into position above FV-140.
- _____ 13. Drain sufficient solution from the caustic drum into FV-140 to bring the liquid level up to the lower mark on the recirculation line (10" from the top of FV-140).
- _____ 14. Agitate the solution for two minutes on _____ off _____.
- _____ 15. Turn off the agitator and add water up to the upper mark on the recirculation line.
- _____ 16. Open valve 19 _____.
- _____ 17. Turn on FV-440.
- _____ 18. Turn off FV-440 pump after solution has been pumped. Close valve 19 _____.
- _____ 19. Add water to FV-440 up to the middle of the bottom ring.
- _____ 20. Hoist a drum of 45% KOH into position above FV-140.
- _____ 21. Drain sufficient solution from the caustic drum into FV-140 to bring the liquid level up to the lower mark on recirculation line.
- _____ 22. Agitate the solution for two minutes on _____ off _____.
- _____ 23. Turn off the agitator and add water up to the upper mark.
- _____ 24. Open valve 19 _____.
- _____ 25. Turn on FV-440.
- _____ 26. Turn off FV-440 pump after solution has been pumped. Close valve 19 _____.
- _____ 27. Add water to FV-140 up to the upper mark on the recirculation line.
- _____ 28. Open valve 19 _____.
- _____ 29. Turn on FV-440 pump.
- _____ 30. Turn off FV-440 after solution has been pumped. Close valve 19 _____.
- _____ 31. Add water to FV-140 up to the middle of the bottom ring.
- _____ 32. Pump this solution to FV-152, then close valve 19 _____ and record LR-4 liquid level _____ inches.
- _____ 33. Close these valves near FV-450 on the roof 3 _____, 13 _____, 14 _____, 16 _____, 17 _____.

- ___ 34. Open valves 11 _____ and 1 _____. (Recirculation line)
- ___ 35. Turn on FV-450 agitator. On at _____.
- ___ 36. Turn on FV-450. On at _____.
- ___ 37. Recirculate solution in FV-152 for 30 minutes.
- ___ 38. Drain about 100 cc from sample valve 14 and discard. Then collect two samples for KOH analysis. Code SM - _____ - 1,2.
- ___ 39. Shut off FV-450. Close valve 11 _____.
- ___ 40. Shut off agitator.
- ___ 41. Proceed to scrubber operation run sheet. (See Part B, Sec. 11.7.2).

NOTE: When FV-152 was not empty, enough KOH was added to bring its strength to ~2 M. So strengthening the KOH was not recommended, however, as mentioned in item a. of Sec. 11.3.2.

11.7.2 Operating Procedure: Development Operations of Scrubber Tower

OST _____

PART A: To prepare the F_2 supply system for operations:

- ___ 1. Shut off the air to FCV-1 and FCV-2 and open V-81. (This isolates nitrogen system from fluorine system.) To close FCV turn right-hand knob FC counter-clockwise until horizontal gage reads zero.
- ___ 2. Record the reading on PR-10 (the F_2 supply) pressure _____. If less than _____ proceed to "Changing Fluorine Tanks".
- ___ 3. Move switch HX-1, near FK-2 on the panel board to the (upper, lower) position. This will give the needed instrument sensitivity for this run.
- ___ 4. Close the following valves on the panel (turn handle "across" the flow line. HX-8 _____, HX-10 _____, HX-11 _____, HX-14 _____, HX-16 _____, HX-20 _____, HX-32 _____ and HX-34 _____.
- ___ 5. Open the following valves on the control panel (turn handle parallel to the flow line). HX-12 _____, HX-35 _____, and HX-36 _____.

PART B: To prepare the scrubber for operation:

- ___ 6. Record LR-4 liquid level _____. If less than _____ inches, proceed to scrubber makeup sheet (50 gal).
- ___ 7. Close the following valves near FV-450 on the roof: 11 _____, 12 _____, 13 _____, 14 _____, 16 _____, 17 _____, 22 _____, 25 _____, 50 _____, and 68 _____.
- ___ 8. Open the following valves near FV-450 on the roof: 1 _____, 2 _____, 3 _____, 27 _____, and 72 _____.
- ___ 9. Turn on FV-450 pump.
- ___ 10. Gradually open each of the following valves checked "open" and adjust the valve position to give the indicated pressure readings. Close valves checked "closed".

	<u>Open</u>	<u>Closed</u>	<u>Gage Reading on</u>

V-4			PI-18 is
V-5			PI-19 is
V-6			PI-20 is
V-7			PI-21 is
V-8			PI-22 is
V-9			PI-23 is
V-10			PI-24 is

11. Connect the sampling apparatus to valve 25.
12. Invert a (500 ml, 1000 ml, 1500 ml, 2000 ml or _____ ml) flask on tapered sample tip.
13. Open glass stopcock on sample flask.
14. To start flow of F_2 to system, turn the right hand valve on FR-2 clockwise until the pen reaches a set point of _____. Time pen reaches set point _____.
15. Five minutes after time in Step 14, open valve on either side of flask on off-gas sampling apparatus. Turn on vacuum pump to evacuate sample flask. Time on _____.
16. Five minutes after vacuum pump was started, close the valve between sample flask and vacuum pump. Time _____.
17. Turn off the vacuum pump.
18. Open valve 25 to let gas into flask.
19. Close valve 25, close the glass stopcock on sample flask, and remove flask from apparatus. Submit to lab. Code OG _____.
20. Drain about 100 cc from valve 50 (near FV-150) and discard in waste pail. Collect two samples for laboratory analysis. Code SO _____ 1,2.
21. Shut off the output air from FR-2 by turning the right hand knob counter-clockwise until the horizontal gage reads zero.
22. Drain about 100 cc of sample from valve 14 (near FV-450) and discard in waste pail. Then collect two samples for laboratory analysis. Code SI _____ 1, 2.
23. Turn off FV-450 pump.

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12.0 PRODUCT SAMPLING SYSTEM

12.1 Introduction

The UF_6 in the product cylinder was sampled in the VPP by flowing a portion of it as liquid into a sample container. Sampling loss was reduced by freezing the liquid UF_6 in the sample container and thermally transferring vapor to the cold container before sealing and disconnecting it. Chemical contamination was avoided by adhering to strict cleanliness and by using controlled atmospheres, either a nitrogen blanket or a vacuum. Principal steps in the operation of this system were:

- a. Coupling the heated cylinder to the sampling rig.
- b. Flowing liquid UF_6 into a tared sample container and freezing it therein.
- c. Weighing the filled container, washing off external α radioactivity, and submitting for analyses.

Masks were worn while sampling to protect personnel from α radioactivity.

12.2 Equipment

Two UF_6 sampling systems have been used:

- a. The K-25 rig shown in Fig. 12.1 in which one sample was obtained ranging in weight from 85 to 125 grams. (Subsequently, this sample was aliquoted to obtain chemical, radiochemical, and isotopic samples.)
- b. The Minisampler (developed by J. E. Bigelow of VPP), shown in Figs. 12.2, 12.3, and 12.4 in which four samples were taken varying in size from 3 to 12 grams each. (The Minisampler was developed to take the aliquots directly from the cylinder.)

The component parts of both the K-25 rig and the Minisampler are described in Table 12.1.

12.3 Operation

12.3.1 Operating Procedure

Steps in the operating procedures were:

a. K-25 Sampler^a

1. Weighing the product cylinder on the Toledo scales checked by SS Accountability.

---

^aAll evacuating was done with FV-420 (Sec. 10.4.12). The sampling time exclusive of cylinder heat-up was ~ 2 hours.

Fig. 12.1. Equipment Arrangement in the Product Sampling System (K-25 Sampler)

Fig. 12.2. Equipment Arrangement in the Product Sampling System (Minisampler)

Fig. 12.3. View of Minisampler as Used in Cell 2.

ORNL
Photo No. 43438 A
UNCLASSIFIED

Fig. 12.4. Close-up View of Minisampler Manifold Connected to a Product Cylinder.

Table 12.1
List of Product Sampling System Components

Components	Drawings and Sketches		Miscellaneous Information	References
	Doc.	Title		
Product Sampler, FY-925	K-25 Dwg.	K-25 Sampler	<u>Period of Use</u> - Through the "C" Run.	<u>This Report</u> - Secs. 12.4.1; 12.3.1, item a; 12.3.2, item a; 12.5; 12.6; 10.4.2. <u>Engineering File Folder No.</u> F-64. <u>Other Literature</u> - 17.
	D-424-K-9028-A	Assembly of Dolly for Sampling Cylinder		
	D-424-K-9028-B	Details of Parts for Sampling Cylinder		
	D-424-K-9028-C	Detail of Sampling Dolly Frame		
	D-424-K-9324	Sampling Manifold		
Sample Container	K-25 Dwg. LW-8-2210	Dogwood Sample Cylinder		
Minisampler, FY-925	See K-25 Dwg. for the K-25 Sampler.	Minisampler	<u>Instrumentation</u> - Two compound (30" Hg vacuum to 30 psi pressure) gauges, FI-25A and FI-25B. <u>Period of Use</u> - "E" and "L" Runs.	<u>This Report</u> - Secs. 12.4.1; 12.4.2; 12.4.2, item a; 12.3.1, item b; 12.3.2, item b; 12.5; 12.6; 10.4.2. <u>Engineering File Folder No.</u> F-64. <u>Other Literature</u> - 21, 23, 24, 17.
	ORNL Photo No. 44993	Minisampler Assembly (Fig. 12.3).		
	ORNL Photo No. 44938	Minisampler Cylinder Connections (Fig. 12.4).		
Sample Container	Fig. 12.5	Fluorothene Sample Tube	<u>Period of Use</u> - "E" and "L" runs Several designs were used with the one shown on Fig. 12.5 preferred. A 250-w. infra-red heat lamp was used for heating the metal nut on the sample container.	<u>This Report</u> - Secs. 12.4.2, item b; 12.5; 12.6. Fig. 12.5. <u>Engineering File Folder No.</u> F-64.
	ORNL Photo No. 44938	Minisampler Cylinder Connections (Fig. 12.4).		
	ORNL Photo No. 44993	Minisampler Assembly (Fig. 12.3).		
Minisampler Trap, FY-125	Fig. 12.2	General Arrangement of Minisampler	<u>Period of Use</u> - "E" and "L" Runs. The trap packing was ~130 g. of 1/8" BeF pellets.	<u>This Report</u> - Secs. 12.4.2, item c; 12.5; 12.6. <u>Engineering File Folder No.</u> F-64. <u>Other Literature</u> - 21.
	ORNL Photo No. 44993	Minisampler Assembly (Fig. 12.3).		
Minisampler H ₂ Line Heater, FY-525, and Minisampler Vacuum Line Heater, FY-525A	Fig. 12.2	General Arrangement of Minisampler	<u>FY-525</u> <u>Control Equipment</u> - Variac and an indicating thermocouple welded to the tubing between adjacent heating cable turns. <u>Period of Use</u> - "E" and "L" Runs. <u>Heater Data</u> - About 30 ft. of industrial heating cable was wrapped helically around the H ₂ line; advance of helix was ~ 1/4" to 3/8" per turn, heater uninsulated; ~400 v at 110 v. <u>FY-525A</u> <u>Control Equipment</u> - Similar to FY-525. <u>Period of Use</u> - "E" and "L" Runs. <u>Heater Data</u> - Similar to FY-525.	<u>This Report</u> - Secs. 12.4.2, item d; 12.4.2, item e; 12.4.2, item f; 22.4.1; 22.4.2; 22.5; 22.6. Table 22.3. <u>Engineering File Folder No.</u> F-64. <u>Other Literature</u> - 17.
	ORNL Photo No. 44993	Minisampler Assembly (Fig. 12.3).		
	D-34895	Wiring Diagram (sheet 9, item "B")		

2. Heating the cylinder to 100°C in FV-526 and the block on the rig to 70°C [items (2) and (5), Sec. 12.3.2a].
3. Having these items on hand prior to removing the cylinder from its furnace:
  - (a) A clean tared sample flask.
  - (b) A new Teflon gasket for connecting the block to the cylinder.
  - (c) Protective clothing and mask for each man in the cell.
  - (d) Dewar flask containing a dry ice-trichlorethylene mixture.
  - (e) Prepo torch and wrenches.
4. Placing the cylinder in the sampler dolly, tilting it up and down four or five times, and then locking it with the valves down and its axis declined at about 15° to the horizontal.
5. Having the light turned on behind block; connecting the upper block line to the cylinder and the lower block line to the sample flask.
6. Leak-testing the assembly by rate of vacuum fall when system was evacuated to a 29⁺ in. of Hg vacuum. (No change in ~30 seconds was acceptable. Leaks greater than this were eliminated before proceeding.)
7. Heating the line between the block and the cylinder with the Prepo torch.
8. Evacuating the block, lines, and sample flask to a 29⁺ in. of Hg vacuum.
9. Closing the lower block valve and valve on sample flask.
10. Opening the cylinder valve about one-half turn, keeping valve open until the receptacle in the block filled about half-full of liquid UF₆, and then closing the cylinder valve.
11. Heating the line between the block and sample flask with the Prepo torch.
12. Opening the lower block valve and the valve to the sample flask thereby allowing UF₆ to run into the flask.
13. Chilling the sample flask with the dry-ice bath.

14. After sample had frozen in sample flask and residual vapor thermally transferred to the flask by playing the torch lightly on the lines (required ~5 to 10 minutes), closing the valves on sample flask, evacuating residual UF_6 from the block to a suitable chemical trap (FV-122, Sec. 10.4.9) and filling the system with N_2 .
15. Removing sample container, weighing, decontaminating external surfaces from α radioactivity, and sending for analyses. (Subsequently, this sample was divided with aliquots being sent to different laboratories for chemical, radiochemical, and isotopic analyses.)
16. Reweighing the cylinder on the Toledo scales.

NOTE: The detailed procedure used is presented in Sec. 12.7.1.

b. Minisampler^a

1. Weighing the product cylinder on the Toledo scales checked by SS Accountability.
2. Heating the cylinder to be sampled to slightly $>100^\circ\text{C}$ in the product cylinder furnace; having FV-125 filled with 130 g of 1/8-in. NaF pellets.^b
3. Having these items on hand prior to removing the cylinder from its furnace:
  - (a) Four clean tared sample flasks.^c
  - (b) A new Teflon gasket for connecting the block to the cylinder.
  - (c) Protective clothing and mask for each man in the cell.
  - (d) Dewar flask containing liquid nitrogen.
  - (e) Prepo torch and wrenches.

---

^a All evacuating was done with FV-420 (Sec. 10.4.12). The sampling time exclusive of cylinder heat-up was ~4 hours.

^b FV-125 was used only during the "L" runs. FV-122 was used to sorb residual UF_6 during the "E" runs.

^c The sample tubes were cleaned with an aqueous Tide solution and dried with C. P. acetone.

4. Placing the cylinder in the sampler dolly, tilting it up and down four or five times, and then locking it with the valves down and its axis declined at about 15° to the horizontal.
5. Connecting the manifold nut to one cylinder valve and attaching one of the sample containers to the manifold.
6. Directing the heat lamp at the metal fitting on the sample tube.
7. Leak-testing the manifold by evacuating, noting the rate of vacuum fall, and eliminating any leaks. (An acceptable vacuum leak-test was essentially no vacuum decrease in ~30 seconds at a vacuum of 29^+ in. of Hg. Greater leaks were eliminated before proceeding.)
8. Evacuating the sampling system.
9. Drawing enough liquid UF_6 to half-fill the tube by proper valving and then closing the cylinder valve.
10. Immersing the lower end only of the sample tube in liquid N_2 until the liquid UF_6 was frozen and vapor had thermally transferred (required about 5 minutes).
11. Evacuating and filling the manifold with N_2 .
12. Removing and plugging the sample tube and then installing a tube for the next sample except for the last sample from a cylinder.
13. Taking three more samples using steps 5 through 12 above.
14. Cleaning and weighing the sample tubes and then submitting for analyses. [Cleaning and decontaminating were done as indicated in item (3) above.]
15. After the cylinder cooled to room temperature, reweighing it on the Toledo scales.

NOTE 1: Originally, the sample for mass spectrometer analysis was to have been collected in a glass tube. This was not done because of the difficulty in adapting the equipment for such sampling, and because of the trouble involved in training operators for such an advanced analytical technique.

NOTE 2: It was not necessary to empty and fill FV-125 (FV-122, Sec. 10.4.9, was used during the "E" runs.) after each sampling. Therefore, usually it was changed after sampling every three or four cylinders.

NOTE 3: The actual procedure used is shown as Sec. 12.7.2.

12.3.2 Critical Operating Steps

a. K-25 Sampler

1. Having the sample flask scrupulously clean and the system leak-tight to avoid contaminating the sample.
2. Having the cylinder at 100°C immediately before sampling to be certain that liquid UF_6 would flow from it.
3. Tilting the cylinder back and forth before placing in the sampling position to avoid vapor-locking the valves.
4. Having the block at 70°C before flowing the liquid UF_6 into it. (A lower temperature froze the UF_6 ; a higher temperature vapor-locked the block and prevented liquid UF_6 from flowing into the block.)^a
5. Using the Prepo torch to heat the lines to and from the block to $>65^{\circ}\text{C}$ because these lines were not otherwise heated.
6. Filling the block only half-full of liquid UF_6 . (More liquid UF_6 than this resulted in a larger sample than needed.)
7. Opening the cylinder valve wide enough so that liquid UF_6 would flow.

b. Minisampler

1. Having the sample tubes scrupulously clean and the system leak-tight to avoid contaminating the samples. (This was especially important for the Minisampler because of the small size of the samples collected.)
2. Having the cylinder at slightly $>100^{\circ}\text{C}$ at the start of sampling. (Since sampling required about one and one-half hours time, the last of 4 samples was sometimes difficult to get if the cylinder was not at the temperature of $>100^{\circ}\text{C}$ initially. Evidently no more samples could be taken after UF_6 started to freeze in the cylinder valve.)^b
3. Tilting the cylinder back and forth before placing in the sampling position to avoid vapor-locking the valves.

---

^aA temperature of 70°C was high enough to keep the UF_6 molten but low enough to prevent the formation of an excessive amount of UF_6 vapor. The triple point of UF_6 was 64°C at 22 psia (17, p. 4). Also Secs. 12.3.1a and 12.4.1.

^bAn initial cylinder temperature of $>100^{\circ}\text{C}$ was essential to keep the UF_6 fluid long enough ($\sim 1\frac{1}{2}$ hours) to take 4 samples. Sec. 12.3.1b and 12.4.2.

4. Using the Prepo torch to heat the cylinder valve and/or cold sections of the manifold and having FV-525 and -525A on to keep the lines $> 65^{\circ}\text{C}$.
5. Filling the sample tube only half-full of liquid UF_6 . (If more liquid than this were initially drawn into the tube, the residual UF_6 in the line between the tube and the cylinder would over-fill the tube.)
6. Opening the cylinder valve wide enough. (For the first sample, it was necessary to open the valve about one-half turn. Later and especially for the last sample, the valve was opened about one full turn.)
7. Being certain to avoid contacting the brass nut on the sample tube with liquid N_2 to prevent cracking it.

12.4 Equipment Evaluation

12.4.1 K-25 Sampler (27, p. 39)^a

The K-25 sampling equipment and operating procedure were satisfactory.^b Using the K-25 sampler had these advantages: (a) Less α contamination was released in Cell 2 than with the Minisampler because only one container was filled, and because the K-25 flask was valved off before disconnecting; (b) less sampling time (one-half hour versus one and one-half hours for the Minisampler) was required; (c) there was less chance of chemically contaminating the sample; and (d) the technique was easier for chemical operators to master. On the other hand, however, using the K-25 sampler offered these disadvantages: (a) increased the sampling cost because it was necessary to aliquot the sample and (b) took about 100 g from a cylinder compared with 30 to 40 g for the Minisampler.

The cylinder clamps on the sampler dolly would not hold some of the cylinders in the dolly because the shoulders on these cylinders were not square all around. The potential seriousness of this situation was realized when a hot cylinder in the sampling position fell out of the dolly. The brunt of the fall was absorbed by one of the valves whose connection to the cylinder was bent. To prevent a recurrence of this, the sleeve which contained the cylinder was drilled and tapped for 8 bolts to hold the cylinder firmly in place without using the clamps.

Records of the analytical data for samples collected with the K-25 sampler are recorded elsewhere for Runs C-3 through -8 (25, p. 27; 27, p. 31; 36, p. 21; and 31, pp. 30, 31). Since the UF_6 in Runs C-9 through -15 was not sampled, the figure for the theoretical U in UF_6 was used (31).

---

^aThe K-25 sampler was dismantled when the Minisampler was fabricated. Unused parts were moved to Burial Ground No. 3 (Sec. 23.4.16b).

^bThat is, analytical samples could be obtained.

12.4.2 Minisampler^a

The disadvantages mentioned in Sec. 12.4.1 were: (a) α contamination of Cell 2 necessitating the wearing of gas masks and subsequently cleaning of the cell, (b) one and one-half hours sampling time, and (c) sample contamination. A record and discussion of the samples collected are given elsewhere (21, 22 and 23).

a. Sampler Dolly, FV-925

The K-25 dolly was successfully used also for the Minisampler. A panel containing V-110, V-111, and PI-29A was added. The bolts had already been added to the sleeve as described in Sec. 12.4.1.

b. Minisampler Sample Container

Several different designs were used with the one shown in Fig. 12.5 being the most satisfactory. While chilling the UF_6 sample with liquid N_2 in this tube, inadvertently immersing the brass nut in the liquid N_2 has caused the brass to crack.

c. Minisampler Chemical Trap, FV-125

This trap used only during the "L" runs was fairly satisfactory. Operational characteristics were:

1. Its small diameter (1-3/8 in.) coupled with its length (11 in.) made discharging difficult except by disconnecting from the system.
2. Forty-nine grams of U were collected while sampling nine cylinders, an average of five grams collected per cylinder sampled. (21).
3. NaF was changed after sampling four or five cylinders.

During the "E" runs, residual UF_6 from the Minisampler was sorbed in FV-122 as previously for the K-25 sampler. Separate data for residual UF_6 from sampling were, therefore, not kept prior to the "L" runs (Sec. 10.4.9).

d. Minisampler N_2 Line Heater, FV-525^b

e. Minisampler Vacuum Line Heater, FV-525A^b

^aAfter the "L" runs, the Minisampler was dismantled and moved to Burial Ground No. 3 (Sec. 23.4.16b).

^bTable 22.3 and Secs. 22.4.1, 22.4.2, 22.5, and 22.6.

Fig. 12.5. Minisampler Sample Container

These heaters operated satisfactorily requiring less than one hour to heat their copper tubing lines to $> 100^{\circ}\text{C}$ with Variacs set at 60 v.

12.5 Summary and Conclusions

Both the K-25 sampler and Minisampler were barely adequate. The sampling times were ~ 2 hours in the K-25 sampler and ~ 4 hours in the Minisampler. Characteristics of the heaters for these samplers are given.

The K-25 sampler was better adapted to sampling in VPP because:

- a. Less α radioactivity was released in Cell 2.
- b. The sampling time was one-half that for the Minisampler.
- c. The technique was less difficult for chemical operators to master.
- d. The probability of sample contamination was less.

In spite of these advantages, more experience was gained with the Minisampler because:

- a. Less UF_6 (30 to 40 g compared with ~ 100 g for the K-25 sampler) was drawn from each cylinder.
- b. One handling step, i.e., taking the 100 g sample, was avoided.

For the K-25 sampler, the critical operating steps of utmost importance were:

- a. Having the cylinder at 100°C prior to sampling.
- b. Keeping the block at $70 \pm 5^{\circ}\text{C}$. Other critical operating steps are listed.

For the Minisampler, the critical operating step of primary importance was having the cylinder at slightly $> 100^{\circ}\text{C}$. Other critical operating steps were recorded earlier.

Two operating personnel and a Prepo torch were required with either rig.

Since α radioactivity was released from the Minisampler, wearing masks was essential. Because masks were worn, communication between the two operating personnel was difficult. This disadvantage also applied to the K-25 sampler although to a lesser degree.

After a cylinder fell from the dolly because the clamps were inadequate, bolts were added to the cylinder-containing sleeve to secure the cylinder.

The operation of the Minisampler chemical trap during the "L" runs was characterized by: (a) being hard to unload because of its i.d. to depth ratio, (b) picking up 5 g of UF_6 per cylinder sampled, and (c) requiring unloading about every four or five cylinders sampled. During the "C" and "E" runs, residual UF_6 from sampling was sorbed in FV-122.

Both the K-25 sampler and Minisampler sample containers were satisfactory. Of the several designs of the latter used, the one described was preferred, but it was possible to crack the brass nut by contacting it with liquid N_2 .

12.6 Recommendations

It is recommended that any product sampling in the future in the VPP be done by the K-25 method.

12.7 Appendix

12.7.1 Operating Procedure: Sampling of Product in K-25 Sampler

SOP _____

- _____ 1. Place product cylinder to be sampled in FV-526 and set TIC-2B-1 controller on 100 C. Gradually increase Variac setting to this controller until automatic control is obtained.
- _____ 2. Turn on switch to heater in sample manifold.
- _____ 3. Turn on switch to strip heaters on inlet and outlet lines to sample manifold, and vacuum header.
- _____ 4. Connect a sample flask to the sample manifold.
- _____ 5. Record time furnace FV-526 (TIC-2B-1) reaches the setpoint _____.
- _____ 6. Close or check to see if closed, valve HX-26 on panel _____, V-53 on vacuum line into FV-420 in Cell 2 _____.
- _____ 7. Open V-30 _____ and 31 _____ near cold trap FV-924.

NOTE: Do not begin the next step unless you have time to complete the entire runsheet.

- _____ 8. Fill FV-924 with dry ice-trichloroethylene mixture.
- _____ 9. Thirty minutes or more after the time recorded in step 5, using asbestos gloves and chain hoist, lift product cylinder from the furnace and lower into the sampling dolly.
- _____ 10. Connect the retaining clamps on sample dolly and also secure cylinder with bolts in cylinder sleeve.
- _____ 11. Invert the product receiver five or six times, pause in the upside-down position, and finally, connect the receiver into the sample manifold.
- _____ 12. Open two valves on sample manifold (blue handles).
- _____ 13. Open the valve on line to vacuum pump (Tees in near V-30.)
- _____ 14. Open valve into sample flask.
- _____ 15. Turn on FV-420 vacuum pump.
- _____ 16. Wait about 3 minutes, close vacuum valve on line from sample manifold.
- _____ 17. Turn off vacuum pump.
- _____ 18. Close lower valve (blue handle) on sampler manifold.
- _____ 19. Partially open the valve on the product receiver and observe the Teflon window.
- _____ 20. Fill the manifold to a depth slightly higher than the center of the Teflon window (approximately 100 g of product).

21. Close the valve on the product cylinder.
22. Open the lower valve in the sampler manifold (blue handle).
23. After solution drains to the sample flask, immerse the flask in dry ice slush (Raise a canful of slush into position around flask.) If difficulty is found in draining, thaw lines with torch.
24. Let system stand with heat on lines and flask cooled for _____ minutes.
25. Close valve on sample flask.
26. Turn on vacuum pump.
27. Three minutes later, close the valve on the line to the vacuum pump.
28. Shut off vacuum pump.
29. Close two valves in sample manifold.
30. Disconnect product receiver and rotate the receiver into its normal upright position.
31. Turn off the heating switches.
32. Submit sample to lab.
Code UP _____ 1.

12.7.2 Operating Procedure: Product Sampling Operation in Minisampler

PSO _____
Time _____
Date _____
Oper. _____

1. Heat cylinder to be sampled to just above 100°C. Time to 100°C _____.
(Cylinder number _____, Run No. _____).
2. Continue to heat for 30 minutes.
3. Assemble the following items before beginning to sample cylinder:
  - a. Four tared sample tubes number _____, _____, _____, _____. Also one glass sample tube if mass spectrometer analysis is desired (Yes _____, No _____).
  - b. Protective clothing and mask for each man in cell _____.
  - c. New Teflon gasket _____.
  - d. Dewar flask full of liquid N₂ _____.
  - e. Wrenches, etc., _____.
4. Place cylinder in sample dolly with left-hand valve (as you face top of cylinder) is pointed downward. Then rotate cylinder clockwise about 5 _____.
5. Clamp and lock cylinder in this position _____. (Two clamps 180° opposed are sufficient to hold).
6. Tilt cylinder up and down four or five times. Pause 5 sec in each position of maximum movement.
7. Lock cylinder in normal sampling position _____. Install one of the sampling tubes _____.
8. Direct infrared lamp at metal fitting on sample tube _____ (just above plastic tube).^a
9. Open three miniature valves (118 A, B, C) _____.
10. Open V-111.
11. Close V-118 A _____. Watch PI-29B _____. If pressure is constant for 30 sec, system is tight _____. If not, check closures and repeat test.
12. Open V-110 to let in N₂ until pressure rises to 10 psig.

^aSee Fig. 12.2 for equipment numbers referred to below.

13. Close V-110 and open V-111.
14. Close V-111 and repeat steps 12, 13 three more times.
15. On final evacuation, leave V-111 open and close V-118 A _____ and V-118 C _____.
16. Personnel should don masks.
17. Open (slightly) cylinder valve (1/4 to 1/2 turn is usually a sufficient opening).
18. Close cylinder valve _____.
19. Open V-118 C carefully. Watch PI-29B and level in sample tube. Close V-118 C in time to fill sample cylinder about half full.
20. Immerse lower end of sample tube in liquid N₂. As container cools, immerse further; however, DO NOT LET BRASS FITTING DIP INTO THE LIQUID N₂.
21. Continue to cool sample tube until PI-29B stops falling.
22. Then, open V-118 A to pump out inert gases.
23. Close V-111 _____ and open V-110 to let in N₂ _____.
24. Close V-110 _____ and V-118 B _____.
25. Remove sample tube and cap off tube.
26. Install fresh sample tube as soon as possible (Vapors are probably HF).
27. Open V-118 A _____ and V-118 B _____. Then begin at step 10 and repeat procedure in steps 10-25.
28. On final sample, retighten closure of cylinder valve _____. Open V-111 _____, V-118 A _____, and V-118 C _____.
29. After pressure has fallen, open V-118 B _____.
30. Close V-118 C _____, disconnect sample manifold _____, replace cap on cylinder valve _____, and set cylinder upright to cool _____.
31. When cylinder is cool, reweigh.
Cylinder No. _____
Run No. _____
Final Gross Wt _____ kgs
32. To clean and dry sample tubes use an aqueous Tide solution, tissue paper, and C. P. acetone. DO NOT GET WATER INTO THREADS.
33. Reweigh sample tubes:
No. _____ grams
_____
_____
_____
_____
34. The second and fourth samples go to Laing, the third sample to Feldman. The first we keep.
NOTE: Samples for mass spectrometer:
35. If a sample is collected for mass spectrometer, it will be drained from the manifold into a glass tube prior to final purging of manifold. A high-temperature flame should be used to seal the neck of the tube.

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13.0 WASTE SALT SYSTEM

13.1 Introduction

Essentially all high-activity level waste material (greater than ninety-nine per cent of the total activity) remained in the fluorinator after fluorination. This waste was eliminated from the process through the waste salt system. The principal steps in the operation of this system were:

- a. Transferring the molten salt from the fluorinator through a freeze valve into a waste receiver.
- b. Removing from the cell (and disposing in the burial ground) the filled waste receiver.

Radiation exposure was minimized by shielding the waste receiver and venting the vapors.

13.2 Equipment

The flowsheet of the waste salt system with the Mark I waste line and the Mark II freeze valve is shown in Fig. 13.1. After the "E" runs, the system was modified to eliminate major difficulties. The revised system with the Mark II waste line and Mark V freeze valve is shown in Fig. 13.2. The components of the waste salt system are listed and described in Table 13.1.

13.3 Operation

13.3.1 Operating Procedure^b

Steps in operating the waste salt system were:

- a. Positioning the waste can and activating the weight recorder to monitor the flow into the waste can.
- b. Maintaining the waste salt temperature at $\sim 600^{\circ}\text{C}$ in the fluorinator.
- c. Heating the entire molten salt line including FV-106, vent lines saddle joints, and waste nozzle to $\geq 570^{\circ}\text{C}$.^c
- d. Establishing fluorinator instrument and vent lines purges at one cfh.

^aSec. 17.4.4 for a discussion of freeze valves.

^bThe waste transfer time from "heat-on" the waste line to salt freezing in FV-106 (i.e., fell to 400°C) varied from 2-16 hr. The "E" run average was 7⁺ hr; the average in the last five "L" runs was 11⁻hr.

^cThat is, $\geq 50^{\circ}\text{C}$ above the melting point of the salt. The composition of most of the salt used was approximately C-30 having an m.p. of 520°C (11).

Fig. 13.1. Equipment Arrangement in the Waste Salt System (Mark I Waste Line)

Fig. 13.2. Equipment Arrangement in the Waste Salt System (Mark II Waste Line)

Table 13.1
List of Waste Salt System Components

Components	Drawings and Sketches		Miscellaneous Information	References
	Doc.	Titling		
Waste Line, MS-100-1 to MS-106-1, (including Freeze Valve, FV-106)	D-22777	Wiring Diagram (Sheet 3, item "G")	<u>Mark I</u> <u>Control Equipment</u> - Pyrovanne (thermocouple welded to the pipeline), Variac, and ammeter. <u>Period of Use</u> - "C" and "E" runs. <u>Heater Data</u> - Heater at 7-8 v. and 450 amp. was supplied (Sec. 21.2; 21.3; 21.3.1; 21.3.2; and Fig. 21.1). The stub (item 3, Dwg. No. C-23198) was removed.	<u>This Report</u> - Secs. 13.4.2; 13.4.1; 13.4.3; 13.5; 13.6; 13.7.2; 5.4.1, item b; 5.4.5, item a; 21.2; 21.4.4; 21.4.5; 21.4.5; and 22.4.2 <u>Other Literature</u> - 19, 21, 42. <u>Engineering File Folder No.</u> - F-22.
	C-23198	Freeze Valve Mark II		
	D-23224	Cell I Piping Details; Sheet No. II		
	C-23225	Freeze Valve Installation		
	D-31350	Flowsheet		
Waste Line	D-23224	Cell I Piping Details (Sheet No. II)	<u>Mark II</u> <u>Control Equipment</u> - Same as for Mark I. <u>Period of Use</u> - "L" Runs and Run M-64. <u>Heater Data</u> - Same power supply as for Mark I. But the waste line current was 170 amp. for half of the line instead of 254 amp. as on Dwg. No. D-34889 because a 3/8 in. NPS line was used rather than the 1/2-in. NPS line of Mark I.	
	D-26994	FV-106 Freeze Valve, Mark V		
	D-31350	Flowsheet		
	D-34889	Wiring Diagram (Sheet 3, item "D")		
	Fig. 13.3	Mark II Nitrogen Supplies to Waste Freeze Valve (FV-106) Vent Lines		
Waste Line	D-31350	Flowsheet	<u>Control Equipment</u> - Pyrovanne (thermocouple welded to pipelines midway of heater) and Variac. <u>Period of Use</u> - All runs. <u>Heater Data</u> - A 2000-w., 230-v. clamshell heater was placed around the first 8 in. of waste line outside of FV-106.	<u>This Report</u> - Secs. 13.4.2, 13.5; 13.6; 21.2; 21.4.4; 21.4.5; 22.4.1; 22.4.2. Table 22.1. <u>Engineering File Folder No.</u> - F-27.
	D-34888	Wiring Diagram (sheet 2, item "B")		
Freeze Valve, FV-506A	D-31350	Flowsheet	<u>Control Equipment</u> - Pyrovanne (thermocouple welded to one of the surge pots midway of the heater) and Variac. <u>Period of Use</u> - "L" Runs and Run M-64. <u>Heater Data</u> - Each enlarged section shown in Dwg. No. D-26994 was covered with a 1000-v. (230-v., 1-phase) clamshell heater. The two clamshells were connected to power as shown on Dwg. No. D-34888.	<u>This Report</u> - Secs. 13.4.2; 13.4; 13.6; 21.4.5; 22.4.1; 22.4.2. Table 22.1. <u>Engineering File Folder No.</u> - F-22.
	D-34888	Wiring Diagram (sheet 2, item "C")		

Table 13.1 (Continued)

Components	Drawings and Sketches		Miscellaneous Information	References
	Doc.	Title		
Vent Lines Lower Heater, FV-506	D-22778	Wiring Diagram (sheet 4, item "D")	<u>Control Equipment</u> - Pyromax (thermocouple welded to pipeline about midway of heater) and Varian.	<u>This Report</u> -
	D-31350	Flowsheet	<u>Period of Use</u> - All runs.	Secs. 13.4.3; 13.4.2; 13.5; 13.6; 3.4.5; 16.4.15; 22.4.1; 22.4.2.
			<u>Heater Data</u> - Semi-Duty Electric Co., Milwaukee, Wis.; Type No. 5012-B; Serial No. 91369; Req. No. W25K-19370-1; X-63903, 2.5 kw, 115/230 v., 1 phase.	Table 22.1. <u>Engineering File Folder No.</u> F-22. <u>Other Literature</u> - 28 (p. 47).
Vent Lines Upper Heater, FV-507	D-22778	Wiring Diagram (sheet 4, item "E")	<u>Control Equipment</u> - Controlled separately by equipment similar to that for FV-506.	Same as for FV-506.
	D-31350	Flowsheet	<u>Period of Use</u> - All runs.	
	D-34890	Wiring Diagram (sheet 4, item "F")	<u>Heater Data</u> - Semi-Duty Electric Co., Milwaukee, Wis.; Type No. M-3012-B; Serial No. 91372; Req. No. W25K-19370-1; X-63903; 2.5 kw, 115/230 v., 1 phase.	
Waste Container, FW-112	D-19706, Rev. 1	Waste Container (Mark I)	<u>Control Equipment</u> - Waste Weighing Device (Sec. 13.4.11).	<u>This Report</u> -
	D-19706, Rev. 2	Waste Container (Mark II)	<u>Period of Use</u> - Mark I: "C" and "E" runs. Mark II: "L" runs and M-64.	Secs. 13.4.4; 13.4.10; 13.4.11; 13.4.13; 13.5; 13.6.
	D-31350	Flowsheet		<u>Engineering File Folder No.</u> : Mark I - F-38. Mark II - F-36. <u>Other Literature</u> - 26 (p. 33).
Waste Nozzle Heater, FV-512	D-31350	Flowsheet	<u>Mark I</u>	<u>This Report</u> -
	D-31368	Wiring Diagram (sheet 8, item "D")	<u>Control Equipment</u> - Wheelco (thermocouple fixed to nozzle underneath heater insulation) and Varian.	Secs. 13.4.5; 13.4.1; 13.4.2; 13.4.12; 13.5; 13.6; 21.4.4; 22.4.1; 22.4.2.
	Fig. 13.4A	Mark I Waste Nozzle and Heater	<u>Period of Use</u> - "C" and "E" runs.	Fig. 13.4A; Table 22.1.
			<u>Heater Data</u> - One 1000-v., 115-v. Calrod was used in one of two ways per Fig. 13.4A: (a) Mark IA as in sketch and (b) Mark IB with the outer nozzle wall removed.	<u>Engineering File Folder No.</u> F-38.
	D-31350	Flowsheet	<u>Mark II</u>	<u>This Report</u> -
	D-34894	Wiring Diagram (sheet 8, item "D")	<u>Control Equipment</u> - Wheelco (thermocouple welded to nozzle) and Varian.	Secs. 13.4.5; 13.4.1; 13.4.2; 13.5; 13.6; 21.4.5; 22.4.1; 22.4.2.
	Fig. 13.4B	Mark II Waste Nozzle and Heater	<u>Period of Use</u> - "L" runs and Run M-64.	Fig. 13.4B; Table 22.1.
			<u>Heater Data</u> - Two 750-v., 115-v. Calrods were bent along the internal surface of the cone.	<u>Engineering File Folder No.</u> F-37.

Table 13.1 (Continued)

Components	Drawings and Sketches		Miscellaneous Information	References
	Key	Titles		
Waste Nozzle Vent Line Heater, FV-512A	D-34888	Wiring Diagram (sheet 2, item "AA")	<u>Control Equipment</u> - Pyrovac (thermocouple welded to pipeline midway of heater) and Variac. <u>Period of Use</u> - "L" runs. <u>Heater Data</u> - Two 750-w. and one 1000-w. Calrods (230 v.) were bent along pipe between the waste nozzle and FV-117.	<u>This Report</u> - Secs. 13.4.5; 13.4.6; 13.5; 13.6; 22.4.1; 22.4.2 Table 22.2 <u>Engineering File Folder No. F-65.</u>
Al ₂ O ₃ Trap, FV-117	Fig. 13.5	Al ₂ O ₃ Trap	<u>Control Equipment</u> - Cf. for FV-517. <u>Period of Use</u> - "L" runs. Trap was packed with Al ₂ O ₃ and arranged in the waste salt system as shown in Fig. 13.2.	<u>This Report</u> - Secs. 13.4.7; 13.4.1; 13.4.8; 13.5; 13.6; 23.4.9; 23.4.10; Fig. 13.2 <u>Engineering File Folder No. F-65.</u> <u>Other Literature</u> - 43.
Al ₂ O ₃ Trap, FV-517 Heater	D-34895	Wiring Diagram (sheet 9, item "E")	<u>Control Equipment</u> - Wheeler (thermocouple placed in trap thermowell) and Powerstat. <u>Period of Use</u> - "L" Runs. <u>Heater Data</u> - Three heaters were connected per Dog. No. D-34895. The two Calrods were wrapped helically around the cylindrical surface of the vessel; the finage-type heater was placed on the bottom surface of the vessel. Total power was 4 kw (230 v.)	<u>This Report</u> - Secs. 13.4.8; 13.4.1; 13.4.7; 13.5; 13.6; 22.4.1; 22.4.2 Table 22.2 Fig. 13.5 <u>Engineering File Folder No. F-65.</u>
Al ₂ O ₃ Trap Inlet Flange Heater, FV-517A	D-34888	Wiring Diagram (sheet 2, item "A")	<u>Control Equipment</u> - Pyrovac (thermocouple welded to one of the inlet flanges) and Variac. <u>Period of Use</u> - "L" Runs. <u>Heater Data</u> - one 1500-w., 230-w. Calrod was wrapped around the inlet flange and connected electrically per Dog. No. D-34888.	<u>This Report</u> - Secs. 13.4.9; 13.4.1; 13.4.7; 22.4.1; 22.4.2 Table 22.2 <u>Engineering File Folder No. F-65.</u>
Waste Dolly, FV-908	D-22493 D-22499 D-22476 D-22480 D-22481	Solid Waste Handling Dolly Assembly Dolly Drive Dolly Details Track Dolly Drive Details (sheet 11)	<u>Control Equipment</u> - None. (Dolly was positioned manually at the waste station) <u>Period of Use</u> - All runs.	<u>This Report</u> - Secs. 13.4.10; 13.4.4; 13.4.13; 13.5; 13.6 <u>Engineering File Folder No. F-37.</u> <u>Other Literature</u> - 32 (p. 34).

Table 13.1 (Continued)

Components	Drawings and Sketches		Miscellaneous Information	Reference
	Doc.	Title*		
Waste Weighing Device, FY-904	D-24487	Waste Handling Weighing Device Weld Assembly	<u>Mark IA</u> <u>Period of Use</u> - "C" and "E" Runs. Weigh element: strain gauge manufactured by Baldwin-Lima-Hamilton; Model U-1; Range: 0-500 lb. P.O. No. W39K-18359; Req. No. D-33984.	<u>This Report</u> - Secs. 13.4.11; 13.4.4; 13.4.5; 13.5; 13.6. <u>Engineering File Folder No. F-37.</u>
	E-24488	Waste Handling Weighing Device Assembly		
	D-24489	Waste Handling Weighing Device Details		
	D-24490	Waste Handling Weighing Device Details	<u>Mark IB</u>	<u>Other Literature</u> - 52 (pp. 54, 55).
	D-24490, Rev. 1	Waste Handling Weighing Device Details	<u>Period of Use</u> : "I" Runs; N-64. The arrangement was the same as for Mark IA except as shown on Dag. No. D-24490, Rev. 1.	
Waste Nozzle, FY-906	C-24475	Waste Discharge Nozzle	<u>Mark I</u>	<u>This Report</u> - Secs. 13.4.12; 13.4.1; 13.4.4; 13.4.5; 13.5; 13.6.
	D-24482	Waste Discharge Nozzle and Shielding Details	<u>Period of Use</u> - "C" and "E" Runs.	
	D-24483	Waste Discharge Nozzle and Shielding Details		
	D-24484	Waste Shutter Sub-assembly		Fig. 13.4A (Mk. I).
	E-24485	Waste Discharge Nozzle and Shielding Assembly		Fig. 13.4B (Mk. II).
	D-24486	Waste Discharge Nozzle Weld Assembly		<u>Engineering File Folder No. F-34.</u>
	D-31350	Flowchart		<u>Other Literature</u> - 52 (p. 35).
	Fig. 13.4A	Mark I Waste Nozzle and Heater		
	Fig. 13.4B	Mark II Waste Nozzle and Heater	<u>Mark II</u>	
	D-24486, Rev. 1	Waste Discharge Nozzle Weld Assembly	<u>Period of Use</u> - "I" Runs; Run N-64. The same shielding was used for Mark II as for Mark I except as shown on Dag. No. D-24486, Rev. 1.	
Waste Carrier, FY-912	E-19704	Waste Container Carrier	<u>Mark IA</u>	<u>This Report</u> - Secs. 13.4.13; 13.4.4; 13.4.10; 13.5; 13.6. Fig. 13.6.
	E-26124	Door Closing Jig	<u>Period of Use</u> - "C" and "E" Runs.	<u>Engineering File Folder No. F-36.</u>
	E-19704, Rev. 3	Waste Container Carrier	<u>Mark IB</u> <u>Period of Use</u> - "I" Runs. This design similar to Mark IA except as shown on Dag. No. E-19704, Rev. 3.	<u>Other Literature</u> - 52 (pp. 2, 33, 50).

- e. Closing HCV-11 to pressurize the waste out of FV-100.
- f. Transferring until the LR-2^a reading was 2 to 4% below the desired FV-100 heel value,^a then stopping the transfer.
- g. Re-establishing nitrogen purges through the freeze valve vent lines, and heating the vent furnaces to ~ 650°C to melt residual salt from the vent lines.
- h. Shutting off heat (about one hour later) to solidify the salt in the freeze valve.

With the Mark I waste line, nitrogen was continuously purged through the fluorinator instrument purges (step d. above) resulting in a pressure transfer which was stopped by opening HCV-11. In addition, the FV-106 freeze valve purges were continued during the transfer to prevent forcing waste salt into the vent lines. With the Mark II waste line, the freeze valve vent lines were kept closed during transfer, resulting in siphoning; HCV-11 was opened when siphoning started; the transfer was stopped by introducing nitrogen into the freeze valve vent lines.

System modifications necessitated additional minor steps in the procedure. For the complete transfer procedure, see Sec. 13.7.

13.3.2 Critical Operating Steps

- a. Maintaining the molten salt at ~ 600°C and the entire waste pipeline at $\geq 570^\circ\text{C}$ to permit flowing the salt through the line to the waste can. The salt transfer usually presented no difficulty when the specified salt and line temperatures were maintained.
- b. Stopping the salt transfer at the appropriate time to retain adequate salt for a fluorinator heel and for sealing the waste freeze valve. Providing a fluorinator heel and a freeze valve seal after each transfer permitted batch measurement, waste line purging, and leak-testing.^b Stopping the transfer was difficult with the Mark I waste line. The troubles encountered are discussed in Sec. 13.4.2.

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^aIn most cases, a 2 to 4% increase in the LR-2 reading occurred after discontinuing the transfer because of salt run-back.

^bBatch measurement could also be done by waste salt weight.

13.4 Equipment Evaluation

13.4.1 Major Design Changes

- a. Waste salt line and freeze valve: The 3/8-in. NPS schedule 40 line in Mark II required about 20% less autoresistance power than did the 1/2-in. NPS Schedule 40 line in Mark I (Table 21.1 and Secs. 13.4.2, 17.4.4, 21.4.4, 21.4.5).
- b. Waste nozzle and heater: The Mark II nozzle was easier to fit with a Calrod than Mark I. In addition, better contact between the conical surface of the nozzle and waste can in Mark II reduced the salt spattering problem (Sec. 13.4.5 and 13.4.12).
- c. Waste can vent line equipment: In the Mark I nozzle design, waste can venting was inadequate because of the can neck-waste nozzle geometry. In Mark II, the better waste-can-to-nozzle fit enabled exhausting the off-gas from the can through a heated Al_2O_3 chemical trap designed to scrub out the ZrF_4 vapor (Secs. 13.4.7, 13.4.8, and 13.4.9).

13.4.2 Waste Line, MS-100-1 to MS-106-1^a

- a. A siphon transfer was more difficult to stop in Mark I than in Mark II. (Sec. 13.4.1). For example, one siphon transfer in Mark I resulted in completely emptying the fluorinator and the freeze valve, thereby eliminating the fluorinator heel and the freeze valve seal. Thus, it was necessary to add barren salt in order to seal the freeze valve before making another run. This siphon was not stopped by opening HCV-11, the usual control measure, and was so fast (5 to 10 sec) that there was insufficient time to break it by adding nitrogen through the vent lines. All siphon transfers in Mark II were interrupted easily.

Waste salt transfer studies were made in the Unit Operations section with equipment simulating a 3/8-in. NPS Schedule 40 pipe system (later installed in the Volatility Pilot Plant) (42). Findings were:

1. A siphon transfer could always be interrupted by increasing the nitrogen purge rate to the freeze valve vent lines.
2. With the design change discussed below, sufficient liquid to seal the freeze valve could be retained even when completely emptying the fluorinator mock-up in an uncontrolled pressure transfer.

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^aSecs. 17.4.4, 21.4.4, and 21.4.5; Table 21.1.

The recommended design change consisted of surge pots in the vertical sections of the freeze valve and a slope toward the freeze valve at each vent-line saddle joint. In operation, liquid was retained by setting the nitrogen purge rates at the end of the transfer. Subsequently equalizing the pressures in the two vent lines by opening V-135 shown in Fig. 13.3 allowed the retained liquid to seek its level.

The surge pots in the Mark V FV-106 (Sec. 17.4.4) required heating by both autoresistance and resistance (FV-506A).^a Autoresistance heating alone was insufficient despite careful wall thickness design per Sec. 21.2. Cold spots evidently resulted from uneven current distribution in the surge pots wall or from overly thick walls at the welds.

The waste transfers made in the Mark II waste line were of the siphon variety. The only difficulties arose from salt plugs in the freeze valve vent lines, near the FV-100 wall, and at the waste nozzle. The vent line plugs resulted from molten salt being sucked into cold sections of these lines by the 10 in. of Hg vacuum created during waste transfer.

- b. Freeze valve vent lines and the waste line at or near the fluorinator wall were susceptible to plugging in both designs. Item b. was related to item a. in that siphoning was accompanied by a vacuum which carried molten salt up into the vent lines (42).^b

Two techniques to prevent vent line plugs during waste salt transfer have been proposed. First, keep a sufficient length (~10 ft) of the vent lines at 600°C to ensure that salt never freezes in these lines. Second, maintain only the first foot of the vent lines at 600°C using the vent furnaces. Salt frozen in the vent lines above the 600°C section would be subsequently melted.

In general, plugs near the FV-100 wall were caused by inadequate heating to maintain the line at $\geq 570^\circ\text{C}$ although one incident of NiF_2 precipitation (Run L-4) did occur.^c Two locations were particularly difficult to heat: (a) above the melt level inside the fluorinator, and (b) in the first 1/2-in. to 2-in. of the waste line just outside the fluorinator shell. The pipe in both of these regions was nickel pipe.

The unheated section of pipe above the melt level inside the fluorinator caused trouble as evidenced by the frozen salt annulus above the interface, indicating that the waste line at this level was below control temperature. To reduce the tendency for annulus formation, heater FV-501 was redesigned as described in Sec. 5.4.5a. The limited data available after this change indicated that this measure was partially effective.

^aTable 22.1 and Secs. 22.4.1 and 22.4.2.

^bFor freeze valves, see Sec. 17.4.4. Line Temperatures were determined with thermocouples placed as described in Sec. 21.4.6.

^cCf. Sec. 5.4.1c and later in this section.

Fig. 13.3. Mark II Nitrogen Supplies to Waste Freeze Valve (FV-106) Vent Lines

Note: Heaters are not shown.
See Fig. 13.2

Fig. 13.5. Al₂O₃ Trap (FV-117)

The first 1/2-in. to 2-in.^a of the waste salt line outside of the fluorinator vessel wall was nickel. Frozen salt plugs formed in this section of the line because lower-resistance nickel was heated to a lower temperature by autoresistance than was Inconel. At 550-600°C, for example, the resistance of nickel is about one-third that of Inconel (Sec. 21.2). These plugs were eliminated by adding the clamshell heater, FV-500A.^b This difficulty was anticipated with the heater addition being preferred over making a dissimilar metal weld (nickel-Inconel) at the vessel wall.

The placement of autoresistance electrodes for the waste line is delineated in Secs. 21.4.4, 21.4.5, and 21.4.6. Although not determined conclusively, the effect on FV-100 wall plugs of moving the FV-100 electrode from the waste line to the FV-100 flange was believed to be beneficial. No disadvantage to flange placement has been found. However, placing the nozzle-end electrode on the nozzle or nozzle vent line did not eliminate the necessity for FV-512 (Sec. 13.4.5).

Most of the waste line plugs were caused by inadequate heating. At least one plug, however, was due primarily to the precipitation of impurities in the salt. This plug (at the end of Run L-4) was caused by NiF_2 which was present both as a corrosion product and as an impurity approaching the solubility limit in the feed. This high-melting waste salt mixture could not be transferred because all dip tubes were plugged. Consequently, removal required jack-hammering and chipping which were very costly, and which resulted in mechanical damage to the fluorinator as described in Sec. 5.4.1c. In a very radioactive operation, such manual methods of waste removal would be impossible.

During the Run C-8 waste transfer, a crack occurred in the nickel section of the Mark I waste line about 1/4 in. away from the fluorinator. It was essential to repair the line before resuming operations. Two factors contributing to this failure were: (a) approximately a half-inch fall of the vessel during prior maintenance work and (b) thermal cycling of the waste line and fluorinator (31, p.34).

- c. Insulation was essential since, when uninsulated, the line reached only ~ 300°C at full power of 4200 watts.

The Mark I waste line was removed after the "E" runs and taken to the burial ground (Secs. 21.4.4 and 23.4.16b).

Corrosion of the Mark II waste line was not severe except near the waste nozzle. The 21-mil attack at the waste nozzle might be attributed to the external heaters FV-512 and -512A which probably augmented the line temperature near the waste nozzle (19).^c

^aTwo inches in the Mark I fluorinator and 1/2-in. in the Mark II vessel.

^bTable 22.1 and Secs. 22.4.1 and 22.4.2.

^cThe waste line in this reference is termed "Mark II-B." After the corrosion specimens were removed from the Mark II waste line, the waste line and auxiliary equipment were discarded in Burial Ground No. 3 (Sec. 21.4.5, 22.4.3, and 23.4.16b). The specimens tested were decontaminated per Sec. 23.4.15b.

13.4.3 Vent Lines Heaters, FV-506 and FV-507

The vent lines were difficult to heat near the saddle joints because the metal walls at the welds were thicker, and because of the chimney effect and conduction of heat to cold portions of the vent lines. Therefore, the lower vent lines heater was placed as close to the top of the freeze valve as possible to adequately heat the lines near the saddle joints. In addition, any opening between the freeze valve insulation and the bottom of the lower furnace or between the two furnaces was eliminated to avoid cooling. The vent lines were electrically insulated from each other to prevent unnecessary autoresistance power leaks. The time-to-650°C was about one hour.

13.4.4 Waste Container, FV-112

The salt spattering problem was reduced but not completely eliminated in the Mark II design. This was attributed to the venting and the better seal between the waste nozzle and the can neck in the Mark II design (Secs. 13.4.10, 13.4.11, and 13.4.13). See Sec. 23.4.16a for activity of the "E" and "L" run waste containers.

13.4.5 Waste Nozzle Heater, FV-512^b

The Mark IA and IB designs differed in that for Mark IA the double-wall of the nozzle was retained whereas for Mark IB the outer nozzle wall was removed as indicated on Fig. 13.4A. Both the Mark IA and IB designs were inadequate, however, for heating the Mark I waste nozzle for two reasons: (a) fitting a heater to the nozzle was difficult and (b) placing the heaters close to the end of the nozzle made them very susceptible to contact with the corrosive molten salt.

The Mark II design (shown in Fig. 13.4B) was capable of keeping the nozzle at 650°C with a 120 v setting on Variac TC-11. The time-to-650°C was < 3 hr. In addition, this heater seemed to be capable of melting a nozzle plug.

13.4.6 Waste Nozzle Vent Line Heater, FV-512A^c

This heater was satisfactory. The time-to-temperature data were similar to those for Mark II FV-512 (Sec. 13.4.5).

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^aThe situation at FV-508 and -509 was similar (Sec. 3.4.5), but that at FV-504 and -505 was dissimilar (Sec. 16.4.15).

^bAll three waste nozzles were heated both by autoresistance and resistance (Table 22.1 and Secs. 13.4.1, 13.4.2, 13.4.12, 21.4.4, 21.4.5, 22.4.1, and 22.4.2).

^cTable 22.2 and Secs. 22.4.1 and 22.4.2.

Mark IA Heater: As Shown

Mark IB Heater: Outer Nozzle Wall Removed

Insulation is not shown on heater.

The N_2 line adjacent to tip for purging tip during waste transfer is not shown.

Fig. 13.4A. Mark I Waste Nozzle and Heater

Fig. 13.4B. Mark II Waste Nozzle and Heater

13.4.7 Al₂O₃ Trap, FV-117

The purpose of this trap shown in Fig. 13.5 (on same page as Fig. 13.3) was to remove radioactive ZrF₄ vapor vented from the waste can (Sec. 13.4.1). The trap was packed with 1/4-in. Al₂O₃ and maintained at 700-800°C. The line between the waste nozzle and the trap^a was kept at 400°C.

Despite the fact that the 700-800°C operating temperature was reportedly low for the Al₂O₃-ZrF₄ reaction, this temperature was selected because of the sheath limitation (815°C or 1500°F) for Calrods (43).^b The necessity for using readily available Calrods to heat FV-117 resulted from the limited construction time before the "L" runs.

Analytical data for samples taken from the Al₂O₃ trap after the "L" runs were:

a. Activity of Al₂O₃:^c

α: None

β-γ: At inlet - ~18 mrem/hr
At outlet - 4 mrem/hr

b. Chemical Analyses:

Essentially no ZrF₄ in the Al₂O₃ bed.

About 1.2% Zr in every light whitish deposit on top flange.
(This material passed the trap.)

From these analytical data, the trap apparently performed no clear-cut function during the "L" runs. In addition, the vapors vented from the approximately equimolar NaF:ZrF₄ waste in the "L" runs evidently presented no serious radioactive hazard although the activity of this waste was ~10 rem/hr. Despite the fact that the trap was not needed for the "L" runs, two considerations for future work seem pertinent. First, higher-activity salt mixtures might result in hazardous amounts of radioactive Zr in the vapors. Second, the 700-800°C operating temperature for the trap and the 400°C operating temperature for the vent line should be increased.

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^aAt that time, a line temperature of 400°C was considered satisfactory to prevent ZrF₄ vapor deposition. Since that time, however, experimental work on entrainment and ZrF₄ vapor in the fluorinator exit gases (Runs M-62, 63 and 64) indicated that ZrF₄ vapor will condense on a 400°C surface.

^bAlso cf. Sec. 13.4.8.

^cAlpha activity was determined with an alpha survey meter (Sec. 23.4.9) and β-γ activities with a cutie pie (Sec. 23.4.10, 16.4.16).

13.4.8 Al₂O₃ Trap Heater, FV-517^a

This heater was satisfactory for keeping the Al₂O₃ trap bed at the operating temperature of 700-800°C. Time-to-750°C was < 3 hours with a 60-80 v setting on Variac, TC-117.

13.4.9 Al₂O₃ Trap Inlet Flange Heater, FV-517A^a

This heater was adequate for maintaining the flange at 400°C. The time-to-400°C was < 3 hours with a 100-v setting on Variac, TC-110.

13.4.10 Waste Dolly, FV-902^b

The principal disadvantage with the FV-902 design was that recesses existed in which spattered salt could collect. These recesses included holes in the chain as well as a number of hard-to-get-at places in the chain support and the can base-plate.

A can of radioactive salt which dropped from the carrier in Run L-8 bent the base-plate. For the one remaining run, the waste system was used without the carrier since the bent base interfered with properly positioning the can-carrier assembly. (Sec. 13.4.13.)

13.4.11 Waste Weighing Device, FV-904^c

The Mark IA and Mark IB waste weighing devices differed only in the size of waste container neck each would accommodate. This instrument was used principally to qualitatively monitor the waste transfer from start to finish. In addition, it was useful in batch measurement.

13.4.12 Waste Nozzle, FV-906^d

The two waste discharge nozzles are shown in Figs. 13.4A and 13.4B. The Mark I nozzle was unsatisfactory because: (a) the nitrogen flow aggravated the plugging tendency by cooling the nozzle; (b) the tip of the nozzle could not be heated to 550-600°C by autoresistance even without nitrogen flow; and (c) a suitable Calrod or clamshell heater could not be fitted to the nozzle design.

Although the Mark II design was superior to the Mark I in that it could be maintained at 650°C, some nozzle plugs still formed. In addition, the poor seal (although better than in Mark I) achieved between the nozzle and the can neck allowed salt to spatter during a transfer.

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^aTable 22.2 and Secs. 22.4.1 and 22.4.2. Also Secs 13.4.1 and 13.4.7.

^bSec. 13.4.4 and 13.4.13.

^cSec. 13.4.4.

^dSecs. 13.4.1, 13.4.4, and 13.4.5.

Two aspects of Mark II waste nozzle plugging are pertinent here. First, it was possible by proper operating technique to prevent plug formation following a waste transfer. This could be done by flowing nitrogen through line LN-106-2 and thence through the waste nozzle long enough after a transfer to ensure the freezing of all residual salt. This prevented a sufficient accumulation of salt to form a plug. However, a plug in line LN-106-2 upstream from the freeze valve would prevent using this technique. Second, the heating of this nozzle was adequate in some cases to melt the nozzle plugs, although experience with the Mark II nozzle was insufficient to determine whether a plug once formed might present a major difficulty.

13.4.13 Waste Carrier, FV-912^a

The waste carrier contained the waste container while filling and until burial.

The Mark IA waste carrier accommodated a larger-neck can than the Mark IB carrier. Two troubles with the waste carrier were experienced. First, the recessed holes in the carrier top tended to collect spattered or spilled salt. Such salt was difficult to remove and, if radioactive, presented a distinct hazard to personnel.

Second, a waste can of radioactive salt was dropped in Run L-8. Before making the next run, the faulty door-closing device causing the incident was altered to prevent recurrence. The equipment flaw which triggered this incident was the inadequate provision for retaining the securing bolt on the door-closing device.^b The before-and-after-the-incident views of this device in Fig. 13.6 show that the lip added to the upper steel strap was the difference between a safe and unsafe situation. The arrow in the beforehand view shows the direction in which an applied force would cause the can to drop from the carrier.

13.5 Summary and Conclusions

The waste salt system was operable. The siphon transfer (Mark II) was preferred to the pressure transfer (Mark I). The average waste transfer time was 7⁺ hours in the "E" runs and 11 hours in the "L" runs. Sources of major troubles were:

- a. Plugs in the piping.
- b. Blowing the freeze valve seal and eliminating the fluorinator heel.
- c. Splattering of salt at the waste can.

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^aSecs. 13.4.4 and 13.4.10; 23.4.16b.

^bThe "as-built" design differed from that shown in ORNL drawing No. E-19704.

BEFORE THE INCIDENT

AFTER THE INCIDENT

Fig. 13.6.. Before- and After-the-Incident Views of the Waste Carrier Door Securing Device

- d. Unsafe door-closing device on the waste carrier.
- e. Crack in waste line during Run C-8 waste transfer.

Plugs formed in almost all cases at cold spots. Therefore, the usual remedy was heating the pipe at the plug to $\geq 570^{\circ}\text{C}$.

Although plug formation occurred in nearly every part of the waste line, the three following regions were particularly susceptible to plugs; (a) waste line adjacent to the FV-100 wall, (b) waste nozzle, and (c) freeze valve vent lines. The flow blocks occurring in the waste line just outside of the FV-100 wall resulted from salt freezing in the short run of nickel pipe at this location because this section of the waste line was incompletely heated by autoresistance. This situation was corrected by adding FV-500A in both the Mark I and Mark II waste lines. Although not assessed, the effect on wall plugs of moving the FV-100 auto-resistance ground lug from the waste line to the FV-100 flange was believed to be beneficial. And no disadvantage to the flange location has been found. In addition, some trouble resulted from plugs in the internal portion of the waste line in FV-100 above the melt level. This situation was improved by redesigning FV-501.

Even though the waste line surge pots were carefully designed for auto-resistance heating, FV-506A was also required to increase temperatures $< 570^{\circ}\text{C}$.

At the waste nozzle, two complementary means of plug elimination were used: (a) flowing nitrogen through the nozzle at the end of each transfer to prevent plug formation and (b) heating the waste nozzle (with FV-512) either to prevent plug formation or to melt a plug which had already formed. This two-fold means of eliminating plugs was more effective in the Mark II design. Placing the ground electrode on the nozzle did not eliminate the necessity for FV-512.

In the freeze valve vent lines, plugs were reduced by avoiding high-pressure (i.e., > 4 to 6 psig) or high-vacuum (i.e., > 10 in of Hg) conditions during transfer and also by melting at the end of the transfer any plugs formed in the vent lines during the transfer. The Mark II design was slightly better in reducing vent line plugs than was Mark I.

The extent of waste line corrosion was determined only on the Mark II waste line. Results indicated corrosion to be severe (21-mil attack) only near the waste nozzle.

Although blowing the freeze valve seal and eliminating the fluorinator heel occurred infrequently, corrective measures were time-consuming. This problem was virtually solved in the Mark II design with the 3/8-in. NPS Schedule 40 waste salt line and the newly developed method of waste transfer.

Salt spattering at the waste station was not eliminated.

The faulty design of the waste carrier door-closure triggered the accidental dropping of the waste can in Run L-8. The remedy was the bolt-retaining lip described.

The study of the vent system containing the Al_2O_3 trap for ZrF_4 vapor removal yielded inconclusive results.

13.6 Recommendations

It is recommended that:

- a. Autoresistance heating continue to be used in waste salt transfer lines with the FV-100 electrode on the vessel flange and the nozzle-end electrode on the nozzle.
- b. 3/8-in. NPS Schedule 40 pipe be used for salt transfer lines.
- c. The use of the siphon transfer be continued.
- d. The freeze valve vent lines be redesigned (either to prevent plug formation or to enable the melting of plugs once formed).
- e. There be no section of nickel pipe between the fluorinator wall and the Inconel waste line in a new-design waste system.
- f. The fluorinator heater be so designed that cold spots cannot occur in the waste line within the fluorinator.
- g. The waste nozzle and surge pots in the freeze valve be heated both by autoresistance and by resistance heaters such as Calrods.
- h. Means be found to eliminate or significantly reduce the spattering problem.
- i. The waste carrier be redesigned to eliminate recesses in which spattered salt might collect and to provide a safe bottom closure.
- j. The waste dolly be redesigned to include a power drive with remote control and to avoid recesses in which salt might collect.
- k. The use of unit shielding at the waste station be continued.
- l. Additional study be done to determine the need for a chemical trap in the waste vent line.

13.7 Operating Procedure: Waste Salt Transfer (Revised June 24, 1958)

WSF _____
Date _____
Time _____
Operator _____

1. Check that waste container is properly in place and ready for use.
2. The following valves in the penthouse should be open: V-27 _____, V-72 _____, V-113 _____, and V-123 _____ or V-124 _____.
3. The following valves in the Penthouse should be closed: V-87C _____, V-88 _____.
4. The following valves on the panelboard should be open: HX-11 _____, HX-35 _____, HX-36 _____.

5. The following valves on the panelboard should be closed: HX-8 _____, HX-12 _____, HX-14 _____, HX-16 _____, HX-32 _____, HX-34 _____.
6. The following valves in the gallery should be open: V-70 _____, V-78 _____, V-79 _____, V-81 _____.
7. The following valves in the gallery should be closed: V-74C _____, V-75C _____, and V-91C _____.
8. Set regulator PV-8 at 12 psig _____.
9. Set regulator PV-44 at 4.5 psig _____.
10. Set the following purges:

Valve No.	FI - No.	Location	Flow Rate	Purge Point
_____	_____	Penthouse	1.2 cfh	DE-2-10
_____	_____	Penthouse	1.2 cfh (ea side)	LE-2
86C	26	Gallery	1 cfh*	LN-106-1
90C	30	Gallery	1 cfh*	LN-106-2

*Enough to raise bob in rotameter.

NOTE: If any of these lines is plugged, notify supervisor.

11. Switch TX-1B _____, EX-1B-4 _____, and TX-1B-7 _____ to position "A".
12. Do not proceed beyond this step without signature of Problem Leader:
_____
Problem Leader
13. Do set these controllers on the setpoints given below:

Controller	Setpoint	Time Set	Time Up
TIC-1B-3A	500°C indicate and control	_____	_____
TIC-1B-7A	650°C indicate <u>only</u>	_____	_____
TIC-1B-4A	650°C	_____	_____
TIC-11	650°C	_____	_____
TIC-108	250°C	_____	_____
TIC-110	400°C	_____	_____
TIC-111	400°C	_____	_____
TIC-112	600°C	_____	_____
TIC-113	620°C	_____	_____
TIC-FV-517	750°C	_____	_____

14. Adjust these Variacs to setpoints shown. ADVANCE TO ABOUT 1/3 OF THE VOLTAGE SHOWN -- WAIT 10 MINUTES -- THEN INCREASE SETPOINT ANOTHER 1/3-- WAIT 5 MINUTES AND RESET TO VALUE SHOWN BELOW.

ET-1B-4	314 amps	TC-111	100 volts
TC-11	120 volts	TC-112	100 volts
TC-108	_____ volts	TC-113	140 volts
TC-110	100 volts	TC-FV-	_____
		517	6 to 30 volts

15. When transfer line and freeze valve are up to temperature, record the following data:

Time _____	WR-1A _____
TR-1B-2 _____	LR-2 _____
TR-1B-4A _____	PR-33 _____
TR-1B-5A _____	DR-2 _____

16. Get approval of shift supervisor before proceeding further.

17. Open V-99 and valve on vacuum line to Cell 1.
18. Have instrument men set vacuum controller on 5 in. water.
19. Set WE-1A between 20 and 30% (adjust hydraulic lifting device on waste receiver).
20. Close V-86A _____, V-86B _____, V-90A _____, V-90B _____.
21. Open V-86C _____ and V-90C _____.
22. Close equalizer valve between V-86C and V-90C.
23. Close ECV-11 _____; when transfer begins watch PI-62 on FI-26 purge line. When this gage reading reaches 10 in. of Hg vacuum the siphon has started -- open ECV-11 _____.
24. As soon as HX-11 is closed and until the transfer is complete, record the following data every two to five minutes.

Time	LR-2	PR-33	PI-38	FI-26	FI-30
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____

25. To interrupt siphon open V-90A fully _____ and then close to give 30% on FI-30 _____.
26. Immediately, open V-86A fully _____ and then close to give 30% on FI-26.
27. Wait until LR-2 levels out and then close V-90A _____.
28. Decrease flow through FI-26 to 8% _____. Open equalizer valve between V-86C and V-90C _____.
29. Increase setpoint on TIC-1B-3A and TIC-1B-7A to 650°C _____. Time _____.
30. One hour later, shut off waste loop heaters.
31. After line cools to $\approx 150^{\circ}\text{C}$, close V-90C _____.
32. Record LR-2 _____%, and WR-1A _____%.
33. Open V-74C _____, V-75C _____, V-87C _____ and V-91C _____.
34. Reset PV-8 to 4.5 psig _____.
35. Set the following purges at 150 cc/min: FI-14 _____, FI-15 _____, FI-26* _____, FI-30* _____, and FI-31 _____ (also FI-27).

* Just enough for float to rise off bottom retainer.

36. When TR-1B-2 cools below 400°C, close HX-11. Time _____.
37. When PR-33 shows 4 psig, open HX-11. Time _____.

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14.0 NITROGEN SYSTEM

14.1 Introduction

The nitrogen system provided the inert gas required for the vessels, instrument purges, and piping in the VPP. Nitrogen instead of air was used as the purge gas for pneumatic-type instruments, and a blanket of nitrogen was maintained in process vessels and pipes at all times as needed. The principal steps in the operation of this system:

- a. Keeping full nitrogen cylinders on the two manifold banks.
- b. Maintaining the nitrogen dryer.

14.2 Equipment

The nitrogen system equipment was arranged as shown in Fig. 14.1. Information regarding components is given in Table 14.1.

14.3 Operation

14.3.1 Operating Procedure

- a. Changing empty nitrogen cylinders (Sec. 14.7.1).^a
  1. Keeping data as required such as cylinder pressures, cylinder numbers, and manifold pressure (after putting on full cylinders) on N₂ Cylinder Log.
  2. Closing cylinder and manifold valves and moving empty and full cylinders as required.
  3. Cracking the valve on each full cylinder for an instant to blow dust out of the valve body.
  4. Opening valve supplying purge nitrogen to the manifold at 5 psig.
  5. Coupling each cylinder in turn by first opening the corresponding manifold and then with N₂ flowing through the pigtail connecting the tubing to the cylinder.
  6. Opening all four manifold valves wide, closing the valve on purge line, and lowering the low-pressure regulator to zero.

---

^a"Empty" cylinder should have >75 psig of N₂ to avoid contamination from the atmosphere (Sec. 14.4.1).

Fig. 14.1. Equipment Arrangement in the Nitrogen System

Table 1A.1
List of Nitrogen System Components

Components	Drawings and Sketches		Miscellaneous Information	References
	Doc.	Title		
Cylinders	D-31349	Nitrogen Flowchart	Period of Use - All Runs Standard high-pressure nitrogen cylinders (2200 psig) were fitted with MCC No. 344 valves labelled "O.R.T.L. - V.P.P." and numbered.	This Report - Secs. 14.4.1; 14.3.1; 14.4.2; 14.4.3; 14.4.4; 14.4.5; 14.4.6, item a; 14.4.6, item b; 14.5; 14.6. <u>Engineering File Folder No. P-52.</u>
Manifold	Q-1679-5-B2 Q-1679-47-R6 D-23220 D-31349	Nitrogen Cylinder Manifold Wiring Diagram Nitrogen Header Station Nitrogen Flowchart	Period of Use - All Runs. Fabricated by National Cylinder Gas Co.	This Report - Secs. 14.4.2; 14.4.1; 14.5; 14.6. <u>Engineering File Folder No. P-52.</u>
Dryer, FV-146	Vendor's 30875-7 Bulletin No. D-200 D-34897	15-8N Grid Dryer Installation - Operation - Maintenance Wiring Diagram (sheet 1, item "J")	Period of Use - Runs C-9 through -15 "E" and "L" runs. "Grid", model 158, serial No. D-2427, supplied by C. M. Keop Manufacturing Co., Baltimore, Md.; P. O. No. 25X-68892, drying agent was Linde Molecular Series, Type SA.	This Report - Secs. 14.4.3; 14.3.1; 14.4.1; 14.4.4; 14.4.6, item c; 14.5; 14.6. <u>Engineering File Folder No. P-58.</u>
Water Analyzer	Vendor's Manual No. 191	Electrolytic Water Analyzer Instruction Manual	Period of Use - Runs C-9 through C-15; "E" and "L" Runs. Model "W" Electrolytic Water Analyzer supplied by Manufacturers Engineering and Equipment Corp., Bethoro, Pa., contained 5 ranges: 0-10, 0-30, 0-100, 0-300, and 0-1000 ppm. P. O. No. 71X - 72634.	This Report - Secs. 14.4.4; 14.4.6, item a; 14.5; 14.6. <u>Engineering File Folder No. P-58.</u>
Transmitter			<u>Main Transmitter Rack</u>	<u>This Report -</u>
Racks	Q-1679-14-R1 Q-1679-15 Q-1679-16	Main Transmitter Rack - Front Transmitter Panels Main Transmitter Rack - Back	Period of Use - All Runs. This rack contained the pressure transmitters and block valves for the pneumatic-type instruments: DE-1 and -2; LE-2, -4, -5, -8, and -9; PE-4, -5, -6, -7, -8, and -9; PE-12, -13, -14, -15, -16, -30, -34, -38, and -45.	Secs. 14.4.5; 14.5; 14.6; 3.4.2; 5.4.7; 8.4.7; 9.4.7, item c; 9.4.7, item d; 9.4.7, item e; 11.4.1, item a; 15.4.2; 16.4.16. <u>Engineering File Folder No. P-52.</u>
	Q-1679-17 D-23220, Rev. 4 D-23228, Rev. 2	Nitrogen Transmitter Rack Nitrogen Header Station Above Cell Piping ¹	<u>Nitrogen Transmitter Rack</u> Period of Use - All Runs. This rack contained the pressure transmitters for these instruments: FE-1, -3, and -4; and PE-5 and -6.	This Report - Secs. 14.4.5; 14.5; 14.6. <u>Engineering File Folder No. P-52.</u> <u>Other Literature - 13.</u>

¹ Airline piping behind Main Transmitter Rack.

7. Opening each cylinder valve in turn by first cracking the valve until the pressure equalized and then opening the valve fully.
8. Checking to see whether the red panelboard light was out. See item e., Sec. 14.3.2.

b. Regenerating Oriad Type Dryer^a

1. Switching main valve slowly from Unit A to Unit B (This put Unit B "on-stream" and Unit A "off-stream" for regenerating.)
2. Turning heat on Unit A and setting the thermostat to maintain a maximum temperature of 400° F. (Maintain a low N₂ flow through the unit being regenerated and, if not in use, also on the spare unit at all times to prevent moist air from getting into the unit.)
3. Cutting off heat to Unit A after 4 to 6 hours.

See Sec. 14.7 for the complete procedure for changing empty nitrogen cylinders and Kemp Manufacturing Company's Bulletin No. D-200 - Book No. 10,033 (corrected) for the electric reactivation of the Oriad type dryer.^b

14.3.2 Critical Operating Steps

- a. Momentarily cracking the valve on each full cylinder to blow out dust.
- b. Purging the pigtail with N₂ while connecting to the cylinder to avoid getting air into the nitrogen system.
- c. Cracking each cylinder valve (after connecting) to equalize the pressure before opening the valve fully.
- d. Marking each cylinder bled to < 75 psig so that the nitrogen supplier will know to pump out the cylinder before refilling.
- e. Checking to see whether the red light at panelboard had gone off after changing a bank of cylinders. (If it is not out, the manifold will not switch automatically to the full bank when the other bank becomes empty.)

---

^aSec. 14.4.3.

^bEngineering File Folder No. F-58. The procedure for the water analyzer which is not recorded here is in Manufacturers' Engineering and Equipment Corporation's instruction manual in Engineering File Folder No. F-58.

14.4 Equipment Evaluation^a

14.4.1 Cylinders (44)

Nitrogen was originally obtained through the ORNL Purchasing Department from National Cylinder Gas Division of Chemetron Corporation (NCG) in Knoxville. By special arrangement, VPP cylinders (identified by painted numbers and markings) to be refilled were bled by NCG to assure that they had a positive nitrogen pressure in them before being refilled. Cylinders that were depleted to < 75 psig were marked by VPP operators for evacuation, purging, drying and pressure testing by NCG before refilling. The NCG filling manifold (Sec. 14.4.2) was checked for positive pressure by bleeding before any VPP cylinders were connected to it. Nitrogen was bled from this manifold and from each cylinder as it was connected to the manifold to prevent entry of air and/or moisture. A similar procedure was followed in changing cylinders at the VPP manifold; pressure reducing valve PV-47 and relief valve PSV-48 allowed low pressure N₂ from full cylinders to purge the section of manifold while cylinders were being changed.

In August, 1958, the nitrogen contract was let to the Welding Gas Products Company in Chattanooga, Tennessee. The handling procedure was unchanged.

A covered outdoor storage area for full and empty nitrogen cylinders was provided on the east dock of Building 3019 near the VPP nitrogen supply manifold. No difficulty was encountered by exposure of this system to out-door temperatures. However, the nitrogen dryer (Sec. 14.4.3), water analyzer (Sec. 14.4.4), transmitter rack (Sec. 14.4.5), valves and purge rotameters (Sec. 14.4.6b) were housed in the heated operating gallery (Dwg. D-23220).

14.4.2 Manifold

Two banks of four cylinders each supplied nitrogen at an initial pressure of ~2000 psig to the manifold through flexible copper tube "pig-tails" and high-pressure valves.^c Only one bank of cylinders was used at a time. Each bank was provided with a pressure-reducing valve (PV-1A or PV-1B, usually set at 20-30 psig), a pressure switch (PX-1A or PX-1B) and a low-pressure alarm (PA-1A or PA-1B). When the pressure in the bank of cylinders in use fell to 100 psig, the pressure switch automatically actuated the alarm and a 3-way solenoid valve (PCV-1) mounted at the junction of the two halves of the manifold. The solenoid valve then closed the depleted bank of cylinders and opened the full bank to the nitrogen header. This system worked quite satisfactorily until the last two weeks of operation, when a worn coil in the alarm annunciator circuit prevented the switch-over. This coil had been in service two years, making some 500 switch-overs.

^aAfter the "L" runs, the nitrogen system equipment downstream of the dryer was dismantled and moved to Burial Ground No. 3 (Sec. 23.4.16b). The remaining equipment was retained for future processing.

^bSecs. 14.3.1 and 14.4.6a.

^cSec. 14.4.1.

The nitrogen supply system was organized as follows (flowsheet D-31349): Main supply header LN-1 from manifold to:

- a. Coolant line LN-510-1 for FV-510 fan.
- b. Blanket header LN-2 for:
  1. Salt charging system (LN-102-1 and -2; LN-108-1 and -2).
  2. Waste nozzle FV-112 (LN-112-1)(line abandoned).
  3. Product collecting and sampling systems (LN-126-1).
- c. Blanket header LN-4 (originally operated at a higher pressure than other blankets because of waste dip leg in FV-100) for:
  1. FV-103 inlet purge (LN-100-1).
  2. FV-106 purges (LN-106-1 and -2).
- d. Penthouse supply header (LN-3) for:
  1. FV-100 sampler blanket (LN-955).
  2. Ring-joint flange leak-detecting system (LN-FLD).
  3. Main instrument transmitter rack (LN-MTR).
- e. Vacuum relief gas to vacuum pump FV-420 (LN-420-1).
- f. $N_2 - F_2$ supply to fluorinator FV-100 and absorbers FV-120 and 121 (LN-121-1).
- g. Purge for F_2 system (LN-FTR-1).

14.4.3 Dryer, FV-146

The Oried type dryer was satisfactory. It maintained the water content of the nitrogen supplied to the VPP at $< \text{one ppm}$ under these conditions.^a

- a. Rate of regeneration - once a week (Sec. 14.3.1b).
- b. Average moisture content of N_2 to the dryer - probably between 10 and 50 ppm (Secs. 14.4.4 and 14.4.6a).
- c. Rate of N_2 usage - about 1000 standard cubic feet per day.^b

^aThe water content of the N_2 was determined with the Model W Electrolytic Water Analyzer (Sec. 14.4.4). No O_2 analyses were made of the N_2 in VPP (Sec. 14.4.1).

^bThis value was derived from these data: (a) During Runs E-1 and E-2 5-1/2 cylinders were used (~ 1100 std cu ft/day) and (b) during Runs L-5 through -9 five cylinders were used (~ 1000 std cu ft/day).

14.4.4 Water Analyzer

The water analyzer apparently worked satisfactorily. This instrument was calibrated by the Instrument Division. It consistently showed that the dried N_2 had a lower moisture content (<1 ppm) than did the cylinder N_2 (10-50 ppm, Sec. 14.4.6a), as would be expected. The instruction manual is in Engineering File Folder No. F-58.

14.4.5 Transmitter Racks^a

The main transmitter and nitrogen transmitter racks were satisfactory (Sec. 15.4.2).

The main transmitter rack (in Penthouse, Fig. 14.2) contained the pressure transmitters, block valves, and "C" clamp rotameters for the pneumatic-type instruments: DE-1 and -2; LE-2, -4, -6, and -8; FE-5, -6, -7,^b -8, and -9; and PE-12, -13, -14, -15, -16, -30, -33, -34, -38, and -45(13). Nitrogen was the purge gas for all instruments requiring purges (except FE-9) and was supplied to this rack from line LN-3. Minor alterations were made in this transmitter rack from time to time. Major troubles were:

- a. F_2 and/or UF_6 getting into the pressure transmitters and purge rotameters. Three measures in addition to those normally exercised in operations were taken to prevent this: (a) installed Hoke No. 413 valves in the instrument lines between the cells and main transmitter rack to give purge shut-off as required; (b) permanently capped off instrument purge lines for FE-5, -6, and -7; PE-12, 13, 14, 15, and -38, after Run E-3 (Secs. 8.4.7, 9.4.7c, 9.4.7d, and 17.4.1; and (c) provided the N_2 - F_2 interlock (Sec. 15.4.4). Fluorine and/or UF_6 backing up into instrument purge lines damaged and/or contaminated lines, rotameters, and instruments. An example of what could happen was demonstrated when F_2 initiated a fire in one of the plastic tubing lines. (Plastic tubing was used extensively in instrument lines carrying air or nitrogen. The plastic tubing was both cheaper and easier to install than copper tubing.)

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^aSec. 15.4.2 for fluorine transmitter rack.

^bThese instruments are discussed briefly in other sections as follows: (a) LE-9 and PE-45 in Sec. 3.4.2 (Salt Charging System); (b) LE-2, DE-2, PE-33, PA-33, PX-33A, PX-33B, and PA-57 in Sec. 5.4.7 (Fluorinating System); (c) FE-5, PE-12, PE-34, and PE-38 in Sec. 8.4.7 (Absorbing System); (d) FE-6, -7, -8; PE-13, -14, -15, and -16 in Secs. 9.4.7c, 9.4.7d, and 9.4.7e (Cold Trapping System); (e) LE-4 and FE-9 in Sec. 11.4.1a (Scrubbing System); and (f) DE-1, LE-6 and PE-30 in Sec. 16.4.16 (ARE Charging System).

Fig. 14.2. Main Transmitter Rack and Scrubbing Equipment in the Penthouse.

- b. Instruments not being removed from service resulting in transmitter bellows damage. This occurred with DT-1 (Sec. 16.4.16). Correctly removing an instrument for service required opening the by-pass valve on the "block" before closing the high- and low-pressure purge line valves on the "block". (Then, correctly putting an instrument into service was done by opening the purge line valves before closing the by-pass valve.)

The nitrogen transmitter rack (in Gallery) received nitrogen from line LN-1 and contained the pressure transmitters for instruments FE-1, -3, and -4; and FE-5 and -6 (13). Two additions were made:

- a. FI-33 to provide a continuous purge through line LN-121-1 when FCV-1 was not in use to prevent F_2 from entering the FR-1 pressure transmitter. (This system still required that FCV-1 be opened slightly.)
- b. FI-34 to meter N_2 in the 0-37 liters/min range since the FR-1 range was so great (0-200 liters/min). (After the range of FR-1 was changed to 0-22 SIM, FI-34 was no longer needed.) Aside from the fact that the range of the first FR-1 was too great, all of these instruments were satisfactory.

14.4.6 General Information

a. Specifications and Procurement of Nitrogen

The liquid-pumped nitrogen stocked in cylinders at ORNL carried only the specification "bone-dry". Over a period of several months in 1957, the following data on this nitrogen were collected:

<u>Analytical Laboratory</u>	<u>O_2 (ppm)</u>	<u>H_2O (ppm)</u>
Reed Division (J. Leonard)	20	3
K-25	100	10
Analytical Chemistry Div. (Laing)	175	70
Y-12 Mass Spectrometer	195	50

It was suspected that the higher impurities reported were caused by sample contamination either in collecting or transferring operations. Although no O_2 removal equipment was installed, no difficulties were encountered that could be assigned to O_2 in the N_2 . A dryer and continuous water analyzer were installed, mainly to scavenge water entering the system from an occasional cylinder with high water content or from careless purging during cylinder changing (Secs. 14.4.3 and 14.4.4). The water analyzer readings on N_2 sampled downstream from the drier consistently read <1 ppm H_2O (Sec. 14.4.4).

b. Materials of Construction

Nitrogen seemed to be compatible at the VPP temperatures (-60°C to 750°C) and pressures (maximum 50 psig) with all the materials used in the Volatility Process. No nitriding of metals was observed. It was considered superior to helium and argon for VPP use because of its lower cost and easier containment.

The original screwed fittings in N_2 service (2000 psig forged steel) were difficult to make leak-tight against gas pressure (0-60 psig); so most were back-welded. Copper tubing and Swagelok fittings were found more satisfactory than steel pipe and screwed fittings for N_2 service. They were easier to install and to alter in arrangement and were more leak-tight. Standard flanged joints with Teflon gaskets were originally used in the cells and on the sleeves through cell walls. Most of these flanged joints were eliminated after one was inadvertently left loose, causing a loss of UF_6 from the fluorinator in Run E-2 (22, p. 21); copper tubing with Swagelok and/or silver-brazed fittings were substituted for some steel pipe and flanged joints in cells. Swagelok fittings were easier to make leak-tight than screwed pipe fittings; silver-brazed fittings were used to minimize the number of joints that could be opened by wrenches (45).

The small purge rotameters (150 cc/min capacity), several larger rotameters (2 scfm capacity), and packed valves (Sec. 17.4.1a) were satisfactory in N_2 service. These had pipe fittings where Swagelok fittings were preferred. However, this fact caused no major difficulty. The glass tubes in several rotameters frosted upon accidental contact with F_2 and/or HF caused by operational errors. The rotameters acted as restrictions to N_2 flow and caused the pressure in vessels, dip lines, and piping downstream from them to be several psig lower than the pressure indicated on the N_2 blanket header gauges. This pressure differential increased the difficulty of maintaining penumatic balance in the process system during certain salt transfers. Bypass valves were, therefore, installed around the N_2 blanket rotameters to reduce the pressure drop. Hoke No. 1197 bellows-sealed valves were installed downstream from the rotameters and their bypasses to provide a means of isolating the N_2 system from the process system during leak tests. This was the "C-valve" arrangement shown in line LN-108-2 and in other lines on Dwg. D-31350.

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^a Sec. 19.

Troubles with N_2 valves as delineated elsewhere were:

- a. Check valve near PI-43 was never satisfactory (Sec. 17.4.1).
- b. Safety valves (Sec. 17.4.1) and packed valves (Sec. 17.4.1) were sources of insignificant continuous leaks.
- c. Pressure regulators such as FV-8 were hard to adjust to low gas pressures because the range was too great (Sec. 17.5).

14.5 Summary and Conclusions

Nitrogen having ~10 to 50 ppm of H_2O and ~100 ppm of O_2 was obtained at an initial cylinder pressure of 2,000 psig² in VPP cylinders. The pressure of this gas was reduced to 20 to 30 psig before entering the plant. The nitrogen then flowed through an Oriad type dryer and into the VPP. A continuous water analyzer indicated that the H_2O content was maintained at <1 ppm. The oxygen was not removed, however. This nitrogen was evidently compatible at VPP temperatures (-60°C to 750°C) and pressures (max of 50 psig) with all materials of construction.

The manifold upstream of the dryer containing two 4-cylinder banks of nitrogen was fitted with an automatic switch-over from an empty (<100 >75 psig) bank to a full bank. This manifold was virtually trouble-free. The Oriad type dryer and water analyzer evidently operated satisfactorily.

The nitrogen transmitter rack containing pressure transmitters for FE-1, FE-3, FE-4; PE-5 and PE-6 required only minor design changes such as adding FI-33 and FI-34.

The main transmitter rack containing pressure transmitters, "C-clamp" rotameters, and "block" valves for most of the remaining pneumatic-type instruments was satisfactory. Major troubles arose from F_2 and/or UF_6 backing up to the rack and from improperly removing instruments from service.

Leaking joints comprised the major difficulty with the nitrogen system. To eliminate some leaks the original screwed fittings were back-welded. In other leaking joints, steel pipe and flanges were replaced with copper tubing and Swagelok fittings as for the loose flange through which the UF_6 leak in Run E-2 occurred. Generally, using copper tubing with Swagelok joints eliminated leaks originally occurring in screwed pipe fittings and standard flanges with flat gaskets.

Rotameters were subject to small continuous leaks as well as to some attack by F_2 and UF_6 . These leaks were considered insignificant. After Run E-2, the "C-valve" arrangement was installed to isolate the N_2 system from the cell piping. The check valve near PI-43 was never satisfactory. The safety valves exhibited insignificant continuous leaks as did the packed valves. The pressure reducing valves such as FV-8 were difficult to adjust to low gas pressures.

14.6 Recommendations

It is recommended that:

- a. All N_2 piping in the Gallery downstream from the N_2 dryer be removed and replaced with suitably sized copper tubing containing Swagelok joints except where flow orifices, pressure reducing valves, and other essential devices require pipe. Tubing should be properly supported to avoid sagging and to protect the joints. A centrally located panelboard for mounting purge rotameters should be provided. Access to the rear of this panelboard is required. A rack to support all N_2 station instrument transmitters should be provided.
- b. A means for keeping F_2 out of N_2 lines be provided. The best of these designs should be used: (a) separate N_2 supply for the F_2 station, (b) more reducing stages in a system similar to the existing one, and (c) N_2 - F_2 interlock similar to that described in Sec. 15.4.4.
- c. The by-pass around FI-34 and V-93 be altered to include the by-passing of FCV-1. In addition to FI-33, this by-pass should include a suitable manually operated valve. This arrangement would make it possible to keep N_2 pressure at all times in line LN-121-1, thereby helping to prevent molten salt plugs in the FV-100 F_2 inlet line when HCV-8 is opened while no F_2 is flowing (Sec. 5.6).
- d. Commercial nitrogen be tried as a substitute for "VPP" nitrogen described herein. "Commercial nitrogen" refers to that stocked in ORNL Stores which differed from VPP nitrogen in two ways; (a) not contained in special cylinders and (b) filled without extra precautions.
- e. The pressure reducing valve PV-8 and others as needed be replaced with valves having a lower output pressure range to facilitate setting low line pressures.

14.7 Operating Procedure: Changing Nitrogen Cylinders

Initial
When Complete

- ___ 1. Record whether north ___ or south ___ bank of manifold is being changed.
- ___ 2. Record nitrogen pressure on that bank: high-pressure gage: ___ psig;
low-pressure gage: ___ psig.
- ___ 3. Record cylinder numbers (left to right): VPP ___, ___, ___, ___.
- ___ 4. Close all four cylinder valves.
- ___ 5. Close the four manifold valves connecting to the cylinder pigtails.
- ___ 6. Using line lever (to prevent twisting tubing) and open-end or crescent wrench, disconnect cylinders.
- ___ 7. Put caps on used cylinders and tag each cylinder. Tags should show (1) VPP cylinder number, (2) pressure, (3) date, and (4) operator's name.

8. Move empty cylinders to north end of storage rack and strap in place.
9. Place four full cylinders of nitrogen in manifold rack. Record cylinder numbers: VPP - _____, _____, _____, _____.
10. Open valve supplying purge N_2 to the manifold at 5 psig.
11. Before connecting each cylinder, crack the cylinder valve for an instant to blow dust out of the valve body.
12. Connect each cylinder in turn by first opening the corresponding manifold, and then while the nitrogen flows through the pigtail, connect the tubing to the cylinder.
13. Open all four manifold valves wide after first cracking to equalize pressures.
14. Close valve on purge line, and lower low-pressure regulator to zero.

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15.0 FLUORINE SYSTEM

15.1 Introduction

Fluorine was used in the Volatility Process as an oxidizing agent for converting UF_4 in the molten salt to UF_6 which passed from the salt as a gas subsequently collecting on NaF in the absorbers. Fluorine was also used to desorb UF_6 from NaF in the absorbers and to leak-test joints. The principal steps in operating the fluorine system were:

- a. Maintaining an adequate supply of fluorine in the tank trailers.
- b. Flowing fluorine at controlled flow rates through the HF trap from the trailers into the VPP.
- c. Scrubbing fluorine from the waste gases.

15.2 Equipment

The general arrangement of the fluorine system is shown in Fig. 15.1. Details of the individual components are given in Table 15.1.

15.3 Operation

15.3.1 Operating Procedure

Steps in operating the fluorine system were:^a

- a. Hooking up the F_2 Tank Trailers and flowing F_2 into the VPP.
  1. Connecting the copper pigtailed to the trailer manifolds, taking adequate precautions against residual F_2 which might be released.
  2. Making certain the scrubber is operating and has sufficient KOH to react with the F_2 to be flowed to the Plant (Sec. 11.3.1).
  3. Purging the trailer manifolds and all connecting lines with N_2 .
  4. Shutting off the air to FCV-2; nearly shutting off the air to FCV-1 and starting a slow purge of N_2 through FI-33; and opening V-81. (This isolated the N_2 and F_2 systems and provided a low N_2 purge through FCV-1 to prevent F_2 from entering this valve.)

^aConditioning (Part e) was necessary only in Part d as indicated. In parts a, b, and c, the system was either not opened or opened only momentarily to air while being blanketed with nitrogen, in which case reconditioning was unnecessary.

Fig. 15.1 Equipment Arrangement in the Fluorine System

Table 15.1
List of Fluorine System Components

Component	Drawings and Sketches		Miscellaneous Information	References
	No.	Title		
Tank Trailers, FV-161 and -162	D-15708	Fluorine Transport Tank	Period of Use - All Runs	This Report -
	D-25229	Fluorine Trailer Handling Facilities	Tanks - Block, Sivalle, & Bryson, Oklahoma City, Oklahoma,	Secs. 4.1.1, 15.4.1, item b, 15.4.3, item b;
	D-31349	Fluorine and Nitrogen Flow Sheets	P. O. No. W3X-15821.	15.5, 15.6, 17.4.3;
	Fig. 15.2	Fluorine Tank Trailer Showing Manifold Valves Trailers	- La Crosse Trailer Corp., La Crosse, Wis.;	Page. 15.2, 15.3, 15.4.
	Fig. 15.3	Schematic Diagram for High Fluorine Flow Shut-Off	P. O. No. W3X-15813.	Engineering File Folder No. F-52.
	Fig. 15.4	Schematic Diagram for H_2 - F_2 Interlock	Off Device High F_2 Flow Switch - Used only during "I" runs. FX-900 flow switch supplied by the Goss Co., Newington, Conn., 115 v, 60 cycles, 10-w lamp load.	Other Literature - 52 (pp. 16, 52).
Fluorine Transmitter Rack	Q-1679-18-80	Back Arrangement and Piping F_2 Transmitter Rack	Period of Use - All runs except as indicated	This Report -
	Q-1679-19-80	Front Arrangement and Piping F_2 Transmitter Rack	Back Date and Components - See revision comments (6-10-18) on Log. No. Q-1679-18-80 in Engineering File Folder No. F-53.	Secs. 15.4.2, item a, 15.4.2, item b; 15.4.2, item c; 15.4.2, item d; 15.4.1, item b; 15.4.4;
	Q-1679-35-80	Fluorine Detector	Surge Pot (FV-160) - installed in early work to increase the system surge capacity in an attempt to reduce the fluctuation in PCV-2 and PCV-10.	15.5; 15.6; 17.4.3.
			Trailer Switching Device - Sec. 15.4.2, item a. Remote Manual Shut-Off - Sec. 15.4.2, item b. Flow Orifice Selector Device - FX-2A and -2B metered F_2 flow rate into the plant with PCV-2 controlling this flow rate. Of. also Sec. 15.4.2, item c. Other Instruments - FX-10 metered F_2 pressure into the plant with PCV-10 controlling this pressure. Cf. Also Sec. 15.4.2, item d.	Engineering File Folder No. F-53. Other Literature - 13.
HF Trap and Heater (FV-563), FV-163	D-28763	Sodium Fluoride Trap for HF Removal	Control Equipment - Wheeler (thermocouple welded to external surface of vessel about midway of heater) and Powerstat.	This Report -
	D-28764	Insulation of HF Trap	Period of Use - "E" and "I" Runs.	Secs. 15.4.3, item a; 15.4.3, item b, 15.5; 15.6.
	C-28766	Flowsheet for HF Trap Installation	Trap Packing - Ca. 10 kg. 1/8 in. NaF pellets.	Engineering File Folder No. F-56.
	D-31368	Wiring Diagram (sheet B, item "B")	Heater Data - Levi-Duty Electric Co., Model 5024, serial No. 94143; 5.5 kw, 230 v, 1 phase, 1950°P, X-66998.	Other Literature - 18.
	Sketch No. L-1	HF Trap Piping		
	Report No. CF-57-2-43	Trap Design		
H_2 - F_2 Interlock	D-34087	Wiring Diagram (sheet 1, item "A")	Period of Use - Runs E-3 through -6; "I" Runs. The differential pressure switches were manufactured by Minneapolis-Rosemount Regulator Co., type No. P606A3X1.	This Report -
	Fig. 15.4	Schematic Diagram for Fluorine-Nitrogen Interlock		Secs. 15.4.4, item a, 15.4.4, item b; 15.4.4, item c; 15.4.4, item d; 15.4.2, item b; 15.4.2, item c; 15.5; 15.6; 15.4.7, 15.4.5; 17.4.10.
				Fig. 15.4, 5.2. Engineering File Folder No. F-53. Other Literature - 13.

5. Selecting a trailer to use which has more than 15 psig of F_2 ; otherwise, the automatic switch-over would switch to the other trailer.
  6. Setting the trailer manifold and other valves for the selected trailer. (Assume that the orifice selector device no longer existed since this was the case in the latter part of the VPP work. Otherwise, it would be necessary to select the orifice for use.)
  7. Energizing the high F_2 flow shut-off device, throwing the F_2 manual emergency shut-off switch to "on," and opening PCV-16 and PCV-2. (Usually PCV-10 was set at 4-1/2 psig with PCV-2 being set at the desired flow rate.)
  8. Preparing the other trailer for switch-over by setting the trailer manifold and other valves and energizing the high F_2 flow shut-off device.
  9. (When the pressure in a trailer was reduced to 15 psig, the automatic trailer switching device took it "off-stream" and put the other trailer "on-stream.") Preparing the 15-psig trailer for shipment by closing trailer manifold valves, de-energizing the high F_2 flow shut-off device, purging trailer manifold valves, disconnecting the copper pigtails, capping the tubing connectors on manifold, and closing the trailer doors.
  10. Cutting off F_2 flow as required by shutting off PCV-10, PCV-2, and then throwing the F_2 manual emergency shut-off switch to "off."
  11. In an emergency, throwing the F_2 manual emergency shut-off switch to "off" stopped F_2 flow.
- b. Using the HF trap.
1. Setting the necessary valves.
  2. Maintaining the center of NaF bed at 100-105°C with FV-563.
  3. Adjusting the water supply to trap heat exchanging coil to maintain the bottom of the NaF bed at 25°C (TR-2E-12).
- c. Regenerating the HF Trap

NOTE 1: During operations, the HF trap was regenerated after about every 5 runs. Tests with N_2 -HF mixtures indicated that HF removal was still essentially complete at an HF loading of > 7 lb. on 9.7 kg of NaF pellets as delineated in Sec. 15.4.3.

NOTE 2: Because of the piping arrangement, FV-163 regeneration was done only when F_2 was not being used.

1. Adjusting valves at F_2 Station, in Penthouse, and at Main Panel-board as required.
2. Throwing the F_2 manual emergency shut-off switch to "on" and setting PCV-10² at 4 psig.
3. Adjusting flow rate of the N_2 supplied to F_2 transmitter rack by setting FI-29 at 25%.
4. Setting TIC-2E-6 to obtain a temperature of 350°C at top of NaF bed (TR-2E-1) and noting the time this temperature was attained.
5. Stopping N_2 flow one hour after item d was completed.
6. Reducing NaF bed temperature at the center to 100°C (TR-2E-6) by adjusting TIC-2E-6 and the gas outlet temperature to 25°C (TR-2E-12) by cutting off steam and starting water through heat exchanging coil to prepare for using trap.
7. Setting valves as needed in system.

d. Filling the HF Trap

NOTE: The frequency of trap charging has not been established. Trap Charge = 10 kg of 1/8-in. NaF pellets.

1. Removing top flange, adding the NaF and replacing lid.
2. Leak-testing lid by soap-bubble test with vessel containing about 15 psig of N_2 .
3. Conditioning NaF bed with internal surfaces of trap with F_2 by using F_2 to slowly sweep out the N_2 (Part e).
4. Leak-testing lid joint by KI-starch test with vessel containing F_2 at ~10 psig.^a

e. F_2 Conditioning

1. Making certain that the entire system is clean, leak-tight and at essentially room temperature, that the scrubber has adequate (Sec. 11.3.1) capacity for the F_2 to be flowed and that the instrumentation is in service.
2. Starting the heated duct heaters and blower and the scrubber.

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^aSec. 17.4.2, e.2.

3. Flowing nitrogen through the various N_2 supply headers, instrument purges, and FI-34 at a combined rate of ~ 15 slm (~ 5 through supply headers and ~ 10 through FI-34).
4. Cutting off FI-34 one hour later but keeping N_2 flowing through the supply headers and instrument purges.
5. Turning on F_2 and adjusting the F_2 flow rate to ~ 4 slm (The resulting N_2 - F_2 mixture contained $\sim 50\%$ F_2 .)
6. Then by valving properly, flowing this N_2 - F_2 mixture for periods of a half-hour each through successively greater portions of the system until it was flowing through the entire system. For example, the first part (portion A) included FV-100, -103, -120, and -124; portion B included portion A plus FV-121, -220, and -222; and so on. In this way, each such portion was contacted first with a mixture of very low F_2 concentration which gradually increased as the residual N_2 was swept out, and which finally became $\sim 50\%$ in F_2 .
7. Next, flowing the N_2 - F_2 mixture through the entire system for 4 hours or less depending on the judgment of the supervisor.
8. Then, turning off the F_2 and again flowing ~ 15 slm of N_2 for 20 minutes.
9. Finally, turning off FI-34 and leaving the header and instrument purges on until the next operation.

Minor changes were made in the operating procedures from time to time, and the necessary records were kept. The actual procedures used are shown as Secs. 15.7.1, 15.7.2, 15.7.3, and 15.7.4.^a The procedure used for starting F_2 flow was incorporated in other procedures such as Secs. 5.7 and 8.7.

15.3.2 Critical Operating Steps

- a. Exercising extreme care at all times when working around F_2 , especially while making or breaking connections. (All lines were purged with N_2 before breaking a connection if there was any doubt as to whether this had already been done.)
- b. Assuring that the scrubber was operating and had required scrubbing capacity before starting F_2 flow.
- c. Throwing the F_2 manual emergency shut-off switch in Penthouse, at F_2 Station,² or at Main Panelboard in an emergency.

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^aSec. 15.7.1 was the original runsheet prepared for changing fluorine tanks. Later the procedure for the purging phase of Tank No. 1 was prepared which is shown here as Sec. 15.7.2. A corresponding runsheet for purging Tank No. 2 was never made.

- d. Keeping the gas exit bed temperature of FV-163 at 25°C and central bed temperature at 100°C.
- e. Using the sense of smell to establish the presence of F_2 in an area and being careful about entering such an area unless the true situation was known.
- f. Using acetone around F_2 -handling equipment cautiously (Sec. 15.4.5b).

15.4 Equipment Evaluation^a

15.4.1 Tank Trailers, FV-161 and -162

a. General Information (44, 46)

Gaseous fluorine was obtained from the K-25 fluorine plant in steel tank trailers at ~ 55 psig. Fig. 15.2 is a photograph of the two trailers with doors opened showing manifold valves. Each trailer held 650 standard cubic feet of gas.^b The K-25 typical composition was 90-98% F_2 (usually 95%), 3-8% HF (usually 5%), and 1-2% N_2 and/or O_2 as determined from a flowing stream sample taken once per day.² No difficulty in the VPP was experienced that could be directly assigned to impurities in the fluorine, although the HF content was believed troublesome in FV-103 (Sec. 6.4.1b), -120, and -121 (Sec. 8.4.1b). The HF was removed with an NaF trap installed prior to the "E" runs (Sec. 15.4.3).

The VPP Fluorine Station was located outdoors on the south side of Building 3019. The two fluorine tank trailer bodies were not protected from weather although the tank valves and connections were housed in a cabinet on the end of the trailer. The F_2 control instruments, valves, and fittings were installed under the Sample Gallery, which afforded some protection from rain. The main difficulty encountered with this system because of weather was the freezing of steam and water lines to the HF trap during one week of severe cold in February, 1958.

The tank trailers were backed into position and parked with their rear wheels against a curb stop and their small front parking wheels resting in steel chocks partially buried in the asphalt pavement. Painted lines on the pavement aided the truck driver

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^aAfter the "L" runs, the equipment downstream of the fluorine transmitter rack was dismantled and moved to Burial Ground No. 3 (Sec. 23.4.16b). The remaining equipment was retained for future processing.

^bTrailer volume is 137 cu ft (R. P. Milford, personal communication) standard conditions: 14.7 psia and 70°F; average pressure and temperature of trailer = 55 psig and 70°F.

Fig. 15.2 Fluorine Tank Trailers Showing Manifold Valves.

in positioning the trailers. No difficulty was experienced in handling the tank trailers. Copper pigtails made from 3/8-in. o.d. tubing formed flexible connections from the tank trailers to the VPP Fluorine Station; a similar arrangement was used at K-25 fluorine filling station (Sec. 15.4.5b). Two copper pigtails, one for the F_2 flow to the process and one for venting the trailers and piping to the F_2 disposal system, were provided for each trailer at the VPP Fluorine Station. Flare-type fittings with freshly annealed inserts were used for connecting the pigtails to the trailers. After a near-accident because of a worn flare fitting, these fittings and the copper tubing were frequently inspected and replaced every four months during operations. In September, 1957, the trailers were repainted because the original paint had partly deteriorated. At that time, the original paint was about 3 years old.

b. Automatic High F_2 Flow Shut-Off

An arrangement actuated by high F_2 flow from the F_2 trailers was installed after approximately half a trailer of F_2 was discharged to the atmosphere because a Teflon gasket burned on a temporary HF trap near the F_2 trailer (Sec. 15.4.6; 47). Since the emergency remote manual shut-off (Sec. 15.4.2b) depended on PCV-2A and PCV-2B which are located a considerable distance downstream from the F_2 trailers, no remote or automatic means of shutting off F_2 at the trailers existed at that time. Some difficulty was encountered in designing such an automatic high F_2 flow shut-off that met these K-25 Safety Department and the K-25 Fluorine Plant specifications: (a) no alteration of trailer valves and piping was permitted and (b) no change could be made that would affect the K-25 trailer filling procedure.

The automatic high F_2 flow shut-off, mounted on both F_2 trailers, is illustrated by Fig. 15.3. Flow switches FX-37 and FX-38 actuated F_2 shut-off valves FCV-37A and FCV-38A whenever the flow through the switches exceeded 2 cfm. The shut-off was accomplished by means of relays and solenoid valves in the air supply to the shut-off valves. The entire installation was added to the existing piping on the F_2 trailers without modifications to the piping or to the K-25 filling procedure. The electrical circuit for the remote manual shut-off system was extended to serve the automatic safety system.

This F_2 safety system worked satisfactorily. Although no emergency arose to actuate the system, its functioning was demonstrated every time F_2 flow was started into the empty F_2 piping, as the flow would exceed the 2 cfm setting of the flow switch, causing it to shut off the F_2 flow. To avoid having an operator hold the re-set button during F_2 start-up, the flow switch was manually bypassed until sufficient back pressure was built up in the piping down-stream from the switches to reduce the F_2 flow to normal. In subsequent operations, the change in-

Fig. 15.3 Fluorine Tank Trailer, FV-161, Gas Manifold Showing High F₂ Flow Shut-off Equipment.

licated at the reset button on Dwg. No. Q-2052-50-R0 will eliminate such flow interruptions. The reset button will be held in manually at the trailer until the pressures equalize, and until the F_2 flow rate is too low to actuate the high F_2 flow shut-off. Then, the desired F_2 flow can be set.

One flow switch (FX-37) developed a leak through its setting adjustment and had to be replaced after several months' service. The Teflon gaskets on both switches tended to creep loose and had to be retained in a special recessed fitting. One gasket was destroyed by F_2 after it had worked loose as mentioned in Sec. 17.4.1. Both switches were subject to the spindle sticking in the "open-circuit" position. The remedy for this was sharply rapping the switch with a small tool.

15.4.2 Fluorine Transmitter Rack^a

a. Trailer Switching Device

The F_2 trailer switching device automatically changed the F_2 feed from a nearly depleted tank trailer to a full trailer when the pressure fell to 15 psig. This was accomplished by pressure switches PX-2C-2 and PX-2C-1, relays and solenoid valves PCV-2C-1 and PCV-2C-2 in the air supply to air-operated F_2 supply valves PCV-2A and PCV-2B. This device was satisfactory, although later control circuit modifications or additions to the F_2 system apparently caused the switch-over to be actuated unnecessarily. The reason for this was obscure, and therefore, needed further study. The trailer switching almost always upset the PCV-2 and PCV-10 combination. Such fluctuations sometimes continued for as much as one-half hour and required close attention to eliminate (Sec. 15.4.2d and 17.4.1).

b. Remote Manual Shut-Off^b

Three hand-operated switches (HX's) were provided for emergency remote manual shut-off of F_2 . One switch was mounted on the Main Instrument Panelboard, one in the Penthouse near the main Instrument Transmitter Rack, and one at the F_2 Station. These switches were wired in series so that all had to be "on" to flow F_2 to the process. The switches actuated solenoid valves PCV-2C-1 and PCV-2C-2 in the air supply lines to F_2 shut-off valves PCV-2A and PCV-2B. This arrangement was satisfactory, and the switches were used for convenience in addition to emergency.

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^aSec. 14.4.5.

^bSec. 15.4.1b for automatic high F_2 flow shut-off.

c. Orifice-Selector Device

Originally a device was installed at the F_2 station to allow fluorine flow meter FE-2 to operate on two ranges of flow, a low range of 0-2 scfm at 10 psig and a high range of 0-10 scfm at 10 psig. Two sets of orifice flanges (FE-2A for low range and FE-2B for high range) and an air-operated shut-off valve for each (HCV-1B-1 and HCV-1B-2) were installed in the main F_2 supply header at the F_2 station. A switch (HX-1) on the Main Panelboard actuated 3-way solenoid valve HCV-1C in the air supply to HCV-1B-1 and HCV-1B-2, opening one and closing the other to select which orifice to flow F_2 through. Simultaneously HX-1 actuated 3-way solenoid valve HCV-1A, whose position determined which signal (that for FE-2A or FE-2B transmitter) was sent to flow recorder FR-2 so that the flow was properly recorded.

The high-range orifice was not needed because a much lower F_2 flow rate was used than was first anticipated. A new low-range orifice was substituted for the high-range orifice in the FE-2B flanges. It was later discovered, however, that HCV-1B-1 allowed F_2 to leak through its seat and through FE-2A so that more F_2 reached the process than FR-2 recorded from FE-2B. A blank installed in the FE-2A flanges corrected this difficulty but trapped F_2 that leaked through HCV-1B-1, interfering with the N_2 - F_2 interlock operation (Sec. 15.4.4). The situation was finally corrected by providing separate, temporary air supply lines to the orifice selector valves to keep HCV-1B-2 open and HCV-1B-1 closed at all times. The air outlet from solenoid valve HCV-1C was capped and HX-1 was removed from the Main Panelboard.

d. Other Instruments

All purge-type instruments depending on N_2 bleeding into the F_2 lines were changed because F_2 diffused or surged back into the instrument transmitters, defeating the purpose of the N_2 purge. Transmitters capable of being exposed to F_2 were substituted for the originals (FT-2A, FT-2B, PT-10, and PT-11), and proved satisfactory. A number of instruments (PE-54, PE-55, PX-2A, PX-2B, etc.) were exposed to F_2 internally at 60 psig, without incident. It was considered hazardous design to mount PX-2A and PX-2B in the "Black Box" control center on the fluorine transmitter rack with all the electrical wiring exposed to F_2 in the event one of the piping connections leaked. No harm came from this, however.

The instruments tended to cycle because of pressure surges in F_2 piping, which seemed to have too small a capacity. Installation of surge pot FV-160 (2-1/2 in.-NPS pipe, 6-ft long) improved this situation, but a larger surge capacity was still needed.

e. Fluorine Detection^a

The utility of this instrument was reduced by:

1. The difficulty of determining which of the three sampling points (Penthouse, Cell 1, and Cell 2) actuated the alarm at the Main Panelboard. Since the signal from each of the sampling points was sent to the Main Panelboard through a common circuit, the source of the high sample could not be determined at the Main Panelboard. This necessitated going to the Penthouse. Even then, positive proof could not be established because the instrument had already switched to another sampling point (The instrument remained on a sampling point for two minutes before switching to the next.). In some cases, such follow-up consumed considerable time, especially when the sensing element was "poisoned."
2. The ease with which the sensing element became "poisoned." Because of the high sensitivity of the sensing element, considerable recovery time was required after receiving a concentrated sample of fluorine.

15.4.3 FV-163, HF Trap and Heater (FV-563)^b

a. General Information

The HF trap and its furnace operated satisfactorily. The only difficulty arose when the steam and water supply lines to the trap burst during a period of severe cold weather. Before the trap was installed, UF_6 broke through the absorber beds prematurely. Calculations indicated that the 3% HF in the F_2 supply from K-25 would cause this UF_6 break-through (Sec. 15.4.1b), this fact initiating the design and installation of the HF trap. After the trap was installed, no UF_6 break-through was experienced, possibly indicating successful HF removal by the trap. The trap was packed with ca. 10 kg of 1/8-in. NaF pellets.

b. HF Removal by HF Trap

During the last "L" runs, attempts were made to determine the amount of HF in the fluorine entering the VPP. The fluorine both upstream and downstream of FV-163 was passed through a copper coil chilled with a dry ice-trichloroethylene mixture. The material collected in the coil was subsequently titrated with a standardized KOH solution. The data were erratic presumably because of the large volume of solution required for rinsing the coil, and because of the uncertainty of whether HF was lost while

^aThe fluorine detector was located at the main transmitter rack in the penthouse.

^bTrap design (48, 29, p. 9).

rinsing. Both weight change and titrating data indicated that some HF had been collected. After the "I" runs, this work was continued using N_2 containing up to 10 volume % HF. HF concentration in the gas streams entering and leaving FV-163 were determined in equipment similar to that used in the Reich test for air- SO_2 mixtures.^a The data showed that:

1. A negligible HF concentration ($< 0.002\%$) downstream of the trap could be obtained under these conditions:
  - (a) Inlet gas HF concentration - 2 to 10%
  - (b) Gas flow rate - 15 liters/min
  - (c) Bed temperature near trap exit (TI-5-19) - varied from 22° to $70^\circ C$.
  - (d) Bed temperature near center of bed (TR-2E-6) - $\sim 100^\circ C$.
  - (e) HF loading of bed - 7 lb of HF on 9.7 kg of NaF pellets (determined by weight differences of HF cylinders).^b
2. An HF concentration of $\sim 0.045\%$ could be obtained under the same conditions except that the bed temperature near exit was held at $150^\circ C$. Thus, the temperature dependence of the HF concentration as well as the negligible HF concentration attainable were demonstrated.

Subsequently the trap was opened to inspect the bed, and this inspection was followed by attempts to regenerate and then a final inspection. The pre-regeneration inspection revealed that the top thermowell was firmly held in place by the bed. After the lid was finally removed, the upper part of the bed was a solid cake containing blow-holes and cracks and evolving HF, indicating it to contain polyfluorides. The trap was then covered with a blank flange, and regeneration was started. The regenerating period was October 31 - November 7, 1958 under these conditions:

1. N_2 flow rate - 15 slm
2. Bed temperature near center of bed - varied from $120 - 320^\circ C$.
3. Exit gases HF concentration - varied from 12 to $2-1/2\%$.

NOTE: Trap was plugged, presumably with polyfluorides, for about one-fifth of regeneration period.

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^a Instead of the standardized iodine solution with a starch indicator for SO_2 , a standardized KOH solution with a phenolphthalein indicator was used.

^b The bed actually contained somewhat more than 7 lb of HF because it had been used prior to this test-work.

Since the HF concentration of the exit gases was 2-1/2% after the week's regeneration period, all of the HF was not removed. The final bed inspection revealed that:

1. The top one-foot of material was a solid mass in which the individual pellets had lost their identities.^a
2. The remainder of bed contained individually identifiable pellets sticking together but easily broken up and some fines.

15.4.4 N₂-F₂ Interlock^b

a. Purpose

The purpose was to prevent entry of F₂ into N₂ lines (N₂ blanket system and instrument N₂ supply) by shutting off F₂ flow:

1. When F₂ (or UF₆) pressure rose to within 0.5 psi of N₂ pressure.
2. When pressure in the fluorinator exceeded a set value (usually 4.5 psig).

b. Components (Fig. 15.4)^c

1. Pressure switch PX-33B, mounted behind the main instrument panelboard and connected to FV-100 vapor space through PE-33, set for actuation at 4.5 psig.
2. Differential pressure switch PX-57, mounted behind the Main Panelboard, its high-pressure side connected to N₂ supply header LN-121-1 (to the fluorinator and absorbers) through PE-6; its low-pressure side connected to F₂ supply header X-160-1 through PE-10 (set for 0.5 psi differential pressure).
3. Differential pressure switches PX-58, PX-59, and PX-60, mounted in the gallery, their high-pressure sides, respectively, connected to N₂ blanket headers LN-112-1 and LN-4 and N₂ supply header LN-MTR-1 (to the Main Instrument Transmitter Rack, Sec. 14.4.5); their low-pressure sides all connected

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^aThe solid mass evidently resulted from incomplete regeneration of the top of the bed because of the low temperature in the top one foot of bed.

^bSec. 14.4.5.

^cSec. 5.4.7.

Fig. 15.4. Schematic Diagram for N_2 - F_2 Interlock

to the F_2 supply header X-121-1 through V-128 in the N_2 line LN-F21-1 that connects to the F_2 supply (set for 0.5 psi differential pressure).

4. Fluorine shut-off valves PCV-2A and PCV-2B (air-to-open, Kel-F disk, bellows-sealed), mounted at the F_2 station.
5. Air supply shut-off valve PCV-57 (3-way solenoid, power-to-open mounted on the F_2 transmitter rack.
6. Pressure alarm PA-57 (activated by open relay circuit), mounted on panelboard.
7. Relays for pressure switches and alarm (power-to-close).

c. Operation

When the N_2 pressure on any of the pressure switches fell to no more than 0.5 psi above the F_2 pressure on the switch, current flowed to a relay, which opened the circuit to PCV-57, shutting off air to PCV-2A and PCV-2B, closing them, and shutting off F_2 flow. The alarm sounded simultaneously.

d. Discussion

The N_2 - F_2 interlock was installed after F_2 had entered the N_2 system several times through vessels and pipes having connections to both gas systems. Instrument purge rotameters and plastic tubes were damaged by F_2 on these occasions. The interlock was effective in preventing recurrence of such incidents. But it could not prevent F_2 at full trailer pressure (55 psig) from entering the entire N_2 system through a N_2 purge valve (V-48 at the F_2 trailers) that once was erroneously left open. A check valve in this N_2 purge line failed to keep the F_2 from backing into the N_2 supply (Sec. 17.4.1).

The interlock required that the blanket N_2 pressure be raised from the 4.5 psig previously maintained to 8-12 psig. This was desirable from the standpoint of protecting the N_2 system from F_2 but undesirable in that gas pressures needed to be kept as low as possible to minimize surges, which tended to force molten salt into unheated areas (e.g., the dip-leg in the fluorinator, Sec. 5.4.7).

The installation of the interlock proved to be awkward, although it pointed up changes needed in the F_2 system.^a As an example, start-up of F_2 flow or switching of F_2 trailers was diffi-

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^aSecs. 15.4.2b and 15.4.2c.

cult because the interlock frequently shut off the F_2 flow at these times. The reason was found to be a F_2 pressure surge caused by PCV-2 shutting suddenly to reduce F_2 flow, that tended to be high (i.e., >2 cfm) when the F_2 was admitted to the empty piping. The surge raised the F_2 pressure above the N_2 pressure, properly actuating the interlock to shut off F_2 flow. The difficulty arose because PCV-57 was installed to shut off air to the flow meter (FR-2) orifice selector valves (HCV-1B-1 and HCV-1B-2) as well as to the F_2 shut-off valves PCV-2A and PCV-2B. This arrangement caused a reversal in the setting of the orifice-selector valves, causing F_2 pressure to be trapped against the blanked low-flow orifice (FE-2A). The F_2 pressure could then be relieved only by leakage or by releasing F_2 through the F_2 Station vent. This situation was corrected temporarily by providing independent air supplies to the orifice-selector valves to hold HCV-1B-1 closed and HCV-1B-2 open all the time (see Dwg. No. Q-1679-19-RQ revised to June 10, 1958, in Engineering File Folder No. F-53).

The operating principle and the basic need for the N_2 - F_2 interlock are valid. However, a study of it is needed to improve its performance.

15.4.5 Materials of Construction

a. Valves and Piping (49)

Piping as originally specified was satisfactory. Teflon-packed hand valves became very difficult to operate after brief F_2 service. Packless valves proved satisfactory as follows.

<u>Valve Types</u>	<u>Sizes</u>	<u>Duty</u>
Crane Co., types SMMD, SSD, and SMD	1/4-in., 1/2-in., and 1-1/2 in. NPS	F_2 supply and vent piping, scrubber F_2 inlet, vacuum pump piping, etc.
Hoke, Inc., types 411, 413 and 415	1/4-in. and 3/8-in. o.d. tube	F_2 sampling, F_2 inlet to instrument transmitters, N_2 supply to molten salt sampler, shut- off between process and instruments.
Hoke, Inc., types 480 (HGP) series	1/4-in. and 3/8-in. o.d. tube.	F_2 sampling, N_2 supply to ring- joint leak test system, HF sampling from F_2 supply.

^aSee Sec. 15.4.2c.

^bSee Sec. 17.4.1 for more information.

Valve Types	Sizes	Duty
Hoke, Inc., types 440 series (1197)	1/4-in. o.d. tube	Shut-off valves between process and N_2 system, F_2 inlet to instruments.

A standard brass swing-check valve in the N_2 purge line to the F_2 station failed to prevent back-flow of F_2 into the N_2 system on one occasion when the SMD valve (V-48) was erroneously left open. The check valve was not examined to determine why it failed to operate, but it is suspected that this type of valve is generally unsuitable for gas service and especially F_2 service (Sec. 17.4.1).

b. Other Materials

Monel, nickel, Inconel, steel, stainless steel, (types 316 and 347), brass, and copper gave satisfactory service in contact with F_2 below $150^\circ C$. Nickel and Inconel only were exposed to F_2 above $150^\circ C$. At elevated temperatures, widely varying amounts of attack occurred on the component materials in the VPP by process gases (7). Specific troubles occurring were:

1. A cast brass tee brazed to a trailer and a cast brass coupling on FV-222 (Sec. 9.4.4) cracked. Because of this, most cast brass fittings were replaced by Swagelok fittings of brass, Monel, or stainless steel (type 316) bar stock, which were suitable for tubing connections in F_2 service except in places where the K-25 use of flare fittings (on tank trailers and UF_6 sample lines) required flares at the VPP. On the tank trailer pigtailed, freshly annealed copper inserts were used when remaking the flare joints to ensure leak-tightness.
2. Brass Swagelok fittings on a Hoke No. 413 valve cracked after about six weeks exposure to F_2 and UF_6 .

A variety of joints were used successfully:

1. Tubing - Swagelok, silver brazed, and flare with the first two preferred.
2. Pipe and vessels - Welds^b and ring-joint flanges with annealed copper rings.

^aSec. 17.4.1, (50, p. 5 ; 7).

^bPer specifications in (51). Also see (52-pp.42-44).

Teflon and Kel-F were partly satisfactory for valve disks and gaskets where soft materials were desirable. The disadvantages of these plastics were their tendencies:

1. To creep or cold-flow under pressure, which resulted in gaskets and washers becoming loose after several weeks of service (several incidents).
2. To burn to a fine ash if the slightest amount of grease (e.g., a fingerprint) was left on them (Sec. 15.4.1b; 17.4.2, f.4; and 17.4.3).

These difficulties were surmounted by designing confining chambers in flanges to prevent gasket creep and by maintaining scrupulous cleanliness on Teflon and Kel-F surfaces to avoid fire danger.^a

All metals, Teflon, and Kel-F subjected to F_2 were found to be more resistant to F_2 attack if they had been "conditioned" by exposure to gradually increasing concentrations of F_2 [Secs. 17.4.2, f.3; and 17.4.2, f.4 (37)]. Before conditioning, all surfaces (metal and nonmetal) to be exposed to F_2 were washed carefully with acetone or other solvent and dried. Incidents occurring while using acetone were:

1. F_2 contacted liquid acetone which had not completely evaporated from a new installation of copper tubing because of severe cold weather. The copper tubing became red hot, and a deposit of soot was spread through the F_2 station piping.
2. Residual F_2 on a metallic surface ignited an acetone-soaked cloth being used to wipe the surface.

Methylene chloride, although not as widely used as acetone, had two advantages over acetone: (a) less susceptible to fire danger from F_2 and (b) lower boiling point (40°C to 58°C for acetone). Although insufficient work with methylene chloride was done to prove that it would not initiate a fire with F_2 , no case is known where such a fire occurred. Danger from using methylene chloride in poor ventilation might occur because of its narcotic nature (mpc = 220 ppm of air) (53). Trichloroethylene has also been used although its boiling point is high (87°C).

VPP practices required that the following be "conditioned" before use with F_2 and/or UF_6 :

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^aFor preconditioning Kel-F disks for HCV's, see Sec. 17.4.2, f.4. Also see below for conditioning of other materials.

1. New equipment.
2. Used equipment.
  - (a) After performing maintenance or operational steps such as recharging with NaF.
  - (b) After being washed out with water or aqueous solutions. Drying out of water was done with the VPP heating equipment.
  - (c) After being opened to the air for more than about an hour.

Troubles occurring which were believed to be associated with poor cleanliness control and/or insufficient conditioning were:

1. Disintegration of Kel-F and Teflon valve parts (Sec. 17.4.2, f.3 and 17.4.3).
2. Destruction of a Teflon gasket (Sec. 15.4.1b).
3. Disintegration of an absorber diffuser ring (Sec. 8.4.1b). The diffuser ring in FV-120 apparently failed when F_2 was suddenly admitted to the vessel, which was being held at $600^\circ C$. The gas distribution ring of 1/8-in. thick Inconel melted over 5 inches of its length. Because the vessel had recently been opened and exposed to the atmosphere, it was suspected that some impurity, probably grease, on the gas distribution ring started the reaction with F_2 . The sudden exposure to F_2 was also thought to have contributed to the failure, as no failure in other instances occurred at $600^\circ C$ upon gradual increase of F_2 concentration to a maximum of 100%.

15.5 Summary and Conclusions

In general, the entire F_2 system worked satisfactorily. Most difficulties that arose were caused by hurried installation and were corrected. No trouble except perhaps from the HF content could be directly assigned to impurities in the F_2 .

Fluorine was received in steel tank trailers filled to ~ 55 psig at the K-25 generating plant. The typical F_2 analysis received from K-25 was: 95% F_2 , < 5% HF, and 1 to 2% N_2 and/or O_2 .

The F_2 station was outdoors on the south side of Building 3019. The two trailers were unprotected from the weather, but tank valves, F_2 control instruments, valves, HF trap, piping and fittings were under the Sample Gallery which afforded some protection from the rain. The only trouble with the weather arose from the freezing of water and steam lines of the HF trap during the severe cold in February, 1958. The trailers were coupled to the Fluorine Transmitter Rack through copper pigtaills.

The trailers were repainted in September, 1957 at which time the original paint was 3 years old.

An automatic high F_2 flow shut-off device was installed after about half a trailer was accidentally discharged to the atmosphere. Although no subsequent emergency arose to actuate this device, high F_2 flows at times when starting F_2 flow proved its efficacy. Several minor troubles occurring with this device are delineated.

The automatic trailer switching device was satisfactory. The difficulties met with it are discussed. One trouble was the cycling of the PCV-2 and PCV-10 combination following trailer switching. Subsequent addition of surge pot FV-160 improved this situation very little.

The remote manual F_2 emergency shut-off which enabled cutting off F_2 at the Fluorine Transmitter Rack, in the Penthouse, or at the Main Panelboard worked satisfactorily.

The orifice-selector device was not needed because a much lower F_2 flow rate was used than anticipated. Later, it was necessary to blank off the valve not in use (HCV-1B-1) because it leaked.

The transmitters on all purge-type instruments depending on nitrogen bleeding into F_2 lines had to be replaced with transmitters capable of F_2 service.

Fluorine detection was hampered by: (a) the difficulty of determining the source of high F_2 and (b) poisoning of the sensing element.

The HF trap and its furnace operated satisfactorily. Its installation apparently prevented UF_6 from prematurely "breaking-through" the absorber beds. Attempts to establish the efficiency of the trap by freezing out HF in the F_2 both upstream and downstream of FV-163 produced erratic results. Later, using N_2 -HF mixtures, the HF concentration downstream of the trap was essentially nil at a 15 liters/min flow rate of the N_2 -HF mixture containing 2 to 10% HF. During this work, the 9.7 kg NaF pellet charge containing >7 lb of HF was held at an exit temperature $\sim 60^\circ C$. Subsequent bed inspection revealed much sintering of pellets and probable polyfluoride formation.

Regeneration of this bed showed that after ~ 6 days the exit stream still contained 2-1/2% HF. Final bed inspection indicated some pellet agglomeration.

Data indicated that even a heavily loaded (7 lb HF in ~ 10 kg of NaF) HF trap will exhaust a gas of negligible HF concentration, and that such a heavily loaded trap required a long period for complete regeneration. In addition, the dependence of the exit gas HF concentration on bed exit temperature was shown.

After installing the N_2 - F_2 interlock, the number of purge rotameters and plastic tubing damages with F_2 were reduced. Although this advantage and the pointing up of changes needed accrued, the interlock was awkward. It required raising the N_2 blanket pressure from 4-1/2 to 8-12 psig which increased molten salt plugs in line X-100-1 to the fluorinator. In addition, starting F_2 flow or switching of trailers was difficult because the interlock frequently shut off F_2 at these times.

The valves suitable for F_2 were Crane's SMD, SSD, and SMD; and Hoke's Nos. 411, 413, 415, 480 (HQP), and 446 (No. 1197). The check valve near PI-43 was unsatisfactory. Piping as originally specified was satisfactory. Other materials of construction included Monel, nickel, Inconel, steel, stainless steel types 316 and 347, brass and copper below 150°C. Nickel and Inconel only were exposed to F_2 above 150°C. Corrosion of these metals occurred above 400°C as probably experienced for Inconel with the FV-120 diffuser ring at 600°C. All metals and Kel-F were more resistant to F_2 attack if previously "conditioned" by exposure to gradually increasing concentrations of F_2 .

Several failures of brass parts in F_2 occurred: (a) a cast brass tee brazed to a trailer flange and a cast brass coupling on FV-222 by cracking after several weeks of F_2 service and (b) Swagelok fittings on a valve by cracking after six weeks' exposure to F_2 and UF_6 .

Suitable joints for F_2 service were: (a) tubing - Swagelok, silver brazed, and flare; (b) piping and vessels - welds and ring-joint flanges with annealed copper rings.

Teflon and Kel-F were partly satisfactory for valve disks and gaskets where soft materials were desirable. Disadvantages were: (a) cold flow under pressure and (b) burning when not scrupulously clean. The effect of cold-flowing was reduced by confining in metallic framework. The fire hazard was diminished by cleanliness and conditioning.

Cleaning agents used on both plastics and metals were acetone, methylene chloride, and trichloroethylene. Of these, methylene chloride was safest although it is a narcotic. Acetone was a fire hazard, and the high boiling point of trichloroethylene made it unattractive.

15.6 Recommendations

It is recommended that:

- a. The F_2 station piping be altered to remove unused sections (FE-2A, HCV-1B-1, HCV-1C and HCV-1A as shown on Dwg. Q-1679-18-RO comments in VPP Engineering File Folder No. 53) and to make permanent the temporary air supply line to HCV-1B-2; a larger surge capacity be built into the piping.
- b. A study of the N_2 - F_2 interlock be made to retain its advantages but remove its disadvantages. See recommendations j. and k. in Sec. 5.6.
- c. A study be made to determine the reason for the unnecessary actuation of the trailer switch-over at times.
- d. Two N_2 cylinders with pressure reducers be provided at the F_2 station for purging the piping (The original design provided this arrangement.) instead of the existing connection to the N_2 blanket system to eliminate the greatest danger of backing F_2 into the N_2 blanket system; a low pressure switch-over and alarm device be provided on these N_2 cylinders to assure an adequate supply at all times; a restriction in the N_2 line be provided to prevent blasting N_2 through the F_2 system

during purging. (A back pressure on the fluorinator was sometimes observed during F_2 station purging, caused by excessive quantities of N_2 , which flowed through V-68, V-72, FV-124 and all Cell 2 piping to FV-160.)

- e. The thermocouple wells in the FV-163 top flange be moved or an NaF fill line be added to facilitate NaF charging and discharging.
- f. Brass be re-evaluated as a material of construction for F_2 service.
- g. More work be done on FV-163 to determine maximum HF loading of NaF, the permissible exit bed temperatures, and efficiency using F_2 -HF mixtures of about 5% HF. (Until this work is done, the bed loading should not exceed 7 lb of HF and the exit gas bed temperature be kept $< 70^\circ C$.)
- h. The F_2 trailers be painted about every 3 years or more often if needed.

15.7 Appendix

15.7.1 Operating Procedure: Changing Fluorine Tanks

CFT _____

When F_2 low-pressure alarm sounds, do the following:

Removal Operation

- 1. Open valves 32 _____, 33 _____, 40 _____, and 41 _____ at fluorine supply tanks. This will give readings on pressure gages, PI-2A and PI-2B.
- 2. Record PI-2A _____ and PI-2B _____ and the time _____.
- 3. The lower reading on the two gages indicates which tank to remove from the system. Record whether #1 or #2 tank reads the lower pressure _____.
- 4. Close valves 32 _____, 33 _____, 40 _____, and 41 _____.
- 5. Check to be sure valve 67, near vent header on the roof, is open _____.
- 6. If tank #1 is to be removed from the system start with step 7; omit steps 12-16 inclusive. If tank #2 is to be removed from the system start with step 12; omit steps 7-11, inclusive.
- 7. For tank #1, close valves 34 _____ and 35 _____.
- 8. Open valves 37 _____, 38 _____, 39 _____, and 84 _____.
- 9. Adjust PV-40 regulator to read 60 psig.
- 10. Open valve 48 for 1 second, close valve 48, reopen for another second, and close. (This is more effective than one opening for 2 seconds.)
- 11. Close valves 37 _____, 38 _____, 39 _____, and 84 _____.
- 12. For tank #2, close valves 42 _____, and 43 _____.
- 13. Open valves 45 _____, 46 _____, 47 _____, and 85 _____.
- 14. Adjust PV-40 regulator to read 60 psig.
- 15. Open valve 49 for 1 second, close valve 49, reopen for another second, and close. (This is more effective than one opening for 2 seconds).
- 16. Close valves 45 _____, 46 _____, 47 _____, and 85 _____.
- 17. Disconnect F_2 tank from header and notify transportation for removal if they are not on a routine schedule.

Reinstallation Operation

18. When replacement trailer arrives from K-25 connect it into the F₂ header.
19. If #1 tank is the unit being connected to the system, start with step 21; omit steps 29-37, inclusive. If #2 tank is the unit being connected to the system start with step 29; omit steps 21-28 inclusive.
20. To install #1 tank, open valves 37 _____, 38 _____, 39 _____, and 84 _____.
21. Adjust PV-40 regulator to read 60 psig.
22. Open valve 48 for 1 second, close valve 48, reopen for another second, and close.
23. Close valves 37 _____, 38 _____, and 84 _____.
24. Check to be sure valve 36 is closed.
25. Open valves 34 _____, 35 _____, 32 _____, and 33 _____.
26. Record PI-2A reading _____.
27. Close valves 32 _____, and 33 _____.
28. Annunciator panel light for "low fluorine pressure" should be off now that the first unit is on "standby". If not, check valving on drawing D-22759.
29. To install #2 tank, open valves 45 _____, 46 _____, 47 _____, and 85 _____.
30. Adjust PV-40 regulator to read 60 psig.
31. Open valve 49 for 1 second, close valve 49, reopen for another second, and close.
32. Close valves 45 _____, 46 _____, and 85 _____.
33. Check to be sure valve 44 is closed.
34. Open valves 40 _____, 41 _____, 42 _____, and 43 _____.
35. Record PI-2B reading _____.
36. Close valves 40 _____ and 41 _____.
37. Annunciator panel light for "low fluorine pressure" should be off now that the second unit is on standby. If not, check valving on drawing D-22759.

15.7.2 Operating Procedure: Purging Fluorine Line # 1

PFL _____

1. Check to be sure V-67, near vent header on roof is open _____.
2. Open V-77 _____.
3. Open V-32 _____, V-33 _____.
4. Record PI-2A _____ and the time _____.
5. Close V-32 _____ and V-33 _____.
6. Close V-34 _____, V-35 _____, and V-36 _____.
7. Open V-37 _____, V-38 _____, V-39 _____, and V-84 _____.
8. Open V-48 for 1 second, close V-48, reopen for another second, and close. (This is more effective than one opening for two seconds.)
9. Open V-35 _____ and V-36 _____.
10. Repeat step 8.
11. Close V-35 _____, V-36 _____, V-37 _____, V-38 _____, V-39 _____ and V-84 _____.

15.7.3 Operating Procedure: HF Trap Operation

HTO _____
 Date _____
 Time _____
 Oper _____

Part A: Charging NaF Pellets to HF Trap

1. Turn off heater TIC-2E-6 _____; close V-107 _____.
2. Check that V-106 is open and allow FV-163 to cool enough for safe handling.
3. Close V-77A, 39, 47, 102, 84, 85, 67, FCV-2. (Check off each one.)
4. Open V-48, 101, 103, 105, 68. (Check off each one.)
5. Open F₂ supply switch and set PC-10 to 4 psig.
6. Set FI-29 (V-77B) for N₂ flow at 25% of scale. Time done _____.
7. After 30 minutes close FI-29 (V-77B), V-103, 105. Time done _____.
8. Remove top flange of FV-163, vacuum out spent NaF pellets and obtain weight. Gross _____ kg, Tare _____ kg, Net _____ kg.
9. Charge about 10 kg of fresh NaF pellets to a level 4 to 5 in. below gas inlet line. Record weight. Gross _____ kg, Tare _____ kg, Net _____ kg.
10. Replace top flange, taking care to work thermocouple-well into pellets without crushing them.
11. Open V-103, 105, and set FI-29 for 25% of scale. Time done _____.
12. After one hour shut FI-29. Time done _____.
13. Proceed to part B, for conditioning of NaF.

Part B: Conditioning NaF Pellets and Trap

1. Close V-102, 67, 84, 85, 39, 47, 77A, FCV-1, FCV-2. (Check off each one.)
2. Open V-48, 101, 103, 105, 68. (Check off each one).
3. Open F₂ supply switch.
4. Set PCV-10 at 4 psig and put on automatic.
5. Set FI-29 (V-77B) for N₂ flow at 25% of scale.
6. Close water valve V-106 and open steam valve V-107 wide, slowly.
7. Set TIC-2E-6 to read 100-105°C on TR-2E-6.
8. Record time 100-105°C is reached on TI-5-19 _____, TR-2E-1 _____, TR-2E-7 _____, TR-2E-12 _____.
9. One hour after 100-105°C is reached in item 8, adjust FE-6 purge rotameters to give 85% reading on FR-8.
10. Crack F₂ valve V-39 to obtain an increase to 87% on FR-8. Time done _____, FR-8 reading _____.
11. Fifteen minutes after Item 10, adjust FE-6 purge to reduce FR-8 to 85% _____, then adjust V-39 to raise FR-8 to 87% again _____. Time done _____.
12. Immediately after Item 11, decrease FI-29 to 20% of scale.
13. Fifteen minutes after Item 11, repeat Item 11. Time done _____.
14. Immediately after Item 13, decrease FI-29 to 15% of scale.
15. Fifteen minutes after Item 13, repeat Item 11. Time done _____.
16. Decrease FI-29 to 10% of scale.

17. Fifteen minutes after Item 15, repeat Item 11. Time done _____.
18. Decrease FI-29 to 5% of scale.
19. Five minutes after Item 17, close FI-29 (V-77B). Time done _____.
20. Fifteen minutes after Item 19, close V-39 _____. Set FI-29 for N_2 flow at 25% of scale _____. Set TIC-2E-6 for 350° F _____. Open V-107 wide, slowly _____. Time done _____.
21. Record time 300-350° C is reached on: TI-5-19 _____, TR-2E-7 _____, TR-2E-1 _____, TR-2E-12 _____.
22. One hour after reaching 300-350° C in Item 21, stop N_2 flow through FI-29 (V-77B). Time done _____.
23. Set TIC-2E-6 to obtain 100-105° C on TR-2E-6 and TR-2E-1 _____. Close V-107 _____ and adjust V-106 to obtain 25° C on TR-2E-12 and TI-5-19 _____.
24. Close V-48, 101, 105, 68, PCV-10. (Check off each one.)
25. Open V-39, 47, 102, 67, FCV-1. (Check off each one.)
26. Hold temperatures as set in Item 23 for use in next pilot unit run.

Part C: Regeneration of HF Trap (Revised April 1, 1958)

1. Close these valves at the F_2 trailers: V-39 _____, 47 _____, 84 _____, 85 _____, 102 _____, and 106 _____.
2. Open these valves at the F_2 trailers: V-48 _____, 101 _____, 103 _____, 105 _____, and 107 (slowly) _____.
3. Close V-67 _____ in Penthouse. Open V-68 _____ in Penthouse.
4. Close PCV-2 _____ at panelboard.
5. Open F_2 supply switch _____ and set PC-10 on 4 psig _____ (and "auto").
6. Close V-77A _____ in gallery. Adjust flow through FI-29 to 25% _____.
7. Raise setpoint on TIC-2E-6 to attain 350° C on TR-2E-1.
8. When TR-2E-1 reached 350° C, record time _____.
9. One hour after time in previous step, stop N_2 flow through FI-29. Due off _____. Actual time flow stopped _____. Record TI-5-19 _____, TR-2E-1 _____, TR-2E-6 _____, TR-2E-7 _____, TR-2E-12 _____, TIC-2E-6 _____, and TC-2E-6 _____.
10. Reduce setpoint on TIC-2E-6 to give 100° C on TR-2E-6. Adjust V-106 to obtain 25° C on TR-2E-12 _____. Record setpoint on TIC-2E-6 _____.
11. Close these valves at the F_2 trailers: V-48 _____, 101 _____, and 105 _____.
12. Open these valves at the F_2 trailers: V-39 _____, 47 _____, and 102 _____.
13. Close V-68 _____ in the Penthouse. Open V-67 _____ in the Penthouse.
14. Shut off the F_2 supply switch _____ and close PCV-10 _____. (Air to close valve.)

15.7.4 Operating Procedure: Fluorine Conditioning Procedure
(Revised November 1, 1957)

FCP _____
Date _____
Time _____
By _____

1. Check that the following pieces of equipment are installed and leak-tight: FV-103 top flange _____, FV-103 side flange _____, FV-114 top flange _____, FV-120 _____, FV-121 _____, FV-124 _____ and FV-126.
2. Turn on EX-FV-522 _____ and EX-FV-650 _____.
3. Set the following nitrogen reducing stations on 4.5 psig. PI-4 _____, PI-6 _____, PI-7 _____, PI-42 _____, PI-44 _____, PI-50 _____ and PI-53 (on 15 psig) _____. (PI-50 and -53 are on nitrogen into FV-100 sampler.)
4. Close all HCV's on instrument panels (except HCV-7 which will be open when HCV-8 is closed).
5. Close all "B" and "C" valves around N₂ purge rotameters.
6. Close V-88 on sampler.
7. Open the following valves near the nitrogen supply header: V-69 _____, 70 _____, 71a _____, 73a _____, 74c _____, 75a _____, 76a _____, 77b _____, 78 _____, 79 _____, 80 _____, 81 _____, 82 _____, 83a _____, 86c _____, 90a _____ and 91a _____.
8. Open V-27 _____, 72 _____, 112 _____ (or 113 _____) in off-gas lines. Close V-25 _____ in off-gas sampler.
9. Start up scrubber _____. Open valve to top fluorine inlet _____ or side inlet _____.
10. Check that the following instruments are in service and their purges flowing: DR-1 _____, LR-2 _____, DR-2 _____, LR-4 _____, FR-5 _____, FR-6 _____, LR-6 _____, PR-8 _____, PR-12 _____, PR-13 _____, PR-16 _____, PR-33 _____, PR-34 _____, PI-38 _____.
11. Read the following rotameters on nitrogen supply lines FI-13 _____, 14 _____, 15 _____, 16 _____, 25 _____, 26 _____, 27 _____, 30 _____, 31 _____, and 33 _____. The readings should be either 1 cfm or 150 cc/min whichever is applicable.
12. Open HCV-8 _____, 11 _____, 14 _____, 16 _____, 35 _____, 36 _____.
13. Open FCV-1 to _____ psig. _____ Set FI-34 to 25% _____. Time _____.
14. One hour later at _____ close off flow through FI-34 _____.
15. Check that fluorine trailer valves are open _____.
16. Set PC-10 on "Auto" _____ and adjust set point to 4 psig _____ (20%).
17. Move orifice-selector switch to "up" position _____. Turn on fluorine supply switch _____. Time on _____. Adjust flow to give 10% on FR-2 _____.
18. If fluorinator, or FV-103 has been opened since the last run, proceed to next step _____. If not proceed to step 20 _____.
19. Thirty minutes after step 18 go to step 20.
20. Open HCV-12 _____, 13 _____, 14 _____, 15 _____, 16 _____, 30 _____, and 34 _____.
21. Open HCV-12 _____. Close HCV-8 _____. Time _____.

22. Twenty minutes later open HCV-18 _____, 19 _____, 32 _____.
Close HCV-15 _____, 34 _____. Time _____.
23. Twenty minutes later open product receiver valves _____, HCV-22 _____,
23 _____, 24 _____. Time _____.
24. Open HCV-23 _____. Time _____.
25. Twenty minutes later open all HCV's except HCV-7 _____.
26. Reduce fluorine flow to 5% on low scale _____. Time _____.
27. Continue up to four hours or whenever requested to stop fluorine
flow by supervision.
28. Open fluorine supply switch (stop flow) _____. Time _____.
29. Close PCV-10 _____. PCV-2 _____.
30. Open PCV-1 to 5 psig.
31. Open V-93 to give 25% on FI-34 _____. (PI-6 on 4.5 psig _____.)
32. Close fluorine trailer valves.
33. Twenty minutes after step 31, close valve 93 _____.
34. Leave purges on until time for next operation.
35. Time completed _____.

By _____

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16.0 ARE CHARGING SYSTEM (5, 22, 23)

16.1 Introduction

The ARE fuel was stored in the dump tank (FV-110) until it was processed in the Volatility Pilot Plant in Fiscal Year 1958. This material was removed from the dump tank and fed to the fluorinator batch-wise from the ARE Charging System. The principal steps in the operation of this system were:

- a. Transferring the entire molten ARE fuel charge into the hold tank (FV-114).
- b. Flowing batches of the fuel as required to the fluorinator (FV-100) for processing.

Radiation exposure was reduced by remote operation.

16.2 Equipment

The general arrangement of equipment for this system is shown in Fig.

16.1. Each piece of equipment is described in Table 16.1.

16.3 Operation

16.3.1 Operating Procedure

Steps in the operation of the ARE Charging System were:^a

- a. Demonstrating Utility of FV-104 Cross-Over and Loop and Sealing Cross-Over (54).^b
  1. Heating to and maintaining at 600°C the FV-114 inlet line, outlet line and vent line and FV-114.
  2. Flowing twelve cans of barren salt into FV-114 through the FV-114 inlet line with N₂ purges on.
  3. Heating to and holding $\geq 570^{\circ}\text{C}$ the three autoresistance heated sections of the charge line (Fig. 16.1).
  4. Filling the FV-104 cross-over with salt by having autoresistance sections A and C at $\geq 570^{\circ}\text{C}$ and then sealing the cross-over by shutting off circuit A.

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^aRadiological safety practices included using the means listed in Sec. 16.4.16.

^bThe flange between FV-110 and -114 was capped.

Fig. 16.1. Equipment Arrangement in the ARE Charging System

Table 16.1
List of A.R.E. Charging System Components

Components	No.	Drawings and Sketches Title	Miscellaneous Information	References
A.R.E. Dump Tank, FV-110	D-22767 D-31350 E-A-3-1-31 D-A-3-1-31-1 through -7 Photo. No. 15044	Cell I Equipment Layout Cell I Engineering Flowchart A.R.E. Fluid Circuit Dump Tank Assembly Miscellaneous Dump Tank Drawings A.R.E. Dump Tank	<u>Period of Use</u> - While transferring the A.R.E. Charge from FV-110 to FV-114.	<u>This Report</u> - Secs. 16.4.2; 16.4.16; 16.5; 16.6; 23.4.10; Figs. 16.3; 16.4; 16.5. <u>Engineering File Folder No. F-30</u> . <u>Other Literature</u> - 22 (p. 5).
A.R.E. Dump Tank Retort, FV-111	Fig. No. 16.5 <u>Misc. Drawg.</u> C-14407 C-14426 C-15166 C-15166A	A.R.E. Dump Tank - Retort-Furnace Assembly Welding Specifications (Monel) Welding Specifications (Inconel) Retort Welding Specification (Stainless Steel)	<u>Period of Use</u> - While transferring the A.R.E. Charge from FV-110 to FV-114; Macos Fabricators, Inc. Detroit (Sub-Contract to A. F. Holden Co.).	<u>This Report</u> - Secs. 16.4.3; 16.4.2; 16.5; 16.6; 3.4.2; Figs. 16.4; 16.5. <u>Engineering File Folder No. F-34</u> . <u>Other Literature</u> - 52 (pp. 6, 7).
A.R.E. Dump Tank Furnace, FV-510	D-22775 <u>Vendor's Drawg.</u> DF516-1 through -6	Wiring Diagram (sheet 1, items "D", "E", "F" and "H") Dump Tank Furnace	<u>Control Equipment</u> - Two interlocking Pyrovens on each of three zones (thermocouples). <u>Period of Use</u> - While transferring the A.R.E. Charge from FV-110 to FV-114. <u>Furnace Data</u> - A.F. Holden Co., Detroit, serial No. 2033; P. O. No. 425X-21435, X-63579; 96 kw., 230 v., 3 phase delta 3 heating zones. Fan and motor supplied with furnace by Industrial Gas Engineering Co., Westmont, Ill. Type FW-355, Class B.	<u>This Report</u> - Secs. 16.4.4; 16.4.3; 16.5; 16.6; 3.4.2; 22.4.1; 22.4.2. Table 22.1. Fig. 16.5. <u>Engineering File Folder No. F-34</u> . <u>Other Literature</u> - 52 (pp. 2, 6, 47, 63).
A.R.E. Dump Tank Furnace Outlet Heater, FV-510A	D-22775 <u>Vendor's Drawg.</u> DF-516-1A, -5 and -6	Wiring Diagram (Sheet 1, item "C") Dump Tank Furnace	<u>Control Equipment</u> - Pyrovene (thermocouple welded to the pipeline midway of the heater) and Variac. <u>Period of Use</u> - While transferring the A.R.E. Charge from FV-110 to FV-114. <u>Heater Data</u> - Supplied with FV-510 by A. F. Holden Co., Detroit; 1 kw., 115 v., 1 phase.	<u>This Report</u> - Secs. 16.4.5; 16.5; 16.6; 22.4.1; 22.4.2. Fig. 16.5. Table 22.1. <u>Engineering File Folder No. F-34</u> .
A.R.E. Molten Salt Drain Pipe Upper Heater, FV-511 and -511A	D-31350 D-31367 Sketch Nos. B-41-2-10 D-25911	Cell I Engineering Flowchart Wiring Diagram (sheet 2, items "A" and "AA") FV-511 and -511A Placement Electrical Details (sheet No. 1)	<u>Control Equipment</u> - Pyrovene (thermocouple welded to pipeline about midway of heater out on inch or more away from the heating element) and Variac for each heater. <u>Period of Use</u> - While transferring A.R.E. Charge from FV-110 to FV-114. <u>Heater Data</u> - For each heater, two Calrods were run along external pipe surface on diametrically opposite pipe elements. FV-511: 3.5 Kw (230 v). FV-511A: 3.0 Kw (230 v).	<u>This Report</u> - Secs. 16.4.6; 16.4.4; 16.5; 16.6; 22.4.1; 22.4.2. Table 22.2. <u>Engineering File Folder No. F-34</u> . <u>Other Literature</u> - 52 (p. 47).

Table 16.1 (Continued)

Components	Drawings and Sketches		Miscellaneous Information	References
	No.	Title		
A.R.E. Molten Salt Drain	D-25911	Electrical Details (sheet No. 1)	Control Equipment - Pyrothane (thermocouple welded to pipeline about midway of heater but an inch or more away from the heating element) and Variac. Period of Use - While transferring ARE charge from FV-110 to FV-114. Heater Data - Two Calrods arranged as for FV-511 and 511A. Total power was 4.0 kw. (230 v).	This Report - Secs. 16.4.7; 16.4.8, 16.5, 16.6; 22.4.1, 22.4.2, Table 22.2. Engineering File Folder No. P-34. Other Literature - 52 (p. 47).
Pipe Lower	D-31367	Wiring Diagram (sheet 2, item "C")		
Heater, FV-513	Sketch No. B-11-5-15	FV-513 and -513A Placement		
A.R.E. Molten Salt Flange Disconnect	D-25911	Electrical Details (sheet No. 1)	Control Equipment - Similar to that for FV-513 Period of Use - While transferring A.R.E. Charge from FV-110 to FV-114. Heater Data - Two Calrods were wrapped around the flange. Total power was 1.5 kw. (230 v).	Same as for FV-513.
Heater, FV-513A	D-31367	Wiring Diagram (sheet 2, item "B")		
	Sketch No. B-11-5-15	FV-513 and -513A Placement		
A.R.E. Hold Tank, FV-114	D-19713	Hold Tank	Control Equipment - Cf. for FV-514. Period of Use - "B" Run. A stainless steel drip pan was built under FV-514 to catch the salt in event of vessel failure.	This Report - Secs. 16.4.8; 16.4.9; 16.4.16; 16.5; 16.6. Engineering File Folder No. P-31. Other Literature - 52 (pp. 2, 3, 6).
	D-23199	Assembly FV-114, -115, and -514		
A.R.E. Hold Tank Furnace, FV-514	D-31367	Wiring Diagram (sheet 2, items "D" and "E")	Control Equipment - Two interlocking Pyrothanes on each zone (each Pyrothane having a thermocouple in a thermowell immersed in the salt and a thermocouple in a thermowell in the annulus between FV-114 and the FV-514 heating elements). Period of Use - "B" Run. Furnace Data - A. F. Holden Co., Detroit; serial No. 2032; P. O. No. W25X-21435; 64 kw, 230 v, 3 phase Delta, two heating zones, heating elements chrome "A".	This Report - Secs. 16.4.9; 16.4.8; 16.5; 16.6; 22.4.1, 22.4.2, Table 22.1. Engineering File Folder No. P-35. Other Literature - 52 (pp. 8, 4, 7, 63).
	Holden Dwg. DF-515-1	Hold Tank Furnace		
	through -5			
A.R.E. Hold Tank Furnace Inlet Heater, FV-514A	D-31367	Wiring Diagram (sheet 2, item "F")	Control Equipment - Pyrothane (thermocouple was welded to FV-114 inlet pipeline about midway of the heater) and Variac. Period of Use - While transferring the A.R.E. charge from FV-110 to FV-114 and also barren salt into FV-114. Heater Data - Supplied with FV-514 by A. F. Holden Co., 1 kw, 115 v., 1 phase.	This Report - Secs. 16.4.10; 16.4.8; 16.5; 16.6; 22.4.1, 22.4.2 Table 22.1 Engineering File Folder No. P-35.
	Holden Dwg. DF-515-1, A			
	and -5	Hold Tank Furnace		
A.R.E. Hold Tank Furnace Outlet Heater, FV-514B	D-31367	Wiring Diagram (sheet 2, item "G")	Control Equipment - Similar to that for FV-514A Period of Use - "B" Run Heater Data - Similar to FV-514A.	This Report - Secs. 16.4.11; 16.4.8, 16.4.13, item b; 16.5, 16.6; 22.4.1; 22.4.2. Table 22.1 Engineering File Folder No. P-35
	Holden Dwg. DF-515-1, A			
	and -5	Hold Tank Furnace		

Table 16.1 (Continued)

Components	Drawings and Sketches	Miscellaneous Information	References
	No.	Title	
A.R.E. Hold Tank Snow Trap, FV-115	C-23194 D-23199 Sketch BOC. B-16-28-15 B-11-1-5	Hold Tank Snow Trap Assembly FV-114, FV-115, and FV-514 Snow Trap, FV-115, Heater Thermocouples and Heaters Below Hold Tank (Fig. 16.6)	<u>Control Equipment</u> - Cf. for FV-515. <u>Period of Use</u> - "E" Runs. <u>This Report</u> - Secs. 16.4.12; 16.5; 16.6; 23.4.10; Fig. 16.6. <u>Engineering File Folder No.</u> - F-35. <u>Other Literature</u> - 52 (p. 9).
A.R.E. Hold Tank Vent Line Heaters FV-515 and -515A	D-22780 Fig. 16.6	Wiring Diagram (Sheet 6, Items "F" and "G") FV-515, -515A and -115 Placement	<u>Control Equipment</u> - FV-515: Pyroverne (thermocouple welded to the heated end of FV-115 adjacent to, but not touching, FV-515) and Variac. FV-515A: Pyroverne (thermocouple welded to the pipe line about midway of the heater). <u>Period of Use</u> - "E" Runs. <u>Heater Data</u> - FV-515: D.E. Caird No. 84419 (790-v, 230-v) wrapped and fitted to FV-115 per Fig. 16.6. FV-515A: Two 1000-w (230-v) clamshell heaters placed end-to-end on pipeline per Fig. 16.6. <u>This Report</u> - Secs. 16.4.13; 16.4.12; 16.5; 16.6; 13.4.5; 22.4.1; 22.4.2. Table 22.2; Fig. 16.6. <u>Engineering File Folder No.</u> - F-32.
A.R.E. Charging Line (Including Marks III and IV Freeze Valves FV-104)	D-23224, Rev. 3 D-31390 D-30890 Fig. 16.7 Fig. 16.8 Fig. 21.2	Cell I Piping Details (Sheet No. 2) Cell I Engineering Flowsheet Wiring Diagram (Sheet 3, Items "E" and "H") Details of FV-104 Vent Line and Heaters View of Vent Line Furnaces for FV-104 Schematic Diagram of the Three Automatic- ally-Heated Lines Between FV-114 and FV-100	<u>Control Equipment</u> - A Pyroverne (thermocouple welded to the pipeline), Variac and ammeter for each molten salt line (NS-114-1 to NS-104-1 and NS-114-1). <u>Period of Use</u> - "E" Runs. <u>This Report</u> - Secs. 16.4.14; 16.3.1; 16.4.15; 16.6; 17.4.1, item g; 21.2; 21.4.2; 21.4.3; 21.4.6; 16.5. Figs. 16.2; 16.5; 16.7; 16.8; 17.4 Table 21.1. <u>Engineering File Folder No.</u> - F-22. <u>Other Literature</u> - 52 (pp. 13, 47).
Vent Line Lower Heater, FV-504	D-22778 D-31390	Wiring Diagram (Sheet 4, Item "A") Cell I Engineering Flowsheet	<u>Control Equipment</u> - Pyroverne (thermocouple welded to the pipe line midway of heater) and Variac. <u>Period of Use</u> - "E" Runs. <u>Heater Data</u> - Hevi-Duty Electric Co., Type No. M-5012S; Serial No. 91371; X-63392; 2.5 kw, 115/230 v, one phase. <u>This Report</u> - Secs. 16.4.15; 16.4.14; 16.5; 16.6; 13.4.2; 22.4.1; 22.4.2. Table 22.1; Figs. 16.7; 16.8. <u>Engineering File Folder No.</u> - F-22. <u>Other Literature</u> - 52 (p. 47).
Vent Line Upper Heater, FV-505	D-22778 D-31390	Wiring Diagram (Sheet 4, Item "B") Cell I Engineering Flowsheet	<u>Control Equipment</u> - Same as for FV-504. <u>Period of Use</u> - "E" Runs. <u>Heater Data</u> - Hevi-Duty Electric Co., Type No. M-5012S, Serial No. - 91374; X-63394; 1.6 kw, 115/230 v, one phase. Same as for FV-504.
A.R.E. Charging System Instrumentation	D-31390	Cell I Engineering Flowsheet	<u>Functions of Instruments</u> DE-1 Liquid Density in FV-114. LE-6 Liquid Level in FV-114. PB-30 Pressure in FV-114 vapor space. PE-45 Sec. 3.4.2. FV-45 Sec. 3.4.2. LI-9 Sec. 3.4.2. Thermocouples were placed in thermowells immersed in molten salt and were welded to pipe and vessel surfaces to maintain the required temperatures. <u>Period of Use</u> - "E" Runs. <u>This Report</u> - 16.4.16; 16.4.2; 16.4.3; 16.5; 16.6; 3.4.2; 14.4.5, 22.4.2. <u>Engineering File Folder No.</u> - F-31. <u>Other Literature</u> - 13.

5. Transferring molten salt across the loop by having auto-resistance sections B and C at $\geq 570^{\circ}\text{C}$ and then sealing the charge line at its lowest point by cutting off auto-resistance power.

b. Charging FV-114 with the ARE Fuel Charge

1. Having already performed item 1 in Part A and having connected the flange between FV-110 and -114.
2. Weighing FV-110 and then placing FV-110 into FV-111.
3. Putting lid on FV-510.
4. Adjusting the necessary valves, setting purges, and continuing the purges for 18 hours to remove all air before starting to melt the salt.
5. Turning on the FV-510 fan and maintaining N_2 flow and addition of dry ice to keep fan bearing cool.
6. Putting necessary instruments into service and synchronizing the charts.
7. Adjusting set-points on temperature controllers to 600°C and setting Variacs on furnaces and heaters to prepare for ARE charge melt-down.
8. Maintaining temperatures of vessels and lines at $\sim 600^{\circ}\text{C}$.
9. Watching liquid level recorder (LR-6) in FV-114.
10. When LR-6 reading reached the maximum, heating FV-510 to 650°C and holding it at 650°C for 15 hours to assure entire drainage of ARE charge to FV-114.
11. Turning off power to the FV-510 fan and all heaters above FV-514 (including FV-514A).
12. Resetting valves and purges.
13. When ARE charging equipment had cooled essentially to room temperature, disconnecting and discarding this equipment and then capping the flange above FV-114 (FV-110 was again weighed before discarding.)

NOTE: The degree of FV-110 drainage was determined in three ways: (a) weighing before and after, (b) activity readings, and (c) LR-6 reading. See Sec. 16.4.2 for more detail.

c. Transferring ~150-kg Batches of Feed Salt from FV-114 to -100.

1. Having ARE fuel at 600°C in FV-114; FV-514B, -515, and -515A at 600°C; HCV-6 open; and the charge line sealed.
2. Heating the charge line circuits B and C to $\geq 570^{\circ}\text{C}$ and FV-100 to 600°C.
3. Closing vent valve (HCV-6) and pressurizing FV-114 to ~10 psig with N_2 .
4. When LR-2 reading in FV-100 reached the desired value, opening HCV-6 to stop the transfer.
5. Adjusting valves and maintaining the required temperatures. (Autoresistance circuits B and C were shut off; all other power was left on.)

d. "Washing-Out" FV-114 and the Charge Line after Run E-6 Charging.^b

1. Having vessels and lines at temperatures indicated in items 1 and 2 in Part c and FV-514A at 600°C.
2. Transferring 39 kg of barren salt "flush" into FV-114 through the capped flange above FV-114.
3. Transferring more than half (~25 kg) of this flush over the FV-104 loop.
4. Then, after disconnecting circuit B and connecting circuit A, completing the "wash-out" salt transfer to FV-100 through the FV-104 cross-over.
5. Shutting off power to circuits A and B to seal charge line at its lowest point.
6. Shutting off all power except for FV-100.

Operating procedures prepared for Parts b and c are shown as Secs. 16.7.1 and 16.7.2. Methods used for Parts a and d were variations of these procedures.

16.3.2 Critical Operating Steps

- a. Demonstrating the operability of the FV-104 loop and cross-over and a tight seal at the cross-over.

^aIn contrast to the salt run-back obtained in waste salt transfers (Sec. 13.3.1), essentially no run-back occurred in the ARE work.

^bRun E-6 charging exhausted the ARE fuel. At that time, the salt level was at about point (3) on Fig. 16.1.

Prior operating experience cast doubt on the advisability of using the molten lead-sealed disconnect flange between FV-110 and -114 instead of a positively sealed joint. Using the original flange design would have made it possible to flow the entire ARE salt charge into Cell 1 should a plug occur in the inlet to FV-114 before the salt transferred to FV-114. Such a mishap would have presented a severe radiation hazard and a complicated uranium recovery problem. Therefore, the molten lead-sealed flange was replaced with a ring-gasketed flanged joint. This joint was easily broken after the salt transfer and capped to provide the desired seal with a gas and/or molten salt connection to FV-114.^a

- b. Designs unfinished which required obtaining operating data and/or available manpower for completion.

The pipeline between FV-110 and -114 was fitted with an "S" shaped expansion loop as shown in Fig. 16.2. Also incorporated in this scheme was the four-section heater comprising FV-511, -511A, -513, and -513A.

The shape of the vent line with the placement of FV-115 and associated heaters, FV-515 and -515A, was determined as shown in Fig. 16.1.

16.4.2 ARE Dump Tank, FV-110

The ARE dump tank containing the radioactive ARE salt fuel (reading 10 rem at 5 ft away) was transferred by truck and mobile crane from Building 7503 to Cell 3 in Building 3019 on July 28, 1955 (Fig. 16.3, 22). This move being very carefully planned was made without difficulty or excessive personnel exposure to radiation. An attempt was made to maintain a nitrogen atmosphere about the salt in the inverted dump tank during its storage in Cell 3. However, the rubber stoppers closing the drain holes fell from the tank after a few months. So this idea was abandoned. Nevertheless, no difficulty from air or water vapor in the salt was experienced during processing. The dump tank was loaded into its furnace on December 2, 1957 without trouble or excessive personnel radiation exposure (Figs. 16.4 and 16.5). The dump tank was weighed (4,370 lb) by means of a strain gauge before being lowered into its furnace and again after the salt had been drained from it to the hold tank (2,275 lb).

The transfer of salt from the dump tank to the hold tank was made on December 5, 1958 without incident. The transfer required 5 hours at ~550°C for the bulk of the salt to drain by gravity. The furnace was heated, however, to 650°C for an additional 15 hours to assure that all of the salt had drained. The level of radioactivity at the surface of the dump

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^aThis flanged joint also required the expansion loop described below in item b.

Fig. 16.3. ARE Dump Tank, FV-110.

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Fig. 16.4. ARE Dump Tank Being Lowered Into the Retort and Furnace.

Fig. 16.5. ARE Dump Tank-Retort-Furnace Assembly

tank furnace decreased from 60 mrem/hr (furnace top) before the transfer to ~2 mrem/hr after the transfer, indicating complete removal of the ARE salt.^a The completeness of the transfer was in doubt before the activity measurements were made because the liquid level (LE-6; Sec. 16.4.16) in FV-114 indicated approximately 10% less salt than was expected from preliminary calculations. The highest activity reading on the dump tank itself decreased from 1500 mrem/hr at contact before the transfer to 30 mrem/hr after the transfer.

16.4.3 ARE Dump Tank Retort, FV-111

This vessel contained the dump tank while transferring the ARE charge to FV-114 (Sec. 16.4.2). A thin coating of dark brown scale covered the stainless steel surfaces of the ARE retort (FV-111) despite N₂ purging of the system for 18 hours prior to heating. (A hygrometer had been installed in the retort vent to determine when all of the water vapor was removed.) Because of this scale it was feared that all of the air in the dump tank had not been removed by the purging. No difficulty was experienced, however, during operation because of O₂ or water vapor in the salt. The same vent arrangement described in Sec. 3.4.2 was used for the ARE dump tank (16.4.16). After the dump tank and its furnace were removed from Cell 1, the vent system was reinstalled on FV-102.

16.4.4 ARE Dump Tank Furnace, FV-510^b

The ARE dump tank furnace was satisfactory. Time requirements were:

- a. Time to 550°C (Pyrovane set-point at 600°C) - 27 hr.^c
- b. Time at 550°C to drain bulk of salt by gravity - 5 hr.
- c. Time from 550°C to 650°C - 3 hr.
- d. Time at 650°C to assure complete salt removal - 15 hr.

The sand seal on the dump tank furnace lid held effectively (as indicated by PR-45) both for the ARE salt transfer and for a preliminary trial transfer of barren salt from 10 cans (Secs. 16.4.3 and 3.4.2).

Difficulty was experienced in keeping the FV-510 fan bearing cool (i.e., below 100°C). Since the N₂ flow for this purpose was inadequate, dry

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^aActivities were determined with a cutie pie (Secs. 23.4.10 and 16.4.16).

^bFig. 16.5; Table 22.1; Secs. 22.4.1 and 22.4.2.

^cThis included the curing time of the furnace as well as the time required to heat the dump tank to 550°C. The time to melt the ARE fuel was ~10 hours.

ice was packed around the bearing to cool it. The effective draining of the dump tank, which was a heat exchanger having 90 two-in. diameter tubes vertically through it, indicated that the FV-510 fan spread the heat evenly through the tubes as intended.

16.4.5 ARE Dump Tank Furnace Outlet Heater, FV-510A^a

After the salt was thought to be molten and before the salt transfer to FV-114 had begun, the power to this heater was set at nearly the maximum since this heater was located near the transition point between the retort and the molten salt pipeline, a point found to inhibit transfer in the salt charging system. Actually, several hours elapsed between the time the salt was presumed to be molten and the time the salt level in FV-114 began to rise. Therefore, concern had developed over whether this or some other heater on the line between FV-510 and FV-114 was inadequate.

Operational data were: Pyrovane set-point - 600°C and Variac setting - 140 v.

16.4.6 Molten Salt Line Heaters Between FV-110 and -114, FV-511 and -511A^b

These heaters were satisfactory.

Operational data were:

a. Pyrovane set-points:	FV-511	-	600°C
	FV-511A	-	600°C
b. Variac settings	FV-511	-	140 v
	FV-511A	-	140 v

Heat-up times were less than the ~10 hours^c required to melt the salt in FV-110.

16.4.7 Molten Salt Line Heaters Between FV-110 and -114, FV-513 and -513A^b

These heaters were satisfactory.

Operational data were:

a. Pyrovane set-points	FV-513	-	600°C
	FV-513A	-	600°C
b. Variac settings	FV-513	-	70 v
	FV-513A	-	140 v

Heat-up times were less than the ~10 hours^c required to melt the salt in FV-110.

^aFig. 16.5; Table 22.1; Secs. 22.4.1 and 22.4.2.

^bTable 22.2 and Secs. 22.4.1 and 22.4.2

^cEstimate based on 27 hrs required to cure furnace and melt the ARE charge (Sec.16.4.4).

16.4.8 ARE Hold Tank, FV-114

FV-114 was a stainless steel tank heated by FV-514 (Sec. 16.4.9). This vessel withstood the additional time-at-temperature^a of about one month after Run No. E-2 with no apparent signs of failure. This vessel has not been visually or otherwise inspected since the ARE recovery runs. In order to assure that no loss of salt would occur in the event of vessel failure, a stainless steel drip pan was built under FV-114.

The instrumentation on FV-114 (DE-1, LE-6, and PE-30) is discussed in Sec. 16.4.16. The technique of bringing the instrument purge lines through the bottom of FV-114 was considered less desirable than bringing them through the top. Not only did this subject a greater length of purge line to the corrosive salt mixture but it also made maintenance work on the probes impossible.

16.4.9 ARE Hold Tank Furnace, FV-514^b

This furnace satisfactorily heated FV-114 (Sec. 16.4.8). To maintain the ARE fuel charge at 600°C, its Pyrovane was set at 600°C.

During installation, it was necessary in order to obtain a proper fit to redrill some of the holes in the base provided for pipelines and instrument purge lines.

16.4.10 ARE Hold Tank Inlet Heater, FV-514A^b

This heater was satisfactory.

Operational data were:

- a. Pyrovane set-point - 600°C
- b. Variac setting - 70 v

This heater was used only when transferring salt into FV-114 (Sec. 16.4.8). Its heat-up time was less than that required to melt the salt to be charged (Sec. 16.4.4).

16.4.11 ARE Hold Tank Outlet Heater, FV-514B^b

This heater was satisfactory.

Operational data were:

- a. Pyrovane set-point - 600°C
- b. Variac setting - 70 v

^aThe salt was maintained at 560°C, about 40° above the melting point, during the one month's period whereas normally it was kept at 600°C while operating.

^bTable 22.1 and Secs. 22.4.1 and 22.4.2.

Since this heater was kept hot while the ARE charge was molten in FV-114 (Sec. 16.4.8), its heat-up time was not operationally significant.

16.4.12 ARE Snow Trap, FV-115

"Snow" removal data included the following: (a) the activity of the mesh varied from 120 mrem/hr at the inlet to 10 mrem/hr at the point half-way along the axis, remaining 10 mrem/hr along the rest of the axis; (b) the deposit was very thin on the first half of the mesh and virtually nonexistent on the second half; (c) the deposit when scraped off the mesh had a grayish cast and read 400 mrem/hr;^a and (d) there was insufficient material for identification analyses.^a These data point toward the conclusion that the snow problem was not serious enough either to form plugs or to render the vent line highly radioactive.

See Fig. 16.6 for location of this trap which was heated by FV-515 (Sec. 16.4.13).

16.4.13 ARE Hold Tank Vent Line Heaters, FV-515A and -515^b

The clamshell heater, FV-515A, was satisfactory,

Operational data were:

- a. Pyrovane set-point - 600°C
- b. Variac setting - 140 v

FV-515, a tightly bent Calrod heating FV-115 (Sec. 16.4.12) failed twice. Since no other Calrod failures under reasonably good conditions of service occurred,^c the reason for trouble with this heater was believed to have been the tight bend required. The usual precautions with bending were taken. However, in spite of these precautions, the final required outside diameter of the bent Calrod was hard to achieve without exceeding the recommended minimum radius of bend. Pyrovane set-point was 600°C.

Since these heaters were kept hot while the ARE charge was in FV-114, heat-up times were not operationally significant.

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^aActivity data were obtained with a cuite pie (Secs. 23.4.10 and 16.4.16).

^bFig. 16.6; Secs. 22.4.1 and 22.4.2; Table 22.2

^cSome trouble with the waste nozzle Calrod, FV-512, was also experienced as cited in Sec. 13.4.5. In this case, however, service conditions were severe, especially since molten salt contacted the Calrod.

Fig. 16.6. Placement of FV-515, -515A, and -115

16.4.14 ARE Charging Line^a

The new-design charge line including the Mark III (no FV number) and IV (FV-104) freeze valves shown in Figs. 16.2 and 17.4 replaced the original design to facilitate "washing-out" FV-114 after the last ARE feed transfer (Part d, Sec. 16.3.1 and Sec. 17.4.4).

The placement of electrodes on the MS-114-1 and MS-114-1 to MS-104-1 transfer lines is delineated in Secs. 21.4.2; 21.4.3; and 21.4.6. The satisfactory placements of two ground electrodes on the corresponding molten salt lines are of specific interest here:

- a. The FV-114 extremity electrode for MS-114-1 on the vent line.
- b. The FV-100 extremity electrode for MS-114-1 to MS-104-1 on the vessel flange.

In addition, the off-molten-salt-line placement of the "hot" electrode on the FV-104 nitrogen line was satisfactory as indicated in the two following paragraphs and in Sec. 16.4.15.

Essential technical design features of FV-104 were:

- a. Placement of the nitrogen line at the top of freeze valve loop equidistant from points (1) and (2) (Fig. 16.2) so that the loop "hot" electrode (H-1) could be placed on the nitrogen line.
- b. At this nitrogen line, proper saddle-joint formation to assure an approximately even split of autoresistance current and adequate heating of the "tee." [This also included the correct wall thickness of the nitrogen line (nickel) for sufficient autoresistance heating at the "tee" per Sec. 21.2.]
- c. Point (2) being placed slightly below point (3) so that the line segment (3)-(2) could stand full of salt with FV-114 empty.
- d. Line segment (1)-(2) being sloped upward toward FV-100 so that pressure would be required to transfer salt from the filled line segment (3)-(2) to FV-100.

The operation of the charge line was without incident except that full current (~500 amp) in Run E-6 was required to transfer the "wash-out" salt to FV-100 through the cross-over. The reason for this was presumed to be cold spot(s) occurring at cracks in the line insulation. The transfer time was normally ~a half-hour, being somewhat longer in the Run E-6 "wash-out."

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^aFigs. 16.6, 16.7, 16.8; Secs. 21.4.2 and 21.4.3; and Table 21.1. Line temperatures were measured with thermocouples placed as delineated in Sec. 21.4.6.

Fig. 16.7. Details of FV-104 Vent Line and Heaters Placement

Fig. 16.8. Vent Line Heaters for FV-104 and Other ARE Equipment in Cell I.

Autoresistance circuits on 1/2-in. NPS schedule 40 Inconel were run at 435 amperes (Table 21.1).

Because of the severe corrosion of the Mark II waste line heated both by autoresistance and resistance (Sec. 13.4.2), four specimens from the ARE charging line were to be tested. These specimens disappeared, however, during the cleaning of Building 3019 following the explosion in November, 1959.

16.4.15 Vent Line Heaters, FV-504 and -505^a

For conventional freeze valves, the joints between the freeze valve and the nitrogen and/or vent lines were the most likely cold spots as delineated in Sec. 13.4.2. This situation was not encountered for FV-104 presumably for the two following reasons: (a) that this joint was auto-resistance heated as well as being partially heated by the resistance heater FV-504, and (b) the wall thickness of the 1/2-in. NPS nickel was correct to ensure autoresistance heating (Sec. 16.4.14); and (c) great care was taken in placing the furnaces, FV-504 and -505. Pyrovane set-points used were 600°C.

16.4.16 ARE Charging System Instrumentation (13)

The instrumentation for FV-114 (DE-1, LE-6, and PE-30) worked satisfactorily. The indication of 10% less salt in FV-114 than expected (Sec. 16.4.2) apparently resulted from a misestimation of the ARE charge rather than an error in LE-6. In Run E-6, the common probe for LE-6 and DE-1 plugged. After this, these instruments could not be used. The resulting high differential pressure across the bellows wall ruptured the density transmitter bellows. This occurrence led to the practice of taking a density instrument from service when the high-pressure probe was thought to be plugged, and when salt was frozen in the fluorinator (Sec. 14.4.5 for procedure of removing an instrument from service).

The same vent arrangement described in Sec. 3.4.2 was used for the ARE dump tank (Sec. 16.4.3).

Means used to detect the presence of radiochemicals were:

- a. Constant air monitor (Sec. 23.4.2).
- b. Portable disk sampler (Sec. 23.4.3).
- c. Alpha hand monitor (Sec. 23.4.6).
- d. Quintector (Sec. 23.4.6).

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^a Secs. 16.7.3, 16.7.5, 22.4.1, 22.4.2 and Table 22.1

- e. Hand-foot counter (Sec. 23.4.6).
- f. Background recorder (Sec. 23.4.7).
- g. α, β smears (Sec. 23.4.8).
- h. Alpha survey meter (Sec. 23.4.9).
- i. β - γ probe meter (Sec. 23.4.9).
- j. Cutie pies (Sec. 23.4.10, 16.4.2, 16.4.12, 13.4.7).
- k. Monitron (Sec. 23.4.10).
- l. Film badges (Sec. 23.4.11).
- m. Dose meters (Sec. 23.4.12).
- n. Urine and fecal samples (Sec. 23.4.13).

Protective devices (Sec. 23.4.14) and decontamination practices (Sec. 23.4.15) were used as required. The UF_6 leak during Run E-2 accelerated the frequency of monitoring for radiochemicals and the use of protective devices. Personnel exposures and air activities during subsequent operation were recorded (Sec. 23.4.1).

16.5 Summary and Conclusions

The ARE salt system operated smoothly and effectively with only minor difficulties. All furnaces and heaters were satisfactory. Heating and cooling times are recorded.

Late design work included:

- a. Changing the location of FV-114 relative to FV-100 to enable transferring batches against a 5-ft instead of a 15-ft head. (This also necessitated moving FV-510 and designing another charge line with the Mark III and IV freeze valves.)
- b. Replacing the molten lead-sealed disconnect flange with a bolted flange to eliminate the possibility of flowing the ARE charge into Cell 1 in the event of a line plug below the flange.
- c. Designing the pipeline between FV-110 and -114, including the expansion joint.
- d. Determining the shape of the FV-114 vent line and the placement of FV-115, -515, and -515A.

Dump tank data included: (a) moved to Cell 3 on July 28, 1955, (b) placed in FV-510 on December 2, 1957, and (c) salt drained out on December 5, 1957.

The bulk of the salt transferred in 5 hours with FV-110 being held at 550°C. Subsequently, the temperature of FV-510 was raised to 650 C and kept at that temperature for 15 hours to ensure complete drainage. The completeness of removal was assured by: (a) tank weights, (b) activity readings, and (c) the salt level in FV-114. Although a thin brown scale formed on FV-111 during the ARE melt-down, no later trouble was experienced with oxygen or water vapor in the salt.

The nitrogen flow for cooling the FV-510 bearing was inadequate. Additional cooling was accomplished with dry ice.

The hold tank successfully held the ARE charge during processing and the one month shut-down between Runs E-2 and -3. Evidently the common instrument probe for LE-6 and DE-1 failed in Run E-6. Otherwise, the FV-114 pneumatic-type instruments (LE-6, PE-30, and DE-1) were satisfactory. Redrilling for pipelines and instrument lines was required to obtain the proper fit for the FV-514 installation.

FV-115 data indicated that the "snow" problem in the FV-114 vent line was minor both regarding plug formation in the line and the radioactivity of the line. The FV-515 Calrod failed twice, apparently because of the close bending required. These Calrods and the earlier one at FV-512 were the only Calrod failures in VPP.

Operation of the charge line and two freeze valves was satisfactory. No plugs formed at the vent line joint similar to those at the FV-106 and -108 vent lines joints. This was attributed to the better design of FV-104. Three autoresistance electrodes were successfully placed off the molten salt lines: (a) on the FV-114 vent line, (b) on the FV-104 nitrogen line, and (c) on the FV-100 flange.

16.6 Recommendations

It is recommended that future salt charges like the ARE be handled similarly and that:

- a. Subsequent pressure-type salt transfers be made against as low a head as possible, preferably 5 ft or less.
- b. Bolted flanged joints be used in salt transfer lines instead of molten lead-sealed flanges.
- c. The expansion joint necessary with bolted flanges (item b above) be an "S" bend as for the ARE work.
- d. Vent lines on future freeze valves be designed as for FV-104 to eliminate cold spots at saddle joints.
- e. No gas-cooled bearing be used in the future unless absolutely necessary and then only if its adequacy is double-checked.
- f. All pneumatic-type instrument probe lines enter through the top of a vessel.

- g. Either autoresistance or resistance (clamshell, Calrod, or furnace) heating be used, the choice being based on the item being heated.
- h. Off-molten-salt line placement of autoresistance ground and hot electrodes as described herein (Sec. 21.4.6) continue to be used.

16.7 Appendix

16.7.1 Operating Procedure: Charging Hold Tank (Revised November 1, 1957)

CHT _____
 Date _____
 Time _____
 Operator _____

This runsheet is to be started after the hold tank is partially charged with barren salt; the FV-104 loop is successfully used; and the cross-over is sealed.

1. Close HX-6 _____; open FV-45.
2. Close V-98 _____ on vent system.
3. Set the following purges: V-74a omit, 75a _____, 76a _____, 90a omit.
4. Turn on the fan in FV-510 _____. Adjust nitrogen flow through V-82 to give small flow over fan bearing _____. Increase flow as required to maintain low bearing temperature and add dry ice to bearing surface.
5. Check that these instruments are in service, purges on, and chart synchronized. DR-1 _____, LR-6 _____, and PR-30 _____.
6. Go to LMV, do steps 16-31 remembering that FV-111 has replaced FV-102, and that the system should be purged with N₂ for 18 hours prior to melting the salt. After the 18-hr purge time, open HX-6.
7. Turn on the following controllers and set for 600°C:

TIC-1 _____	TIC-109 _____
TIC-2 _____	TIC-110 _____
TIC-101 _____	TIC-111 _____
TIC-102 _____	TIC-112 _____
TIC-103 _____	TIC-113 _____
TIC-105 _____	TIC-1A-9 _____
TIC-106 _____	TIC-1A-10 _____
TIC-107 _____	TIC-1A-11 _____
TIC-108 _____	TIC-1A-12 _____
Time _____	

8. Set the following Variacs:

Variac - TC	Set point	Variac - TC	Set Point
108	40	112	65
109	40	113	65
110	65	1A-12	65
111	65		

Time _____

9. Three hours after time in 7, reset the Variacs to these settings:

TIC	Set Point
108	70
109	70
110	140
111	140
112	140
113	140
1A-12	140
Time	

10. Two hours after time in 8, set the Variacs only as high as is necessary to maintain 600°C on the line. Time _____.
11. Record data on the appropriate sheets.
12. Watch LR-6 for maximum liquid level. Wait twenty minutes, then record final liquid level reading _____. NOTE: FV-510 was subsequently heated to 650°C and held at 650°C for 15 hr to insure draining entire charge into FV-114. This was not contemplated when the runsheet was written (Secs. 16.3.1b and 16.4.2).
13. Turn off the following controllers: _____
TIC-101 _____	TIC-109 _____
TIC-102 _____	TIC-110 _____
TIC-103 _____	TIC-111 _____
TIC-105 _____	TIC-112 _____
TIC-106 _____	TIC-113 _____
TIC-107 _____	
TIC-108 _____	Time _____
14. Lower the following Variacs to zero: TC-108 _____, TC-109 _____, TC-110 _____, TC-111 _____, TC-112 _____, and TC-113 _____.
15. When furnaces has cooled off turn off fan in FV-510 _____. Close V-82 _____.
16. Riggers will remove dump tank and furnace from cell area. If this is a cold run, see log for additional instructions.

16.7.2 Operating Procedure: ARE Feed Salt Transfer (Revised November 1, 1957)

FST _____
 Date _____
 Time _____
 Oper _____

1. Open the following valves on the control panel: HX-6 _____, HX-11 _____, HX-35 _____, and HX-36 _____.
2. Close the following valves: HX-8 _____, HX-12 _____, HX-14 _____, HX-16 _____, HX-34 _____.
3. Valves V-27 _____ and V-72 _____ and fluorine inlet valve _____ should be open near the scrubber.
4. Switch EX-1B-4 to "C" position.
5. Adjust the setpoints on the following temperature controllers:

Controller TIC	Set Point	Time set
1A-5	660°C	_____
1A-6	610°C	_____
1A-8	650°C	_____
1B-4	650°C	_____
1B-8	650°C	_____
1B-10	650°C	_____
1B-11	700°C	_____

6. Set the following Variacs:

Variac - TC	Set Point	Time set
1B-4	{about 170}	_____
1B-8	{about 140}	_____
1B-10	{about 70}	_____

7. Check these instrument purge rates to be correct, then record these readings DR-1 _____, DR-2 _____, LR-2 _____, LR-6 _____, PR-30 _____, PR-33 _____.

8. When all these points are above 600°C, go to the next step. TR-1A-5 _____, TR-1A-6 _____, TR-1A-8 _____, TR-1B-4C _____, TR-1B-10 _____, and TR-1B-11 _____.

9. Record pertinent data on runsheets provided.

10. Set PI-8 on 10 psig _____.

11. Close HX-6.

12. Watch LR-2 closely. When LR-2 reaches _____%, open HX-6 _____. Time _____.

13. Wait 5 minutes, then lower these set points to zero: TIC-1B-4 _____, and TIC-1B-8 _____.

14. Reduce these Variacs settings to zero TC-1B-4 _____, and TC-1B-8 _____.

15. Reduce the set point on TIC-1B-10 to _____°C.

16. Record these readings when TR-1B-4C and 5C fall below 200°C:

DR-1 _____, DR-2 _____, LR-2 _____, LR-6 _____, PR-30 _____ and PR-33 _____.

17. Reset PI-8 to 4.5 psig.

18. Proceed to "Feed Salt Fluorination" runsheet.

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17.0 VALVES

17.1 Introduction

The manually and automatically operated valves in the Volatility Pilot Plant provided desired "on" or "off" conditions in pipelines and gave suitable controlled flow in both process and service piping.

This treatment lists the valves used under various service conditions, troubles encountered, tests and developmental work, and finally a list of valves recommended for future work. In some cases, advantages and disadvantages of widely used valves are given.

17.2 Equipment

Process valves comprised four categories:

- a. More than one hundred conventional manually operated valves to give shut-off and controlled flow.
- b. Twenty-six remotely operated "on-off" valves (commonly called HCV's) to isolate sections of the process piping.
- c. Ten remotely operated control valves to control nitrogen and fluorine flow rates as well as line pressures.
- d. Freeze valves in molten salt lines - an ORNL innovation - to seal molten salt lines.

The treatment of remotely operated control valves is limited to the valves themselves. The mechanics of each control valve system is discussed in the appropriate system. Service valves were used in air, water, and nitrogen lines. Valve specifications were drawn up prior to VPP operations (49).

17.3 Operation

The operations of specific valves were parts of the plant operating procedures.

17.4 Equipment Performance - Process Valves

17.4.1 Conventional Manually Operated Valves

Table 17.1 is a compilation of the design features and service conditions for the bulk of the process valves. The valves listed under each service notation were successfully used in the specified service conditions. Detailed descriptions of the design features for these valves are available in existing valve literature. Noteworthy items from experience in the Volatility Pilot Plant follow.

Table 17.1

MANUALLY OPERATED VALVES USED IN VPP PROCESS WORK

Identification Data			Materials of Construction			No.	Size	Remarks
Designation	Type	Manufacturer	Body	Disk	Stem Seal	Used	Used	
<u>Service:</u> UF ₆ , F ₂ , and N ₂ Mixtures (Temperature = ~ 66-150°C; Pressure = 15-25 psia)								
No. K-4140 SMMD ⁵	Globe	Crane	Monel	Monel	Bellows	10 24 3	1/4" 1/2" 1 "	Inner Bellows, Phosphor bronze Outer Bellows, 80-20 Brass; Used principally with F ₂ -N ₂ mixtures near room temperature.
No. 5665	Angle Cylinder valve	Superior	Bridge- port Alloy E 3712	Monel	Teflon packed	15	1 "	Bridgeport No. 112 (Cast) (Al:275- 4.25; Si: 0.75-1.25; Bal. Cu) was presumed to be similar to E-3712. Used at pressures up to 65 psia. Used with 100% F ₂ for conditioning and leak-testing ² only.
No. 411	Globe Angle	Hoke	Monel	Dura- Nickel	Inconel Diaphragm	4	1/4"	Small ports were subject to plugging in UF ₆ service.
No. 1193	Globe	Hoke	Brass	Brass	Asbestos Packed	1	1/4"	Small ports were subject to plugging in UF ₆ service.
No. 1197	Globe	Hoke	S.S.	Teflon	18-8 S.S. Bellows	8	1/4"	Used at room temperature to isolate N ₂ portion of system from process portion and not for flowing UF ₆ and F ₂ .
No. 1198	Globe	Hoke	316 S.S.	Teflon	18-8 S.S. Bellows	1	1/2"	Used at room temp. to isolate N ₂ portion of system from process portion and not for flowing UF ₆ and F ₂ .
Fig. 1 No. 1300	Gate	Jenkins	S.S.	S.S.	Packed	3	1/2"	
No. 1103	Angle	Discon- tinued Hoke	Monel	Monel	Monel Bellows	3	1/8"	Used with 100% F ₂ for conditioning and leak-testing ² only.
No. 413	Globe	Hoke	Monel	Dura- Nickel	Inconel Diaphragm	14	1/4"	Small ports were subject to plugging in UF ₆ service.

Table 17.1 (Continued)

MANUALLY OPERATED VALVES USED IN VPP PROCESS WORK

Identification Data			Materials of Construction			No.	Size	Remarks
Designation	Type	Manufacturer	Body	Disk	Stem Seal	Used	Used	
<u>Service:</u> AQ KOH and KF ² (Temperature = Room ³ to ~ 100°C; Pressure = Atmospheric).								
2900-B	Saunders Diaphragm	Hills-McCanna	Monel	Neoprene Diaphragm	Neoprene Diaphragm	5 1 12	2" 1" 1/2"	The diaphragm seals also.
No. 731	Needle	Aloyco	S. S.	S. S.	Packed	2	1/2"	
Fig. No. 518 ¹	Gate	Powell	Brass	Brass	Packed	2	1/2"	
No. 2493	Globe	Powell	Monel	Monel	Packed	1	2-1/2"	
<u>Service:</u> AQ.KOH (~ Room Temperature; Pressure = Atmospheric).								
Fig. No. 1709-8 ¹	Globe	Lunkenheimer	Casual	Monel	Packed	1 2 1	1-1/2" 1" 3/4"	
No. 3602KW	Gate	Crane	Steel	Steel	Packed	1	3/4"	
<u>Service:</u> Off Gas ⁴ (~ Room Temperature; Pressure = Atmospheric).								
No. 1070 A	Needle	RPandC	S. S.	S. S.	Packed	2	1/4"	
Cat. No. 18840	Gate	Crane	S. S.	S. S.	Packed	3	1"	
Cat. No. 18851	Globe	Crane	316 S.S.	316 S.S.	Packed	2	2"	
Fig. No. B-16 ¹	Globe	Stockham	Brass	Brass	Packed	1	1"	

Table 17.1 (Continued)

MANUALLY OPERATED VALVES USED IN VPP PROCESS WORK

Identification Data			Materials of Construction			No.	Size	Remarks
Designation	Type	Manufacturer	Body	Disk	Stem Seal	Used	Used	
Service: Vacuum (~ Room Temperature; Pressure = 5-15 psia)								
SSD	Globe	Crane	Steel	Teflon	Bellows	4	1-1/2"	Inner Bellows Phos. Bronze Outer Bellows, 80-20 Brass
						4	1"	
No. U-0228	Gate	Fairbanks	Brass	Brass	Packed	3	1"	Vacuum Service Lines for Discharging Chemical Traps
Cat. No. 431	Gate	Crane	Brass	Brass	Packed	1	1/4"	
Service: N ₂ (Room Temperature; Pressure = 0-40 psig)								
No. 96417	Globe	Fulton Sylphon	Monel	Teflon	Phos. Bronze Bellows	20	1/4"	
Fig. No. 150	Gate	Powell	Brass	Brass	Packed	3	1/2"	
Fig. 2608	Globe	Powell	Brass	Brass	Packed	2	1/8"	
Fig. 1708	Globe	Powell	Brass	Brass	Packed	15	1/4"	
Fig. 708	Globe	Powell	Brass	Brass	Packed	6	1/2"	
No. 11	Globe	Walworth	Brass	Brass	Packed	4	1/2"	
No. 704	Gate	RPandC	Brass	Brass	Packed	4	1/4"	
No. 212P	Gate	Crane	Brass	Brass	Packed	1	1/2"	
						1	1/4"	
No. 108 HD	4-Way	Imperial	Brass	Brass	Packed	1	1/4"	
Fig. 556 ¹	Globe	Jenkins	Brass	Brass	Packed	1	1/4"	
No. 73 P	Globe	Lunkenheimer	Brass	Brass	Packed	1	1/2"	
No. 0501	Globe	Fairbanks	Brass	Nickel	Packed	1	1/4"	
No. 1193	Globe	Hoke	Brass	18-8 S.S.	Packed	20	1/4"	
No. 333	Globe	Natl. Cyl. Gas Co.	Brass	Brass	Plastic Gasket	10	1/2"	Pressure = 0-2000 psig
No. 334	Globe		Brass	Brass		~60	3/4"	Used on all N ₂ cylinders. Pressure = 0-2000 psig.
No. 1734	4-Way Lub. Plug	Walworth	Steel	Steel	Packed	2	1/2"	

Table 17.1 (Continued)

MANUALLY OPERATED VALVES USED IN VPP PROCESS WORK

Identification Data			Materials of Construction			No.	Size	Remarks
Designation	Type	Manufacturer	Body	Disk	Stem Seal	Used	Used	
<u>Service:</u> N ₂ (Room Temperature; Pressure = 0-40 psig) (Continued)								
Fig. No. 150 ¹	Pressure Safety	Scott	Brass	Brass	Metal to Metal	2	1/2"	
Fig. No. 1020 ¹	Pressure Safety	Farris	Brass	Brass	Metal to Metal	2	1/2"	
Model KDP	Pressure Safety	Loneghan	Brass	Brass	Metal to Metal	1	1/2"	
Sho-Rate "50"	Flo-Mite (Rotameter)	Brooks	Anodized Aluminum	S. S.	Teflon	30	1/8"	Maximum Pressure was ~ 10 psig
No. 132-G	Flow-Rator	Fischer-Porter Co.	Zemak	S.S.	Plastic Gasket	11	1/8"	Maximum Pressure was ~ 10 psig.
<u>Service:</u> Freon-11 (Temperature = ~-60 to 120°C; Pressure = 15-35 psia).								
No. 2031	Globe	Henry	Bronze Alloy	Bronze Alloy	Packed	4	1-3/8"	
No. 6261	Diaphragm	Henry	Bronze	Spauldite or Micarta Insert	S.S. Diaphragm	1	1/4"	
No. R-2402	Diaphragm	Kerotest	Bronze	Bronze	S.S. Diaphragm	2	1/4"	

1 Available catalogs in the pilot plant do not show this figure number.

2 From 2 M KOH to 1 M KOH and 1 M KF.

3 Room temperature varied from ~ 5°C to 35°C.

4 Off Gas: N₂ with < 100 ppm F₂.

5 (46, pp. 16, 51).

a. Crane SMMD (52, p.16) and SSD

These valves were used extensively, SMMD's in fluorine service and SSD's in nitrogen and vacuum service. The only difficulties with them arose because the gas stream contained NaF fines.

1. Difficulties

- (a) In one SMMD (V-72), NaF fines eroded the disk sufficiently to necessitate replacement. Repair involved replacing the entire stem assembly because of the integral construction of the stem assembly.
- (b) Seat leakage in an SSD resulted from NaF fines depositing internally so as to prevent normal valve closure. The deposit was removed by vacuuming out the fines. Proper valve orientation to avoid recesses subject to deposits would have prevented this happening.

2. Advantages

- (a) Excellent for gas and vacuum service.
- (b) Amenable to lapping disk into seat to reduce leakage.^a (SMMD disk could be used whereas SSD would require an auxiliary disk.)

3. Disadvantages

- (a) Socket-weld ends.^b
- (b) Replacing either the disk or bellows would require changing the entire bonnet assembly.
- (c) Subject to closure interfering deposits if misorientated.

b. Hoke No. 1197

One of these valves did not pass gas even in the fully opened position. Inspection revealed that no opening through the valve existed because one of its ports had been incompletely drilled at the factory.

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^aNo maximum permissible leak-rate standard was established for these valves although 2 cc/min, that for the HCV's, could probably have been achieved.

^bButt-weld joints were preferable to socket-weld joints.

c. Check Valve Near PI-43

This valve was on the nitrogen supply to the fluorine station and was supposed to prevent fluorine from entering the nitrogen system. A tight shut-off of such a valve cannot evidently be expected at the low ΔP 's (~ 5 -50 psig) existing across the valve.^a Consequently, another means to obtain tight shut-off should be tried. In late work, manually operated "on-off" valves were used in this line in addition to the check valve. This scheme gave the desired tight-shut-off but did not offer the versatility of a check valve.

d. Safety Valves in Nitrogen Service.

A small leak in these valves could never be eliminated. So far, the small leak^b has been considered economically insignificant. It is probable, however, that a more elaborate and consequently more expensive safety valve would give a tighter closure if a nearly no-leak situation were necessary.

e. Valves on Nitrogen Purge Line Rotameters

These valves were inadequate in that they (1) did not afford a nearly no-leak shut-off, and (2) were unsuitable for both UF_6 and fluorine. The experiences with these purge line rotameter valves are given here as a record only since neither a no-leak shut-off nor compatibility with UF_6 and fluorine was expected.

All cases in which UF_6 and/or fluorine flowed into a purge rotameter resulted from maloperation and necessitated replacing the rotameter. In fact, at one stage of the operation early in the "E" runs such occurrences were so prevalent that control measures were taken to eliminate them. The first such step was installing Hoke No. 413 valves at the Penthouse floor on the process-side of the purge rotameters which could be used as needed to prevent process gases from reaching the purge rotameters.^c Since this step proved to be less positive than that required, certain instrument connections were capped off in the heated duct.^d

f. Valve on a UF_6 Cylinder (Superior No. 5665)

One of these valves developed a leak after filling with UF_6 . The Teflon packing in this valve was successfully replaced using two precautionary measures: (1) the cylinder was maintained cold with dry

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^aPrivate communication.

^bProbably < 5 cc/min.

^cFailures occurred in leaded yellow brass fittings used to install these valves as delineated in (50,7).

^dInstruments put out-of-service were: FE-5, -6, and -7; PE-12, -13, -14, -15, and -38 (Secs. 9.4.7c and 9.4.7d).

ice to reduce the vapor pressure of UF_6 ; (2) the void space above the UF_6 in the cylinder was filled with nitrogen to reduce the contact of UF_6 with air.

In several cases, closing the UF_6 cylinder valves was prevented by UF_6 solidifying internally in the valve. These situations were remedied by heating the valves. In early work, heat was supplied with a torch. In later work after the situation had been established as a chronic one, heating was done with heat lamps (Sec. 10.4.4).

g. Four Valves Developing Plugs in UF_6 Service

1. A Hoke No. 413 valve in the product pigtails plugged. This valve developed a port plug during product collection in the early stages of pilot plant work even though the temperature of the valve was kept above 65°C . The plug was probably caused by the deposition within the valve of reaction products of UF_6 with air and/or water vapor. Very likely the small diameter of the Hoke No. 413 opening rendered this valve particularly amenable to such a plug. Further study of this situation, however, was not made.
2. A Hoke No. 413 (V-126) valve in the FV-103 bypass also plugged. This valve presumably became plugged with UF_6 reaction products and/or nonvolatile fluorides during fluorination in the early "L" runs. In this case, the conditions of use were somewhat uncertain because the early "L" runs had frequent operational troubles. As for the Hoke No. 413 in the product pigtails the valve plugged in spite of being maintained above 65°C with industrial heating cable.^a The probable remedy was to replace this valve with one having a larger opening. Consequently, a 1/4-in. SMMD valve was substituted for it. However, the SMMD valve had not been service-tested because no subsequent occasion to use the FV-103 bypass arose.
3. Both a Hoke No. 1193 and a Superior No. 5665 valve developed plugs during the cold trap and absorber studies. Little information exists regarding these plugs. Observations included, however, three noteworthy items: (a) the plugs could not have been pure UF_6 because the valves were kept above 65°C ; (b) more than 75 pounds of UF_6 had already been transmitted through both of these valves before the plugs formed; (c) once plugging started the flow of UF_6 from that cylinder was never again successfully maintained although several attempts were made to do so. These observations seem to point toward one conclusion, that the UF_6 cylinder contained UF_6 reaction products and/or foreign materials. Two likely sources of the extraneous materials were the cylinder "as received" from K-25 and water vapor from air entering the cylinder and forming UO_2F_2 during temperature cycling while the first 75 pounds of UF_6 was^a being used.

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^aThat is, safely above the sublimation point of UF_6 (57°C at 14.7 psia) (17, p. 4).

The packed valves in N₂ service were sources of insignificant continuous leaks. Although these were considered economically insignificant in VPP, they might become significant at a higher manifold pressure or in a system containing more valves.

After the "L" runs, most of the conventional manually operated valves were taken to the burial ground along with associated pieces of equipment (Sec. 23.4.16b).

17.4.2 Remotely Operated "On-Off" Valves (HCV's)

a. Description of HCV's

Eighteen 1/2-in. and eight 1-in. HCV's were used in the VPP^a. These valves were manufactured by the Associated Valve Engineering Company, Chicago, Illinois. Since the designs of the 1/2-in. and 1-in. were similar, the cross-sectional view of the 1-in. valve shown in Fig. 17.1 gives the essential design features. Certain good and poor design items are apparent.

b. Good Design Features

1. All Monel construction provides for corrosion resistance in UF₆ and fluorine.
2. The disk-seat closure is metal-to-metal and of the line-contact variety. Line contact between disk and seat is capable of providing low leak rates in gas service.
3. The bellows eliminated using stem packing which might be attacked by UF₆ or fluorine.

c. Poor Design Features

1. Disk and seat of the same material. On closure one would not deform the other, a situation which may tend to aggravate the tight-closure problem.
2. Inadequate provision for guiding the stem. This may allow the valve disk to contact the seat in a variety of positions. Such a situation can produce a number of leak-rates, one in one position, another in a second, and so on.
3. Undesirable main bonnet joint seal and unnecessary additional joints. The main bonnet seal scheme involved tightening threads to provide contact between a circular raised face and a flat annulus. This design was considered less desirable than bolted flanges with a suitable

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^aSee Drawings Nos. D-33761 and D-31350 for location of HCV's and VPP Engineering File Folders Nos. F-13 and F-14 for other pertinent information. (52, p. 51).

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gasket such as copper.^a In addition to the main bonnet seal, a joint above the bellows also existed, the seal for which depended on tight contact of flat annuli. This seal could probably have been more positively made by welding.

4. Cast body. Because of the notorious porosity of castings, bar stock is preferred for body construction.
5. Valve welded into the pipeline. Welding the valves into the system is superior to flanging from the leak-possibility standpoint. But welding in this case forced body and seat maintenance in the field because of the difficulty and expense involved in removing and replacing the valves. Therefore, the desirability of welding the valves in place is questionable.
6. Threaded joint between disk and stem with no locking mechanism. The threaded joint between the disk and the stem is a poor method of fastening the two together. Such a joint may work loose during operation. Then, after working loose, any subsequent valve operation may result in the disk striking the seat in a variety of ways. In such seating, either a variety of contact rings is worn on the disk or uneven wear of the seat may effect seat leakage.

d. Initial Installation of HCV's

The initial installation of HCV's brought some unexpected problems:

1. Several valves leaked either through the seat or through the body.^b The cause(s) of the seat leakage was not readily ascertained. On the other hand, however, body leakage resulted from the porosity of the cast bodies.
2. Welding the valves into the system was thought capable of warping the valve because of the heat developed in welding. Such warpage might ruin the entire valve. Therefore, the following two precautions were taken in welding: (a) the bonnet assembly was removed and (b) the valve body was cooled with dry ice or a damp cloth. Since all of the valves were later rendered leak-free, these precautions were apparently effective in eliminating valve warpage from welding-in-place.

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^a Kel-F or Teflon are not specified because both reacted with pure fluorine under ORNL test conditions. (For a description of these tests, see 17.4.2,f.3, and 15.4.1b.)

^b These leaks were detected by maintaining 30 psig air pressure on one side of the valve with the valve closed and immersed in water.

3. The anticipation of seat leakage resulting from the expected gradual loosening of the disk was responsible for the decision to weld the disks to the stem. This step was deemed risky in view of the possible concomitant disk warpage. In spite of this disadvantage, however, and since Mr. Schubert of Aveco reported satisfactory experience with welding the disks, this welding was done (55).

e. Developing and Maintaining HCV's

HCV maintenance work was divided into two parts based on the crafts performing the maintenance. Initially, for the first half-year, maintenance was done by a machinist and a pipefitter. Later and until the present, these valves were serviced by a second machinist and an instrument mechanic.

The work in these periods was to some extent developmental. During the first period, there was little experience at ORNL with this particular remotely operated valve. During the second period, although a few of the earlier maintenance practices were continued, independent thinking and somewhat different techniques were used in order to gain a wider experience with the valves.

1. Initial Period

In the initial maintenance work, three schemes were tried more or less simultaneously to render the valves leak-free and to lengthen the leak-free period:

- (a) The disks which had been welded to the stems were cut off, turned to the original conical shape on a lathe, and fastened to the stem as before welding. This step was taken because the warpage occurring in the conical surface during welding was apparently effecting seat leakage.
- (b) Disks with integral bottom guides (B. and F. in Fig. 17.2) were used. Such a disk kept the stem axis nearly on a vertical straight line as the valve opened and closed by the action of the guides in the valve seat. In this way, the disk always contacted the seat in very nearly the same way thereby eliminating a number of different disk-seat contact lines. This technique tended to eliminate one of the original objections to the valves, that is, the inadequacy of the "as received" stem guiding.

Care was exercised in designing the guided disk to prevent an excessive pressure-drop through the valve. This was achieved by cutting out passageways through the guides as for B. in Fig. 17.2. The pressure-drop limit per valve was arbitrarily set at 1/2 psi for both 1/2" and 1" valves. At these pressure drops, the flow rates indicated in Table 17.2 were obtained. The flow rate of about 5 scfm obtained with

1-in.
Valve

A

B

C

D

1/2-in
Valve

E

F

G

H

I

Fig. 17.2. HCV Disks.

Table 17.2

Flow Rates Through 1/2-in. and 1-in. HCV's with a 1/2-psi Pressure Drop

Flow Rate, scfm

Gas Flowing	Disk with no Guide	Disk with Cut-Out Guide	Disk with Solid Guide
N_2	20 ¹	$\frac{1'' \text{ HCV's}}{11}$	7
UF_6^2	Not determined	6	4
N_2	12	$\frac{1/2'' \text{ HCV's}}{8}$	8
UF_6^2	6	4	4

1. With no ΔP .

2. The data were obtained with nitrogen. To obtain UF_6 data, N_2 data were converted to UF_6 data by calculation.

the guided disks in both the 1/2" and 1" HCV's was adequate by a several-fold factor for use in the plant; therefore, the pressure drop through one valve during normal operation (<1 scfm) would be much less than the selected figure of 1/2 psi.

- (c) Line contact between the disk and the seat was re-established each time a valve was lapped. In this method, two fluted cutters for heavy cuts and two grades of lapping powder for the finishing work were used. One of the cutters contained cutting edges at 30° to the horizontal while the other contained 45° blades. The lapping procedure was:
- (1) By using the two fluted cutters properly centered with a jig, a circular edge was made on the valve seat.
  - (2) The Monel disk retained on a shaft centered by a jig as for the cutters was lapped lightly into the seat using the coarse compound,^a 400-mesh diamond dust compounded with a suitable vehicle.
  - (3) The disk was relapped similarly into the seat using the fine compound,^a 600-mesh diamond dust mixed with a suitable carrier.
  - (4) The excess lapping compound was wiped off the seat and disk with acetone.
  - (5) After the residual acetone had been allowed to vaporize, the valve was reassembled.
  - (6) The spring-tension,^b 14 psig for 1" valves and 12 psig for 1/2" valves, was set, and the valve was leak-tested in place in the plant.

Valve leak-testing consisted of two parts: (a) The seat-leakage was determined. (b) After seat-leakage was satisfactory, bonnet leakage was investigated.

For both tests, arbitrary specifications were established. For seat-leakage, this specification was <2 cc/min; for bonnet-leakage, the criterion was that no Freon could be detected with the Freon flame detector while the valve contained Freon at 30 psig.

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^aBoth compounds were labeled "Dymo" as prepared by the Abrasives Division of the Elgin National Watch Company, Elgin, Illinois. Both were available in ORNL Stores.

^bLower spring tensions seemed to be incapable of giving the desired seat leak rate. Spring tension was that pressure of air supplied to the diaphragm required to completely open the valve.

The seat-leakage test was performed with the valve closed as follows:^a

- (1) Nitrogen at a predetermined pressure was applied against one side of the valve while the other side of the valve was at atmospheric pressure.
- (2) A soap film was placed on a 1/2" or 3/8" tubing nipple connected to the atmospheric pressure side at some convenient location.
- (3) The size of bubble blown by this tube in a given length of time was noted.
- (4) The valve was opened to allow N₂ escape through the nipple to confirm that N₂ pressure had actually been against the valve seat during the test.
- (5) Leak rate was calculated from bubble diameter and time data.

Although Step (4) might seem unnecessary, it was deemed necessary as a check because of the complexity of the plant piping. In fact, on several occasions test data were discarded when this check revealed that no pressure had been against the valve seat.

The frequency of HCV maintenance during this period can be determined only through data taken between series of runs because valve maintenance was done only between run series. Usually each series covered five to ten runs, and each series was started with all of the valves leak-tight by the specification given above. After the series of runs and before water-washing the system, generally about half of the valves would leak. After water-washing, usually a few more of the valves leaked than before. In a few cases during a series of runs, valves were suspected of developing leaks. Therefore, some of the valves which were found to leak at the end of run series apparently developed leaks before the end of the series.

2. Later Period

During this period, the HCV development and maintenance program was altered. Changing the program was an effort to utilize the experience of Instrument Division personnel in developing and maintaining the HCV's. Several innovations and alterations were initiated:

---

^a"T" connections were provided at several points in the system so that the valves could be leak-tested in place.

- (a) The guided disk was replaced with an unguided one, and a new design disk having Kel-F contained in Monel framework as for disks C and G in Fig. 17.2 was used extensively.^a
- (b) The lapping procedure was altered. Materials employed during lapping were: first, an HCV stem and bellows subassembly for supporting and centering the disk; second, a Monel disk for the lapping surface. (This disk was the one to be subsequently used in the valve when the valve contained a Monel disk, or this disk was a Monel disk with a relatively smooth conical surface when the valve contained a Kel-F disk.); and finally, lapping compounds - for roughing No. 303-1/2 and for finishing No. 39-1200, both compounds being manufactured by United States Products Company, 518 Melwood Avenue, Pittsburgh, Pennsylvania.^b

The lapping procedure was:

- (1) The bonnet was removed with the valve open to avoid seat-disk damage.
- (2) Both seat and disk were cleaned, usually with acetone but sometimes with methylene chloride to reduce the fire hazard existing when rubbing a fluorinated surface with an acetone-soaked cloth (Sec. 15.4.5b).
- (3) The Monel disk was fixed onto the stem and bellows subassembly and then a small amount of roughing compound was smeared on the conical surface of the disk and on the surface of the seat.

NOTE: The roughing compound was used only on valves having badly scored seats.
- (4) This stem-bellows-disk subassembly was fitted to the valve body as usual by engaging the bonnet threads.
- (5) Then, by placing one hand on top of the valve stem to hold the disk against the seat, the seat and disk surfaces were rubbed against each other by moving the stem back and forth with the other hand through an angle of about 15 to 30 degrees. Occasionally, to get evenness in lapping, this back-and-forth motion was stopped and the stem was lifted and moved through an angle of from 45 to 180 degrees.

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^a Kel-F disk designs were available as ORNL Drawing No. Q-1679-44-R0 for 1/2" HCV's and Q-1679-45-R1 for 1" HCV's.

^b Neither of these preparations was available in ORNL Stores.

Then, the lapping was resumed as before. To determine the progress of lapping, the excess lapping compound on the seat was removed with the solvent. At this time, the machinist decided whether to resume the roughing operation or to proceed to the finishing step. The length of roughing time depended primarily on the judgment of the machinist.

- (6) In finishing, the same general method was used as in roughing.
- (7) Following the finishing operation, the seat and disk were cleaned as before. It was very important, moreover, that this cleaning be scrupulous because if the valve did not leak after assembly it was then conditioned with fluorine and put into fluorine service, (Sec. 15.4.5b).
- (8) The valve was then reassembled and the spring tension set at 12 psig. Before assembly, the Monel disk was replaced with a preconditioned Kel-F disk if the valve was to contain a Kel-F instead of a Monel disk.^a On the other hand, if the valve was to contain a Monel disk, the lapped-in disk was used. See Sec. 17.4.2, e.1, for the definition of spring tension. Extreme care during lapping was exercised to avoid enclosing foreign materials in the system. The term "foreign materials" embraced any substance such as cellulosic matter, finger prints, greasy spots, metal turnings, and the like. The two main reasons for this precaution were: first, fluorine reacts violently with many materials, especially those of organic origin and second, any hard material, for example, metal turnings, might be blown through the system and possibly at some time interfere with valve closure and/or score valve seats. (At least one case in which metal turnings have scored a valve disk has occurred.)
- (c) The KI-starch method of leak testing was used on the valve bonnet.^b The procedure was:
  - (1) A fresh KI-starch solution was made by dissolving about one-third of a teaspoon of soluble starch and the same amount of C. P. KI crystals in a half-pint of demineralized water. (Note: The water should not contain free chlorine or other substances which liberate free iodine from KI; otherwise, the color of the solution will be midnight blue. Such a coloration renders the solution useless since it indicates a positive test.)
  - (2) The valves for bonnet checking were opened, and the pertinent portion of the system was pressurized dynami-

^aCf. Sec. 17.4.2, f.4 regarding pre-conditioning Kel-F disks.

^bThis method was developed by G.I. Cathers and S. Mann for use generally with joints in the VPP. The necessary care to avoid the false indication by Fe^{3+} reacting with KI should be exercised.

cally^a to 10 to 15 psig with fluorine.

- (3) A strip of soft tissue paper such as Kleenex long enough to entirely cover the bonnet joint and wetted with the KI-starch solution was placed on the joint.
- (4) After this strip had been in place for about a minute, it was removed. A midnight bluish coloration of the paper indicated a leak. In the event that the piece of tissue was not entirely discolored, a leak existed only over a portion of the joint. In such cases, the exact location could be fairly well ascertained. No work directed at determining the sensitivity of this test has been attempted. It was found, however, to be as sensitive as the soap bubble method with 100 psig N₂ (Sec. 19.4.2). The joint to be tested should be clean to avoid the interference of extraneous materials with the test.

The frequency of lapping was on the order of every 6 to 9 runs during the second maintenance period. The wear on the Kel-F disks in 6 to 9 runs was about half-way through the Kel-F or as indicated by disks D and H in Fig. 17.2.

It was necessary to replace a bellows in only one HCV valve. The bellows failure evidently resulted from poor maintenance practice such as trying to remove the bonnet with the valve shut. The repair work necessitated replacing the entire stem assembly with that from another valve.

HCV-22 developed a bonnet leak in Run E-2 (Sec. 7.4.2). This leak was eliminated by lightly dressing the bonnet contact surfaces and then replacing the bonnet.

f. Tests with Kel-F and Monel Disks in HCV's^c

Several tests using HCV's 18, 34, and 36 fitted with Kel-F disks were made during the later period of valve maintenance and testing. The purpose of this work was three-fold: first, to determine the expected life of Kel-F disks; second, to determine whether a Kel-F fitted valve will start to leak as the Kel-F wears, and third, to determine whether Kel-F disks would self-heal by cycling the valve.

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^aDynamically instead of statically to avoid pressure leak-off while leak-testing. Otherwise, the system should be monitored continuously while testing.

^bCf. Sec. 17.4.2f for details of the wear obtained during Kel-F disk test-work.

^cThe results of the first endurance tests on Kel-F disks which were made by L. H. Chase of the Instrument Division are not reported here.

1. Test Conditions and Procedures

The following endurance tests were made:

- (a) Test 1_a was performed at room temperature with unconditioned disks.
- (b) Test 2 was performed at normal heated duct temperatures with unconditioned disks._{a,b}
- (c) Test 3 was performed at normal heated duct temperatures with preconditioned disks._{b,c}

The valves tested at room temperature simulated conditions for valves outside of the heated duct and those tested at normal heated duct temperatures simulated conditions in the heated duct. A test at room temperature with preconditioned disks was not made because wear under such conditions was considered less severe than that for preconditioned disks at normal heated duct temperatures.

The procedure in all endurance tests was:

- (a) The valves were fitted with Kel-F disks and rendered leak-tight at the temperature of the test and at the specified test pressures.
- (b) Each valve was fully opened and closed, or cycled, 100 times and retested for seat-leakage.
- (c) Each valve was cycled 100 more times and again tested for seat leakage.
- (d) Each valve was disassembled for disk and seat inspection. A seat inspection was done only in Test 1.

One self-healing test (Test 4) was made by cycling leaking HCV's fitted with Kel-F disks. Periodically, the cycling was stopped to check the seat leakages.

2. Test results and discussion. The results of the valve tests are presented in Tables 17.3, 17.4, 17.5, and 17.6.

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^aThat is, disks without preconditioning with fluorine.

^bNormal average heated duct temperature data were: (1) 130°C near HCV-18, (2) 150°C near HCV-34, and (3) 145°C near HCV-36.

^cPreconditioning was done per procedure given in Sec. 17.4.2,f.4.

Table 17.3

ENDURANCE TESTS OF UNCONDITIONED KEL-F DISKS AT ROOM TEMPERATURE^aTest 1

Spring Tension: 9 psig

Dates: 9-18, -19, 1957

← Valve Data →

HCV No.	Size	Test Pressure psig	← Leak Rate, cc/min, ^b at Indicated → Number of Cycles		
			Zero Cycles	100 Cycles	200 Cycles
→ 18 ^c	1/2"	50	4	7	9
← 18 ^d		50	<2	<2	<2
→ 34	1"	10	<2	<2	<2
← 34		10	<2	<2	<2
→ 36	1/2"	10	<2	<2	<2
← 36		10	<2	<2	<2

Appearances of Disks After Test 1

The actual wear was difficult to assess, probably being $<1/64$ "; little cracking, splintering, or peeling of Kel-F was apparent. Disk photographs were made as follows: HCV-18, ORNL No. 41723 and No. 41724; HCV-34, ORNL No. 41721 and No. 41722; and HCV-36, ORNL No. 41725 and No. 41726.

Appearances of Seats After Test 1

HCV-18 had a rough seating surface but not as rough as for HCV-36; HCV-34 had a smooth seating surface; and HCV-36 had a rough seating surface with nicks. Photographs of the seats were not made.

^aRoom temperature: $\sim 10-30^{\circ}\text{C}$.

^bWhen leak rate is given as <2 cc/min, this indicates a soap bubble of $<1/2$ " diameter formed in 30 sec.

^c→ 18 indicates that leak-test was made in same direction as arrow on HCV-18 valve actuator at Main Panelboard pointed when valve was open. A similar scheme was used for the other valves.

^d← 18 indicates that leak-test was made in the reverse direction to that which arrow on HCV-18 valve actuator at Main Panelboard pointed when valve was open. A similar scheme was used for the other valves.

Table 17.4

ENDURANCE TESTS OF UNCONDITIONED KEL-F DISKS AT NORMAL HEATED DUCT TEMPERATURES^aTest 2

Spring Tension: 9 psig

Date: 10-16-57

HCV No.	Size of Valve	Test Pressure psig	Temperature in Vicinity of Valve, °C	Leak Rate, cc/min, ^b At Indicated Number of Cycles		
				Zero	100	200
$\overrightarrow{18^c}$	1/2"	50	130	<2	<2	<2
$\overleftarrow{18^d}$		50		<2	<2	<2
$\overrightarrow{34}$	1"	10	150	<2	<2	<2
$\overleftarrow{34}$		10		<2	<2	<2
$\overrightarrow{36}$	1/2"	10	145	<2	<2	<2
$\overleftarrow{36}$		10		<2	<2	<2

Appearances of Disks After Test 2

Wear was much worse than in Test 1, being ~1/16 in. with some evidence of cracking, splintering, or peeling of Kel-F. Disk photographs made were: HCV-18, ORNL NO. 41889; HCV-34, ORNL No. 41890; and HCV-36, ORNL No. 41891.

Appearances of Seats After Test 2 were not determined.

^aNormal average heated duct temperature data were: (1) 130°C near HCV-18, (2) 150°C near HCV-34, and (3) 145°C near HCV-36.

^bWhen leak rate is given as <2 cc/min, this indicates a soap bubble of <1/2" diameter formed in 30 sec.

^c $\overrightarrow{18}$ indicates that leak-test was made in same direction as arrow on HCV-18 valve actuator at Main Panelboard pointed when valve was open. A similar scheme was used for the other valves.

^d $\overleftarrow{18}$ indicates that leak-test was made in the reverse direction to that which arrow on HCV-18 valve actuator at Main Panelboard pointed when valve was open. A similar scheme was used for the other valves.

Table 17.5

ENDURANCE TESTS OF PRECONDITIONED KEL-F DISKS AT NORMAL HEATED DUCT TEMPERATURES ^a

Test 3

Spring Tension: 9 psig

Dates: 10-29, 30 - 57

HCV No.	Size of Valve	Test Pressure psig	Temperature in Vicinity of Valve, °C	Leak Rate, cc/min, ^b at Indicated Number of Cycles		
				Zero	100	200
$\overrightarrow{18^c}$	1/2"	50	130	<2	<2	<2
$\overleftarrow{18^d}$		50		<2	<2	<2
$\overrightarrow{34}$	1"	10	150	<2	<2	<2
$\overleftarrow{34}$		10		<2	<2	<2
$\overrightarrow{36}$	1/2"	10	145	<2	<2	<2
$\overleftarrow{36}$		10		<2	<2	<2

Appearance of Disks After Test 3

Wear was probably a little worse than in Test 2, being $\geq 1/16$ " with much more evidence of cracking, splintering, or peeling of Kel-F than in Test 2, especially for HCV-18. Disk photographs made were as follows: HCV-18, ORNL No. 41935; HCV-34, ORNL No. 41936; and HCV-36, ORNL No. 41937.

Appearances of Seats After Test 3 were not determined.

^aNormal average heated duct temperature data were: (1) 130°C near HCV-18, (2) 150°C near HCV-34, and (3) 145°C near HCV-36.

^bWhen leak rate is given as <2 cc/min, this indicates a soap bubble of <1/2" diameter formed in 30 sec.

^c $\overrightarrow{18}$ indicates that leak-test was made in same direction as arrow on HCV-18 valve actuator at Main Panelboard pointed when valve was open. A similar scheme was used for the other valves.

^d $\overleftarrow{18}$ indicates that leak-test was made in the reverse direction to that which arrow on HCV-18 valve actuator at Main Panelboard pointed when valve was open. A similar scheme was used for the other valves.

Table 17.6

SELF-HEALING DATA AT ROOM TEMPERATURE ON LEAKING HCV'S
FITTED WITH UNCONDITIONED KEL-F DISKS^a

Test 4

Spring Tension: 9 psig			Disks: Unconditioned Date: 9-18-57						
HCV No.	Size of Valve	Test Pressure, psig	Leak Rate, cc/min, at Indicated Number of Cycles						
			Zero	5	10	20	40	80	160
$\overrightarrow{14^c}$	1"	50	26	51	51	51	51	17	26
$\overleftarrow{14^d}$		20	2	51	0	0	0	0	0
$\overrightarrow{18}$	1/2"	50	17	15	26	15	15	21	N.D. ^b
$\overleftarrow{18}$		50	34	34	26	34	34	34	N.D.
$\overrightarrow{22}$	1/2"	20	~300	170	170	170	170	170	170
$\overleftarrow{22}$		50	>500	>500	170	170	170	170	64

^aRoom temperature: ~10-30°C.

^bN.D. = Not Determined.

^c $\overrightarrow{18}$ indicates that leak-test was made in same direction as arrow on HCV-18 valve actuator at Main Panelboard pointed when valve was open. A similar scheme was used for the other valves.

^d $\overleftarrow{18}$ indicates that leak-test was made in the reverse direction to that which arrow on HCV-18 valve actuator at Main Panelboard pointed when valve was open. A similar scheme was used for the other valves.

(a) Results of Test 1, 2, and 3

In hot and cold tests with both conditioned and unconditioned Kel-F disks, 1/2-in. as well as 1-in. valves remained leak-tight for 200 cycles with one exception. This exception was HCV-18 in Test 1, which was not leak-tight initially and which leaked worse after cycling.

The wear on the disks was most severe (probably $\geq 1/16$ -in. deep) for the preconditioned disks in the hot test, Test 3. In spite of the wear suffered by these disks, no disk failure occurred. Some cracking, splintering, or peeling of the Kel-F did take place, however, this spalling of the disks was apparently worst for the preconditioned disks in the hot test. The desired leak rate of < 2 cc/min was achieved with both smooth and rather rough-looking seats in Test 1.

(b) Results of Test 4

The self-healing test results from Test 4 were erratic. For instance, HCV-14 exhibited healing in the reverse direction only. In addition, HCV-22 seemed to show a tendency to heal whereas HCV-18 showed no tendency to heal.

(c) Discussion of Test Results and Additional Information

The results of Tests 1, 2, and 3 indicated that both 1/2-in. and 1-in. HCV's with either preconditioned or unconditioned disks used at room temperature or at $\sim 150^\circ\text{C}$ will remain leak-free for more than 200 valve cycles. The wear on the Kel-F during 200 valve cycles was most severe for the preconditioned disks at $\sim 150^\circ\text{C}$. Even at this worst wear rate, the useful life of a disk should probably be several times the 200 cycles used in these tests.

This expected life is based on the $\sim 1/16$ " wear in Test 3 and on the thickness of the Kel-F at the point of wear which is $1/8$ " for both 1/2-in. and 1-in. disks. In this projection of wear, it was assumed that:

- (1) Wear varies linearly with the number of valve cycles.
- (2) Wear would not exceed one-half of the Kel-F thickness available for wear.
- (3) The valve would not leak before this "half-thickness" of Kel-F available for wear was worn through.

In practice during an average series of about nine runs, the worst wear on Kel-F disks was about the same as that in Test 3 as shown by disks D and H in Fig. 17.2. This fact might indicate that the most

used valve was cycled about 200 times during a run series. But a cross-check from runsheets revealed that the maximum number of cycles during a series of nine runs would be about 80 (with 100% excess cycling). Thus in a 9-run series to obtain wear equivalent to 200 cycles would require either a greater wear rate in practice or about 500% excess cycling. Either of these explanations is possible. But the main consideration here is that data obtained both through endurance testing and experience indicated the adequacy of the Kel-F disks for a 9-run series.

The cracking, peeling, or splintering of the pre-conditioned disks might effect seat leakage earlier than otherwise because fissures might form across the seat contact line. There was no concrete evidence during operations, however, that spalling reduced disk life.

The conditions of the valve seats in Test 1 might lead to the conclusion that using Kel-F disks would enable obtaining leak-free valves with both smooth and rough seats. This conclusion has not been substantiated.

The data in Test 4 most likely indicated that valves with Kel-F disks which leaked originally would not heal on cycling.^a In spite of the fact that little work along this line has been done, additional effort is not recommended because the test results were not encouraging.

The variability of some of the data in Tests 1 and 4 may cause a question of the reliability of the leak-testing procedure. The point merits mention when the two following factors which may influence the results are considered: (1) the possibility of unknown sources of gas flow in such a complex piping system and (2) the difference in technique used by different operators. Even though the existence of either of these two factors may produce spurious results, only the second factor is believed to be of any significance in these four tests. The effect of this factor is difficult, if not impossible, to assess. The reason that the adverse effect of unknown gas sources on the leak-rate data is discounted here is that such gas supplies usually produced abnormally high leak rates which were not observed in these tests.

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^a That is, the leak would not disappear.

3. Monel and Kel-F Disks in a Variety of Service Conditions

During the pilot plant operations, the HCV's with either Monel or Kel-F disks were maintained leak-free in a variety of service conditions. The service variables were: material handled, temperature, and time. The materials handled were UF_6 (in solid, liquid, or gaseous state) F_2 , and N_2 ; the temperatures varied from ambient cell temperatures of around $10-30^\circ C$ up to about $150^\circ C$; the exposure time ranged from about 35 hr to 150 hr.

Both Monel and Kel-F valve disks were used in all combinations of the service variables with one exception.^b This exception concerned using Kel-F disks at heated duct temperatures ($\sim 150^\circ C$) in pure fluorine supplied at room temperature. In HCV-12 which was exposed to pure F_2 at times, for instance, two Kel-F disks have disintegrated. The first such occurrence initiated the preconditioning of the disks with fluorine prior to installing in the system.^c At first such preconditioning was thought to be the remedy. However, subsequent operating experience in which a supposedly preconditioned Kel-F disk was destroyed in HCV-12 led to the conclusion that Kel-F was unsuitable for pure F_2 service regardless of whether preconditioned. A qualification is necessary because it is possible that the preconditioned Kel-F disk might have had some foreign material on its surface which reacted with fluorine, thereby initiating the reaction between fluorine and the Kel-F. At any rate, in the VPP work, replacing the disintegrated preconditioned disk was costly enough to take no further chances. In future work, only Monel disks were used in pure fluorine service.

4. Preconditioning of Kel-F Disks with Fluorine

While preconditioning the first batch of Kel-F disks, several disks disintegrated when contacted with fluorine as soon as the temperature of the disks reached $100^\circ C$. To avoid this, it was necessary to hold the disks a length of time at the preconditioning temperature of $100^\circ C$ before exposing them to fluorine. The necessary length of the holding time was never determined exactly. Practice revealed, however, that 1 to 2 hours was adequate. Figure 17.3 shows the effect of preheating conditions on the reaction of Kel-F disks with fluorine.

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^aEstimated exposure time during a 9-run series for little used HCV's such as HCV-34 and for more frequently used HCV's such as HCV-18 or 19, respectively.

^bThe use of Kel-F in liquid UF_6 at K-25 was reportedly unsatisfactory. Our results indicated, however, that Kel-F disks were compatible with liquid UF_6 in the Volatility Pilot Plant.

^cSec. 15.4.6 and 17.4.2,f.4.

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Preheating Prior to F_2 Addition:
on Attaining $100^\circ C$.

Preheating Prior to F_2 Addition:
2 or more hours at $150^\circ C$.

-300-

Fig. 17.3. Effect of Preheating Conditions on the Reaction of Kel-F Disks with Fluorine.

The initial reaction leading to the disintegration during pre-conditioning was apparently that of fluorine and the plasticizer in the Kel-F. This reaction, in turn, produced sufficient heat to cause the fluorine and Kel-F to react. Regardless of whether the reaction partly or completely destroyed the Kel-F portion of the disk, the disk was useless. Probably the reason that the holding time at 100°C rendered the disk compatible with fluorine was that a smaller amount of the plasticizer was retained at the surface to react with fluorine.

Although the optimum preconditioning scheme has not been worked out, the following procedure has been used successfully:

- (a) Degrease disks briefly with C. P. carbon tetrachloride and allow excess solvent to vaporize.
- (b) Place disks in a container suitable for use with fluorine and start an inert gas such as nitrogen sweeping across the disks and thence to vent.
- (c) Heat container to 100°C and hold it at this temperature for 1 to 2 hours.
- (d) While continuing to maintain the container at 100°C, shut off N₂ flow gradually while starting F₂ flowing through the container at a slow rate.
- (e) Maintain fluorine flow and the container at 100°C for one-half hour.
- (f) Shut off heat allowing container to cool to room temperature while maintaining the fluorine flow.
- (g) When container is at room temperature, cut off fluorine and sweep chamber with N₂ for one hour before removing disks.
- (h) Remove disks with care to avoid contaminating Kel-F surfaces with foreign materials or fingerprints. Store disks in a dry place at room temperature until ready for use. Subsequent handling of disks should be with the same care as during unloading disks.

Preconditioning the disks as recommended apparently produced warpage or uneven shrinkage of the Kel-F. This warpage caused the conical surface of the Kel-F to depart from that of a right cone. This situation can best be described through the following test made on a lathe. With the disk mounted on the live center, the disk was rotated such that a feeler gage would describe a circle on the conical surface of the Kel-F if the Kel-F surface were truly conical. The described circle was near the place at which the seat would normally strike the disk. The results of this test indicated

that a true circle could not be described on many of the preconditioned disks. In fact, the described circle for some of the disks failed to contact the Kel-F surface by as much as 7 to 10 mils.

This situation was remedied by truing the conical surface on a lathe without any lubricant; otherwise, if a lubricant had been used, the disk would have had to be preconditioned again after machining.

Before warpage of the Kel-F was found to be the difficulty, much concern was expressed regarding the inability of preconditioned Kel-F disks to give a tight shut-off. And, since this difficulty came chronologically after tight shut-off with Kel-F disk had initially been attained, this problem was one of the most puzzling encountered in the HCV program.

The following factors in preconditioning Kel-F disks seem to be important: rinsing in carbon tetrachloride, time and temperature of preconditioning, and surface machining. The exposure to carbon tetrachloride should be brief so as to decrease the amount of this solvent sorbed by the Kel-F. This is important because both sorbed carbon tetrachloride and plasticizer must be removed during preconditioning. The time and temperature of preconditioning govern the depth of conditioning, the degree of surface warpage, and the amount of machining necessary to "true" the conical surface. The envisioned goal in preconditioning is to render the disks compatible with fluorine so that little or no machining is required. Such a scheme would necessitate minimal rinsing in carbon tetrachloride, time-at-temperature, and preconditioning temperature.

g. Additional Comments on HCV's

In the future, the following three considerations regarding HCV's might become very important:

1. Replacement of body or body components to eliminate leak(s). The HCV's lend themselves reasonably well to any type of replacement except for body replacement if spare parts are stocked. The body replacement would be difficult because installing the new body would require making two fully inspected Heli-arc welds.
2. Further attempts to prevent deposits on internal parts and salt backflow such as have occurred in HCV-7 and -8. Just what form future attempts to reduce the internal deposition of materials and/or to prevent salt backflow might take is unknown. Installing the CRP trap and water-washing the system periodically were attempts at decreasing the internal deposition of solids in the system generally. Various fluorinator design changes as well as perfecting operating techniques were aimed at preventing salt backflow near the fluorinator.

3. Replacement of HCV's with other valves. Whether to replace the HCV's with different valves is the most important decision in the field of remotely operated "on-off" control valves. The following three factors are of prime importance in making this decision:

First, whether the future experience with HCV's can be expected to parallel the past experience. Past experience with HCV's indicated that relatively satisfactory plant operation has resulted through a large expenditure of money for development and maintenance. The "relatively satisfactory" qualification refers to the fact that some valves developed leaks during a given run series, and that the "integral" nature of the valve made it more desirable to operate with the leaks rather than repair or replace the valves. In addition to the troubles in past experiences with HCV's, some of the poor design features not yet causing trouble might cause difficulties in the future.

Second, whether the advantages of maintaining valves in a shop rather than in the field would make replacing all HCV's attractive. New valves might be flanged into the system and, therefore, could be handled as units.

Third, whether the combined initial and maintenance costs for replacement valves would be less than that for maintaining the HCV's in the future. To resolve this consideration depends on the answer to the first factor, and on the maintenance requirement of the new valves.

The above considerations involved in replacing the HCV's have been raised without resolution because that decision is beyond the scope of this report.

After the "L" runs, the HCV's were decontaminated and delivered to the Instrument Division for reuse, (Sec. 23.4.15a and Table 23.4).

17.4.3 Remotely Operated Control Valves

Remotely operated control valves were used in the Volatility Pilot Plant to control F_2 and N_2 flow rates and also to control pressures at several points in the system. Table 17.7 lists construction data and service conditions for each control valve as well as an operational evaluation. The mechanics and design of each control mechanism are covered in the description of the VPP system containing that control mechanism.

Most of the remotely operated control valves were left in place after the "L" runs. If any are removed with equipment in the future, decontamination as for the HCV's (Sec. 23.4.15a) is planned along with subsequent delivery to the Instrument Division.

Table 17.7

REMOTELY OPERATED CONTROL VALVE DATA AND REQUIRED OPERATIONAL ATTENTION (46, p. 51)

Valve Number	Size	Manufacturer	Materials of Construction				Ease of Operation	Service Conditions		
			Body	Disk	Stem Seal	Bonnet Seal		Temp., °C	PSIG Pressure	Chemical Component(s)
FCV-1	1/2"	Research Controls	Forged Steel	316 S.S.	Monel Bellows	Teflon Gasket	Good	10 to 30	0-20	N ₂
FCV-2 Model No. F-75M	1/2"	Research Controls	Monel	Monel	Monel Bellows	Teflon Gasket	Poor ^a	-10 to +30	~0-30	F ₂ , N ₂
FCV-37A and -38A Model No. 3919 ABS	3/16"	Hammel Dahl	Brass	Kel-F	Brass Bellows with Teflon Gasket on one end and other end welded	Teflon Gasket	Good	-10 to +30	15-60	F ₂
PCV-2A and -2B Model No. 3919 ABS	1/2"	Hammel Dahl	Brass	Kel-F	on one end and other end welded	Teflon Gasket	Poor ^b	-10 to +30	15-60	F ₂
FX-37 and -38 Model FI-900	3/8"	The Gems Co.	Brass	Brass	None	Teflon Gasket	Good	-10 to +30	0-55	F ₂ , N ₂
PCV-10 Model No. F-75M	1/2"	Research Controls	Monel	Monel	Monel Bellows	Teflon Gasket	Poor ^a	-10 to +30	0-60	F ₂
PCV-39 Fig. 3978 ABP9	1-1/2"	Hammel Dahl	Monel	Monel	Monel Bellows	Teflon Gasket	Good	~30 to 50	~ 0	Scrubber Off Gases
PCV-45 FIG. 3978 ABP9	1-1/2"	Hammel Dahl	Monel	Monel	Monel Bellows	Teflon Gasket	Good	10 to 30	~ 0	N ₂
PCV-61 Type No. V-4	3/4"	Foxboro	304 S.S.	18-8 (304 S.S.)	Teflon V-Ring Packed	S.S. Gasket	Good	~ 50 to 100	5" H ₂ O Vac.	Waste Can Off Gases

^aCycling of the FCV-2 and PCV-10 combination caused considerable flow variation most of the time.^bSwitch-over from PCV-2A to PCV-2B or vice versa accentuated cycling of FCV-2 and PCV-10.

Operational difficulties occurring were:

- a. Components' failures in PCV-10 and FX-37. The two components' failures in PCV-10 and FX-37 are evidence that fluorine will react with both Kel-F and Teflon although the conditions existing in these cases cannot be specified.^a At any rate, the Kel-F disk of PCV-10 was destroyed by fluorine. In addition a Teflon washer in FX-37 disintegrated when a leak developed through a faulty seal in the switch. In both situations, pure fluorine was being handled. For PCV-10, the valve was replaced with one of all Monel construction. For FX-37, since the switch was replaced with a spare, Teflon was successfully used after the incident. In neither case has further trouble been experienced.

Another difficulty with both FX-37 and -38 was that a sudden high flowrate of fluorine caused the switch spindle to stick in the "open-circuit" position. In such a case, fluorine flow could not be resumed until the spindle was freed by rapping the switch sharply with a small tool. This situation was principally annoying but also potentially time consuming. For instance, without knowing that this might happen, much operating time might have been lost while investigating other possible causes of fluorine flow interruption.

- b. Undue cycling of the PCV-10 and FCV-2 combination. The cycling of the PCV-10 and FCV-2 combination was a problem during the entire processing period. Since there were always unsolved instrumentation problems of a more serious nature, this condition was never corrected. Early in the pilot plant work (i.e., before the "C" runs) a surge pot (FV-160) was added to the system in an attempt to reduce the fluctuations by adding surge capacity. This was a step in the correct direction in that it seemed to diminish the fluctuations. The effect was slight, however, and subsequently no further remedies were tried.

The automatic trailer switch-over through the action of PCV-2A and PCV-2B usually disrupted the operation of the PCV-10 and FCV-2 combination and almost invariably necessitated re-establishing the desired readings on PCV-10 and FCV-2. In many cases, leveling out the PCV-10 and FCV-2 readings required almost constant attention for a half-hour or more.

- c. The KOH backflow through PCV-39 was countered in two ways. First, jack-legs, an over-flow drum (FV-151), and a slope in the vent gases line were incorporated in the system as described elsewhere.^c Second, a

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^aSee Secs. 15.4.1b, 15.4.6, and 17.4.2,f.4 for other cases of Kel-F or Teflon reacting with fluorine. Details for the Kel-F fitted PCV-10 are not given in Table 17.7.

^bCf. Sec. 17.4.2f relative to using Kel-F disks in pure fluorine service.

^cSec. 11.4.1b.

modified PCV-39 system designed to control the vent line pressure was installed (Sec. 11.4.1b). The relative merits of one of these actions are unknown. But another KOH backflow has not occurred since these changes were made.

17.4.4 Freeze Valves

A freeze valve was a loop or bend in either 3/8-in. or 1/2-in. Schedule 40 NPS Inconel pipe with the seal being achieved by freezing salt in the low portion of the loop. The valve was "opened" by melting the salt, that is, by heating the entire autoresistance line in which the freeze valve was located to $\geq 570^{\circ}\text{C}$ (Sec. 21.3.2). The valve was "shut" by discontinuing flow and freezing the entrapped molten salt by cooling the entire autoresistance heated line to room temperature. The four different designs used with indicated regions of sealing are shown in Fig. 17.4. In addition to autoresistance heating, the 3/8-in. NPS valve (Mark V, Sec. 13.4.2) also required resistance heating to maintain the expanded sections at the desired temperature. The expanded sections were added to this valve to increase liquid retention for sealing purposes, as discussed under Sec. 13.4.2.

The Mark I (pot-type) freeze valve design tested in the Unit Operations section was never used in the VPP. The Mark II (Bull Moose) design, however, was thoroughly tested in the Unit Operations section and was used as both FV-106 and -108 in the VPP. One valve of this type has been tested through 225 cycles of freezing and thawing with a gas pressure of 20 psig applied after each cycle without leakage (determined by gas collection) or dimensional change (determined by micrometer). Leakage was also monitored with a halogen leak detector and Freon gas with no leakage being found even at pressures up to 100 psig (56). Three other Bull Moose valves were tested 15 times each using 20 psig gas pressure without leakage (determined by gas collection) (57). The "fin" of the Bull Moose valve was removed in VPP (Sec. 21.4.1). No leakage tests except those done in the VPP and described below have been made for the Mark III, IV, and V freeze valves.

Two difficulties with freeze valves found in the Unit Operations section were: (a) having vents which are too small and (b) plugging of the vents with salt forced up into them (56). Having the vents too small resulted in siphoning which sometimes completely emptied the freeze valve. In only one case in the VPP, however, did the Bull Moose valve completely empty itself (Sec. 13.4.2). Siphoning in the Mark V freeze valve could be stopped (Sec. 13.4.2).

Operationally, two aspects regarding freeze valves were of prime importance in the VPP:

- a. The valve should have an unobstructed flow path when open. The normal time required to flow a fluorinator batch (about 52 liters) through a freeze valve varied from about a minute to an hour or more. The fast transfers (pressure or siphon types) were made through the freeze valves FV-104 (Sec. 16.4.14) and -106 (Sec. 13.4.2).^a In both types of transfers, the rapidity and/or success of the transfer depended largely on whether all of the salt throughout the entire transfer line was molten.

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^aSlow transfers (by gravity) were made through FV-108 (Sec. 3.4.1).

Fig. 17.4. Four Freeze Valve Designs with Approximate Regions of Sealing

b. It was essential that a tight shut-off be achieved. Leak rate data for a freeze valve alone were never obtained in the Volatility Pilot Plant because of system complexity. The evidences of tight shut-off of the three freeze valves bounding the fluorinator were as follows:

1. The fluorinator leak-test which was run prior to feed salt fluorination almost always indicated a leak rate less than the minimum arbitrarily set.
2. There was no evidence of F_2 leaking through FV-104 or FV-108 during a feed salt fluorination nor during desorption of product.^a

The fluorinator leak-test mentioned in item 1. above actually tested for leak tightness the Mark III (no FV number), FV-104, -106, and -108 freeze valves as well as V-88, HCV-7 or -8, HCV-11, the FV-100 top flange, and the FV-103 top flange.^b The leak rate found was the aggregate leak rate through these valves and flanges. Because of the sundry possible leak sources, the arbitrary criterion that the total leak rate should be less than 125 cc/min with a system pressure of 4.5 psig seemed reasonable. From measurements made during runs, the aggregate leak rate was less than 125 cc/min, an indication that the leak rate through any one freeze valve was much less than 125 cc/min.

This type of leak-test in which the leak rate of one item is presumed from the aggregate leak rate of a number of items is non-specific and at best a rough indication of the leak rate. Consequently, almost simultaneously with this type of leak-testing another sensitive type of leak-testing was performed during every feed salt fluorination and during many of the product desorption steps. This type of leak-testing utilized the odor of fluorine as the criterion. For example, small quantities of F_2 leaking through the FV-108 freeze valve or through the fluorinator or snow trap flange would have probably been detected by personnel in Cell 1 and/or the Penthouse.

Although objections might be raised to the two types of leak-testing used for freeze valves in VPP, the results indicated very little, if any, leakage --- a leak rate consistent with the "no leak" found in Unit Operations tests.

After the "E" runs, the Marks II (FV-106), III, and IV (FV-104) freeze valves were removed from the plant and sent to the burial ground. After the "L" runs, the Mark V (FV-106) freeze valve was removed and sent to the burial ground (Sec. 23.4.16b); Mark II (FV-108) was left in Cell 1.

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^aThat is, for the runs in which HCV-11 was left open during desorption of product.

^bLater the two flanges were incorporated into the flange leak-detector system described in Sec. 18.

17.5 Equipment Performance - Service Valves

The various service valves such as those used on water and air supplies were satisfactory. No tabulation of these valves is needed here because a wealth of experience already exists.

The pressure regulators used in the nitrogen supply system are listed in Table 17.8. The regulators given were satisfactory for the specified pressure range and presented no known leakage problems. In some cases, however, such as for PV-8 (Mason-Neilan Model No. 33-22), the range of 0-20 psi was so large that it was difficult to set a pressure of 5 psi as was sometimes desired.

17.6 Summary and Conclusions

A list of valves which were suitable for indicated service conditions in the Volatility Pilot Plant is given in Table 17.9. The list given includes all Volatility valves except service valves on water and air lines. The criterion for selection was that several valves of a particular kind had been successfully used in the listed service conditions. Since the over-all performance of several valves was assessed instead of only one, there is reasonable assurance that each valve is suitable as indicated.

17.6.1 Conventional Manually Operated Valves

Recesses in the bodies of both SMMD and SSD valves trapped NaF fines when placed in certain positions in lines carrying NaF-laden gases. Because of the integral nature of the SMMD and SSD stem assemblies, replacing either the disk or bellows required changing the entire stem assembly.

The ports in Hoke Valve No. 413 and 1193 plugged in UF_6 service presumably with the reaction products of UF_6 with extraneous materials. Valves with larger ports appeared more promising for UF_6 service, but similar plugs in the Superior No. 5665 valve indicated that a large port valve will also plug with UF_6 reaction products.

In VPP, an insignificant quantity of N_2 apparently leaked through packed valves.

17.6.2 HCV's

The HCV's were successfully used in operating the Volatility Pilot Plant with no major difficulty which could be assigned directly to these valves.

The frequency of repair paralleled the lengths of the different run series; that is, valve repair occurred between series of runs. In fact, after a series of runs, usually 6-9 runs, approximately one-half of the 26 HCV's leaked through the seats. Just when these valves started leaking during the run series was not ascertained although evidences of leaking valves were found during a run series. These leaks were never serious enough to cause shut-down, even though in some cases leaking valves reduced the versatility of the plant. A bonnet leak in Run E-2 was repaired without difficulty.

Table 17.8

PERTINENT DATA FOR PRESSURE REDUCING VALVES USED IN THE VPP NITROGEN SYSTEM

Type or Style	No. in Use	Manufacturer	Manufacturer's Number	Outlet Pressure Range (PSI)	(PSI) Maximum Inlet Pressure	Material of Construction					Service	Pipe Size
						Spring	Filter	Trim	Body	Diaphragm		
Style# 5902	2	National Cylinder Gas	-	0-400	4000	Steel	Sintered Bronze	-	Brass	S.S.	N ₂	1/4"
Model # 33-22	2	Mason-Neilan	-	0-20	200	Steel	-	S.S.	Bronze	Rubber	N ₂	1/2"
Type 2A4	3	C.A.Norgrene	-	0-125	125	Steel	-	Brass	Brass	Oil resistant synthetic rubber with nylon cord	N ₂	1/2"
Type 67	1	Foxboro	R 222	0-50	250	Steel	-	S.S.	Aluminum	Nylon-Cord Buna-N	N ₂	1/4"
Model # 71	1	Mason-Neilan	-	0-250	250	S.S.	-	Composition disk	Brass	Neoprene	N ₂	3/8"

Table 17.9

RECOMMENDED VALVES FOR VARIOUS SERVICE CONDITIONS IN THE VOLATILITY PILOT PLANT

Service Conditions				
Chemical Components	Pressure, psia	Temperature, °C	Recommended Valves	Remarks
UF ₆ , F ₂ , N ₂ mixtures in all proportions (except 100% F ₂) ^a	15-25	66-150	Crane SMMD; Hoke No. 411, 413, 1197, 1198; Jenkins No. 1300; HCV's with either Monel or Kel-F disk	Small ports in Nos. 411 and 413 were subject to plugging in UF ₆ service. Nos. 1197 and 1198 have been used for isolating N ₂ portion of system from pro- cess portion and not usually for flowing UF ₆ and F ₂ . No. 1300 was also used to isolate portions of system from F ₂ and UF ₆ and not to flow F ₂ and UF ₆ .
F ₂ and N ₂ mixtures in all proportions (except 100% F ₂) ^a	~0-25	0-150	Crane SMMD; HCV's with either Monel or Kel-F disk; PCV-2, PCV-10, PCV-2A and -2B.	
F ₂	15-30	0-150	Crane SMMD; HCV with Monel disk	
F ₂	15-30	0-40	PCV-2; PCV-10; PK-2A and -2B	PCV-2A and -2B; PCV-37A and -38A are not recommended be- cause of Kel-F disks.
UF ₆ and N ₂ in all proportions	0-65	66-150	Superior No. 5665; HCV's with Monel or Kel-F disk	Small ports in Hoke No. 411, 413, and 1193, would be sub- ject to plugging with solid UF ₆ .

^aUsing Kel-F or Teflon parts in contact with pure F₂ is questionable.

Table 17.9 (Continued)

Service Conditions				
Chemical Components	Pressure, psia	Temperature, °C	Recommended Valves	Remarks
Equimolar NaF-ZrF ₄ with or without up to 5 M % UF ₄	15-25	550-600	Inconel freeze valves Mark I, II, or III	See Sec. 17.4.1f
Freon 11	~15-35	-55. to +100.	Henry No. 2031, 6261; Kerotest No. R-2402	
Aq. soln. of KOH + KF (from 2 M KOH to ~1M KOH and ~1 M KF)	~15	~20. to 60.	Saunders No. 2900B	
Aq. soln. of 2 M KOH	~15	Room Temp.	Lunkenheimer No. 1709-8	
Off-Gas (N ₂ with ≤ 100 ppm F ₂)	~15	Room Temp.	Crane No. 18840, No. 18851; PCV-39, PCV-61	
N ₂	15-50	Room Temp.	Fulton Sylphon No. 96417; Powell: Figs. No. 150, 708, ^a and 1708. Walworth No. 11, ^a RP and C No. 704 ^a and Hoke No. 1193.	A variety of packed valves were used satisfactorily but probably leaked at packing; two types of rotameters were used satisfactorily but tended to leak through joints and seats; all safety valves tried leaked slightly.
N ₂	15-2000	Room Temp.	National Cylinder Gas Co., No. 333 and 334	

^aThese were the most frequently used packed valves, especially the Hoke No. 1193. A small leak through the packing was suspected. Since the leak if existing at all was negligible in our work, these valves were considered satisfactory for N₂ service in the nonprocess portion of the plant. In the process portions, Hoke No. 411, 413, or 1197 with diaphragms or bellows seals were suitable.

Water-washing the system between series of runs increased the number of leaking valves.

A leaking HCV was defined as one which when closed passed nitrogen through its seat at >2 cc/min. During leak-testing, the upstream pressure applied to the valve varied from 10 to 50 psig of nitrogen depending on location in the VPP; the downstream was at atmospheric pressure. Other details are given for leak-testing and repair. No irreparable valve was encountered.

The lapping procedures and the various disk designs discussed were satisfactory in both periods of maintenance.

Both Monel and Kel-F disks were suitable for a variety of service conditions with one exception: The Kel-F disks were not always used successfully in pure F_2 .

Both unconditioned and preconditioned Kel-F disks were capable of sustaining the maximum wear imposed on a disk in a 9-run series.

Kel-F disks which leaked showed signs of healing on cycling the valve, but such healing was never successfully demonstrated.

Preconditioning Kel-F disks should have reduced the probability of fluorine attacking the disk in the system although preconditioning did not eliminate the possibility of subsequent disintegration in pure F_2 even though using the preconditioned disks in all other plant service conditions was safe.

A suitable procedure for preconditioning Kel-F disks has been given, a treatment which should be followed by "truing" the Kel-F surface as discussed. Whether "truing" the Kel-F surface might render the disk unconditioned by removing the external layer of preconditioned Kel-F is unknown. Although not demonstrated, using the proper minimal combination of surface degreasing and time and temperature of preconditioning might render the Kel-F surface safe for use in fluorine without warpage.

17.6.3 Remotely Operated Control Valves

Most of the valves and associated control systems were satisfactory as designed and, therefore, would require no change before further processing. The particular service conditions in which specific remotely operated control valves were suitable are given in Table 17.9.

The disintegration of fluorinated plastics in PCV-10 and FX-37 corroborated the experience with some Kel-F HCV disks in pure fluorine service. This is further evidence that neither Teflon nor Kel-F is suitable for pure fluorine although exceptions to this generalization are on record.

Sticking of the FX-37 or FX-38 spindle in the open-circuit position could be remedied by rapping the flow switch sharply with a small tool.

The cycling of the PCV-10 and PCV-2 combination was troublesome and time consuming as was the effect of a trailer switch-over on this valve combination.

No KOH solution entered the FV-150 off-gas line after modifying the PCV-39 system and altering the piping.

17.6.4 Freeze Valves

Inconel freeze valves of the Mark II, III, IV, and V types were suitable for transferring molten salts encountered in the Volatility Pilot Plant as indicated in Table 17.9. Since there were differences in the configurations of these types of freeze valves, probably other configurations would work as well depending principally on whether the entire valve could be maintained at $\geq 570^{\circ}\text{C}$ and whether sufficient liquid could be retained to form a seal after using the valve. Tests made in the Unit Operations section on the Mark II valve showed that no leakage occurred using 20 psig gas pressure, and that the valve could withstand 225 freezing and thawing cycles without dimensional change. VPP leak-tests with the Mark II, III, IV and V valves in combination with other valves and flanges were apparently consistent with the Unit Operations work. Size of vents and plugging of vents were also mentioned.

17.6.5 Service Valves

Service valves on water and air lines were satisfactory. In some cases, the ranges on pressure regulators was too great.

17.7 Recommendations

It is recommended that:

- a. The valves listed in Table 17.9 be used within the specified service conditions as needed in future Volatility processing.
- b. Consideration to be given to replace the existing HCV's with flanged valves designed to avoid as many as possible of the design flaws in the existing valves with maintenance being done in the shop rather than in the field. (The flanges on these valves should probably be incorporated into a flange leak-detection system as described in Sec. 18.)
- c. An even more elaborate system of plant leak-testing connections for testing HCV's be installed for future work than in past work.
- d. Disks made of Kel-F and Teflon not be used in pure fluorine service.
- e. SMMD and SSD valves be placed so as not to trap solid particles; steps be taken to replace SMMD and SSD disks and bellows without having to change the entire stem assemblies or equivalent valves of other makes be found in which this can be done.

- f. Valves with small ports such as Hoke Nos. 411 and 413 not be used in UF₆ service.
- g. All Kel-F disks for HCV's be preconditioned and, if necessary, be "trued" by machining before use.
- h. Steps be taken to eliminate the cycling of the PCV-10 and PCV-2 combination and the adverse effects of a trailer switch-over on these valves.
- i. In new freeze valve design, emphasis be placed on maintaining the entire valve at $\geq 570^{\circ}\text{C}$ and on retaining sufficient liquid to seal the valve after it is operated.

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18.0 FLANGE LEAK-DETECTING SYSTEM

18.1 Introduction

In the flange leak-detecting system, the leak rates of ten ring-joint flanges were determined. In addition, the grooves of leaking flanges could be dynamically pressurized with nitrogen to ensure in-leakage rather than out-leakage. The design and operation of this system were patterned after the one conceived by HRT personnel.

18.2 Equipment

A schematic diagram of the nitrogen header and the list of ring-joint flanges monitored are given in Fig. 18.1. The changes necessary in a pair of flanges and its ring gasket are shown in Fig. 18.2. As shown, only one of the flanges required altering.

After the "L" Runs, the nitrogen header and tubing lines were removed from the plant and taken to the burial ground (Sec. 23.4.16b).

18.3 Operation

18.3.1 Operating Procedure

Steps in operating the flange leak-detecting system^a were:

- a. Ensuring the leak-tightness of the flange leak-detecting manifold and the tubing run to each flange.
- b. Determining the M value for each flange which depended on the length of the tubing run.
- c. Closing valve B and opening valve A.
- d. Adjusting the nitrogen supply pressure by setting the appropriate pressure reducing valve and observing gage X.
- e. Cracking valve C and filling the tubing run for the flange to be tested until the desired pressure was indicated on gage Y.^c

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^aFig. 18.1 for locations of valves and gages.

^bSec. 18.7.

^cFlanges connected to this system were tightened carefully using Bemol on the threads and a torque wrench. The final torque varied with the bolt size, being 150 ft-lb for the 3/4-in. absorber bolts and less for smaller bolts.

Legend

1. FV-121 (Inlet, Outlet and Top Flanges). (Secs. 8.4.1a and 8.4.1b)	7. FV-103 Top Flange (Sec. 6.4.1)
2. FV-120 (Inlet, Outlet and Top Flanges). (Secs. 8.4.1a and 8.4.1b).	8. Spare
3. Leak-Test (Penthouse)	9. FV-103 Exit Flange (Sec. 6.4.1)
4. Spare	10. FV-100 Hand-Hole Flange (Sec. 5.4.1b)
5. Spare	11. FV-100 Top Flange (Sec. 5.4.1b)
6. FV-103 Fill Line Flange (Sec. 6.4.1).	

Fig. 18.1. Equipment Arrangement in the Flange Leak-Detecting System

Fig. 18.2. Details for Incorporating the First Flange in Line H-103-1 into the Flange Leak-Detecting System

- f. Closing valves A and C.
- g. Opening valve B to vent gage X and then closing valve B.
- h. Recording readings on gages X and Y periodically.

Operating details including M values for the tubing runs and the equation for calculating the leak rate are shown in Sec. 18.7.

18.3.2 Critical Operating Steps

- a. Being certain that the leak-detecting manifold or tubing runs did not leak.
- b. Making certain that the ring-joint gasket was in good condition and contained a pressure equilization hole; that the flange bolt threads were lubricated with Bemol; and then that the bolts were tightened properly using a torque wrench.
- c. Ensuring that the manifold section containing gage X was bled to atmospheric pressure and then valve B closed to make it possible to monitor leaks in valves A and C.

18.4 Equipment Evaluation

18.4.1 Flange Leak Rate Data

The data available are shown in Table 18.1. These data are not necessarily listed in chronological order and should not be used to prove that the leak rate changed noticeably with small temperature changes (i.e., $\sim 50^{\circ}\text{C}$). It is true, however, that the leak rates of the absorber top flanges were greater near room temperature than at 400°C .

18.4.2 Discussion of Data

Flange leak rates ranged from zero on several flanges to 40 cc/hr on the FV-121 top flange. The leak rates on the absorber top flanges were greater near room temperature than at $\sim 400^{\circ}\text{C}$. All attempts to reduce high leak rates by retorquing the bolts failed. Therefore, for those exhibiting high leak rates, ~ 30 psig of N_2 was dynamically applied to ensure leakage into rather than out of the system. No permissible leak rate was established.

Accounts of joint and over-all system leak-testing are recorded in Secs. 19 and 20, respectively.

Table 18.1

FLANGE LEAK-DETECTING SYSTEM DATA

Test Point	Pressure	Leak Rate ^a cc/hr	Duration of Test	Vessel Temperature °C
FV-120 Inlet (Secs. 8.4.1a and 8.4.1b)	30.2	0	1 hr	~100
	30.0	.12	1 hr	~100
	30.0	<3.4	1 hr	Room
	30.0	1.7	1 hr	~100
FV-120 Outlet (Secs. 8.4.1a and 8.4.1b)	30.0	0	1 hr	~100
	30.0	0	1 hr	~100
	30.0	<3.4	1 hr	Room
	30.0	<3.4	1 hr	~100
FV-120 Top (Secs. 8.4.1a and 8.4.1b)	30.0	3	1 hr	400
	30.0	0	1 hr	430
	30.0	10	1 hr	Room
	30.0	<4.1	1 hr	400
	24.0	30	1 hr	100
FV-121 Top (Secs. 8.4.1a and 8.4.1b)	30.2	1	1 hr	400
	24.2	10	Not Available	Not Available
	30.0	4.3	1 hr	Room
	30.0	<4.3	1 hr	400
	24.0	40	1 hr	100
FV-121 Inlet (Secs. 8.4.1a and 8.4.1b)	30.0	0	1 hr	~100
	30.0	<3.6	1 hr	Room
	30.0	1.8	1 hr	~100
FV-121 Outlet (Sec. 8.4.1a and 8.4.1b)	30.2	0	1 hr	~100
	30.0	.2	1 hr	Not Available
	30.0	<3.6	1 hr	Room
	30.0	1.8	1 hr	~100
FV-103 (Sec 6.4.1)	30.0	0	1 hr	390
	25.2	0	Not Available	Not Available
	30.0	<2.4	1 hr	Room
	30.0	<2.4	1 hr	390
	25.0	<2.4	1 hr	100
All Lines open	50.0	2.3	1 hr	Room
	30.0	1.08	1 hr	Operating Temp., cf. above

^aEffect of ambient temperature change was nil.

NOTE: Data are not available on these flanges: (a) FV-103, Sec. 6.4.1; (b) FV-103 Exit Line, Sec. 6.4.1; (c) FV-100 Hand-Hole, Sec. 5.4.1b; and (d) FV-100 Top Flange, Sec. 5.4.1b.

18.5 Summary and Conclusions

No operational difficulty was met with the flange leak rates obtained which ranged from zero to 40 cc/hr. The leak rates of the absorber top flanges were greater near room temperature than at $\sim 400^{\circ}\text{C}$. Retorquing leaking flanges failed to reduce the leak rates. Dynamic N_2 at ~ 30 psig was applied to leaking flanges to ensure in-leakage rather than out-leakage. No permissible leak rate was established.

18.6 Recommendations

It is recommended that:

- a. A flange leak-detector be used in VPP during future work with all process flanges being incorporated therein.
- b. Permissible leak rates for flanges be specified.

18.7 Operating Procedure: Operation of Flange Leak-Detecting System

NOTE: Gage and valve notations may be found on Fig. 18.1.

- a. To fill leak-detector system:
  1. Close valve B.
  2. Open valve A.
  3. Observe gage X. If N_2 supply pressure is equal to, or greater than the desired test pressure, proceed to step 4. Otherwise adjust PV-1A or PV-1B to give the desired pressure.
  4. Crack valve C and fill leak test system until desired pressure is indicated on gage Y.
  5. Close valves A and C.
  6. Open valve B to vent gage X.
  7. Close valve B.
- b. To make test:
  1. Observe gages X and Y at least once each shift. An indication of pressure on gage X shows valve A or valve C leaks. Pressure drop on gage Y may be correlated with leak rate from the flange system:

$$\text{leak rate} = \frac{\Delta P}{\text{Unit time}} \times M = \text{std cc/unit time}$$

where M is a multiplier characteristic of each leak-test line. The multipliers for all lines for which the valves are open should be added together in computing the leak rate.

M VALUES FOR FLANGE LEAK-DETECTING SYSTEM

Manifold		M = 3
FV-103		M = 9
FV-120	Line to valves	M = 13
	Inlet	M = 0.7
	Outlet	M = 0.7
	Top	M = 3.6
	All three	M = 18
FV-121	Line to valves	M = 15
	Inlet	M = 0.7
	Outlet	M = 0.7
	Top	M = 3.6
	All three	M = 20
All lines open		M = 50

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19.0 JOINT LEAK-TESTING

19.1 Introduction

Following the UF₆ leak in Run E-2 and before Run E-3, joints in the VPP were catalogued and confirmed to be leak-free (22).^a

19.2 Equipment

Before joints were catalogued, as many semipermanent joints as possible in the process portion of VPP were made into permanent joints.^b A "semipermanent joint" was one which could be broken and remade in a short time such as a flanged or a screwed joint. A "permanent joint" was one which required more than merely screwing or bolting the parts back together to remake such as welded or silver brazed joints. Not all joints could feasibly be made permanent because of the expense involved for some joints as well as the fact that normal operations required the frequent making and breaking of other joints.

In the joints catalogue, VPP was divided into six areas. Each area was assigned a series number such as 1000, 2000, etc. Where possible the first digit of the series number for an area was the same as the second digit in the equipment numbering system which indicated locations inside or outside of Building 3019 (8). In each series, one thousand joints could be assigned numbers in sequence starting with the series number. The areas, series numbers, and number of joints in each series were:

<u>Area</u>	<u>Series Number</u>	<u>Number of Joints</u>
Cell 1	1000	66
Cell 2	2000	73
Main Transmitter Rack (Penthouse)	3000	133
Nitrogen Transmitter Rack (Operating Gallery)	4000	227
Penthouse	5000	These joints were grouped with those in the 3000 series.
Fluorine Transmitter Rack	6000	89

A total of 588 semipermanent joints were catalogued. This included all process and some nonprocess joints.^c

^a"Leak-free" meant that the KI-starch test was negative (Sec. 19.3.1a, Test 2).

^bThe "process" portion of VPP included equipment and piping in Cells 1 and 2, in the Penthouse, and at the Fluorine Station. The process portion ended on the N₂ lines at the "C" valves in the Operating Gallery and at FE-8 in the Penthouse. All other parts of VPP were termed "nonprocess."

^cFlanges incorporated in the flange leak^d-detector system were not included. See Fig. 18.1.

19.3 Operation

19.3.1 Operating Procedure

a. Initial Leak Tests

Two tests were made on the catalogued joints by:

1. Test 1: Soap-Bubble Test

- (a) Pressurizing the portion of the system containing the joint to ~ 30 psig with N_2 .
- (b) Applying bubbles from a soap solution with a leak being indicated if the number of bubbles increased.^a

NOTE: Joints were tested, repaired, and retested until a "no-leak" test was obtained by this method before proceeding with Test 2.

2. Test 2: KI-Starch Test^b

- (a) Pressurizing the portion of system containing the joint to 10 to 15 psig with F_2 .
- (b) Making KI-starch test as described in Sec. 17.4.2,e.2.

Leak-test data were kept on all of the catalogued joints (Sec. 19.2). Before plant start-up, it was necessary that the final test on each of these joints be negative.

b. Later Leak Tests

After start-up, a red tag system for all openings into the system was set up. Under this system, when it was necessary to break a joint, a joint tag was filled out for that joint. The original tag was tied to that joint, and its carbon copy was filed in the shift supervisors' office. Before the original tag could be removed from the joint, it was essential that the joint be remade, and that a negative test by the KI-starch method be obtained. Upon its removal, the tag was initialed by the person making the test and clipped to the carbon copy. Then, both were filed. No VFP operating work was to be done while any of these red tags was tied to a joint in the system.

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^aAt times, pressure-drop data while the system was pressurized with N_2 were taken to determine sections of the plant leaking badly.

^bInitially, the halide (Freon-flame) leak-detection method was used. But that method was more time-consuming than the KI-starch method developed by G.I. Cathers and S. Mann.

19.3.2 Critical Operating Steps

- a. Making certain that the required system pressure was in the system during the test. (This was done by monitoring with appropriate pressure instruments the system pressure during the test, and observing the fall of the system pressure on subsequently bleeding down the system.)
- b. Ensuring that foreign materials such as dirt or grease did not interfere with the KI-starch test.
- c. Being careful to avoid being burned by F_2 while doing the KI-starch test by placing the hand over a leak, or by attempting to tighten a leaking joint with F_2 in the system.
- d. Keeping the red tag system up-to-date as specified.

19.4 Equipment Evaluation

19.4.1 Preliminary Work

Prior to the leak-tests made before Run E-3 was started, the following were done:

- a. Welded joints were tested as made either by dye-checking or by both dye-checking and X- or gamma-ray testing.

Three classes of welded joints were encountered: (1) those in non-critical sections of the plant (These were dye-checked only), (2) those in critical sections but so located as to be impossible to X- or gamma-ray test (These were also dye-checked only), and (3) those in critical sections with good accessibility (These were dye-checked and X- or gamma-ray tested). Weld test data were recorded, and copies were filed in VPP Engineering File Folder No. 62 (58). In most cases, flaws appearing in X- or gamma-ray tests were eliminated by remaking the welds. But in some cases this was not feasible. Such situations were handled individually, and some of these welds were accepted with the flaws. None of the welds accepted with flaws gave trouble, however.

- b. Some semipermanent joints were replaced with permanent joints, that is, either welded or silver brazed joints.^a Most of these joints were on N_2 lines on the process-side of the "C" valves.
- c. Silver brazed joints were dye-checked. Since X- or gamma-raying these joints was virtually impossible because of their inaccessibility, these were dye-checked. Although dye-checking silver brazed joints had not been common practice, it was apparently successfully done here. Test data are contained in VPP Engineering File Folder No. 62.

^aSec. 19.2. Silver brazing specifications for various joints were made in (5).

19.4.2 Joint Leak Tests - Red Tag System

The 588 joints were tested first by the soap-bubble method and then by the KI-starch method. Leaking joints were repaired and retested until no joint leaked. This work required approximately four days. Pertinent records were filed in VPP Engineering File Folder No. 68.

The only work done on the sensitivity of the KI-starch test indicated that a joint leak giving a moderate indication by that method could also be detected with the soap-bubble test using N_2 at 100 psig although not at lower N_2 pressures.

After the plant was cleared of leaking joints, the over-all system test as described in Sec. 20.3 was made.

Although some flanges were leak-tested by the KI-starch method, most of the ring-joint flanges were tested with the flange leak-detecting system as set forth in Sec. 18.3.

The red tag system (Sec. 19.3.1b) was satisfactory. Complete records were kept in the shift supervisors' files.

It is significant that after incorporating the various leak-tests and the red tag system no serious UF_6 leak as in Run E-2 occurred, and that the frequency of minor UF_6 leaks was reduced sharply.

19.5 Summary and Conclusions

Prior to the leak-tests made before Run E-3, permanent joints (welded and silver brazed) had all been dye-checked and some X- or gamma-rayed. Results of dye-checking and X- or gamma-ray tests were filed.

A total of 588 semipermanent joints were catalogued. These joints were in both the process and nonprocess portions of the VPP. The cataloguing system was patterned partially after that for vessel numbering.

Leaks in the catalogued joints were checked by both the soap-bubble and KI-starch methods. Leaks so found were repaired until all joints showed no leak by the KI-starch method. The sensitivity of the KI-starch method was of the same order as that obtained with the soap-bubble test using 100 psig N_2 . A complete record of KI-starch tests were kept.

After the catalogued joints were leak-free, the red tag system described was set up for controlling subsequent openings into the system. Under this system, additional openings into the system were closed and shown to be leak-tight by the KI-starch method before any subsequent processing was done.

Some flanges were leak-tested by the KI-starch method. But, generally, leak-detection on ring-joint flanges was performed as described in Sec. 18.3.

After eliminating all joint leaks detectable by the KI-starch method, over-all system tests delineated in Sec. 20.3 were made.

In subsequent processing, no large UF_6 leak occurred. The frequency of small UF_6 leaks was reduced sharply.

19.6 Recommendations

It is recommended that all process joints in the VPP pass the KI-starch test before any future work is done.

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20.0 SYSTEM LEAK-DETECTING

20.1 Introduction

Before Run E-3, leak rate data on the system were made to determine whether the system was sufficiently leak-free for start-up.

20.2 Equipment

The portion of the VPP system leak-tested was that shown in Fig. 20.1. This portion was isolated from connecting lines by indicated valves (RCV's, FCV's, manual valves, and freeze valves).

The leak rate was determined from the change in the PR-26 or -33 reading using 240 liters as the estimated volume of the system.

20.3 Operation

20.3.1 Operating Procedure

Major operating steps were:

- a. Isolating the system^a as shown in Fig. 20.1 with all bounding valves shut off except those on the N₂ feed line.
- b. Putting PE-26 or PE-33 in service.
- c. Pressurizing the system to 30 psig with N₂ and then closing the bounding valves (FCV-1 and V-93) on the N₂ feed line.
- d. Noting the change in PR-26 or in PR-33 and in ambient temperature with time.

20.3.2 Critical Operating Steps

- a. Ensuring the leak-tightness of bounding valves and system joints.^b
- b. Monitoring the ambient temperature so that its effect could be assessed.

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^aAll heaters were shut off, and the entire system was at ambient temperature.

^bCf. Sec. 17.4.2, e.1 for methods of leak-testing valves and Sec. 19.3 for the methods of leak-testing joints.

20.4 Equipment Evaluation

Prior to over-all system leak-tests, joint leak-tests were made as delineated in Sec. 19. These joints included some of the flanges. Ten ring-joint flanges were leak-tested as described in Sec. 18.

The test made from February 22-24, 1958, indicated a leak rate of 8.4 cc/min. The portion of the system tested is shown in Fig. 20.1 with these bounding valves: freeze valves FV-104, -106, and -108; "C" valves V-74C, V-75C, and V-86C; HCV's -12, -14, -16, -25, -26, -35, and -36; manual valves V-93 and V-103; and FCV-1. The pressure-drop was determined with PE-26. On February 22 at midnight, the PE-26 reading was 20 psig. At midnight on February 24, the PR-26 reading was 18.5 psig. The uncertainty in reading PR-26 was $\sim \pm 0.1$ psi.

This leak rate of 8.4 cc/min was believed to be pessimistic because it included the seat-leakage of all the valves blocking off the system. The temperature change during the test was insignificant. Because of the many bounding valves in the system, this value should not be compared with the standard ($\sim 0.1\%$ of the contained volume per day) for reactor-containment vessels (59).

The effect that a temperature change can have in short-time tests, however, is a serious limitation to using this method. Consequently, long-time tests (i.e., 48 hours duration or longer) are preferred. But economic considerations associated with an idle plant become important in long-time tests.

Although there was no suitable standard with which to compare the result obtained, the work done is important because:

- a. The result is a check against the joint leak-tests, possibly showing that no large leak was overlooked.
- b. It pointed up just how difficult such tests are.

20.5 Summary and Conclusions

The leak rate of 8.4 cc/min obtained on the equipment in Cells 1 and 2 was considered pessimistic and should not be compared with the standard for reactor-containment vessels. The mechanics of the test and some of the limitations are discussed.

Flange and joints leak-tests were made as described in Secs. 18 and 19 respectively.

20.6 Recommendations

It is recommended that system leak-tests be made, and that the rates obtained be compared with an established permissible leak rate before every processing series in the future.

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21.0 AUTORESISTANCE HEATING

21.1 Introduction

Five molten salt transfer lines in VPP have been heated by autoresistance.

21.2 Theory and Equipment

In autoresistance heating, the item to be heated is the current carrier and, therefore, the resistance in the electrical circuit. Heat is generated in the resistance by the relationship, $\text{heat} = i^2rt$ where:

i = electrical current
 r = electrical resistance
 t = time

The heat produced either heats the resistance or escapes by radiation, conduction, or convection. Therefore, the fraction of heat available for heating the resistance can be increased by thermally insulating the resistance. Since the resistance of a conductor depends upon its cross-sectional area, the temperature attained by the material can be changed by altering the cross-sectional area. Consequently, different materials in a given circuit can be heated to a uniform temperature by suitably adjusting the cross-sectional areas. This fact was successfully used in the design of MS-114-1 to MS-104-1 because the molten salt line was made of Inconel and the vent line at the top of FV-104 was of nickel (Secs. 16.4.14 and 21.4.6).

In the VPP, all autoresistance heating was of pipelines insulated with 2-in. Superex to reduce heat losses. The joints between pieces of Superex were sealed with asbestos cement. Usually the entire outer surface of the insulation was covered with Thermatex "B" to minimize the sloughing off of powdery material. Sometimes, then, a layer of aluminum foil was put over the Thermatex "B" to further reduce heat losses (Sec. 21.4.6).

Specific resistances in microhms per cm of the three pertinent materials of construction at temperatures of interest are:

	<u>0°C</u>	<u>20°C (61)</u>	<u>600°C (61)</u>
"L" Nickel	8.3 (60)	---	39 ^a
Inconel		120 ^a	120 ^a
INOR-8		120 ^a	120 ^a

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^aMemorandum from A. P. Litman to F. W. Miles, "Electrical Resistivity of Nickel, Inconel and INOR-8", 10-23-59.

A wiring diagram of a typical electrical circuit is presented in Fig. 21.1. Power at 480 vac is supplied through a 20 amp contactor to a 460-v Variac with a 10-amp capacity. The output of the Variac, which was mounted on the panelboard, was fed into two step-down transformers located in the Pipe Tunnel just outside Cell 1. These transformers having a turns ratio of 400/6 were each capable of carrying 450 amp in the secondary winding. The low-voltage, high-current output of the step-down transformers was directly impressed upon a length of process piping. The "hot" sides of the transformers were connected by two 500 MCM cables to the center leg on the heated line (point K in Fig. 21.1).^a The "ground" sides of the transformers were connected to points J and L by one 500 MCM cable each, connected as near as possible to the ends of the pipe. The secondary winding of the step-down transformers was grounded at the transformers in the design. However, in installation it was found more convenient to connect together the points J and L (shown by dotted line in Fig. 21.1) and also connect ground there.

The five molten salt lines sketched in Figs. 3.1 (MS-102-1 to MS-108-1), 16.1 (MS-114-1 to MS-104-1),^b 13.1 (MS-100-1 to MS-106-1 with Mark II FV-106) and 13.2 (MS-100-1 to MS-106-1 with Mark IV FV-106) have been used. Pertinent data are presented in Table 21.1.

21.3 Operation

21.3.1 Balancing Procedure

The balancing procedure for each of the three autoresistance circuits given in Sec. 21.7. is typical and, therefore, valid for all such autoresistance lines. The critical operating step was welding the hot electrode at the center of the line to obtain an even current split. Fine adjustments could be made in the adjustable electrodes. There was, however, no coarse adjustment short of cutting off the hot electrode and rewelding it on the line. Making coarse adjustments was avoided because of the experience with removing a heat-bleed lug described in Sec. 21.4.1. Lug designs are shown on drawings Nos. D-26999 and D-27536.

21.3.2 Heating Procedure

Curing the insulation prior to use was done as for resistance-heated lines described in Sec. 22.3b.

The procedures for heating autoresistance lines are incorporated in the procedures for the systems involved. Generally, optimum practice dictated gradually rather than abruptly increasing the current (EI-1B-4 reading for MS-102-1 to MS-108-1, MS-114-1 to MS-104-1, and MS-100-1 to MS-106-1 and EI-1B-8 reading for MS-114-1). Any insulation cracks required

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^aIt was essential to have a nearly equal current split at the center leg so that the two halves of the process pipe would be heated to nearly the same temperature. The voltage drop in each half was 7 to 8 volts.

^bAnd also MS-114-1.

Fig. 21.1. Autoresistance Heating Circuit

Table 21.1

DATA FOR AUTORESISTANCE-HEATED MOLTEN SALT LINES

Molten Salt Line	References	Freeze Valve	Length (ft)	Normal Operating Data			Operating Temperature Extremes (Average Extreme Temperatures during Salt Transfer)			
				Voltage (Volts)	Current (Amps)	Power (Watts)	Molten Salt Line		Freeze Valve	
							Maximum	Minimum	Maximum	Minimum
MS-102-1 to MS-108-1	Secs. 21.4.1; 17.4.4; 3.4.1	Mk.II (FV-108)	14-1/2	N.A. ^b	435	----	690	570	630	530
							(Note: Data are for Runs L-1 and L-7)			
MS-114-1 to MS-104-1 (Over loop)	Secs. 21.4.2; 17.4.4; and 16.4.14	Mk.III (no FV number)	8	N.A.	435	----	600	550	780	680
							(Note: Data are for Runs E-1 and E-5)			
MS-114-1 to MS-104-1 (Cross-over)	Secs. 21.4.2; 17.4.4; 16.4.14	Mk.III (no FV No.) Mk.IV (FV-104)	5	N.A.	435	----	640	620	570	550
							(Note: Data are for Runs E-1, -2, & -3)			
MS-114-1	Secs. 21.4.3; 17.4.4; 16.4.14	Mk.III (No FV number)	16-1/2	N.A.	435	----	590	560	---	---
							(Note: Data are for Runs E-1, -2, & -3)			
MS-100-1 to MS-106-1	Secs. 21.4.4; 17.4.4; 13.4.2	Mk.II (FV-106)	28	7-8	435	~3300 ^c	770	660	610	580
							(Note: Data are for Runs E-1 and -5)			
MS-100-1 to MS-106-1	Secs. 21.4.5; 17.4.4; 13.4.2	Mk. V (FV-106)	28-1/2	7-1/2	340	~2600	620	550	620	540
							(Note: Data are for Runs L-1 and -7)			

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^aThe current was approximately divided into two equal parts at the "hot" electrode.^bN.A. = Not Available.^cBased on 7-1/2 volts.

patching, and current leaks needed to be minimized. The goal was to heat the entire line to $\geq 570^{\circ}\text{C}$, a temperature satisfactory for transferring fused salt compositions 30, 31, and 108 (11). Normal operating currents are given in Table 21.1.

21.4 Equipment Evaluation

21.4.1 MS-102-1 to MS-108-1

This molten salt line included the Mark II freeze valve FV-108 (Sec. 17.4.4) and is shown in Fig. 3.1; operational data are given in Table 21.1. Its operation was satisfactory although some difficulties occurred. Temperature extremes were: (a) molten salt line - maximum, 690°C and minimum, 570°C and (b) freeze valve - maximum, 630°C and minimum, 530°C . This 530°C value was lower than the 570°C desired. It is not known whether transfers could be made at true line temperatures of ~ 530 to 540°C or whether the indicated temperatures were lower than the true line temperatures. Line temperatures were determined with thermocouples placed as described in Sec. 21.4.6.

Slow transfers resulted in part from the gravity-type transfer used but mainly from cold spots at the vent lines joints and insulation breaks as mentioned in Sec. 3.4.1.^a One of the plugs (Sec. 3.4.1) was caused by Zircon ore falling into the molten salt line. The removal of the heat-bleed lug from the Mark I freeze valve eliminated a cold spot at that point. While cutting off this lug with frozen salt in the freeze valve, a hole formed in the pipe wall. This necessitated replacing the entire freeze valve and indicated the difficulty of performing work at cutting or welding temperatures on lines containing salt. Placing the ground lug at the vessels for both FV-100 and FV-102 avoided troubles as delineated in Secs. 5.4.1a and 3.4.1, respectively. See Sec. 21.4.5 for placement of other electrodes. This molten salt line was left in place after the "I" runs.

21.4.2 MS-114-1 to MS-104-1

This molten salt line included the Mark III (no FV number) and Mark IV (FV-104) freeze valves (Sec. 17.4.4) and is shown in Fig. 16.1. Operational data are given in Table 21.1. Two flow paths were possible: (a) over the freeze valve and (b) through the cross-over. The operation of both was satisfactory and much less troublesome than for MS-102-1 to MS-108-1 or MS-100-1 to MS-106-1. Note that the ground electrode at the FV-100 extremity was welded to the vessel flange (Fig. 16.1 and Sec. 16.4.14), and that the "hot" electrode for FV-104 was placed on the nitrogen line (Fig. 16.1 and Secs. 16.4.14 and 16.4.15). See Sec. 21.4.6 for placement

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^aA "cold spot" was a point below a temperature of 570°C (Sec. 21.3.2).

of other electrodes. Temperature extremes for the path over the loop were: (a) molten salt line - maximum, 600°C and minimum, 550°C^a and (b) freeze valve - maximum, 780°C and minimum, 680°C. Temperature extremes for the path through the cross-over were: (a) molten salt line - maximum, 600°C and minimum, 530°C^a and freeze valve - maximum, 740°C and minimum 650°C. Line temperatures were determined with thermocouples placed as described in Sec. 21.4.6.

The cold spot trouble at the vent line joint was absent. The cold spots at the insulation cracks were fewer than for MS-102-1 to MS-108-1, MS-100-1 to MS-106-1 (Mark II FV-106), or MS-100-1 to MS-106-1 (Mark V FV-106). It was necessary to increase the current to ~500 amperes to flow salt through the cross-over in Run E-6 (Sec. 16.4.14).

This molten salt line was removed from the plant after the "E" runs and taken to the burial ground (Secs. 16.4 and 23.4.16b).

21.4.3 MS-114-1

This molten salt line included the Mark III freeze valve (no FV number) (Sec. 17.4.4) and is shown in Fig. 16.1. Operational data are given in Table 21.1. Temperature extremes were: maximum, 590°C and minimum, 560°C^a. Note that the ground electrode at the FV-114 extremity was welded to the vent line (Fig. 16.1 and Sec. 16.4.14). See Sec. 21.4.6 for placement of other electrodes. Line temperatures were measured with thermocouples placed as described in Sec. 21.4.6. The low point in the line retained liquid and served as the freeze valve in this molten salt line. Operational troubles with this line were nearly nonexistent.

This molten salt line was removed from the plant after the "E" runs and taken to the burial ground (Secs. 16.4 and 23.4.16b).

21.4.4 MS-100-1 to MS-106-1

This molten salt line included the Mark II freeze valve FV-106 (Sec. 17.4.4) and is shown in Fig. 13.1. Operational data are given in Table 21.1. Its operation was satisfactory. Temperature extremes were: (a) molten salt line - maximum, 770°C and minimum, 660°C and (b) freeze valve - maximum, 610°C and minimum, 580°C. Line temperatures were measured with thermocouples placed as described in Sec. 21.4.6. Although its configuration was similar to that of MS-102-1 to MS-108-1, transfers were made under pressure or by siphoning instead of by gravity. These transfer techniques resulted in rapid transfers at times and in one case an uncontrolled transfer (Sec. 13.4.2). In addition to this difficulty, the line was subject to cold spots inside the fluorinator, at the vent lines joints, at breaks in the insulation, and at the waste nozzle. As for MS-102-1 to MS-108-1, the heat-bleed lug was removed, and the ground electrode

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^aComment on temperatures in Sec. 21.4.1.

at FV-100 was put on the vessel (Sec. 13.4.2). See Sec. 21.4.6 for the placement of other electrodes. Insulation was necessary for this line since when uninsulated it heated to only $\sim 300^{\circ}\text{C}$ (Sec. 21.4.5). Auxiliary heaters (FV-500A of Sec. 13.4.2 and FV-512 of Sec. 13.4.5) were necessary for satisfactory line temperatures.

In two instances, incomplete heating of this line resulted from accidentally grounding a vent line:

- a. A vent line flange was improperly insulated.
- b. A ladder had been placed so as to ground a vent line to other piping.

The Mark I waste line was removed from the plant after the "E" runs and taken to the burial ground (Sec. 13.4.2 and 23.4.16b).

21.4.5 MS-100-1 to MS-106-1

This molten salt line included the Mark V freeze valve FV-106 (Sec. 17.4.4) and is shown in Fig. 13.2. Operational data are given in Table 21.1. Temperature extremes were: (a) molten salt line-maximum, 620°C and minimum, 550°C ^a and (b) freeze valve - maximum, 620°C and minimum, 540°C ^a. Line temperatures were determined with thermocouples placed as described in Sec. 21.4.6. Although its operation was superior to the 1/2-in. NPS line previously used (Sec. 13.4.2), cold spots retained were: (a) inside the fluorinator, (b) at breaks in the insulation, (c) at the vent lines joints, and (d) at the waste nozzle.^b As for the previous MS-100-1 to MS-106-1, the FV-100 ground lug was placed on the vessel flange (Sec. 13.4.2). See Sec. 21.4.6 for the placement of other electrodes. Insulation was essential for this line since, when uninsulated, it also attained only $\sim 300^{\circ}\text{C}$ (Sec. 21.4.4.). As anticipated, the power requirement of ~ 2600 watts for this 3/8-in. NPS line was less than that (~ 3300 watts) for the 1/2-in. NPS line. This lower power requirement coupled with good operating characteristics resulting from an advanced design made this line superior to the 1/2-in. NPS line discussed in Sec. 21.4.4.

The Mark II waste line was removed from the plant after the "L" runs and taken to the burial ground (Sec. 13.4.2 and 23.4.16b).

21.4.6 Discussion

Designs of suitable autoresistance electrodes for 3/8-in. and 1/2-in. NPS Inconel are shown on drawings No. D-26999 and D-27536. Welding and installation details are also included. These have been satisfactorily used in both 3/8-in. and 1/2-in. Schedule 40 Inconel. Although these electrodes have never been used on INOR-8 autoresistance heated lines the resistance of Inconel relative to INOR-8 indicates Inconel electrodes to be satisfactory for INOR-8 pipelines (Sec. 21.2).

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^aComment on Temperatures in Sec. 21.4.1

^bAn auxiliary waste nozzle heater (FV-512 of Sec. 13.4.5) was necessary for satisfactory temperatures. Other auxiliary heaters required were FV-500A and FV-506A of Sec. 13.4.2.

Electrode placement on autoresistance lines were determined during balancing (Sec. 21.3.1). While some of the electrodes were welded to the molten salt lines, others were put in off-the-line locations integral with the corresponding lines. Usually such electrode placement avoided salt plugs at vessel walls and pipeline tees. Examples were:

- a. For autoresistance lines starting at or ending at an equipment piece or vessel such as FV-100, FV-102, FV-112, or FV-114, the "ground" electrode at that extremity of the line was welded to a convenient flange integral with the equipment piece (Secs. 21.4.1, 21.4.2, 21.4.4, and 21.4.5 for FV-100, FV-102 and FV-112),^{a,b} or to a vent line (Sec. 21.4.3 for FV-114).^c
- b. For MS-114-1 to MS-104-1 with an N₂ line "teed" into FV-104, the "hot" electrode was welded to the N₂ line about three feet from the tee (Sec. 21.4.2).^d

The current distribution in the two halves of a balanced autoresistance line was usually such that the currents in the two halves differed by about 10%.

The only study of corrosion in autoresistance-heated lines was made on the Mark II waste salt line (Sec. 13.4.2). Results indicated that:

- a. Relatively insignificant corrosion occurred on most of the line.
- b. Severe attack took place near the waste nozzle, the attack being attributed to the expected higher line temperatures effected by FV-512 and -512A.

A failure occurred in MS-100-1 to MS-106-1 (with Mark II FV-106) near the fluorinator wall, (Sec. 13.4.2). This failure apparently resulted from stresses rather than from corrosion.

The one case in which dissimilar metals were used in an autoresistance heated line was in the vent line of the Mark IV freeze valve. The freeze valve and molten salt line were made of Inconel; the vent line was made of nickel. The thickness of nickel pipe required was determined using the equation in Sec. 21.2. In addition to properly sizing the wall of this line, it was heated with vent line furnace FV-504. This arrangement worked satisfactorily.

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^aSecs. 5.4.1a and 13.4.2.

^bThe waste nozzle also required resistance heaters (Sec. 13.4.5).

^cSec. 16.4.14.

^dSecs. 16.4.14 and 16.4.15.

Thermocouples were indispensable on autoresistance-heated lines. Both of the methods used for attaching thermocouples to autoresistance-heated pipes were satisfactory. The preferred method was welding the hot junction to the external pipe surface. In this method, the insulated leads were run along the pipe and strapped to it for several inches before being brought through the Superex insulation to avoid unduly stressing the thermocouple-pipe joint. The other method utilizing a strap-on thermocouple has been very little used. Thermocouples attached in both ways evidently gave readings of the desired $\pm 10^\circ\text{C}$ accuracy.^a As to choice of method, the welded joint was preferred because of more experience with it, and because good contact with the pipe was almost always ensured. In addition, some of the temperatures obtained with strap-on thermocouples have been low. This possibly indicated inadequate contact of the thermocouple hot junction with the pipe. Other methods of placing thermocouples in VPP are given in Sec. 22.4.2.

Three difficulties have been met with welded thermocouples:

- a. Current leaks from the line through thermocouples which touched the outer aluminum foil wrapping. Such leaks reduced the line temperature and startled nearby personnel at times, thereby being possible safety hazards. The remedy was to prevent the thermocouple wire from touching the aluminum foil.
- b. Interference with Pyrovanes. The trouble was from AC pick-up by the Pyrovane thermocouple which affected the Pyrovane reading. The remedy was to tie a capacitor across the leads inside the Pyrovane case and then to ground one of the leads.
- c. Low temperature readings. Low temperatures usually indicated that the welded joint between the thermocouple and the pipe was cracked, or that the thermocouple was partially shorted. Use of such a thermocouple was discontinued.

Although no work on the variability in the wall thickness of autoresistance-heated lines has been done, no trouble directly assignable to thin or thick walls has occurred. Care has always been exercised to select pipe appearing to be good by visual examination, to avoid unduly thinning or thickening pipe walls in bends, and to prevent weld deposits of either extreme.^b In the future, however, tests of "as-received" stock and "as-bent" configurations may be essential to prevent either variability in wall thickness or ovality of pipe.

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^aThis accuracy was established by comparing the recorder reading with the melting point of the molten salt mixture. Also cf. Sec. 22.4.2.

^bWeld inspection has included: (a) dye-checking and (b) X- or gamma-ray testing (Sec. 19.4.1). Cf. (58) for examples of weld inspections and (52).

21.5 Summary and Conclusions

The following were found to be essential for the operation of auto-resistance-heated molten salt lines with Compositions 30, 31 and 108:

- a. Sufficient power to maintain the entire pipeline at $> 570^{\circ}\text{C}$ although successful transfers have been made with line temperatures as low as $\sim 540^{\circ}\text{C}$. For 1/2-in. NPS Inconel about 28 feet long, power required was ~ 3300 watts. For 3/8-in. NPS Inconel about 28-1/2 feet long, power required was ~ 2600 watts.
- b. Continuous^a 2-in. thick Superex insulation covered with Thermatex "B" to prevent sloughing off of fine material.
- c. Avoiding the pipeline touching itself or other objects.
- d. Adequate heating of joints at any vent or nitrogen line.
- e. Proper balancing of two halves of circuit such that the currents in the halves were within 10% of each other.
- f. Adequate placing of autoresistance electrodes on the line where possible or off-the-line as discussed to avoid salt plugs at vessel walls or pipeline tees.
- g. Either avoiding a heat sink such as the waste nozzle or providing additional resistance heating for the heat sink.
- h. Either avoiding dissimilar metals in the molten salt line or suitably adjusting the wall thicknesses of dissimilar metals.
- i. Proper electrode design to avoid a cold spot on the line at the electrode connection.
- j. Absence of protruding lugs such as the heat-bleed lugs on early design freeze valves.

The extent of corrosion on Inconel autoresistance-heated lines is probably minor except possibly in high-temperature zones. The one failure occurring evidently resulted from stresses rather than from corrosion.

Cutting off a heat-bleed lug from FV-108 with the line containing frozen salt necessitated replacing the entire freeze valve. This occurrence raised a question as to the advisability of doing work at welding or cutting temperatures on Inconel lines containing frozen salt.

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^aThat is, containing no cracks to cause plugs at cold spots.

Thermocouples attached to the pipe both by welding and by strapping-on have evidently given temperatures within the desired $\pm 10^{\circ}\text{C}$ variability. The welded joint was preferred since only three minor difficulties with welded joints have been experienced.

Intensively inspecting pipe "as-received" and "as-bent" has never been done. Molten salt lines fabricated without such inspection have performed adequately although care has been exercised in selecting and bending pipe to avoid abnormal wall thicknesses. In addition, welding inspection included visual examination as well as dye-checking and X-raying. This inspection avoided poor welds and either thick or thin weld-metal deposits.

21.6 Recommendations

It is recommended that:

- a. Insulated autoresistance-heated molten salt lines of either 1/2-in. or 3/8-in. Schedule 40 NPS Inconel with welded thermocouples continue to be used.
- b. Balancing of such lines continue to be done as described herein.
- c. Vent or nitrogen lines be redesigned to provide adequate heating at joints with the molten salt line.
- d. Electrodes continue to be placed as described herein.
- e. A heat sink at either end of a molten salt line be avoided or, if it cannot be avoided, be heated also with resistance heating.
- f. Suitable wall thickness design be worked out for any lines of dissimilar metals built in the future.
- g. Electrodes as designed be used on Inconel and probably on INOR-8 lines.
- h. No heat-bleed lugs be incorporated in future freeze valve or molten salt line designs.
- i. No future molten salt line be extended unheated into a vessel as the waste line in the two fluorinators built to date.
- j. Corrosion study in progress be followed up as needed.
- k. Welding inspection of molten salt line joints be continued as described herein.
- l. The inspection of pipe "as-received" and "as-bent" be studied and appropriate recommendations be made.

21.7 Operating Procedure: Balancing Autoresistance Circuits

NOTE: Prior installation of power leads in correct lengths was assumed. Four 500 MCM copper power cables were required for each autoresistance-heated line. Two of these cables were "hot" leads and the other two "cold" leads. As shown on Fig. 21.1, the "hot" leads were connected to point K, one "cold" lead to point J, and the other to point L. To avoid a significant difference in the voltage drop in either the "hot" or "cold" leads, the lengths of the "hot" leads were made within a foot of each other and likewise for the "cold" leads. The difference in voltage drop so imparted in either the "hot" or "cold" leads was insignificant because the ratio of the resistance of Inconel pipe to the 500 MCM cable was 155:1 for 3/8-in. NPS and 104:1 for 1/2-in. NPS.

The three sections of line MS-114-1 to MS-104-1 were balanced separately. See Fig. 21.2.

To balance line ABCDE, one ground lead of the double circuit was connected to the lug (type C on drawing No. D-27536) welded to the fluorinator at point E, and a double hot lead to the lug (sketch AR-3) welded to the freeze valve vent pipe at point C. One "tuning fork" electrode was then clamped to the Inconel pipe at point A according to item No. B-1 on drawing No. D-27536 and with the adjustable clamp set in midposition. The circuit was then turned on to about 100 amp. Amprobe readings were taken on lines BFD, BC, and CD. The current on line BFD should have been zero. If it was not zero, the electrode at point A was moved along the pipe as follows. If the current in CD was greater than the current in BC, the electrode at point A was moved closer to FV-100. If the current in BC was greater than the current in CD, the electrode at point A was moved away from FV-100, closer to FV-114. Next, a check was made to see that no current was flowing through LN-104-1. When the circuit was sufficiently well balanced that with 100 amp flowing to the hot lead $BD = CD$, and $BFD = 0$ within the precision of the Amprobe, the cycle was repeated at currents of 200, 300, and 400 amp. When the circuit was balanced at 400 amp, the electrode was welded in place as shown for type B on drawing No. D-27536. Then another "tuning fork" electrode was welded directly opposite point A, and the adjustable clamp was set in midposition.

To balance line ABFDE, the hot lead was removed from C and connected to the lug (type A on drawing No. D-27536) fastened at point F, midway between B and D. With 100 amp flowing to the hot lead, the currents in lines BF, FD, and BCD were measured. The current in line BCD should have been zero. If it was not zero, the electrode at point F was moved along the pipe as for the corresponding electrode for line ABCDE above. When the circuit was sufficiently well balanced that with 100 amp flowing to the hot lead, $BF = FD$ and $BCD = 0$ within the precision of the Amprobe, the cycle was repeated at currents of 200, 300, and 400 amp. When the circuit was balanced at 400 amp, the electrode at point F was welded in place as shown for type A on drawing No. D-27536.

To balance line AJK, a "tuning fork" electrode (item B-1, drawing No. D-27536) with adjustable clamp set in midposition was welded at point K. The hot lead (type A on drawing No. D-27536) was clamped to this line at point J.

With 100 amp flowing to the hot lead, the currents in lines AJ and JK in the vicinity of the hot electrode were measured. If the currents were not equal, the electrode at point J was moved along the pipe as for corresponding electrodes for lines ABCDE and ABFDE. When the circuit was sufficiently well balanced that with 100 amps flowing to the hot lead $AJ = JK$ within the precision of the Amprobe, the cycle should be repeated at currents of 200, 300, and 400 amp. When the circuit was balanced at 400 amp, the electrode was welded in place as shown for type A on drawing No. D-27536.

After the line was insulated and allowed to cure, fine adjustments were made in the "tuning forks" at points A and X to equalize line temperatures.

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22.0 RESISTANCE HEATING

22.1 Introduction

All of the heated vessels and most of the heated pipelines in the VPP were heated by resistance heaters to temperatures varying from ~ 100 to 750°C . This heating equipment was arbitrarily divided into two classes: (a) heater (wattage < 5 kw) and (b) furnace (wattage > 5 kw).

22.2 Equipment

Pertinent data for the resistance heaters are recorded in Tables 22.1, 22.2, and 22.3.^a Table 22.1 contains heaters which were not in contact with the heated surfaces, and which heated these surfaces to 400° - 650°C . Table 22.2 includes heaters which were in contact with the heated surfaces, and which heated these surfaces to 400° - 750°C . Table 22.3 gives heaters which were in contact with the heated surfaces, and which heated these surfaces to $\sim 100^{\circ}\text{C}$.

Disposition of heaters after use was:

- a. Removed from the plant and taken to the burial ground after the "E" runs: FV-510, -510A, -514, -514A, -514B, -511, -511A, -513, -513A, -515, -515A (Sec. 16.4).
- b. Removed from the plant and taken to the burial ground after the "L" runs: FV-500, -504, -505, -506, -507, -508, -509, -501A,^c -503, -512A, -517, -517A, -500B, -503A, -525, -525A, -520A, -521A, -520B, -521B.
- c. Sold to J. A. Martin, Technical Services Dept., Building K-1401 at K-25 after the "L" runs: FV-520 and -521.
- d. Left in the plant after the "L" runs: FV-502, -502A, -527, and -563.

See also appropriate report section indicated in Tables 22.1, 22.2, and 22.3 and Sec. 23.4.16b.

22.3 Operation

Heater operating procedures were incorporated in sections of this report devoted to the pertinent systems. The procedures given presupposed that the heater and its insulation had already been cured. Curing in VPP might be arbitrarily divided into three classes:

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^aFV-522 did not fit into any of these tables; cf. Sec. 7.4.3. Also cf. Engineering File Folder No. F-75.

^bHot enough to prevent the condensation of UF_6 . Sublimation point of UF_6 was 57°C at 14.7 psia (17, p. 4).

^cAnd also FV-501.

Table 22.1^a

VESSEL OR PIPE NOT IN CONTACT WITH HEATER: OPERATING TEMPERATURE = 400-650°C

Heater Number	Report Section Number	Approximate Operating Temperature, °C	Surface Area Seeing Heating Element (sq. ft.)	Container FV No. or Pipe Line Number	Wattage	Watt Density ₂ (Watts/ft ²)
FV-500	5.4.3	650	8.18	FV-101	30,000	3665
FV-502	3.4.3	600	14.45	FV-102	36,000	2500
FV-502A	3.4.4	650	0.234	MS-102	1,250	5000
FV-504	16.4.15	600	0.344	LN-104	1,600	4650
FV-505	16.4.15	600	0.344	LN-104	----	----
FV-506	13.4.3	600	0.688	LN-106	2,500	3630
FV-507	13.4.3	600	0.361	LN-106	2,500	6925
FV-508	3.4.5	600	0.688	LN-108	2,500	3635
FV-509	3.4.5	600	0.361	LN-108	1,600	4430
FV-510	16.4.4	650	45.57	FV-111	96,000	2100
FV-510A	16.4.5	650	0.344	MS-111	1,000	2900
FV-514	16.4.9	600	39.42	FV-114	64,000	1625
FV-514A	16.4.10	600	0.344	MS-111	1,000	2900
FV-514B	16.4.11	600	0.615	MS-114	1,000	6060
FV-520	8.4.2	400	8.941	FV-120	15,000	1675
FV-521	8.4.2	400	8.941	FV-121	15,000	1675
FV-563	15.4.3	100-350	1.767	FV-163	5,500	3110

^aFV-500A, -506A, and -512 were omitted because these heaters were used on the waste line which was also heated by autoresistance (Secs. 13.4.2 and 13.4.5).

Table 22.2^a

VESSEL OR PIPE IN CONTACT WITH HEATER: OPERATING TEMPERATURE = 400-750°C

Heater Number	Report Section Number	Approximate Operating Temperature, °C	Surface Area Seeing Heating Element, Sq.Ft.	Container FV Number or Pipe Line Number	Wattage	Watt Density (Watts/Ft ²)
FV-501 ^b	5.4.5	600	4.88	FV-100	9,500	1950
FV-501A	5.4.6	600	4.88	FV-100	9,500	1950
FV-503	6.4.2	400	3.40	FV-103	6,000	1750
FV-511	16.4.6	650	2.064	MS-111	3,500	1700
FV-511A	16.4.6	600	1.767	MS-111	3,000	1700
FV-512A	13.4.6	600	0.868	V-112	2,500	2890
FV-513	16.4.7	600	1.032	MS-111	4,000	3875
FV-513A	16.4.7	600	1.245	MS-111	1,500	1200
FV-515	16.4.13	600	0.239	V-115	750	3135
FV-515A	16.4.13	600	0.688	V-115	2,000	2900
FV-517	13.4.8	750	1.758	FV-117	4,000	2275
FV-517A	13.4.9	650	0.295	V-112	1,500	5080

^aFV-520C and -521C were omitted because of ineffectiveness (Sec. 8.4.5). FV-500A was omitted because of the difficulty in determining the surface area.

^bBoth Marks I and II.

Table 22.3^{a,b}VESSEL OR PIPE IN CONTACT WITH HEATER: OPERATING TEMPERATURE = $\sim 100^{\circ}\text{C}$

Heater Number	Report Section Number	Surface Area Seeing Heating Element, Sq.Ft.	Container FV Number or Pipe Line Number	Wattage	Watt Density (Watts/ft ²)
FV-500B	6.4.4	0.742	H-100-2	~ 400	540
FV-503A	6.4.3	2.170	H-103	~ 400	185
FV-520A	8.4.3	1.30	H-103	~ 400	300
FV-520B	8.4.4	1.30	H-120	~ 400	300
FV-521A	8.4.3	1.30	H-120	~ 400	300
FV-521B	8.4.4	1.30	H-121	~ 400	300
FV-525 ^c	12.4.2d	0.424	LN-125	~ 240	560
FV-525A ^c	12.4.2e	0.530	VS-125	~ 400	750
FV-526A ^c	10.4.3	0.994	H-220-2 and V-126	~ 400	400
FV-526B ^c	10.4.4	0.846	(Pigtails) H-220-2 and V-126	~ 400	475
FV-527	9.4.3	0.688	H-121 and H-220	~ 400	550

^aThe heaters for FV-220 and -222 were omitted because of the difficulty in determining the surface areas (Secs. 9.4.3 and 9.4.6, respectively).

^bFV-526 was omitted because FV-126 did not contact the heating elements (Sec. 10.4.2).

^cHeater was uninsulated.

- a. Curing 400-600°C furnaces such as FV-102. This procedure was to keep at:

1. 400°F for the first 8 hours or overnight,
2. 700°F for the next 6 hours or overnight,
3. 1000°F for the next 4 hours, and
4. 1300°F for the next 4 hours;

then use at any temperature up to 1600°F maximum.^a Such slow curing prevented cracking the furnace lining.

- b. Curing 2-in. Superex insulation joined with asbestos cement^b on lines heated to ~600°C. Usually such lines were heated for 16 to 24 hours at ~200°C to dry out the water. Sometimes during this curing holes opened in the insulation which required patching before the line could be heated to operating temperature. Fiberfrax, aluminum silicate vitreous ceramic fiber, was used for such patchwork.
- c. Curing 2-in. Superex insulation joined with asbestos cement on lines heated to ~100°C. Such lines needed little or no curing.

22.4 Equipment Evaluation

22.4.1 Watt Density Data

Operating characteristics of the heaters are given in pertinent sections of this report.

From Tables 22.1, 22.2, and 22.3, the following generalizations about the heaters are made:

- a. Vessel or pipe not in contact with heater (Table 22.1). The design watt density varied from ~1625 to 6925 watts per square foot. The values over ~4000 were primarily for heaters on small diameter pipelines such as FV-507 and FV-514B.^d Values for the large furnaces were between ~1625 and 4000. Two of the large furnaces (FV-500 and -510) heated material through a double wall. In addition to FV-500 heating through a double wall, this furnace fitted around only about half of FV-100, possibly explaining why its watt density of 3665 was the highest for the large furnaces. Although no detailed study was intended from these watt density data, the figures compare favorably with those for ORNL-designed heaters listed in Table 22.2.

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^aAs recommended by the Hevi Duty Electric Company.

^bJohns-Manville No. 450 insulating cement.

^cORNL Stores Cat. No. 15-002-3000.

^dThat is, pipelines less than 1-in. NPS.

- b. Vessel or pipe in contact with heater (Table 22.2). The watt density for these heaters varied from ~1200 to 5080. Those with the higher values were more than double the accepted design criterion of 1500 watts per square foot. One heater (FV-517) heated its vessel to 750°C with a watt density of 2275.

All of the heating elements used to heat vessel walls or pipelines to temperatures in the 400-750°C range were G. E. Calrods with 1500 F sheaths.^a The only trouble with this brand of tubular heater occurred with FV-512 and FV-515 as described in Secs. 13.4.5 and 16.4.13, respectively.

- c. Vessel or pipe in contact with heater at operating temperature of ~100°C (Table 22.3). These values are given primarily as a contrast to those in the other two tables. The watt densities were much lower varying from 185 to 750 watts per square foot. All of these heaters were fabricated of industrial heating cable as described in Sec. 22.4.2.

Pertinent design factors which have not been considered here are: (a) amount of insulation, (b) heat losses, (c) materials of construction of heated surfaces, and (d) the distance between the heated surface and the heating element.

22.4.2 Heater Construction

The design and construction of the heaters listed in Table 22.1 were performed by the manufacturer. All of these heaters were satisfactory and trouble-free.

For heaters in Tables 22.2 and 22.3, the design and fabrication were done at ORNL. The chief design and construction criteria for heaters in Table 22.2 were: (a) using watt density of ~1500 watts/sq ft, (b) attaching element firmly to the heating surface, (c) insulating with 2-in. Suprex with joints being filled with asbestos cement,^b and (d) covering outside surface of insulation with Thermax "B".

Calrods were covered or "canned" with stainless steel shimstock before insulating. This "canning" allowed dissipation of heat from the Calrods whereas permitting the insulation to touch the Calrods would inhibit heat removal -- a situation effecting Calrod burn-out.^c All of the Table 22.2 heaters were satisfactory and relatively trouble-free.

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^aThe TNK and Calrod tubular heaters used in FV-522 were satisfactory in that service (Sec. 7.4.3).

^bJohns-Manville No. 450 insulating cement.

^cSome trouble with FV-515 was experienced as discussed in Sec. 16.4.13.

The major design criteria for Table 22.3 heaters were: (a) wrapped 30-to 50-ft lengths of industrial heating cable helically around line to be heated with the advance of the helix being 1/4-to 3/8-in.^a for 3/8-in. o.d. tubing and 3/8-to 1/2-in. for 1/2-to 1-1/4-in. NPS and (b) insulated only lines over 1/2-in. NPS as in item c. for Table 22.2 heaters followed by the Thermotex "B" covering in item d.^b All of these heaters were satisfactory and trouble-free.

Thermocouples were indispensable in the VPP. Chromel-Alumel thermocouples were used in the 20° to 800°C range and those of copper-Constantan in the -60° to 0°C range. The accuracy of temperatures indicated with Chromel-Alumel thermocouples was believed to be $\pm 10^\circ\text{C}$. This accuracy value was arrived at by comparing the recorded temperatures at which UF_6 stayed in the gaseous state in the heated duct (Sec. 7.4.3) with the sublimation point of UF_6 , i.e., 57°C.^c The accuracy of copper-Constantan thermocouples was not determined.

Thermocouples for resistance-heated equipment were usually welded or silver brazed to the surfaces of vessels (FV-501 in Sec. 5.4.5b), pipelines (FV-511 in Sec. 16.4.6), or tubing (product pigtails in Sec. 10.4.4). When the Calrod temperature was also used for control as with FV-501, the controller thermocouple was welded to the Calrod. Other satisfactory placements of thermocouples were:

- a. In thermowells for protecting from molten salt (FV-100 melt, Sec. 5.4.1), NaF, or gaseous mixtures of F_2 and UF_6 (FV-120 and -121, Sec. 8.4.1).
- b. In contact (alloy-sheathed thermocouples) with NaF or gaseous mixtures of F_2 and UF_6 (FV-103, Sec. 6.4.1).
- c. In the heated duct air stream (bare thermocouples, Sec. 7.4.2).^d

Thermocouples on unheated equipment were also welded or silver brazed to the surfaces of vessels (FV-150, Sec. 11.4.1) or pipelines (F_2 trailer manifold, Sec. 15.4.1).

^aThirty feet for 3/8-in. o.d. tubing and 50 ft for 1/2-to 1-1/4-in. NPS. Fifty feet was the ideal length for 120.v supply. Heating cable data: G. E. No. CMCH-58758 Industrial Cable, No. 20 solid Nichrome wire with Monel shielding and asbestos sheath; resistance = 0.635 ohm/ft.

^bFV-520A, -520B, -521A, and -521B were canned in stainless steel shimstock before insulating as described in Table 8.1 (Secs. 8.4.3 and 8.4.4).

^c(17, p.4). Also Sec. 21.4.6.

^dSec. 21.4.6 for thermocouple placement on autoresistance-heated lines.

22.4.3 Corrosion

Corrosion data were obtained on the following heated surfaces:

- a. FV-100 (Sec. 5.4.1d).
- b. The heated duct piping (Sec. 7.4.2).
- c. The Mark II waste line (Sec. 21.4.6).

22.5 Summary and Conclusions

All of the heaters were satisfactory. Observations made on the three classes of heaters used were:

- a. Those not in contact with vessel or pipe (operating temperature = 400-650°C). Watt densities varied from ~1625 to 6925 watts per square foot with those of large furnaces ranging from ~1625 to 4000. These heaters were designed and built away from ORNL.
- b. Those in contact with vessel or pipe (operating temperature = 400-750°C). Watt densities varied from ~1200 to 5080. These heaters were ORNL designed and fabricated using 1500 watts per square foot as the main design criterion. Construction details are given. G. E. Calrods with 1500°F sheaths were satisfactory. The only trouble with these heaters occurred at FV-512 and FV-515.
- c. Those in contact with the vessel or pipe (operating temperature = ~100°C). Watt densities ranged from 185 to 750. These heaters were designed and built at ORNL. Construction details are given.

Corrosion studies were made on some heated surfaces.

22.6 Recommendations

It is recommended that:

- a. Watt densities of furnaces purchased in the future to heat vessels in the 400-650°C range be at least 2000.
- b. Design and construction criteria given herein continue to be used on ORNL-built heaters in contact with the heated surfaces with 1500°F sheath G. E. Calrods as the heating elements for high temperatures (400-750°C) and industrial heating cable for low temperatures (~100°C).

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23.0 RADIATION SAFETY INCLUDING CRITICALITY^a

23.1 Introduction

In the VPP, radiation safety consisted of detecting radiation hazards, determining personnel exposures, decontaminating equipment and its environment and preventing critical incidents. These efforts resulted in personnel exposures and air activities below the permissible levels.

23.2 Radioactive Hazards and Detecting Equipment

The radioactive hazards in VPP were enriched uranium and fission products. Enriched uranium as UF_6 was the most serious hazard because, being a gas, it was difficult to contain. The fission products were also serious hazards because of their high activities (e.g., the waste container read as high as ~10 rem/hr), but these were well contained within the equipment.

The hazardous radioactive operations in the VPP are listed in Table 23.1. Air-borne α was the most serious result of an operation because the tolerance in air was low, and because the subsequent decontamination was so difficult and time-consuming wearing assault masks. Contamination from some of these operations was greatly reduced by design and operating techniques, but little improvement was realized in: (a) the salt spatter at the waste station, (b) the release of UF_6 during product sampling, and (c) the spreading of dust while unloading FV-103.

Personnel exposure and air activity data are discussed in Sec. 23.4.1. Details of the equipment and means used to detect radiation hazards and to determine personnel exposure are recorded in Table 23.2 and described in Secs. 23.4.2 through 23.4.13. Protective devices, decontamination, and waste disposal are listed in Table 23.3 and in Secs. 23.4.14, 23.4.15 and 23.4.16, respectively.

Relative to a critical incident, three possible situations were considered:

- a. Hold-up of a large mass of UF_6 (several kilograms) in the Cell 2 equipment. This was prevented by comparing after each run the cumulative amount of product collected with the cumulative uranium fed into the system since the last system wash-out. The difference between these two figures was never greater than 5 kg.
- b. High concentration of uranium in aqueous solution during system wash-out. The aqueous wash solution draining from the Cell 2 equipment into 30 gallon drums was analyzed for uranium periodically. A concentration of 10 g/l was considered a safe upper limit. The highest concentration in the samples taken was ~4 g/l during the wash-out after the "L" runs.
- c. Storage of product cylinders. Each individual cylinder was stored in a critically safe container (Sec. 12.4.1).

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^aRadiation exposure and survey instrument data are given in "rem" throughout although it is recognized that survey instruments read directly in "roentgen."

Table 23.1

RADIOACTIVELY HAZARDOUS OPERATIONS IN THE VPP

Operation	Hazardous Agent	Principal Radioactivity
Product Sampling	UF ₆	Air borne α
Molten Salt Sampling	Fission Products in Salt UF ₆ (at times)	β-γ Air borne α
Emptying Chemical Trap: a. FV-103 b. FV-122 c. FV-124	Fission Products UF ₆ .3NaF UF ₆ UF ₆ .3NaF UF ₆ .3NaF	β-γ α Air borne α α α
Waste Removal ^a	Fission Products	β-γ
Maintenance Around FV-100	Fission Products UF ₆	γ Air borne α
Product Collecting	UF ₆	Air borne α Critical Incident
System Wash-out	Critical Incident	

^aSpatter contaminated part of Cell 1A (Sec. 13.4.4).

Table 23.2
List of Equipment and Means for Detecting Radiation¹

Components	Type of Activity Detected	Tolerance	Detecting Components	Scales and Ranges	Miscellaneous Information	References
Constant Air Monitor (CAM) (Mobile Air Monitor) Model Q-6b7 Serial No. 10; ORNL Instrument Dept.	α , β , and γ	1×10^{-8} $\mu\text{C}/\text{cc}$ for β, γ 5×10^{-11} $\mu\text{C}/\text{cc}$ for α See Sec. 23.4.2 for Instr. Tolerance	β - γ : Geiger-Mueller Tube α - Filter Disk is read in Bldg. 3550 on 64 scaler and Photomultiplier tube.	2 K Scale (0-2,000 c/min.) 10 K Scale (0-10,000 c/min.) 20 K Scale (0-20,000 c/min.) Instrument Shifts Scales Automatically (See. 23.4.2)	Period of Use - During "E" and "L" Runs. Monitoring times for both α and β - γ activities were (a) immediately, (b) after 4 hrs., and (c) after 72 hrs. Monitoring was stopped when the activity was zero. <u>Medium Sampled</u> - Air. <u>Air Sampling Rate</u> - 5 c.f.m. <u>Areas Used</u> - Cells I and II; Penthouse; and Room 100.	<u>This Report</u> - Secs. 23.4.2; 23.4.1; 23.5; 23.6; 12.3.2, item b; 16.4.16; figs. 23.2 and 23.1.
Portable Disk Sampler, Model No. Q211; Gask Mfg. Corp. Benton Harbor, Mich.	α , β , and γ	Same as for CAM.	Head for holding filter disk was counted in 3019 on 64 scaler and photomultiplier tube for α ; on 64 scaler and end-window type Geiger-Mueller tube for β - γ .	None	Period of Use - "C", "E", and "L" Runs. Monitoring times were the same as for the CAM. The sampling period could be varied as desired, being usually 30 min. <u>Medium Sampled</u> - Air. <u>Air Sampling Rate</u> - 1 c.f.m. <u>Areas Used</u> - As needed in plant.	<u>This Report</u> - Secs. 23.4.3; 23.4.2; 23.5; 23.6; 9.4.4; 12.3.2, item a; 12.3.2, item b; 16.4.16.
Alpha Air Monitor (Linear Count Rate Meter) Model Q-1451; Serial No. 12; X-69565; X-71251; ORNL	α , β , and γ	Same as for CAM.	α - Photomultiplier tube in instrument; β - γ can be counted as for portable disk sampler if necessary.	None	Period of Use - "L" Runs. Monitoring times were the same as for CAM. The sampling period could be varied from 5 min. to 10 hrs. <u>Medium Sampled</u> - Air. <u>Air Sampling Rate</u> - ~2 c.f.m. <u>Areas Used</u> - Cells I and II.	<u>This Report</u> - Secs. 23.4.4; 23.4.2; 23.5; 23.6; 12.3.2, item b.
β - γ Monitor (10 point) Model 685; Victoreen Instrument Co.	β - γ	Indicated by set point on each individual compartment	Air ionization chamber	0-1 Rm/hr. 0-10 Rm/hr. 0-100 Rm/hr. The 0-1 Rm/hr. range was used on all compartments.	Period of Use - "L" Runs. Alarm sounded and a red light shown when the activity was above the set-point; the set-point could be set anywhere within its range. Instrument was calibrated daily by S.P. Division Personnel. <u>Medium Sampled</u> - Radiation. <u>Areas Used</u> - One used at each of the following locations: (1) Cell I, (2) Cell IA, (3) Cell II, (4) PV-320, (5) Gallery, (6) PV-150, and (7) pipe tunnel.	<u>This Report</u> - Secs. 23.4.5; 23.5; 23.6; 16.4.16.
Alpha Read Monitor	α	Any Detectable amount, i.e., 250 counts/min.	Photomultiplier Tube and crystal	0-600 counts/min. 0-6000 counts/min. 0-60,000 counts/min.	Period of Use - "C", "E", and "L" Runs <u>Areas Used</u> - Outside Health Physics Office and VPP Main Panelboard.	<u>This Report</u> - Secs. 23.4.6; 23.5; 23.6; 16.4.16.

¹VPP Engineering File Folder No. P-76.

Table 23.2 (Continued)

Components	Type of Activity Detected	Tolerance	Detecting Components	Scales and Ranges	Miscellaneous Information	References
Quintector Model No. Q-1267, Serial No. 16; ORNL Instrument Dept.	B-γ	Hands - 0.3 mrem/hr Shoes - 2.5 mrem/hr Clothing - 0.75 mrem/hr	Geiger-Mueller tube for each part checked	---	Period of Use - "C", "E", and "L" Runs. Instrument enabled checking both hands, both shoes, and clothing, contained a separate alarm for each sensing element. <u>Areas Used</u> - Before entering lunch room, Bldg. 3019.	<u>This Report</u> - Secs. 23.4.6; 23.5; 23.6; 16.4.16.
Hand-Shoe Counter Model No. 1939A-1B0 Serial No. 5, X-72492, ORNL	B-γ	Posted at counter	Geiger-Mueller tube	---	Period Used - "C", "E", and "L" Runs. <u>Medium Sampled</u> - Radiation. <u>Area Used</u> - One located in clothing Change House, Bldg. 3019.	<u>This Report</u> - Secs. 23.4.6; 23.5; 23.6; 16.4.16.
Background Recorder	B-γ	The cause for any increase was sought.	Geiger-Mueller tube	0-2000 counts/min. 0-10,000 counts/min. 0-20,000 counts/min.	Period of Use - "E" and "L" Runs. <u>Medium Sampled</u> - Radiation. <u>Area Used</u> - Located in hall outside Health Physics office, Bldg. 3019.	<u>This Report</u> - Secs. 23.4.7, 23.5; 23.6; 16.4.16.
α, β Scanners	α, β	For plant floors - 10 counts/min./100 cm ² α [*] <u>For Materials</u> Moved to un-contaminated areas - 10 counts/min./100 cm ² and 20 counts/min./100 cm ² β	α - Photomultiplier tube and 64-scaler. β - end-window type Geiger-Mueller tube and 64-scaler. Contamination collected on amber paper and read in 3019 on above instruments.	0-30,000 counts/min.	Period of Use - All Runs. Scaler frequencies during operation were: (a) Plant floors - intermittently. (b) Panelboard Area - once a week. (c) Offices - once a week. (d) Other locations - when suspected of having activity. <u>Medium Sampled</u> - Loose surface contamination. <u>Area Used</u> - All plant areas and all items of equipment.	<u>This Report</u> - Secs. 23.4.8; 23.4.9; 23.4.10; 23.4.15, 23.5; 23.6; 9.4.4; 12.3.2, item a, 12.3.2, item b; 16.4.16.
Alpha Survey Meter Model No. B5; Serial No. 368; Radioactive Products, Inc., Detroit, Mich.	α	150 counts/min. per 100 cm ²	Air ionization chamber	50-2000 counts/min.	Period of Use - All Runs. The surface area of the instrument was 100 cm ² . <u>Medium Sampled</u> - Both loose and adhered surface contamination. <u>Area Used</u> - All items of plant equipment.	<u>This Report</u> - Secs. 23.4.9, 23.4.8; 23.4.15; 23.4.16, 23.5; 23.6; 13.4.7, 16.4.16.
β-γ Probe	B-γ	0.25 mrem/hr	Geiger-Mueller tube	0-80,000 counts/min. or 0.2 - 20 mrem/hr	Period of Use - All Runs <u>Medium Sampled</u> - Radiation <u>Area Used</u> - All areas and all items of equipment	<u>This Report</u> - Secs. 23.4.9, 23.4.8, 23.4.15, 23.4.16, 23.5; 23.6; 16.4.16.

*And 100 counts/min/100 cm² β.

Table 23.2 (Continued)

Components	Type of Activity Detected	Tolerance	Detecting Components	Scales and Ranges	Miscellaneous Information	References
Cutie Pie (Soft Shell) ORNL Instrument Dept.	All β and γ	60 mrem/day 300 mrem/week (Sec. 23.4.10)	Air ionization chamber	0-100 mrem/hr 100-1,000 mrem/hr 1,000 - 10,000 mrem/hr	Period of Use - "E" and "L" Runs Medium Sampled - Radiation Areas Used - All Areas and all items of equipment	This Report - Secs. 23.4.10; 23.5; 23.6; 13.4.7; 16.4.2; 16.4.12; 16.4.16.
Cutie Pie (Hard Shell) Victoreen Instrument Co.	γ and high-energy β	Same as for soft shell	Air ionization chamber	0-1 mrem/hr 1-10 mrem/hr 10-100 mrem/hr		
Monitron (Remote) Model No. Q-1154 Serial No. B8; X-50370; ORNL Instrument Dept.	γ and high-energy β	Alarm when set-point is reached; can be set anywhere within range.	Air ionization chamber	0-125 mrem/hr	Period of Use - "E" and "L" Runs. Medium Sampled - Radiation Areas Used - Thorax vessel off-gas; set to alarm at 7.5 MR/HR.	This Report - Secs. 23.4.10; 23.5; 23.6; 16.4.16.
Film Badges	3 β , γ Readings: 1. DM. 2. LM. 3. BM.	All three were same as for Cutie Pie.	Three films in type 553 pocket shielded as follows: 1. Unshielded. 2. Plastic shield. 3. Cd shield.	1. All β and γ . 2. All β and γ damaging to lens of eye. 3. High-energy β and all γ .	Period of Use - All Runs. Picture Film - Monitored quarterly or in an emergency at any frequency. Special Film - Monitored weekly or in an emergency at any frequency. Medium Sampled - Radiation Areas Used - Picture Badges: All the time in all areas. Special: Expected high areas.	This Report - Secs. 23.4.11; 23.4.12; 23.4.10; 23.5; 23.6; 16.4.16. Fig. 23.1.
Dose Meters	High - Energy β and all γ	Same as for Cutie Pie	Air ionization chamber	Pencil (or Pocket) meters; 0-300 mrem/hr Indicating Dosimeters; 0-200 mrem/hr	Pencil (or Pocket) Meters: Period of Use - "E" and "L" Runs. Data were normally taken daily but, in an emergency, at any frequency. Medium Sampled - Radiation. Areas Used - All VPP areas during operations. Indicating Dosimeters: Period of Use - "E" and "L" Runs. Data were normally taken daily, by the job, or at any desired frequency. These meters were zeroed by Health Physics Division personnel in 3019 Building. Medium Sampled - Radiation. Areas Used - As needed during operation.	This Report - Secs. 23.4.12; 23.5; 23.6; 16.4.16.

Table 23.2 (Continued)

Components	Purpose	Collection Intervals	Scales and Ranges	Miscellaneous Information	References
Urine Samples	To determine ingested or inhaled radiochemicals (Bulk: U, Sr).	Any time exposure occurred, or if in full operation, every 2 months - sometimes once a month if previous sample showed activity.	---	Period of Use - "E" and "L" Runs or at any time exposure occurred.	<u>This Report</u> - Secs. 23.4.13; 23.5; 23.6; 9.4.4; 12.3.2, item 4; 12.3.2, item 5; 16.4.16.
Fecal Samples	To determine ingested or inhaled radiochemicals (Th, Pa)	Anytime exposure occurred.	---	Period of Use - "E" and "L" Runs or at any time radioactive exposure occurred.	<u>This Report</u> - Secs. 23.4.13, 23.5; 23.6, 16.4.16.

Table 23.3

PROTECTIVE DEVICES, DECONTAMINATION, AND WASTE DISPOSAL INFORMATION

Item	Materials Used and/or Procedures	Remarks												
Protective Devices	Unit shielding, lead bricks, assault masks, protective clothing (including coveralls, safety shoes, caps, and gloves), and shoe covers	Secs. 23.4.14, 13.4.4, 13.4.13.												
Decontamination	<table><tr><th colspan="2">Surfaces Not in Contact with Molten Salt</th></tr><tr><th>Floors & Walls</th><th>External Surfaces of Equipment, e.g., HCV's</th></tr><tr><td>Three pounds of sulfamic acid and two pounds of Tide per 100 sq ft; add sufficient water to scrub thoroughly with brooms; rinse sufficiently to remove suds; allow to dry before smearing. Usually one or two such cleansings reduced α and β smear data below tolerances.</td><td>Five treatments: a. Bristle brushed with a warm aq solution of Tide and Bab-O (2 cups Tide per 30 gal; Bab-O sprinkled on each valve while brushing). b. Same as in a. c. Same as in a. with 5 lb $(\text{NH}_4)_2\text{C}_2\text{O}_4$ added per 30 gal. d. Same as in c. e. Same as in a.</td></tr><tr><th colspan="2">Small Metallic Surfaces Contacted by Molten Salt (Corrosion Specimens)</th></tr><tr><th colspan="2">Inconel Pipe^a</th></tr><tr><td colspan="2">a. Removing as much fused salt as possible mechanically, exercising care not to scar the piece. b. Gently boiling for one hour in 0.3 M aq $(\text{NH}_4)_2\text{C}_2\text{O}_4$ solution. c. Gently boiling for one hour in an aq 5% HNO_3 and 5% $\text{Al}(\text{NO}_3)_3$ solution.</td></tr></table>	Surfaces Not in Contact with Molten Salt		Floors & Walls	External Surfaces of Equipment, e.g., HCV's	Three pounds of sulfamic acid and two pounds of Tide per 100 sq ft; add sufficient water to scrub thoroughly with brooms; rinse sufficiently to remove suds; allow to dry before smearing. Usually one or two such cleansings reduced α and β smear data below tolerances.	Five treatments: a. Bristle brushed with a warm aq solution of Tide and Bab-O (2 cups Tide per 30 gal; Bab-O sprinkled on each valve while brushing). b. Same as in a. c. Same as in a. with 5 lb $(\text{NH}_4)_2\text{C}_2\text{O}_4$ added per 30 gal. d. Same as in c. e. Same as in a.	Small Metallic Surfaces Contacted by Molten Salt (Corrosion Specimens)		Inconel Pipe ^a		a. Removing as much fused salt as possible mechanically, exercising care not to scar the piece. b. Gently boiling for one hour in 0.3 M aq $(\text{NH}_4)_2\text{C}_2\text{O}_4$ solution. c. Gently boiling for one hour in an aq 5% HNO_3 and 5% $\text{Al}(\text{NO}_3)_3$ solution.		Sec. 23.4.15a for activity data.
Surfaces Not in Contact with Molten Salt														
Floors & Walls	External Surfaces of Equipment, e.g., HCV's													
Three pounds of sulfamic acid and two pounds of Tide per 100 sq ft; add sufficient water to scrub thoroughly with brooms; rinse sufficiently to remove suds; allow to dry before smearing. Usually one or two such cleansings reduced α and β smear data below tolerances.	Five treatments: a. Bristle brushed with a warm aq solution of Tide and Bab-O (2 cups Tide per 30 gal; Bab-O sprinkled on each valve while brushing). b. Same as in a. c. Same as in a. with 5 lb $(\text{NH}_4)_2\text{C}_2\text{O}_4$ added per 30 gal. d. Same as in c. e. Same as in a.													
Small Metallic Surfaces Contacted by Molten Salt (Corrosion Specimens)														
Inconel Pipe ^a														
a. Removing as much fused salt as possible mechanically, exercising care not to scar the piece. b. Gently boiling for one hour in 0.3 M aq $(\text{NH}_4)_2\text{C}_2\text{O}_4$ solution. c. Gently boiling for one hour in an aq 5% HNO_3 and 5% $\text{Al}(\text{NO}_3)_3$ solution.														

^aThese procedures were suggested by G. I. Cathers.

Table 23.3 (Continued)

Item	Materials Used and/or Procedures	Remarks
Decontamination (Contd.)	<u>Nickel Pipe^a</u> Steps a. and b. for Inconel pipe only. (Using step c. would have resulted in Ni dissolution.)	See Sec. 23.4.15b for activity data.
	<u>Nickel Plate^a</u> a. Step a. for Inconel pipe. b. Bristle brushing with a warm aqueous solution of Tide. (also used Turco No. 4501, Sec. 23.4.15b).	See Sec. 23.4.15b for activity data.
	<u>Vessels</u> a. Air sparging an aq solution of $0.4 \text{ M } (\text{NH}_4)_2\text{C}_2\text{O}_4$ for 6 hr in the vessel, keeping the solution at $\sim 100^\circ \text{C}$ with FV-500 and a level of 27 in. by adding make-up water. b. Same as a. c. Air sparging an aq solution of $0.7 \text{ M } \text{H}_2\text{O}_2$, $1.8 \text{ M } \text{KOH}$, and $0.4 \text{ M } \text{Na}_2\text{C}_4\text{H}_4\text{O}_6$, at room temperature keeping the liquid level at 27 inches.	See Sec. 23.4.15c for activity data.
Waste Disposal	<u>Waste Containers</u> a. <u>Preparatory</u> 1. Digging 16-in. dia. auger hole to a depth just above water table. 2. Pouring a one-foot concrete pad in hole. b. <u>Burial</u> 1. Removing waste container in shielded carrier to burial ground. 2. Pouring pitch or concrete into can to fill rest of way. 3. After pitch or concrete solidified, dropping can into hole. 4. After hole had almost been filled with waste containers, finishing filling hole with concrete. <u>Plant Equipment</u> Storing discarded plant equipment having a maximum activity of 500 mrem/hr on the surface of ground in Burial Ground No. 3.	See Sec. 23.4.16a. Sec. 13.4.13 Sec. 23.4.16b

^aThese procedures were suggested by G. I. Cathers.

23.3 Operations

23.3.1 Operating Procedures

Since most of the equipment was operated and maintained by Health Physics Division personnel, the procedures used are beyond the scope of this report. Decontamination and waste disposal procedures, which were done by VPP personnel, are given in Table 23.3 and in Secs. 23.4.15 and 23.4.16, respectively.

23.3.2 Critical Operating Steps

Responsibility was assumed by the VPP Shift Supervisor in charge to:

- a. Obtain the necessary assistance from Health Physics personnel.
- b. Take the required precautions when doing work in hazardous areas.

23.4 Equipment Performance

23.4.1 Personnel Exposure and Air Activities

The average weekly β - γ personnel exposure is plotted from Runs E-3 through the last "L" run in Fig. 23.1.^a The data are taken from the special film badges (Sec. 23.4.11). The highest value of 49 mrem/man-wk is below the weekly tolerance of 300 mrem (Sec. 23.4.10).

The average α activity in Room 100 air is plotted in Fig. 23.2 from Run E-3 through the last "L" run.^b The average α activity was continuously below the tolerance value of 5×10^{-11} μ c/cc.

The average β - γ activity in Room 100 air is plotted in Fig. 23.3 for Run E-3 through the last "L" run.^b The average β - γ activity was maintained below the tolerance value of 1×10^{-8} μ c/cc.

23.4.2 Constant Air Monitor

Constant air monitors were used in Cells 1 and 2 during the "E" runs and in the Penthouse as needed during both the "E" and "L" runs (Secs. 12.3.2b and 16.4.16). This instrument primarily monitored air borne β and γ .

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^aThese data were unavailable before Run E-3 because the special film badges were not worn prior to February, 1958 (Sec. 16.4.16).

^bData collection started in February, 1958 or just prior to Run E-3 (Sec. 16.4.16).

Fig. 23.1. Plot of Average β - γ Exposure to VPP Personnel vs. Week Ending Date, 1958

Fig. 23.2. Plot of Average α Activity in Room 100 Air vs. Week Ending Date, 1958

Fig. 23.3. Plot of Average β - γ Activity in Room 100 Air vs. Week Ending Date, 1958

Air was drawn through the filter in the instrument at a flow rate of 5 cfm. A continuous record in counts per minute of the β - γ activity collected on the filter was made. The instrument contained three activity scales: (a) 2K scale (0-2000 counts/min), (b) 10K scale (0-10,000 counts/min), and (c) 20K scale (0-20,000 counts/min). The 2K scale was used when starting the instrument. Shifting of scales was automatic, i.e., when the instrument indicated full scale on the 2K scale it shifted automatically to the 10K scale and when full scale on the 10K scale was attained the instrument shifted to the 20K scale. On the 2K scale, there was no flashing light or alarm. But an alarm sounded briefly when the instrument switched from the 2K to the 10K scale. And, while on the 10K scale, a yellow light flashed. On switching from the 10K to the 20K scale, an alarm sounded continuously until acknowledged. In addition, a red light flashed while on the 20K scale.

The instrument tolerance varied with the calibration, the value being posted on the instrument when it was put in service. This value was a certain number of chart divisions (about 5 divisions or half-scale) on the 20K scale. The tolerance was considered exceeded when, after switching to the 20K scale, the recorder pen reached the tolerance value in 30 minutes or less. This signified that the β - γ activity of the air being sampled was above the tolerance of 1×10^{-6} μ c/cc. The filter was then removed by Health Physics personnel and counted as follows: (a) immediately for α (tolerance = 5×10^{-11} μ c/cc), (b) after four hours for α and also β - γ , and (c) after 72 hours again for α and also β - γ . Whether this monitoring schedule was carried the full 72 hours depended on the half life of the radiochemicals involved, i.e., the monitoring was stopped when the activities became zero.

This instrument was useful for continuously monitoring air borne activities. The data in Figs. 23.2 and 23.3 were obtained with the constant air monitor (Sec. 23.4.1).

23.4.3 Portable Disk Sampler

The portable disk sampler was used intermittently for monitoring air borne activities in the VPP especially during the "E" and "L" runs (Secs. 9.4.4, 12.3.2a, 12.3.2b, 16.4.16). This instrument pulled air through a filter disk at a rate of one cfm. The sampling time was varied as desired, usually being about 30 minutes. After collection, the sample was monitored in Building 3019 for α and/or β - γ activities. The α and β - γ data were obtained at the same time intervals and were referred to the same tolerances stated in Sec. 23.4.2. This instrument was useful for spot-checking plant areas and for confirming data obtained with the constant air monitor or α air monitor.

23.4.4 Alpha Air Monitor (Linear Count Rate Meter)

After the "E" runs, a semi-continuous α air monitor was used for Cells 1 and 2. Permanent sampling lines were run to this instrument such that either cell individually or both cells together could be monitored by proper valving. The sampling rate was 2 cfm, and the sampling period could

be set as desired from 5 min to 10 hr. After a sample was taken, that sample was checked for α while the succeeding sample was being collected. Alpha activities after 4 hours and after 72 hours and β - γ data were determined as delineated in Sec. 23.4.2.

The counting portion of the instrument had four ranges: zero, 500 (500 counts/min), 2K (2000 counts/min), and 5K (5000 counts/min). The range selecting knob was connected to a milliammeter (0-1.0 ma range) and a recorder. The milliammeter had three pointers: a red stationary pointer for setting zero, a red stationary pointer for setting the trouble alarm value, and a black indicating pointer. The zero pointer was set at zero; the trouble pointer was usually set at full scale (1.0 ma); and the black pointer indicated the machine milliamps, a value proportional to the count rate of the sample. On each of the three higher scale ranges (500, 2K and 5K), both the milliammeter and the recorder were at full scale when the sample counted 500, 2000, and 5000 counts/min, respectively.

The machine had both trouble and machine failure alarms connected to individual pilot lights. The ringing of either alarm was accompanied by the lighting of its respective pilot light, making possible the immediate determination of why the machine alarmed. In each circuit, a toggle switch was provided to turn off the alarm. A common reset button enabled re-setting the alarm. The trouble alarm indicated that the milliammeter reading had exceeded the setting of the trouble set-point. When the trouble alarm had been actuated, turned off, and reset, it was necessary for the black indicating pointer to have been below the trouble set-point for several seconds before the trouble alarm switch could be cut on again without re-actuating the alarm. The machine failure alarm was actuated on machine failure and could be reset when the cause of failure had been remedied.

Determining that tolerance had been exceeded depended on the longevity of the radiochemical species as well as the count rate value. Consequently, some time was required to make such a determination.

This instrument was useful for continuously monitoring α activity in the cells, especially during product sampling as well as during plant disturbances (Sec. 12.3.2b). In addition, β - γ data could be obtained from the samples as desired.

23.4.5 β - γ Monitor

This instrument which was located at the Volatility Panelboard remotely indicated the β - γ radiation striking a sensing head at each of the seven locations indicated in Table 23.2. Readings at all locations were indicated continuously. Each individual compartment within the instrument contained a dial, red light, and alarm. The dial had two pointers, one a set-point and the other an indicating pointer. The set-point was placed at the acceptable radiation background for that area. When the indicating pointer exceeded this value, the red light came on, and the alarm sounded. The light and alarm could be canceled by pushing a reset button. Each compartment was adjusted for a dial range of zero to one rem/hr but could have been set for other ranges.

This instrument was used only during the "L" runs. It enabled saving operating time and reducing personnel exposures because adequate precautionary measures could be taken prior to entering a β - γ field.

23.4.6 Monitors for Personnel and Clothing^a

An α hand monitor was located on the bench outside of the Health Physics office in Building 3019. This instrument was useful in maintaining the acceptable hand tolerance of any detectable amount (~ 250 cpm).

A Quintector was provided just outside the lunchroom in Building 3019 for β - γ monitoring of hands, shoes, and clothing. The tolerances were: 0.3 mrem/hr for hands, 2.5 mrem/hr for shoes, and 0.75 mrem/hr for issued clothing. This instrument contained three alarms, each of which was actuated by an above-tolerance value in its category.

A hand and shoe counter for β - γ monitoring was located in the change room of Building 3019. The background and hand and shoe tolerance values were posted at the instrument.

23.4.7 Background (β - γ) Recorder

A continuous background (β - γ) recorder was located above the bench outside of the Health Physics office in Building 3019 (Sec. 16.4.16). This instrument kept a continuous record of the β - γ background in that vicinity and also indicated the background count rate by "pops" on its audio system. The rate of "pops" increased with the background until an alarm sounded. The alarm set-point was usually one mrem/hr.

23.4.8 α - β Smears^b

Smears which were subsequently monitored for both α and β contamination were taken on:

- a. Surfaces in the operating areas, especially floors and walls. These smears helped to prevent the spread of radioactivity and to avoid personnel exposure to α and β activities. The protective devices mentioned in Sec. 23.4.14 also helped to accomplish these objectives. Decontamination was done as delineated in Sec. 23.4.15.
- b. Pieces of equipment and samples, especially those to be subsequently worked on in uncontaminated areas. Decontamination practices are discussed in Sec. 23.4.15.

The α tolerance was 10 counts/min/100 sq cm both for plant floors and for materials being moved to uncontaminated areas. Corresponding β tolerances were 100 and 20 counts/min/100 sq cm, respectively. Smears represented α and β activities which could be wiped off surfaces while the total activities were detected as mentioned in Sec. 23.4.9.

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^aSec. 16.4.16.

^bSecs. 9.4.4, 12.3.2a, 12.3.2b, 16.4.16.

The criterion or frequency for making α - β smears was:

- a. Plant floors - intermittently.
- b. Panelboard area - once a week.
- c. Offices - once a month.
- d. Other areas - when suspected of containing activity.
- e. Tools, samples, and materials - when moved to an uncontaminated place.

23.4.9 Alpha Survey and β - γ Probe Meters

The α survey meter was used to detect the total α activity on tools, samples (Sec. 23.4.8 for smear activities and Secs. 13.4.7, 16.4.16), clothing, and pieces of equipment. In all cases, the α activity detected was that which could be removed by smearing plus that firmly adhering to the surface. The tolerance was 150 counts/min/100 sq cm. (The surface area of the meter was 100 sq cm.) Materials surveying below tolerance could be moved to uncontaminated areas, while those above tolerance required additional decontamination (Sec. 23.4.15) or burial (Sec. 23.4.16).

The beta-gamma probe data were taken on pieces of equipment and samples (cf. item b. in Sec. 23.4.8 and Sec. 16.4.16). Subsequent decontamination practices are described in Sec. 23.4.15. The tolerance was 0.25 mrem/hr. As for α surveying, materials below tolerance could be moved to uncontaminated areas. Those above tolerance required additional decontamination (Sec. 23.4.15) or burial (Sec. 23.4.16).

23.4.10 Cutie Pies and Monitron

Both soft-and hard-shell cutie pies were used as needed to detect β - γ activities (Sec. 13.4.7, 16.4.12, 16.4.16). Since these instruments were portable and were used largely by operators, they were immediately available and, consequently, the most useful β - γ instruments. Cutie pie data were used to determine allowable working time such that a man would receive an exposure of no more than 60 mrem/day, 300 mrem/week, 3 rem/yr, or 5 rem/yr.^a In using the data for determining exposure, the next higher exposure rate takes precedence over the exposure for a given period, e.g., if an employee had already received 300 mrem in a given week, an additional 60 mrem exposure in that week would overexpose him.

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^aThese data were obtained from relationship, $5(n-18) = \text{Total acceptable lifetime exposure in rem.}$

A Monitron continuously surveyed the Thorex vessel off-gas line which runs behind the Main Transmitter Rack in the Penthouse (Sec. 16.4.16). The alarming device of this instrument was set at an activity of 7.5 mrem/hr.

23.4.11 Film Badges

Both picture film and special film badges detected exposure to the three combinations of β - γ radiations indicated in Table 23.2. The picture film badge was worn continuously while in the X-10 area and was generally monitored quarterly by Health Physics personnel.^a Special film badges were worn after February, 1958 by all operating personnel while processing radioactive materials in the "E" and "L" runs (Sec. 16.4.16). These badges were usually checked weekly by Health Physics personnel. The average weekly exposure data so obtained are plotted in Fig. 23.1 (Sec. 23.4.1). The tolerances observed are given in Sec. 23.4.10.

23.4.12 Dose Meters

Pencil meters were worn by all operating personnel while processing radioactive materials (Sec. 16.4.16). These meters provided daily monitoring of β - γ radiation. The data were obtained and recorded by Health Physics personnel. At times, indicating dosimeters were also used to obtain immediate checks on personnel exposure to β - γ radiation. Although these meters were zeroed by Health Physics personnel, the data were generally used only by operating personnel and were valuable in limiting radiation exposure.

23.4.13 Urine and Fecal Samples^b

Urine and fecal samples were collected for these purposes and at these intervals:

a. Urine.

Purpose - To detect the presence of the bulk of ingested and inhaled radiochemicals such as U and Sr.

Sampling Criterion and/or Frequency - Any time exposure occurred and also, if in full operation, at least every two months.

b. Fecal.

Purpose - To detect the presence of ingested and inhaled Th and Pa.

Sampling Criterion - Any time exposure occurred.

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^aMore frequent checking was done where deemed necessary.

^bSec. 16.4.16.

For both urine and fecal samples of VPP operating personnel, indications of radiochemicals were sometimes found. There were, however, never enough associated exposures to increase significantly the film badge exposures.

23.4.14 Protective Devices^a

Assault masks, protective clothing (including coveralls, safety shoes, caps, and gloves), and shoe covers were worn in contamination zones. Assault masks protected against air-borne activities and were always worn when the air contained $>5 \times 10^{-11}$ $\mu\text{c/cc}$ of α or $>1 \times 10^{-8}$ $\mu\text{c/cc}$ of β - γ .^b Protective clothing and shoe covers were always worn along with the assault masks. In addition, protective clothing and shoe covers were worn when floors and surfaces in the area smeared higher than the α and β tolerances (Sec. 23.4.8). For above-tolerance areas, shoe cover stations were provided to prevent spreading contamination. During operations, shoe cover stations were usually necessary at each cell, in the Penthouse, and in the Pipe Tunnel.

For β - γ radiation, both shielding and limited exposure time were used to control personnel exposure. Unit shield was either provided in the original design (e.g., the waste station) or lead bricks were stacked as required. The indicating dosimeter was especially useful in limiting personnel exposure.

23.4.15 Decontamination^c

a. Surfaces not in Contact with Molten Salt

Floors were washed as described in Table 23.3. Walls and external surfaces of equipment were decontaminated similarly (cf. Sec. 23.4.8 for tolerances).

Seventeen HCV's were decontaminated and returned to the Instrument Division for subsequent distribution and credit to the VPP account (Sec. 17.4.2g). The five decontamination treatments along with α - β smear data are listed in Table 23.4.^d Three α - β smears were made on each valve: (a) on body and on nipple ends, (b) on stem and underneath the valve operator, and (c) on top of the operator.

Minisampler sample tubes were decontaminated per Sec. 12.3.1b.

^aSecs. 9.4.4, 12.3.2a, 12.3.2b, and 16.4.16.

^bThe building ventilation system and cell covers helped to reduce the level and spreading of air-borne activity.

^cSec. 16.4.16 (62).

^dThe treatments are also recorded in Table 23.3.

Table 23.4

DECONTAMINATION OF 17 HCV'S

NOTE: Initial activities were not determined; β - γ probe data were obtained only after the fifth decontamination. Alpha and beta smear tolerances were: <10 and <20 counts/min, respectively.

a. First Decontamination and Survey

Treatment - Bristle brushed with a warm aqueous solution of Tide and Bab-O. (2 cupfuls of Tide per 30 gal; Bab-O sprinkled on each valve while brushing).

α - β Smear Data - All three smears on five valves were below the α tolerance.^a Alpha smears on the other twelve valves averaged: (a) 290, (b) 18, and (c) 36 counts/min.^b Smear data were essentially below β tolerance.

b. Second Decontamination and Survey

Treatment - Same as for the first decontamination.

α - β Smear Data - More than half of the α data for the 12 contaminated valves in item a. were below tolerance. Smear data were essentially below β tolerance.^c

c. Third Decontamination and Survey

Treatment - Same as for the first decontamination with about 5 lb $(\text{NH}_4)_2\text{C}_2\text{O}_4$ added per 30 gal of solution.

<u>α-β Smear Data</u>	<u>HCV NO.</u>	<u>α Smear Data, Counts/min</u>			
		(a)	(b)	(c)	
	11	31	<10	<10	All three smears for HCV's 14, 16, 23, 26, 30, 33 and 36 were below α toler- ance
	17	19	<10	14	
	22	101	<10	<10	
	24	90	<10	<10	
	25	1233	<10	12	
Smear data were essentially below β tolerance.					

^aThe only further work on these valves were β - γ probe tests which were approximately background, i.e., <0.25 mrem/hr.

^bMeanings of "a", "b," and "c" are given in Sec. 23.4.15a.

^cSmear data on individual valves were not available.

Table 23.4 (Continued)

d. Fourth Decontamination and Survey

Treatment - Same as for the third decontamination.

<u>α-β Smear Data -</u>	<u>HCV No.</u>	<u>α Smear Data, Counts/min</u>		
		(a)	(b)	(c)
	11	20	<10	<10
	25	35	<10	<10

All three smears for HCV's 17, 22, and 24 were below α tolerance.

Smear data were all below β tolerance.

e. Fifth Decontamination and Survey

Treatment - Same as in the first decontamination.

<u>α-β Smear Data -</u>	<u>HCV No.</u>	<u>α Smear Data, Counts/min</u>		
		(a)	(b)	(c)
	25	12	<10	<10

All three smears for HCV 11 were below α tolerance.

All valves probed background, i.e., <0.25 mrem/hr.

b. Small Metallic Surfaces Contacted by Molten Salt (Corrosion Specimens)

Procedures for Inconel pipe, nickel pipe, and nickel plate are recorded in Table 23.3.

Data for decontaminating corrosion specimens using these procedures were:

1. Inconel pipe specimens required four procedures to decontaminate to no α activity, ~ 3 counts/min of β , and < 0.25 mrem/hr probe reading (background).
2. A nickel pipe corrosion specimen (1/2" NPS, Schedule 40) required four procedures to decontaminate as follows:
 α - none, β - none, and probe - < 0.25 mrem/hr (background).^b
3. Two nickel plate specimens^c required one procedure to decontaminate from 24 and 60 counts/min β activity, respectively, and 0.5 and 14 mrem/hr probe reading, respectively, to no β activity on either and 0.5 and 14 mrem/hr probe reading, respectively. Then, one procedure for nickel pipe produced no improvement in the probe reading. Finally, treating the 14 mrem/hr specimen with Turco No. 4501 at 100°C for 1-1/2 hr reduced the probe reading to 1.0 mrem/hr.

c. Vessels

Aqueous decontamination data are available only for the decontamination of FV-100 after Run L-4. As mentioned in Sec. 5.4.1c, the bulk of the solidified salt was removed by jack-hammering and chipping. Then the following work was done:^d

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^aThese specimens were seven 2-in. pieces of 3/8-in. NPS, Schedule 40 from the Mark II waste line. Activity data prior to decontaminating were not obtained. After two of the four procedures, activity data were: 3 counts/min of α , 30 counts/min of β , and < 0.25 mrem/hr probe reading (background). See Sec. 13.4.2 for corrosion results.

^bThis specimen was about a 3-in. section of the F_2 inlet line near the draft tube from the Mark IIA fluorinator. Activity data were not obtained prior to decontamination.

^cThese specimens came from the Mark IIB fluorinator wall (3/8-in. nickel plate originally). There was no initial α activity.

^dTreatments are also recorded in Table 23.3.

1. Cutie pie survey of internal surfaces.

Point 1 (just below upper flange) - 1.5 rem/hr.
Point 2 (level of waste line exit) - 6 rem/hr.
Point 3 (level of charge salt line entry) - 6.5 rem/hr.

2. First decontamination and survey.

Air sparged an aqueous solution of 0.4 M $(\text{NH}_4)_2\text{C}_2\text{O}_4$ for 6 hours in the vessel keeping the solution at a temperature of $\sim 100^\circ\text{C}$ with FV-500 and a level of 27 inches by adding make-up water. After draining the solution and rinsing the vessel, the cutie pie survey was:

Point 1 - 0.9 rem/hr.
Point 2 - 1 rem/hr.
Point 3 - 1.5 rem/hr.

3. Second decontamination and survey.

The same decontamination procedure described in item b. was used after which the cutie pie survey was:

Point 1 - 20 mrem/hr.
Point 2 - 40 mrem/hr.
Point 3 - 40 mrem/hr.

4. Third decontamination and survey.

Air sparged an aqueous solution 0.7 M H_2O_2 , 1.8 M KOH, and 0.4 M $\text{Na}_2\text{C}_4\text{H}_4\text{O}_6$ at room temperature keeping the liquid level at 27 inches. After draining and rinsing, the cutie pie survey was:

Point 1 - 40 mrem/hr.
Point 2 - 30 mrem/hr.
Point 3 - 30 mrem/hr.
Bottom of vessel - 80 mrem/hr.

Some loosely adhering material still clung to the vessel wall, and some material was still adhered tightly to the bottom.

23.4.16 Waste Disposal Practices

a. Waste Containers

Details of burial ground preparation and can burial are given in Table 23.3. The limit on the number of cans per hole was such that three to four feet of concrete was between the neck of the can top and the ground level.

The highest activity of the waste containers for the "E" and "L" runs was ~ 10 rem/hr as determined with a hard-shell cutie pie on contact with the container wall.

Personnel exposure while burying waste containers was monitored by Health Physics personnel.

b. Plant Equipment

Discarded plant equipment such as the two fluorinators, the ARE hold tank, molten salt lines, and miscellaneous piping and tubing have been stored above ground in Burial Ground No. 3.^a The maximum activity of this equipment was ~500 mrem/hr at the time of storage.

23.5 Summary and Conclusions

Enriched uranium as UF_6 was the most serious hazard in the VPP because, being handled largely in the gaseous state, it was difficult to contain. The fission products, although at times producing activities up to 10 rem/hr, were less hazardous because of better containment and shielding.

The critical incident was potentially the most serious consequence of VPP operations. Procedures used to avoid a critical incident were listed in the previous sections.

A list of radioactively hazardous VPP operations is given. The worst consequence resulting from any VPP operations was air-borne α because of the low air tolerance and the difficulty of subsequent decontamination. Although work could be done in an area having an air-borne α activity above tolerance by wearing assault masks, the work times for various operations were lengthened considerably. Some reduction in the hazards of some operations was realized through better design and improved operating techniques, but little reduction occurred for: (a) the salt spatter at the waste station, (b) the release of UF_6 during product sampling, and (c) the spreading of dust while unloading FV-103.

Plots are given showing that the average weekly β - γ personnel exposure and both the air-borne α and β - γ in Room 100 were below established A.E.C. tolerances for Runs E-3 through E-6 and also for the "L" runs.

Information pertinent to VPP personnel is given for all of the instruments and means used to detect radioactive hazards. These include: constant air monitor, portable disk sampler, α air monitor, β - γ monitor, α hand monitor, Quintector, hand and shoe counter, background recorder, α - β smears, α survey and β - γ probe meters, cutie pies, Monitron, film badges, dose meters, and urine and fecal samples. These instruments and monitoring procedures were the responsibility of Health Physics Division personnel. Data obtained were evaluated largely by Health Physics Division personnel.

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^aSecs. 3.4, 4.4.1, 5.4.1, 7.4.2, 8.4.1, 9.4, 10.4, 11.4, 12.4.1, 12.4.2, 13.4.2, 14.4, 15.4, 16.4, 17.4.1, 17.4.3, 17.4.4, 18.2, 21.4.2, 21.4.3, 21.4.4, 21.4.5, and 22.2.

Protective devices and practices are also discussed.

The decontamination procedures used are given for: (a) surfaces not in contact with molten salt, (b) metallic surfaces contacted by molten salt, and (c) vessels. Activity data indicated that the procedure for decontaminating floors and walls was satisfactory; that those for HCV's and metallic surfaces contacted by molten salt were too slow; and that the procedure for FV-100 was fairly satisfactory. The need for more work on decontamination is evident.

Waste disposal practices were recorded in the final part of the section.

23.6 Recommendations

It is recommended that all past practices in radiation exposure and control delineated herein be continued in the future and that:

- a. Effort be expended to eliminate the salt spatter at the waste station.
- b. UF_6 sampling be done by the K-25 method and preferably elsewhere than in Cell 2 to avoid the possible personnel exposure to α activity and also the contamination of Cell 2 equipment.
- c. A better means of emptying FV-103 be devised.
- d. Better decontaminating procedures for metallic surfaces not contacted by molten salt and for Inconel and nickel pipe, nickel plate, and vessels contacted by molten salt be devised.
- e. Waste disposal practices be reviewed to confirm their adequacy.
- f. Two new radiation alarming dose meters, one for dose rate and the other for total dose, be tried in VPP.
- g. Any new pieces of equipment be evaluated as criticality hazards.

24.0 ACKNOWLEDGMENT

For a report of this nature, the authors' desire to acknowledge the contribution of others must be tempered by the fact that a complete listing would be prohibitively long. The major contributors are listed in ORNL-2918 (6, p. 38), and the authors wish to acknowledge the contributions of all those listed in ORNL-2918. In addition, special thanks are due to J. C. Bressee, F. N. Browder, J. H. Gibson, Jr., R. B. Lindauer, R. B. Waters, C. L. Whitmarsh, and A. V. Wilder for invaluable assistance in the preparation of the report.

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