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SUBJECT: A Series of Seven Flowsheet Studies with Nonradive
Salt, Volatility Pilot Plant Runs C-9 through C-15

TO: F. L. Culler

FROM: C. L. Whitmarsh

ABSTRACT

A series of seven flowsheet demonstrations of the Volatility Process was performed using non-irradiated material. In this series (a) the Pilot Plant was proven to be operable but not sufficiently reliable for remote operation; (b) the feasibility of a bed of NaF pellets as a trap for Cr and Zr fluorides was demonstrated; and (c) the most desirable fluorine flow rate for fluorination of molten salt in the existing equipment was determined to be in the range 10-30 std. liters per min.

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1.0 INTRODUCTION

The Fluoride Volatility Process was developed on a laboratory scale to recover uranium from fused salt reactor fuels. The process consists of volatilization of UF_6 from molten salt by fluorination of UF_4 , and additional decontamination of the UF_6 from volatile or entrained fission product fluorides by absorption and desorption in fixed beds of granular NaF.

A pilot plant scale study of the process was started in December, 1956. Design of the plant was based on a batch process, each batch of salt containing approximately 10 kg of highly enriched uranium. Equipment shakedown and process runs with both nonirradiated and irradiated salt are necessary to determine the operational feasibility of the volatility process.

A series of seven runs (C-9 through C-15) was made primarily to demonstrate the operational reliability of the pilot plant and the feasibility of repeated use of an absorber bed. Additional studies included in the runs were the effectiveness of a NaF bed as a Cr and Zr trap, and the effect of F_2 flow rate during the fluorination operation. Each of seven runs consisted of a complete volatility flowsheet demonstration with non-irradiated material. Fluorine flow rates of 7 to 33 slm* during fluorination of the salt were studied.

2.0 SUMMARY

Operation of the Volatility Pilot Plant was demonstrated in a series of seven flowsheet demonstrations to be feasible but not sufficiently reliable for highly radioactive material. During the fluorination and waste salt transfer steps the process was susceptible to line plugs resulting from solidification of salt. These plugs caused interruptions in the normal procedure and required direct maintenance for removal. Leakage in that part of the system exposed to gaseous UF_6 also presented a problem.

The same absorber bed was used for seven absorption and desorption cycles. Desorption losses--uranium remaining on the bed after desorption--were shown not to be cumulative for consecutive runs. However, absorption losses--uranium reaching the chemical trap downstream from the absorber during fluorination--were excessive ($>0.1\%$ of the feed) in all but one run. Whether this was due to repeated use of a bed, valve leakage, or some other cause was not determined.

* Standard liters per minute.

A study of the effect of fluorine flow rate during fluorination of UF_4 (lq) to UF_6 (g) indicated that a flow rate in the range 10-30 slm was desirable. Below 10 slm mixing of the molten salt was inadequate, and above 30 slm corrosion (and erosion) in the fluorinator and entrainment might become excessive.

The use of an NaF bed at $400^\circ C$ to selectively remove Cr and Zr from the gas stream (CRP trap) was demonstrated to be possible, although the present setup was replete with problems. The bed removed approximately 50 g of Cr and approximately 40 g of Zr per run from the gas stream, but it also retained excessive amounts of U and was susceptible to plug formation.

The product collected represented 84.37% of the feed and, a gross recovery (product plus recoverable losses*) of 95.13% of the feed was achieved. CRP trap operation, having had no previous development, caused most of the recoverable losses (7.15% of the feed). Of the uranium put into the system, only 95.16% could be accounted for. The missing 4.84% presumably may be attributed to one observed UF_6 leak (product cylinder valve during Run C-11) and several suspected leaks.

3.0 URANIUM MATERIAL BALANCE

The UF_6 product recovered in product receivers represented 84.37% of the uranium in the feed salt. The poor recovery is tempered somewhat by the fact that 10.76% of the feed was trapped on NaF beds and may be considered recoverable by additional processing.² Therefore, the gross recovery was 95.13%. The CRP trap loss (7.15%) was in excess of that expected in ordinary usage due to the experimental method of operation used in these runs, the primary concern being plant operability and not maximum recovery.

A total of $95.16 \pm 2.44\%$ of the feed was accounted for, which represents a deficit of 1.9 to 4.0 kg of uranium. The only observed loss during processing which could not be measured occurred in Run C-11 when the product cylinder leaked UF_6 . However, after completion of the series, close inspection of the system revealed several spots where small UF_6 leaks had occurred.

The uranium material balance for Runs C-9 through C-15 is tabulated in Table 3.1.

* UF_6 absorbed on NaF.

Table 3.1. Uranium Material Balance

Run No.	Feed, g U	Product, g U	Product, % of Feed	Recoverable Losses, % of Feed					Non- Recoverable Loss, % of Feed (In Waste)	Total Accounted For, % of Feed
				CRP Trap	Absorption	Cold Trapping	Product Transfer	Total		
C-9	7,886	5,638	71.49	14.53	0.84	0.11	1.66	17.14	0.06	88.69
C-10	7,918	7,080	89.42	7.33	0.05	0.05	0.37	7.80	0.01	97.23
C-11	7,372	5,768	78.24	-	0.16	0.01	0.07	0.24	0.01	78.49
C-12	7,833	8,243	105.23	3.03 ^b	1.30	0.08	1.52	5.93	0.04	111.20
C-13	7,947	5,592	70.37	20.12	0.88	0.20	0.89	22.09	<0.01	92.47
C-14	8,779	7,215	82.18	4.36	3.34	0.03	1.07	8.80	0.02	91.00
C-15	7,797	7,316	93.83	0.32	2.24	-	1.19 ^c	3.75	0.04	97.62
Clean-out ^a								1.26		
TOTAL	55,532	46,852	84.37	7.15	1.30	0.07	0.98	10.76	0.03	95.16 ^d

a. "Clean-out" shows recovery following runs; includes 620 g recovered by water-flushing of pipes, 46 g caught in heated duct chemical trap, and 32 g remaining on absorber NaF after desorption.

b. Includes C-11 and C-12 losses.

c. Includes cold trapping and product transfer losses.

d. Limit of uncertainty is $\pm 2.44\%$ of feed.

Note: All FV-500 series
are furnaces or heaters.

Fig. 4.1

VOLATILITY PILOT PLANT CELL I FLOWSHEET

FV-No.	Service
100	Fluorinator
101	Furnace Liner
102	Charge Melt Vessel
103	CRP Trap
104	Freeze Valve
106	Freeze Valve
108	Freeze Valve
110	ARE Dump Tank
111	Retort
112	Waste Can
114	ARE Hold Tank
115	Snow Trap

FV-No.	Service
902	Carrier Dolly
912	Shielded Carrier

Note: All FV-500 series
are furnaces or heaters

Fig. 4.2

VOLATILITY PILOT PLANT CELL 2 FLOWSHEET

FV- No.	Service
120	First Absorber
121	Second Absorber
124	Chemical Trap
126	Product Receiver
220	5" Cold Trap
222	6" Cold Trap

4.0 DESCRIPTION OF PROCESS

A batch of fused fluoride salt was melted and drained by gravity flow through a freeze valve and into the fluorinator. Then the UF_4 in the melt was oxidized to volatile UF_6 by bubbling fluorine through the salt. The gas stream from the fluorinator was passed through a NaF bed maintained at $400^\circ C$ (CRP trap) to remove CrF_5 , ZrF_4 , and entrained salt, and then through another NaF bed maintained at $65-150^\circ C$ (first absorber) to absorb the UF_6 . The unabsorbed gas (primarily F_2) was routed through another NaF bed at ambient temperature (FV-124, chemical trap) to trap out any residual UF_6 , and subsequently through a KOH scrubber tower to neutralize the fluorine before being exhausted to the atmosphere.

After completion of the fluorination, the UF_6 was desorbed from the NaF in the absorber when the bed was heated to approximately $400^\circ C$ in an atmosphere of fluorine sweep gas. The gas stream was then directed through the second absorber (FV-121 in system for Run C-15 only), two cold traps in series ($-40^\circ C$ and $-55^\circ C$, respectively), the chemical trap (FV-124), and the KOH scrubber tower. The UF_6 was condensed out of the gas stream as it passed through the cold traps. Any UF_6 that remained in the gas stream, due to incomplete condensation and/or equilibrium vapor pressure was absorbed by the NaF in the chemical trap.

The product transfer system was then evacuated down to an absolute pressure of < 0.5 mm Hg. The cold traps were isolated from the system and heated to approximately $80^\circ C$ to transform the UF_6 to a liquid. Liquid UF_6 drained to the heated (approximately $80^\circ C$) product cylinder. After which the product cylinder was cooled to approximately $0^\circ C$ and a thermal transfer of any residual UF_6 vapors was effected.

The waste salt left in the fluorinator after fluorination was pressure-transferred through a freeze valve into a waste container.

Losses attributed to various stages of the process were determined by dissolution and analysis of the contents of the various chemical traps.³ All of these traps were fixed beds of 1/8-in. NaF pellets.

When the above procedure had been repeated seven times, the absorbers were desorbed at a higher temperature ($600^\circ C$), and then sampled and analyzed. The system downstream from the fluorinator was washed with water to recover any uranium deposited on the pipe walls, and the system upstream was flushed with barren salt.

The equipment flowsheet for the Volatility Process is shown in Figs. 4.1 and 4.2.

5.0 PROCESS MODIFICATIONS

As a result of experience gained in previous runs and the developmental nature of the present runs, the following modifications were instituted in the Volatility Process.

The FV-103 vessel (previously the ZrF_4 trap, now the CRP trap) was modified in order that CrF_3 as well as ZrF_4 might be removed from the gas stream leaving the fluorinator. Nickel mesh, the previous packing in FV-103, was replaced with 1/8-in. NaF pellets. The bed was supported by a Nichrome wire screen. Calrod heaters were built around the trap so that it could be maintained at $400^\circ C$ during the fluorination operation in order to minimize UF_6 absorption. However, adequate heating was not supplied until Run C-14.

A molecular sieve type nitrogen dryer (Kemp Model 15-E) was installed in the nitrogen supply line to reduce the corrosion hazard from water vapor and the plug hazard from the formation of high melting oxides in the molten salt. Subsequently, the H_2O concentration in the nitrogen to the process never exceeded 2 ppm whereas the nitrogen supply contained up to 60 ppm.

An HF trap⁴ was installed in the fluorine supply line prior to Run C-14 to remove the 2 to 5% HF present in the F_2 . The desirability of preventing HF from entering the system became evident in previous runs. HF absorption on NaF reduces the UF_6 capacity of the absorbers, and at temperatures $< 100^\circ C$ HF-NaF form liquid compounds which represent a problem from the standpoint of plug formation.

An air blast was directed onto the first absorber during absorption of UF_6 to minimize the resulting temperature rise. Since the concentration of UF_6 in the exit gas from the absorber is a function of bed temperature, the lower bed temperature should result in lower UF_6 losses (Fig. 12.25).

Both instrument and blanket nitrogen supplies were adjusted to a pressure of 4.5 psig in order to minimize the possibility of plug formation resulting from molten salt being forced into unheated lines. Also, the F_2 pressure was reduced to approximately 4 psig to minimize salt movement by pressure differentials.

6.0 FEED SALT TRANSFER

6.1 Plant Operation

In all runs the transfer of salt from FV-102 to FV-100 proceeded

smoothly. Four cans of salt were melted in FV-102 and combined with the heel of salt in FV-100 from the previous run. About 20 kgs of waste salt was left in FV-100 after each run (zero heel after Run C-12) to assure adequate submergence of the draft tube in the succeeding run.

In Runs C-9 and C-13 the level of the salt charge was only slightly above the draft tube, and less mixing of the melt by fluorine could be expected. This, in turn, would tend to decrease the efficiency of fluorination.⁵ The magnitude of the salt charge for the various runs is listed in Table 6.1.

Table 6.1. Salt Charge in the Fluorinator

Run	Heel,* kg	Charge, kg	Total Feed		Height Above Top of Draft Tube, in.	U in Feed, g
			Wt., kg	Vol, liters		
C-9	16.5	113.2	129.7	38.7	1	7,886
C-10	65.1	116.5	181.6	55.9	10	7,918
C-11	55.7	109.6	165.3	51.7	7	7,372
C-12	50.9	117.2	168.1	52.5	8	7,833
C-13	**	117.6	128.8	39.0	1	7,947
C-14	47.0	117.1	164.1	49.7	7	8,779
C-15	48.8	108.4	157.2	47.6	6	7,797

* From previous run.

** Level recorder does not indicate < 2 liters.

6.2 Feed Salt Composition

The feed salt was of the type, $\text{NaF} + \text{ZrF}_4 + \text{UF}_4$. Since this material had been previously used in loop studies, it contained a significant amount of corrosion products prior to entering the Volatility Pilot Plant. Table 6.2 lists the general composition of the feed salt.

Table 6.2. Feed Salt Composition

Component	Weight, %	Component	Weight, %	Composition*	Weight, %
U	4.4 - 6.2	Cr	0.06 - 0.10	UF_4	5.8 - 8.0
Zr	35 - 44	Ni	0.08 - 0.18	ZrF_4	65 - 80
Na	9.0 - 10.4	Fe	0.01 - 0.02	NaF	18 - 19
F	40 - 42	Si	0.002 - 3.4		
		Ti	0.01 - 0.60		

* Calculated on basis of U, Zr, and Na analyses.

7.0 FLUORINATION AND ABSORPTION

7.1 Plant Operation

The major operational difficulties encountered during fluorination--line plugs and salt sampling--caused deviations from the standard procedure.

Plugs resulting from molten salt rising into unheated parts of the fluorine inlet line to the fluorinator and then freezing (m. p. approximately 530°C) were very troublesome. Generally, such situations were caused by a pressure surge in the fluorinator when fluorine was not flowing. After Run C-11 valves on the lines into the fluorinator were rearranged to decrease the possibility of pressure surges. When a plug did develop, it was necessary to heat the line with a "Prepo" torch and/or rod out the salt. Such techniques are not amenable to remote operation and thus present a significant hindrance to the processing of radioactive materials.

Numerous plugs in the UF₆ outlet from the fluorinator occurred in the CRP trap (Sect. 7.4). Additional troubles resulted from inadequate bed support by the wire screen, with a resulting obstruction to gas flow caused by the NaF pellets which fell into the line downstream from FV-103.

Sampling of molten salt in the fluorinator continued to be less than perfect.⁵ The ladles hung in the sample line on several occasions and the line became completely plugged (probably salt) at other times. This required lowering the ladles through the low pressure probe of the liquid level indicator during sampling in several of the runs. To alleviate the problem of removing salt samples from the ladles, some samples were taken in copper instead of graphite ladles. Attempts by analytical personnel to dissolve the copper ladles simultaneously with the salt samples, and then analyze the entire solution were unsuccessful due to the presence of interfering ions in the solution. Subsequently, the salt samples were removed en masse by heating the ladle, which then expanded and permitted the salt to drop out. Samples from later runs were pulverized in the ladle and poured out as a powder. In this form the salt could be dissolved more easily.

The density indicator for the salt in the fluorinator ceased operating during Run C-15. When the fluorinator was disassembled at the conclusion of the series of runs, the low pressure density probe was found to be severed about one foot above the molten salt level (Fig. 7.1). The probe, 3/8" schedule 40 nickel pipe, had undergone severe corrosion.

A summation of the various operational difficulties is presented in Table 7.1.

Fig. 7.1. Gas Dispersion Assembly Removed from the Fluorinator After Seven Flowsheet Demonstrations.

Table 7.1. Operational Problems during Fluorination

Run	F ₂ Inlet to FV-100	FV-103	Other
C-9	Was discovered to be plugged when fluorination was attempted; relieved by heating line above FV-100 flange.	Could not attain uniform temperature throughout NaF bed (390°C at middle and 175°C at bottom).	Cu sample ladles were not amenable to complete dissolution.
C-10	---	---	Obstruction developed in sample line to FV-100. Low pressure probe for LR-2 was used for sample line. Salt was removed from Cu ladles by heating.
C-11	---	---	---
C-12	---	Plugged 17 min after F ₂ was started ($\Delta P = 3$ psig). Top layer of pellets (slightly caked) was removed to relieve pressure.	---
C-13	Was discovered to be plugged after FV-103 was recharged. Soft deposit was rodded out.	Bed of 12-20 mesh NaF plugged 36 min after F ₂ was started ($\Delta P = 7.5$ psig). During a period of no F ₂ flow ΔP was relieved. Plugged again 6 min after F ₂ was restarted ($\Delta P = 2$ psig). Entire bed was replaced with fresh NaF, 1/8-in. pellets.	---
C-14	Was discovered to be plugged prior to fluorination; probably occurred when N ₂ supply ran out. Line was rodded out.	Plugged 40 min after F ₂ was started ($\Delta P = 3$ psig). Replacing used part of bed with fresh NaF did not relieve ΔP . Plug was eliminated when approximately 100 g of pellets were removed from line between FV-103 and FV-120.	---
C-15	Was discovered to be plugged after last plug in FV-103. Had to be heated with torch and then rodded out. Became plugged again during sampling.	Plugged 48 min after F ₂ was started ($\Delta P = 2.6$ psig). Relieved ΔP by heating bottom of FV-103 with torch. Several subsequent plugs interrupted F ₂ flow, but were relieved by heating. After the last plug the vessel was opened and the bed found to be caked hard.	High ΔP (2.4 psig) developed near FV-120. Sample ladles hung in line and could not be retrieved.

7.2 Corrosion

After the waste salt transfer in Run C-15 the fluorinator was disassembled and inspected. The gas dispersion assembly (including instrument probes and corrosion specimens) is shown in Fig. 7.1. All surfaces that were below the molten salt level were shiny and clean, but had definite evidence of pitting. Those areas exposed to the vapor phase were covered with salt scale and corrosion products. A study of the corrosion was made by Metallurgy Division personnel.⁶

The interior of the fluorinator is shown in Fig. 7.2. Scale (fluoride salt and/or corrosion products) covered the surfaces above the molten salt level. Below this level the walls were clean. A ring of material adhered to the wall at the salt level (about half way down in Fig. 7.2).

Samples of scale were taken from various locations and analyzed (Table 7.2). Apparently, considerable corrosion resulted from fluorination of the nickel vessel (FV-100) to form NiF_2 .

Table 7.2. Fluorinator Scale Analysis

Origin of Sample	Component, Wt. %						Formula (by X-ray Diffraction)
	U	Na	Ni	Cr	Zr	F	
Underside of flange	1.95	0.78	45.54	0.79	0.72	39.60	90% NiF_2 10% $\text{NaF} \cdot \text{NiF}_2 \cdot 2\text{ZrF}_4$
From F_2 inlet tube, 25 cm above salt level	0.98	5.10	33.76	0.09	0.64	41.15	60% NiF_2 30% $\text{NaF} \cdot \text{NiF}_2 \cdot 2\text{ZrF}_4$ 10% $\beta_2 \text{ 2NaF} \cdot \text{ZrF}_4$
From F_2 inlet tube, at salt level	0.13	6.64	8.36	0.02	1.18	43.30	---
Feed salt	4.96	9.95	0.11	0.07	40.0	40.86	---

7.3 Fluorine Utilization

Fluorine flow rates from 6.7 to 33.4 slm during fluorination were investigated in order to discover optimum conditions for the reaction $\text{F}_2 + \text{UF}_4 \longrightarrow \text{UF}_6$. Flow rates below 10 slm appeared undesirable due to inadequate mixing of the molten salt. Of the other flow rates tested, none showed any particular advantage over the others. However, the possibility of excessive corrosion and erosion in the fluorinator and entrainment of particles in the gas stream becomes greater at the higher flow rates. Flow rates in the range of 10 to 30 slm are therefore recommended.

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Fig. 7.2. Interior of the Fluorinator After Seven Flowsheet Demonstrations.

It seems reasonable to assume that fluorine utilization is affected by flow rate, quantity of salt, uranium concentration, and the quantity of impurities in the salt. However, the data from Runs C-9 through C-15 are somewhat scattered and show no apparent correlation (Table 7.3 and Fig. 7.3). The breakthrough points for UF_6 and F_2 --when UF_6 began to volatilize from the melt and when fluorine was present in excess in the melt--were considered indications of the effectiveness of fluorination.

Table 7.3. Fluorine Utilization

Run	F_2 Flow Rate, slm	Total F_2 , moles F_2/U^*	Breakthrough		Uranium Volatilized, % of Initial
			UF_6 , moles F_2/U^*	F_2 , moles F_2/U^*	
C-9	6.7	5.42	1.06	2.62	99.94
C-10	27.2	5.81	0.91	2.18	99.99
C-11	10.0	6.76	0.89	2.22	99.99
C-12	33.4	5.88	0.75	1.64	99.96
C-13	20.7	6.50	0.90	2.00	99.99 ⁺
C-14	22.9	5.49	0.67	1.54	99.98
C-15	12.3	6.95	1.02	2.30	99.96

* Moles of initial uranium.

Excessive fluorine used in the runs may be attributed to the method of operation. After fluorination of the uranium was essentially complete, as determined by a minimum salt density being attained, fluorine flow was stopped so that the molten salt could be sampled. During the waiting period for analysis of the sample (2 to 4 hr), excess fluorine was bubbled through the melt to assure complete removal of uranium from the salt.

7.4 CRP Trap Performance

Performance of the CRP trap was unsatisfactory when considered from the standpoint of operational difficulties, uranium retention, and removal of Cr and Zr from the gas stream. However, the basic idea (NaF pellets to remove ZrF_4 and CrF_5) appears sound, and a redesign of the trap could improve the situation.

For Runs C-9 through C-15 the former ZrF_4 vapor trap (FV-103) was packed with 1/8-in. NaF pellets (approximately 11 kg) instead of nickel mesh in order that other volatile or entrained fluorides in the gas stream (especially CrF_5) might be removed. Prior to Run C-9, the deposition of chromium fluorides in valves downstream from FV-103 was considered one of the principal causes of valve leakage.

Fig. 7.3. Fluorination of fused salt

Table 7.4. CRP Trap Data

Run	Component g			Position of Major Conc. in Bed			Bed Temperatures, ** °C*	
	U	Cr	Zr	U	Cr	Zr	Midpoint	Bottom
C-9	1146	*	19	top (gas inlet) and bottom (gas outlet)	--	throughout	400	165
C-10	580	*	23	bottom	--	throughout	400	170
C-11 + C-12	237	*	16	bottom	--	throughout	400	170
C-13, a	1470	1	18	--	--	--	405	260
C-13, b	129	67	30	bottom	top	top	420	260
C-14, a	379	8	24	top	top	top	390	370
C-14, b	4	36	16	throughout	top	throughout	410	400
C-15	25	59	46	top	top	throughout	450	425

* Cr analyses were considered unreliable due to possible contamination from stainless steel dissolution drums.

** Since temperatures were measured at the vessel wall, it is probable that the central portion bed was somewhat cooler.

Table 7.4. CRP Trap Data (Continued)

Comments	
Operational	Bed Appearance When Removed
	Upper 1/3 was pink, which turned to brown upon exposure to air. Bottom 1/3 was yellow and was caked hard.
	Upper 1/3 was pink, which turned to brown upon exposure to air. Bottom 1/3 was yellow and was caked hard.
	Pink to brown pellets were scattered throughout the bed.
Plugged when UF ₆ started volatilizing. Bed was 12-20 mesh NaF.	Top 1 in. was relatively dense and was pink to brown in color. Bottom 1/2 was yellow.
	Bed was caked. Upper 1/2 was primarily pink to brown. About 15% of pellets in bottom 1/10 were yellow.
Plugged when UF ₆ started volatilizing. Support screen was out of place and pellets plugged outlet line.	No caking was evident. About 10% of pellets were light green.
	Upper 1/3 was medium green. Bottom 1/5 was light green.
Plug which occurred when UF ₆ started volatilizing was relieved by increasing temperature from 425 to 490°C.	Bed was very firmly packed. Pellets in upper part were pink, which turned to brown upon exposure to air.

Considerable difficulty was encountered with the operation of FV-103 in almost all runs. Frequent plugs occurred which necessitated interruptions of the fluorine flow until the resulting pressure could be eliminated. Various methods were used to reduce this pressure drop, e. g., additional heating, replacement of the bed with fresh NaF, and nitrogen back pressure. Plugs quite often developed immediately after UF_6 started volatilizing from the melt. At the present operating conditions, the bed appeared to be incapable of handling the initial surge of gas without plugging.

Uniform bed temperatures were particularly difficult to attain. Although the midpoint of the vessel wall was maintained at 400°C , not until Run C-14 could the temperature in the bottom of the bed (gas outlet) be increased to 400°C , due to insufficient insulation and heating equipment. Lower temperatures favor the absorption of UF_6 on NaF, and consequently, considerable uranium was retained on the bed in most of the runs (Table 7.4).

Cr tended to collect at the gas inlet (top of bed) which would indicate some sort of sorption reaction of CrF_5 with NaF. Zr apparently was removed from the gas stream by a filtering action since it deposited throughout the bed.

Sections of the beds, which were shown by chemical analysis to be relatively concentrated with Cr, were composed of pellets which were pink immediately after the bed was removed from the trap. Upon exposure to air the color of the pellets turned progressively from pink to brown or green. These colors are indicative of successively lower Cr oxidation states, i. e., CrF_5 , CrF_4 , and CrF_3 or CrF_2 . The pellets in those sections of the bed containing U were predominately yellow.

7.5 Absorber Performance

7.5.1 Uranium Losses

Absorption losses-- UF_6 which passed through (or possibly through piping around) the absorber bed and was absorbed on NaF in the chemical trap--were excessively high for most of the runs (Table 7.5). These losses are dependent on several factors: (1) gas dispersion in the bed, (2) leaky valves, (3) temperature of the bed, and (4) total gas throughput. Repeated thermal cycling of the NaF bed could have resulted in parts of the bed becoming fused and channeled. When the bed was removed after the final desorption (at 600°C), it was caked throughout but no channels were visible. Valve tests performed after Run C-15 indicated that most of the process valves were leaking. Since the reliability of a valve would decrease as its service time increased, greater losses would be expected in the later runs. This general trend is indicated in Table 7.5.

Table 7.5. Absorption Losses

Run	Uranium in FV-124	
	g of U	% of Feed
C-9	66	0.84
C-10	4	0.05
C-11	12	0.16
C-12	102	1.30
C-13	70	0.88
C-14	293	3.34
C-15	175	2.24

Temperature affects both the rate of the absorption reaction and the equilibrium concentration of UF_6 in the gas leaving the bed. The reaction rate has been shown to be proportional to $\log T$ ($< 125^\circ\text{C}$),⁷ and the UF_6 concentration is a function of vapor pressure, which increases with temperature (Fig. 12.25). Thus, lower UF_6 loss would be favored by a higher temperature in one case and a lower temperature in the other. Heretofore, reaction rate has been of little consequence due to the low flow rates (approximately 10 slm), but at increased flow rates (> 30 slm) it could become a factor.

7.5.2 Bed Temperatures

Control of the NaF bed temperatures was necessary to prevent (a) excessive UF_6 losses to the exit gas and (b) deposition of solid UF_6 at the absorber gas inlet. Temperatures of various parts of the FV-120 bed during absorption of UF_6 are shown in Figs. 12.2 through 12.8. Compressed air, circulated in the annular space between the first absorber and its furnace (FV-520), reduced the temperature rise resulting from the UF_6 -NaF absorption reaction.* During absorption of UF_6 in the series of runs the maximum bed temperature attained was 150°C .

A small temperature rise (approximately 5°C) that occurred in the top of the bed in Runs C-14 and C-15 was indicative of UF_6 absorption in that region. With good gas dispersion, the bed capacity and reaction rates were such that all the UF_6 should have absorbed in the lower half of the bed. Sudden temperature rises occurred in the chemical trap prior to salt sampling in Runs C-9 and C-14 and shortly after the previously mentioned temperature rises in the top of the bed. These may be considered indications of absorption losses.

8.0 DESORPTION OF UF_6

8.1 Plant Operation

Few operational problems arose during the desorption phase of the process. Occasionally, an excessive pressure rise occurred in the gas line to the scrubber. NaF fines in the gas stream were coagulated by KOH spray, and deposited at the gas inlet to the scrubber tower. The resulting obstruction could be relieved easily by rodding out the plug.

Two incidences of improper valving procedure occurred. Three hours after fluorine was started for desorption in Run C-12, a valve which allowed the fluorine supply to flow to either the fluorinator or the absorber was found open. In Run C-13 a valve which allowed the gas stream from the absorber to go to either the fluorinator or the cold traps was mistakenly left open and was discovered two hours after the start of fluorine flow.

The second absorber was delayed for structural repairs and could not be installed in the system until Run C-15. In the previous runs the desorption gas stream from FV-120 was routed directly to the cold traps.

8.2 Absorber Performance

Over-all performance of the first absorber during desorption, with the same NaF bed being used for seven consecutive runs, was satisfactory. Both absorber beds were sampled and analyzed by the standard technique³ after the additional, high temperature desorption.

8.2.1 Desorption Losses

Uranium which remained on the absorber beds after desorption was considered a desorption loss. This loss for Runs C-9 through C-15 is shown in Table 8.1 and Fig. 8.1.

Table 8.1. Uranium Retention in Absorbers

After Run C-15	<u>After Extra Desorption</u>	
<u>Desorption</u>	<u>75° - 400°C</u>	<u>400° - 600°C</u>
32 g	25 g	18 g

The loss was considered acceptable (0.06% of the total feed) for the desorption phase of the process. Smaller relative losses may be expected with larger uranium feeds since the absolute loss was shown not to be additive. Previous desorption losses in single runs; C-6, C-7, and C-8, were 22, 21, and 13 g of uranium, respectively.

881 023

Fig. 8.1. Uranium distribution in the first absorber bed after desorption

Fabrication difficulties delayed the installation of the 2nd absorber until Run C-15, and therefore it was exposed to UF_6 for only one run. All the uranium found after the extra desorption (Table 8.1) was in the 1st absorber. About 78% of the uranium in FV-120 remained on that part of the NaF bed below the gas diffuser ring (Fig. 8.1). Possibly the absence of adequate fluorine sweep gas in that region inhibited the desorption reaction, which suggests the use of an inert filler material, e. g., nickel shot or CaF_2 pellets, for the bottom layer of the absorber bed.

8.2.2 Physical Condition of the Absorber

When the absorbers were examined and sampled after the final desorption (600°C), the entire bed in FV-120 was found to be fused. However, no channels through the bed were visible. Dark green and brown pellets were present in the top layer (approximately 2 in.) of NaF, and a few black pellets were on top of the center pipe. Enough pale green flakes (approximately 3 in. by 4 in.) from the interior wall of the absorber had fallen onto the top of the bed to obscure it from view. These flakes were analyzed and shown to be $> 93\% \text{NiF}_2$. The interior of FV-120 after the bed had been removed is shown by Fig. 8.2.

The second absorber showed few effects due to its short term operation. No caking or channel formation in the bed was apparent, and about 10% of the pellets in the top NaF layer were yellow or orange.

8.2.3 Absorber Temperatures

The temperatures existing in the NaF bed of FV-120 were recorded for the desorption operation and are included in the Appendix (Figs. 12.9 through 12.17). The various temperatures followed the same general pattern for all runs except Run C-13, in which the desorption time was relatively short. Generally, a period of 10 to 12 hours was required for complete desorption, and in every run the absorber wall temperature was allowed to exceed 400°C^* in order to decrease the time required to attain a uniform bed temperature of approximately 400°C . A plateau in the temperature curves of the middle and the lower sections of the bed was evident at temperatures of 300 to 350°C . This plateau denoted the period when the rate of UF_6 desorption was greatest**. In Run C-13 only 7.5 hours were required to attain a bed temperature of 400°C , and the temperature curve plateaus were of relatively short duration. It might also be noted here that the greatest uranium loss after the desorption operation*** occurred during Run C-13 (Sect. 8.3).

* Recommended maximum allowable temperature in absorber.

** $\text{UF}_6 + 3\text{NaF} \xrightarrow[\text{heat}]{\text{F}_2} \text{UF}_6 + 3\text{NaF}, \quad \Delta H = +23.2 \text{ kcal.}$

*** These losses were designated "cold trapping losses" because the U in the desorption gas stream should have condensed out in cold traps before reaching the chemical trap.

Fig. 8.2. Interior of First Absorber After Removal of the NaF Bed.

8.3 Chemical Traps

Analysis of NaF bed in FV-124 after desorption indicated that the quantity of uranium which passed through the cold traps without condensing (cold trapping loss) was tolerable for Runs C-9 through C-15. This loss is itemized in Table 8.2.

Table 8.2. Cold Trapping Loss

Run	Uranium	
	Weight, g	% of Feed
C-9	9	0.11
C-10	4	0.05
C-11	1	0.01
C-12	6	0.08
C-13	16	0.20
C-14	3	0.03
C-15	*	*
Total	39	0.07

* Mistakenly not sampled after desorption.

8.4 Process Data

Pertinent process data obtained during the desorption operation are listed in Table 8.3 and Figs. 12.9 through 12.17.

Table 8.3. Process Data for Desorption

Run	Flow Rate, slm F ₂	Temperature, °C				Pressure, psig	
		First Absorber		Cold Traps		Scrubber Solution	Maximum Δ P Downstream from FV-120
		Wall	Bed	FV-220	FV-222		
C-9	6.6	70-460	60-390	-45	-55	31	0.02
C-10	6.5	85-435	60-390	-43	-53	29-42	0.04
C-11	6.8	65-470	50-420	-41	-53	39	0.04
C-12	6.6	60-465	40-410	-41	-53	36-47	0.40
C-13	6.8	90-465	60-410	-41	-48	36-46	0.10
C-14	5.4	70-430	40-390	-42	-51	41	1.80*
C-15	5.5	90-435	70-395	-42	-46	34-46	0.04

* Pressure buildup resulting from a plug at the gas inlet to the scrubber necessitated interrupting the desorption in order to remove the plug.

9.0 PRODUCT TRANSFER

9.1 Plant Operation

The principal problem pertaining to the product transfer operation was leakage of gaseous UF_6 . Leaks generally occurred at or near the two product cylinder (FV-126) valve connections to the product drain line. After the completion of product transfer in Run C-9, the product receiver valve could not be completely closed. Additional heating of the valve was required to eliminate the trouble, which apparently was a deposit of solidified UF_6 . During Run C-11, a UF_6 leak occurred at the product receiver valve connection to the drain line. From visual observation the leak was judged to be slight, but its duration was unknown. The leak was discovered about 20 min after that part of the system was exposed to UF_6 and another 30 min were required to repair it.

Equipment trouble was experienced in Run C-10 when a breakdown of the vacuum pump delayed completion of the product transfer procedure approximately 32 hr. Solids which formed in the MFL pump oil (possibly a reaction with UF_6) caused the pump to clog. A spare pump was installed to complete the operation and remained in the system through product transfer for Run C-11.

A possible change in the product transfer procedure was investigated after Run C-14. Purging of the product transfer lines and the FV-126 connectors was shown to be unnecessary after the thermal transfer. Prior to the final purging step in the normal procedure, the cold traps were rechilled to cause any residual UF_6 vapors in the line to migrate back to the cold traps. After a 4-hr waiting period any residual UF_6 was swept out of the lines by a N_2 purge. It was decided that the quantity of UF_6 (13 g) remaining in the lines was sufficiently small to warrant elimination of the purging step.

9.2 UF_6 Product

Product from each run was collected in a standard shipping cylinder (25 kg UF_6 capacity) and then weighed. The various product collections are itemized in Table 9.1.

9.3 Chemical Traps

UF_6 caught in two chemical traps, FV-122 and FV-124, before and after product transfer was considered excessive for all runs except C-11 (Table 9.2). This material, termed product transfer losses, resulted from evacuation of the system prior to liquid transfer and the N_2 purge of the lines after thermal transfer. The large loss in Run C-9

Table 9.1. UF_6 Product Recovery

Run	Product Collected in FV-126		
	UF_6 , kg	U*, g	% of Feed
C-9	8.34	5,638	71.49
C-10	10.47	7,080	89.42
C-11	8.53	5,768	78.24
C-12	12.19	8,243	105.23
C-13	8.27	5,592	70.37
C-14	10.67	7,215	82.18
C-15	10.82	7,316	93.83
Total	69.29	46,852	84.37

* Based on theoretical quantity of U in UF_6 .

Table 9.2. Product Transfer Losses

Run	Uranium in FV-122, g	Uranium FV-124, g	Total Uranium	
			Wt., g	% of Feed
C-9	47	84	131	1.66
C-10	17	12	29	0.37
C-11	<1	5	5	0.07
C-12	<1	119	119	1.52
C-13	55	16	71	0.89
C-14	85	9	94	1.07
C-15	15	73	93	1.13
Total			542	0.98

probably resulted from UF_6 in the cylinder being re-volatilized into the line when the FV-126 valve was heated to facilitate closure (Sect. 9.1). Excessive loss in Run C-14 may be attributed to incorrect valving procedure. A valve upstream from the vacuum pump was mistakenly left open which permitted the pump (which runs continuously) to pull gas through FV-122 much longer than was done ordinarily. This gas contained UF_6 , the quantity being dependent on the UF_6 vapor pressure at the time.⁸

An empirical relation between the quantity of uranium trapped in FV-122 and the time required to evacuate the system prior to liquid transfer was obtained. In Fig. 9.1 this is shown to be $U = 0.9 t^{-1.1}$, where U is grams of uranium and t is evacuation time in seconds.

9.4 Process Data

Pertinent process data recorded during the product transfer operation are plotted in Figs. 12.18 through 12.24. Several of the product cylinders were not equipped with thermocouple wells and therefore this information is missing for the respective runs. The sudden dip in the cold trap temperature which invariably occurred during the heating up period may be considered the first indication of liquid UF_6 in that region.

10.0 WASTE SALT TRANSFER

10.1 Plant Operation

Inability to initiate a waste salt transfer on the first attempt in Runs C-9, C-10, and C-15 indicated the unreliability of this procedure. This can be attributed largely to partial or total blockage of transfer and instrument lines with salt. Control of the salt flow was also uncertain as evidenced by inability to leave a predetermined quantity of salt in FV-100. However, a rearrangement of valve connections on FV-100 after Run C-12, alleviated the situation somewhat in that the liquid level instrument then gave a more accurate indication of the salt level during transfer.

A serious defect in the heating of the transfer line was discovered at the waste salt outlet nozzle. The temperature of the nozzle could not be maintained high enough with the autoresistance heating to prevent salt solidification. Thus, salt had to be drilled and/or rodded out of the nozzle prior to the transfers.

10.2 Process Data

The data in Table 10.1 represent average values recorded during salt transfer. The temperatures were maintained for only 10 to 30 min since the actual transfer required approximately 5 min.

Fig. 9.1. Uranium loss incurred during evacuation of product transfer system

Table 10.1. Process Data for Waste Salt Transfer

Run	Pressure Required to Start Transfer, psig	Temperature, °C					
		Freeze Valve			Molten Salt Piping		
		max	min	avg	max	min	avg
C-9	8.0	705	595	690	815	625	790
C-10	6.5	610	525	600	700	625	650
C-11	4.6	625	585	600	755	620	720
C-12	5.2	*	*	*	*	*	*
C-13	8.0	610	580	600	795	650	700
C-14	4.8	630	600	610	780	610	730
C-15	7.2	625	585	620	825	625	780

* Temperature recorder was not operating properly.

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12.0 APPENDIX

12.1 Run Schedule

Table 12.1. Run Schedule

Run No.	Date Started	Time Started	Date Ended	Time Ended	Total Time (hours)
C-9	7- 8-57	0815	7-12-57	0600	94
C-10	7-13-57	0005	7-16-57	1300	85
C-11	7-16-57	0240	7-18-57	1715	62.5
C-12	7-22-57	0630	7-24-57	1125	53
C-13	7-23-57	1950	7-26-57	0930	59.5
C-14	7-26-57	1700	7-30-57	2040	63.5
C-15	7-30-57	1805	8- 3-57	0123	55.5

12.2 Accuracy of Calculations

The various measurements on which the material balance calculations were based are subject to certain limits of accuracy (Table 12.2).

Table 12.2. Measurement Accuracy

Calculation	Measurement	Limit of Uncertainty
Feed	Level recorder in fluorinator	$\pm$ 0.9 kg
	Potentiometric analysis for U	$\pm$ 0.5 %
Product	Weight	$\pm$ 20 g
Losses	Sampling plus analysis for U	$\pm$ 10 % (est)

The variance for a specific calculation, involving more than one measurement, was determined by the square root of the sum of the squares technique. These variances represent a minimum because of additional measurements for which there is no basis for estimating limits of error. These measurements include sampling of the molten salt and the purity of the product, which was assumed to be 100% UF_6 .

12.3 Uranium Holdup in System

After the conclusion of the seven flowsheet demonstrations and the subsequent high-temperature desorption, the salt-handling part of the system was flushed with barren salt, and the remainder of the system (everything downstream from the CRP trap) was flushed with water.

Analyses of the resulting solutions indicated the uranium holdup in the system (Table 12.3). After three salt flushes and five water flushes uranium recovery appeared to be essentially complete. A more detailed presentation of the water flushes is shown by Fig. 12.1.

Table 12.3. Uranium from Flush of System

No. of Flush	U Recovered by Salt Flush, g	U Recovered by Water Flush, g
1	*	478
2	67	149
3	2	97
4		59
5		31
FV-120**		1
FV-121**	-	3
	—	—
Total	69	818

* No analysis of salt was made.

** Wash of specific pieces of equipment

From the above total only 620 g of uranium were attributed to Runs C-9 through C-15 because the line from FV-103 to FV-120 had been exposed to UF_6 in previous runs (C-4 through C-8). The assumption was made that only half the uranium in that region could be attributed to the latest series of runs.

12.4 Process Data

Data recorded during processing are shown in Figs. 12.2 through 12.24.

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Fig. 12.1. Uranium recovered by water flushing the UF_6 handling part of the system

Fig. 12.2. Temperatures in the first absorber during absorption of UF_6 in run C-9

Fig. 12.3. Temperatures in the first absorber during absorption of UF₆ in run C-10

Fig. 12.4. Temperatures in the first absorber during absorption of UF₆ in run C-11

Fig. 12.5. Temperatures in the first absorber during absorption of UF₆ in run C-12

Fig. 12.6. Temperatures in the first absorber during absorption of UF₆ in run C-13

Fig. 12.7. Temperatures in the first absorber during absorption of UF₆ in run C-14

Fig. 12.8. Temperatures in the first absorber during absorption of UF₆ in run C-15

Fig. 12.9. Temperatures in the first absorber during desorption of UF_6 in run C-9

Fig. 12.10. Temperatures in the first absorber during desorption of UF_6 in run C-10

Fig. 12.11. Temperatures in the first absorber during desorption of UF_6 in run C-II

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Fig. 12.12. Temperatures in the first absorber during desorption of UF_6 in run C-12

Fig. I2.13. Temperatures in the first absorber during desorption of UF_6 in run C-13

Fig. 12.14. Temperatures in the first absorber during desorption of UF_6 in run C-14

Fig. 12.15. Temperatures in the first absorber during desorption of UF_6 in run C-15

Fig. 12.16. Temperatures in the second absorber during desorption of UF_6 in run C-15

050 188

Fig. 12.17. Temperatures in the first absorber during extra desorption of UF_6 after completion of run C-15.

Fig. 12.18. Product transfer conditions in run C-9

Fig. 12.19. Product transfer conditions in run C-10

881 053

Fig. 12.20. Product transfer conditions in run C-II

Fig. 12.21. Product transfer conditions in run C-12

Fig. 12.22. Product transfer conditions in run C-13

Fig. 12.23. Product transfer conditions in run C-14

881 057

Fig. 12.24. Product transfer conditions in run C-15

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Fig. 12.25. Equilibrium Partial Pressure of UF₆ Over UF₆ · 3NaF Complex as a Function of Temperature.

GLOSSARY

EQUIPMENT CODE NUMBERS USED IN THE VOLATILITY PROCESS

FV-100 Fluorinator
FV-102 Charge melt vessel
FV-103 Complexible Radioactive Products (CRP) Trap
FV-106 Freeze valve in line from FV-100 to FV-112
FV-108 Freeze valve in line from FV-102 to FV-100
FV-112 Waste salt container
FV-120 First absorber
FV-121 Second absorber
FV-122 Chemical trap for product purge system
FV-124 Chemical trap in main process system
FV-126 Product receiver
FV-150 Scrubber for removal of F_2 from off-gas
FV-152 Caustic surge tank
FV-158 Chemical trap for heated duct system
FV-220 First cold trap (5 in. dia)
FV-222 Second cold trap (6 in. dia)
FV-420 Vacuum pump
FV-500 Fluorinator furnace
FV-502 Charge melt vessel furnace
FV-506 Freeze valve furnace, for FV-106
FV-508 Freeze valve furnace, for FV-108
FV-520 First absorber furnace
FV-521 Second absorber furnace
FV-526 Product receiver heater

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