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SUBJECT: Equipment Checkout Prior to ARE Processing,
Volatility Pilot Plant Run B-1

TO: F. L. Culler

FROM: C. L. Whitmarsh

ABSTRACT

The head-end system for the ARE salt was tested with barren salt and shown to be operable. Transfer of the final batch of salt by gravity flow from the hold tank was shown to be feasible with a redesigned feed salt freeze valve. The CRP trap was also redesigned but the remote dumping mechanism did not function properly.

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4-1-59 *H. L. Culler*
DATE SIGNATURE

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1.0 INTRODUCTION

The Fluoride Volatility Process was developed on a laboratory scale¹ to recover uranium from fused salt reactor fuels. The process consists of volatilization of UF_6 from molten salt by fluorination, 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 flowsheet demonstration runs have been made with nonirradiated salt and thereby have demonstrated that the plant is operable.

The Volatility Pilot Plant Run B-1 was performed to check the operability of the head-end equipment for the fuel from the Aircraft Reactor Experiment. This included a dump tank furnace and liner, a salt hold tank, and the redesign of a freeze valve. Modification of the freeze valve was necessitated by the probability that the transfer of the last batch of salt from the hold tank during ARE processing would blow the seal, and thus hinder the following fluorination operation. In addition, the CRP trap (FV-103) was redesigned and relocated to facilitate remote discharge of its NaF bed.

Barren fluoride salt from a portable furnace was pressurized into the hold tank and drained to the fluorinator to check the operability of the modified freeze valve. Later, when the dump tank furnace and its liner had been installed, salt was melted in this vessel and drained to the hold tank. Seven pressure transfers of the salt were made from the hold tank to the fluorinator. Salt which accumulated in the fluorinator was periodically transferred to the waste receiver. To check the CRP trap, pellets were loaded into the vessel, exposed to N_2 sparging and heating cycles, and then dumped into the fluorinator.

2.0 SUMMARY

Performance of the head-end equipment for handling the ARE salt was satisfactory. Operation of the modified freeze valve illustrated that the last batch of salt in the hold tank could be drained by

gravity flow to the fluorinator. Salt (approximately 340 kg) which was melted in the dump tank furnace liner drained to the hold tank. Seven pressure transfers from the hold tank to the fluorinator were successfully made and controlled, as were the three from the fluorinator to the waste receiver. Some difficulty was encountered in starting the initial transfer from each vessel but the remaining transfers proceeded smoothly.

The remote discharge mechanism for the CRP trap failed to operate successfully, primarily because of sintering of the NaF pellets.

3.0 PLANT OPERATION

To check out the modified (FV-104) freeze valve, molten salt was transferred from a portable furnace to the hold tank, from which it was drained intermittently by controlling the freeze valve temperature. Twelve additional cans of salt were then melted in the dump tank furnace liner (FV-111) and drained into the hold tank (FV-114). The salt was pressure transferred in seven batches into the fluorinator (FV-100). Transfer of the salt to the waste container (FV-112) was performed when the capacity of FV-100 was approached, and at the end of the run.

3.1 Freeze Valve Checkout

The FV-104 freeze valve was modified preparatory to handling the ARE salt (Fig. 3.1). The first batch of salt was melted with a portable furnace arrangement and then pressure transferred into the hold tank (maintained at approximately 650°C), from which it drained through FV-104 (cross-over pipe maintained at 700°C) and into the fluorinator. Salt which remained in the cross-over pipe and in the line to FV-114 was then frozen to form a seal.

A second batch of salt was transferred from the portable furnace into FV-114, where it was held by the seal in FV-104. In order to test the feasibility of melting the seal in the cross-over pipe, the transfer line was heated in such a manner that the cross-over pipe was brought to temperature after the remainder of the line. When the seal melted, the transfer from FV-114 to FV-100 was quite rapid, apparently in less than 30 sec. The transfer line was allowed to freeze to re-establish the seal in FV-104.

Fig. 3.1. ARE salt handling equipment in the Volatility Pilot Plant

The loop of FV-104 was then heated while the salt in the cross-over pipe was left frozen. This was accomplished by changing the hot electrode for the autoresistance heating from the cross-over to the loop. While the entire transfer line, except for the cross-over, was at a temperature (approximately 650°C) greater than the salt melting point, a third batch of salt was pressurized into FV-114. Since no salt reached FV-100, the seal in the cross-over pipe was shown to be adequate.

3.2 Salt Transfer from Dump Tank Furnace

Twelve cans of salt (337 kg) were loaded into FV-111 and heated with the dump tank furnace (FV-510). The salt began to melt when its temperature reached approximately 530°C and drained as it melted. The total charge transferred to FV-114 within 1.2 hr. The temperature of the salt was increased to 625°C (after 2.1 hr) and held for 8.5 hr to assure complete drainage. Later, FV-111 was opened and inspected. All surfaces were clean of salt, indicating complete melting and drainage.

3.3 Feed Salt Transfer

Three attempts were made to pressurize salt out of FV-114 before a transfer could be initiated. The pressure in FV-114 was allowed to build up (from nitrogen purges) to 11 psig in one case and to 13 psig in another case with no transfer resulting. It was suspected, although not proved, that a cold spot existed somewhere in the line. On the third attempt the transfer occurred without incident. Six additional transfers were made with the remaining salt in FV-114 (Fig. 4.1).

3.4 Waste Salt Transfer

Three transfers of salt from FV-100 to FV-112 were performed (Fig. 4.1). Two unsuccessful attempts were made (FV-100 pressure = 7 psig) before the first transfer was initiated. The other two transfers proceeded with no trouble. However, some splatter of salt was observed around FV-112 after the last transfer.

3.5 CRP Trap Performance

The CRP trap was redesigned and relocated to facilitate remote discharging of the NaF bed directly into the molten salt in the fluorinator. The trap was moved to the top of the fluorinator, thus causing the direction of flow to be from bottom to top. A dumping mechanism and a syntron electric vibrator were added to FV-103. By

remote discharging of the NaF charge the radioactive materials caught in the trap could be dissolved and removed with the waste salt, thereby decreasing personnel exposure.

The first attempt to discharge the NaF bed (4 kg of 1/8" pellets) was unsuccessful. About 99% of the pellets remained in the CRP trap. Failure apparently resulted from considerable caking of the pellets, which was discovered later when the trap was emptied by vacuuming out the pellets. A second attempt was also unsuccessful, this time with 8 kg of NaF pellets. Misalignment of the retaining cone caused the annular space between the pellet retaining cone and trap wall to be excessive, thus allowing the pellets to pass into the fluorinator as they were loaded.

4.0 DATA AND RESULTS

Table 4.1. Salt Composition

Component	Weight %	Formula	Weight %
Na	9.80	NaF	17.9
Zr	44.0	ZrF ₄	80.7
F	41.64		
Other: Cr, Fe, Ni, Ti, C	0.15		

Table 4.2. Equipment Temperatures

A. Freeze Valve (FV-104) Checkout

	1st Batch from Portable Furnace	2nd Batch from Portable Furnace	2nd Batch from FV-114 to FV-100	3rd Batch from Portable Furnace
Hold tank	650°C	600-650°C	610°C	610-650°C
Transfer line (1 ft below FV-114)	650	565	600-575	600
Transfer line (5 ft from FV-114)	650-690	25	580	625
Transfer line (at junction of upstream end of cross-over pipe)	525-650	35	500-575	580
Freeze valve (center of cross-over pipe)	~680	--	--	--
Freeze valve (loop)	30	30	30	600
Transfer line (at junction of downstream end of cross-over pipe)	650	40	500-605	560
Fluorinator	640	640	640	645

Table 4.2. Equipment Temperatures (Continued)

B. Feed Salt Transfer

	Transfer No.						
	1	2	3	4	5	6	7
Temperature ($^{\circ}\text{C}$) of							
FV-100, Fluorinator	648	645	640	640	645	635	640
FV-104, Freeze Valve	785	765	695	745	770	800	760
FV-114, Hold Tank	610	605	610	608	590	600	615
MS-104, Salt Line	615	620	575	590	625	635	625
MS-114, Salt Line	635	635	635	640	635	625	645

C. Waste Salt Transfer

	A	B	C
Temperature ($^{\circ}\text{C}$) of			
FV-100, Fluorinator	600	600	605
FV-106, Freeze Valve	585	600	590
MS-100, Salt Line	695	685	690
MS-106, Salt Line	700	710	710

Fig. 4.1. Transfers of molten salt in the Volatility Pilot Plant Run B-1

Fig. 4.2. Pressure required to transfer molten salt out of the hold tank

6.0 APPENDIX

6.1 Schedule of Operations

Table 6.1. Operating Schedule

Salt Transfers	Date	Time	Duration, min
FV-104	11-2-57	0145	< 5
		1610	5
FV-111 - FV-114	11-25-57	1225 - 1337	72
FV-114 - FV-100	11-29-57	0330	< 5
		0400	< 5
		0835	5
		1617 - 1645	28
	12-2-57	0345	5
		0845 - 0932	47
		1430 - 1455	25
FV-100 - FV-112	11-29-57	2230 - 2244	14
	12-2-57	1117 - 1217*	20
	12-5-57	0620	5

*Transfer was interrupted for 40 min.

6.2 Comparison between Level Indicators

The hold tank and fluorinator were each equipped with differential pressure-type level indicating instruments, LR-6 and LR-2, respectively. Salt transferred from FV-114 obviously should be equivalent to salt transferred into FV-100 minus that which remains in the transfer line. A comparison between LR-6 and LR-2 should indicate the reliability of these instruments (Table 6.2).

Table 4.3. Salt Balance

	Weight, kg
FV-104 Checkout	
To system (FV-104 open)	38.8
Drained to FV-100	34.6
Remained in transfer line	4.2
To system (FV-104 sealed)	29.4
Transfer Tests	
To system (FV-104 sealed)	337.2
Pressurized to FV-100	358.8
Heel in FV-114	7
Unaccounted for	< 1

5.0 DISCUSSION

The FV-104 freeze valve design was modified in order that the transfer of the last batch of salt from the hold tank (FV-114) could be accomplished by gravity flow. Thus, the final transfer could be made with little risk of "blowing" the seal. Three transfers of salt from the portable furnace to (or through) FV-114 were used to check the operability of FV-104. The first showed that molten salt could be drained from FV-114, into the cross-over pipe, and frozen there to form a seal. The second showed that the previous seal was tight (prevented any salt from reaching FV-100) and that the salt in the cross-over pipe could be melted and refrozen to form a seal. The third indicated that this seal was tight.

The CRP trap operation indicated the feasibility of remote dumping, although this could not be done reliably with the present design. More data pertaining to caking of the pellets when exposed to actual run conditions are needed before evaluation of the dumping mechanism can be made.

Table 6.2. Comparison of Level Indicators in FV-114 (LR-6) and FV-100 (LR-2)

Transfer	Weight Loss in FV-114 by LR-6, kg	Weight Gain in FV-100 by LR-2, kg	Deviation, %
1	12.8	9.6	29
2	2.8	9.8	101
3	44.0	47.7	8
4	63.9	61.1	4
5	68.2	67.6	1
6	99.4	93.1	7
7	67.2	70.3	4
Total	358.3	359.2	<1

The precision of the two instruments was very good over a wide range although large discrepancies occurred in small transfers. The level indicators agreed within 0.8% of the total salt weight (356 kg) obtained by kilogram scales.

GLOSSARY

FV-100	Fluorinator
FV-103	CRP trap
FV-104	Modified freeze valve in feed salt line
FV-111	Dump tank liner
FV-112	Waste receiver
FV-114	Hold tank for feed salt
FV-510	Dump tank furnace

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