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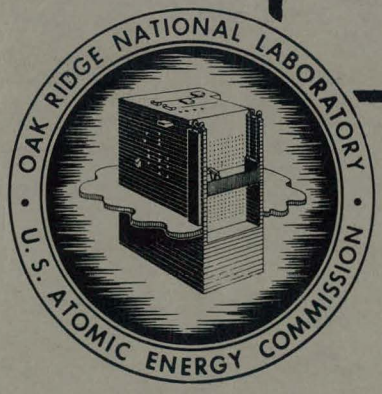
AIRCRAFT NUCLEAR PROPULSION

FLUORIDE FUEL PREPARATION FACILITY

JUNE 1, 1954



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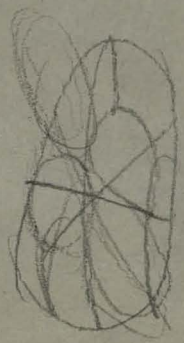

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AIRCRAFT NUCLEAR PROPULSION FLUORIDE FUEL PREPARATION FACILITY

June 1, 1954

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A DIVISION OF UNION CARBIDE AND CARBON CORPORATION
Oak Ridge, Tennessee

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FOREWORD

This report has been written for the information of new personnel associated with the ANP Fluoride Fuel Preparation Facility located in Building 9201-3 of the Y-12 Area. It is not intended herein to cover the complete design of the facility nor to cover the many changes which have been made to bring about the present mode of operation. It is hoped that the material presented here will serve as a training aid in future operations.

The basic research and development of the process was carried out by the members of the ANP Reactor Chemistry Section under W. R. Grimes of the Materials Chemistry Division. A group under the direction of G. J. Nessle of the ANP Reactor Chemistry Section with the assistance of members of the Experimental Engineering Section of the ANP Division was responsible for the design and installation of the facilities. For the past eight months the facility has been operated by Mr. Nessle's group to produce experimental lots of fused salts for the ANP Division. With increased demands for large quantities of fused salts, some members of the Pilot Plants Section of the Materials Chemistry Division are now assisting with the operations.

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SECRET

THE FLUORIDE FUEL PREPARATION FACILITY

Purpose

The Fluoride Fuel Preparation Facility in Building 9201-3 is concerned mainly with the production of 250-pound units of the principal fluoride compositions (Compositions 30, 31, 45, and 43)[1] which are used in various aircraft nuclear propulsion tests. These units are "batched down" into smaller containers and are dispensed to the various requesters in the ANP Division and to the Pratt & Whitney Aircraft Company.

Equipment

The Facility consists of an arrangement of three cubicles. Cubicle No. 1 contains equipment for the weighing and mixing of the loose fluoride salts. Cubicles 2 and 3 each contain a reactor where the mixture is treated, and a receiver to which the purified melt is transferred for storage. The flow diagram of the system is given in Figure 1. Controls for each are located on central panel boards (see Figure 2).

Procedure

General operating procedure consists first of weighing the proper amounts of NaF, ZrF_4 and UF_4, as required, in a tared hopper. The loose fluorides are transferred to a Patterson Kelly blender where they are allowed to mix for one hour until a uniform mixture is obtained. This fluoride salt mixture is transferred to a portable dolly and loaded into the reactor vessel. The equipment is pressure tested with helium to insure a gas tight system.

Heat is applied to the reactor, receiver, and transfer lines; and the system is evacuated to remove the excess air and oxygen. A helium flush is used to remove the last traces of air. After the initial flush the system is pressurized with HF to obtain an HF atmosphere and is then placed under five inches of vacuum and the salt mixture allowed to melt. The reactor is heated to 1500°F while the receiver and transfer lines are heated to 1300°F. After the mixture has melted it

---

1. Composition

	30	31	43	45
NaF	50 M%	50 M%	66.66 M%	53 M%
ZrF_4	46 M%	50 M%	0	47 M%
UF_4	4 M%	0	33.33 M%	0

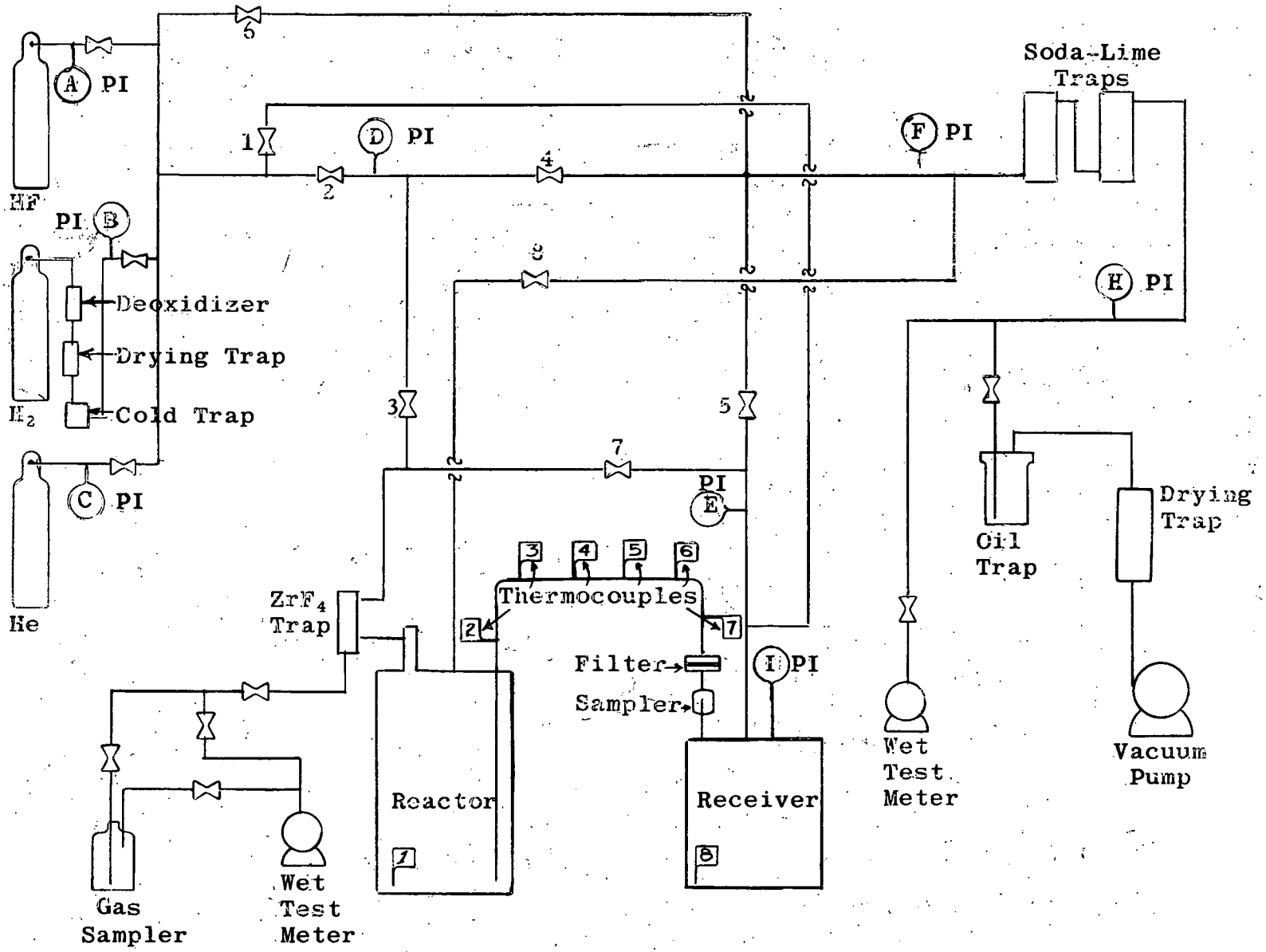

FIGURE 1: FLOW DIAGRAM OF THE FLUORIDE PRODUCTION FACILITIES IN 9201-3

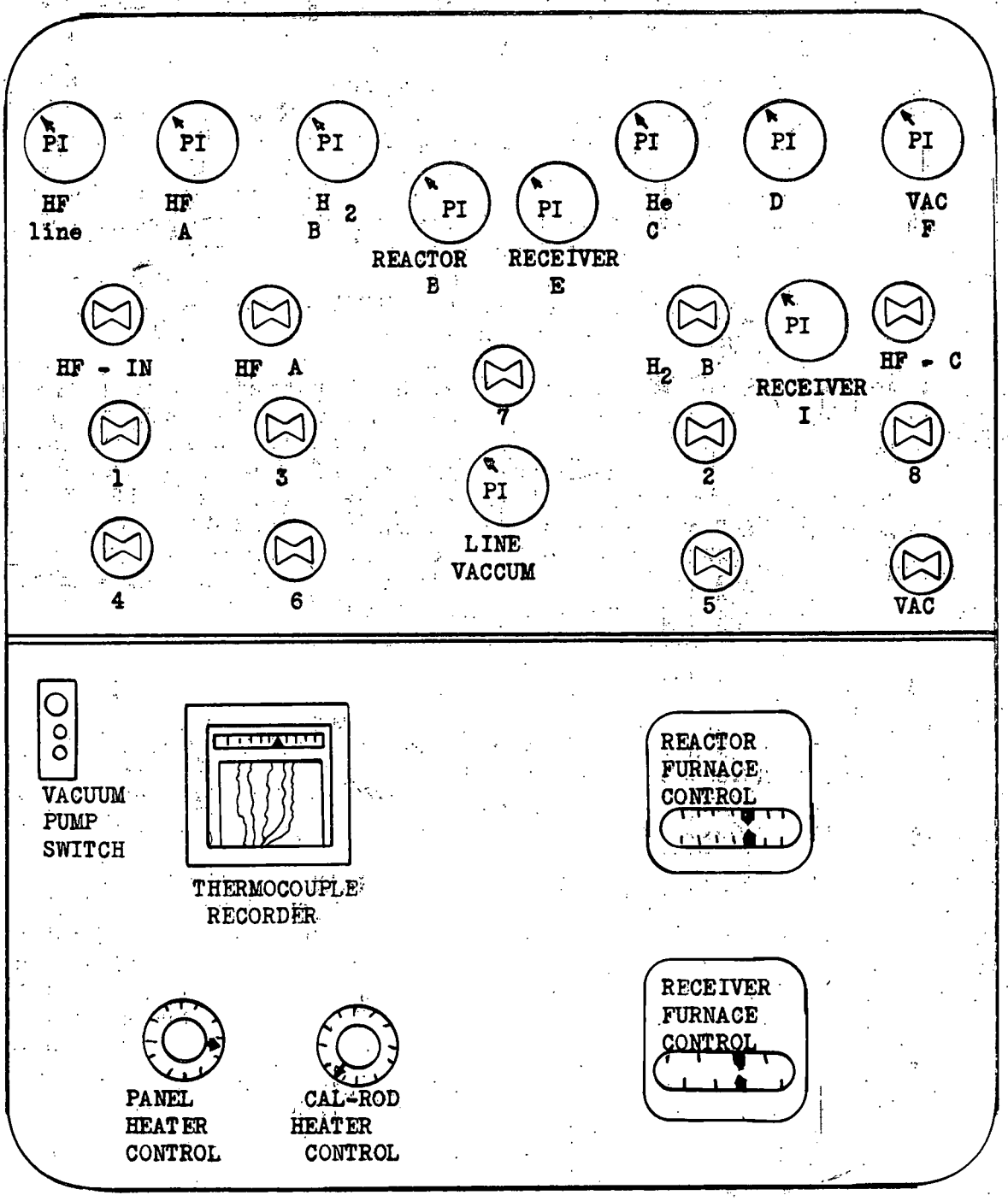

FIGURE 2: CONTROL PANEL LAYOUT

is treated with hydrogen for one hour and then HF for one and one-half hours. This treatment reduces and fluorinates the impurities in the melt.

The system is then flushed with hydrogen at a rate of 3 to 5 liters per minute until the hydrogen fluoride content of the effluent gas is 1×10^{-4} mols hydrogen fluoride per liter. During the final H_2 flush the impurities, Ni and Fe, are reduced to the metal and are plated out on the walls of the reactor. After completion of the hydrogen flush the melt is transferred to the receiver through a sintered nickel filter. The receiver is allowed to cool under a helium atmosphere. After the receiver has cooled it is removed to the storage area and stored under a helium blanket.

The sample is removed from the sampler and a portion is used for chemical analysis, and the remainder is held for reference. If the chemical analysis indicates that the batch will be acceptable the receiver is then assembled as shown in Figure 3. The salt mixture is melted and transferred into the smaller shipping containers. Samples are taken from each container. The "batching down" operation is necessary, as the users of the fluoride salt mixtures do not have the facilities to handle the 250-pound containers.

Discussion

In determining the length of time required to complete a single batch, a summary of runs 1 through 14 is given in Table 1. This gives a fairly good indication of the type of operation which can be expected in the future. The only important trend noted thus far was the increase and leveling off of the time and quantity of H_2 flush required after the hydrogen fluoride treatment. (See Figure 4.) This variation can best be explained by assuming a gradual build-up of metallic impurities in the reactor. The impurities increase to a point where they are not completely fluorinated by the hydrogen fluoride treatment. After this point has been reached a further increase in impurities in the reactor does not appreciably increase the H_2 required. The time required for the H_2 flush seems to have leveled off at about 35 hours.

Difficulties that are encountered in the various runs are generally due to plugs in the system. These plugs may occur in the dip leg, transfer line, loading tube, filter, or ZrF_4 trap. Whenever a plug occurs it is necessary to flush the system with helium before any work may be done on the equipment. The helium flush means an increase of 1 to 3 hours on the time required to complete a batch. The above troubles may be corrected without necessarily shutting down the unit.

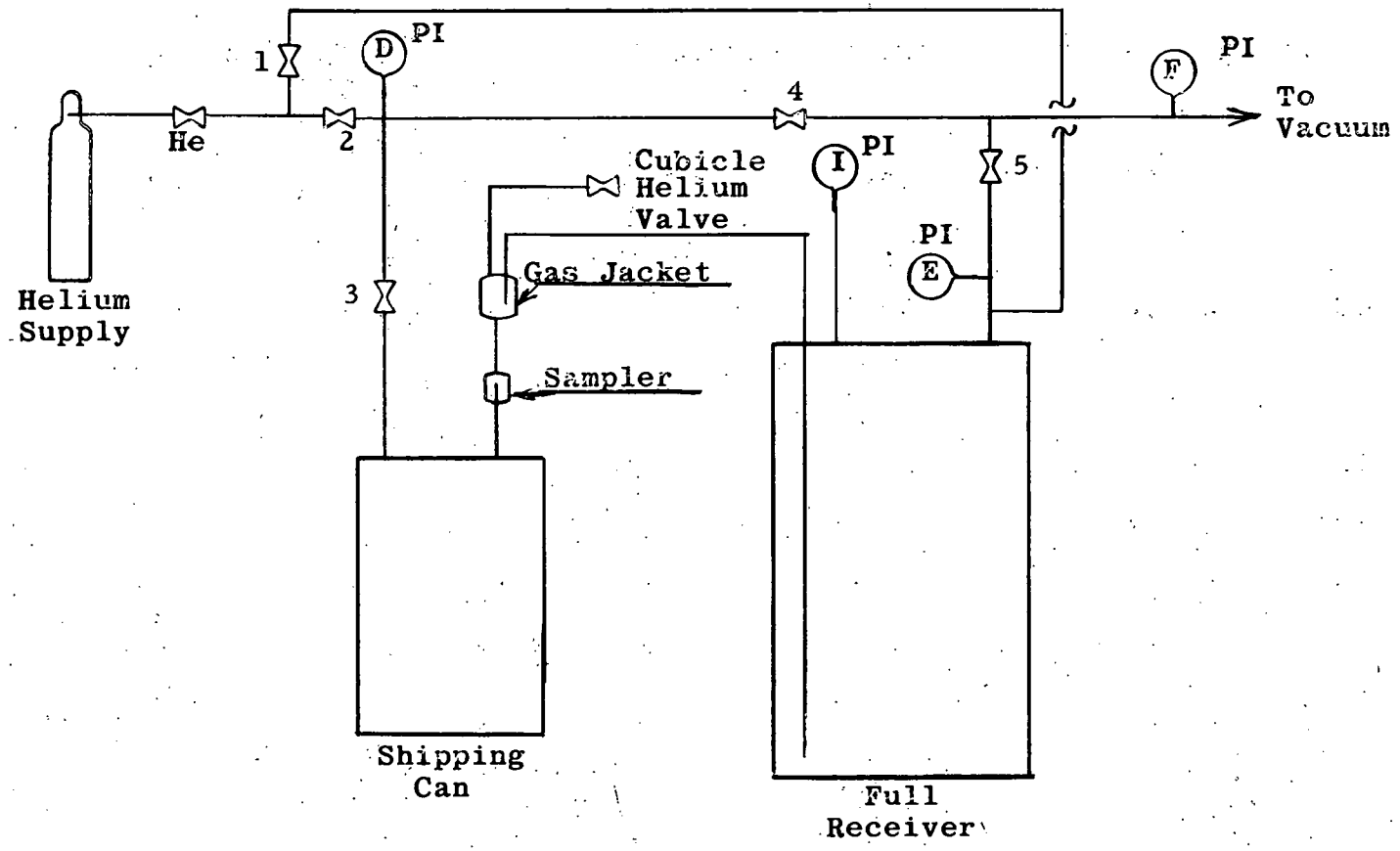

FIGURE 3: FLOW DIAGRAM OF BATCHING DOWN OPERATIONS

Table 1

Time Required for Processing

Operation	Cubicle No. 2 Time Range (hours)	Cubicle No. 2 Average Time (hours)	Cubicle No. 3 Time Range (hours)	Cubicle No. 3 Average Time (hours)
Melting	1.60 - 3.07	2.08	1.67 - 3.00	2.33
H_2 Treatment	1.00 - 1.08	1.02	1.00 - 1.67	1.13
HF Treatment	0.67 - 2.50	1.85	1.50 - 2.00	1.75
H_2 Flush	12.36 - 35.98	29.0	16.92 - 38.37	30.59
Transfer	0.20 - 0.60	0.32	0.25 - 0.33	0.28
Cooling	4.00	4.00	4.00	4.00
Total Time Per Batch	22.50 - 45.00	39.69	26.00 - 73.00	43.93
H_2 Flush (Volume in liters)	2560 - 7975	6530	3915 - 8264	6770

Note: The above table is based on results obtained from runs 1 through 13.

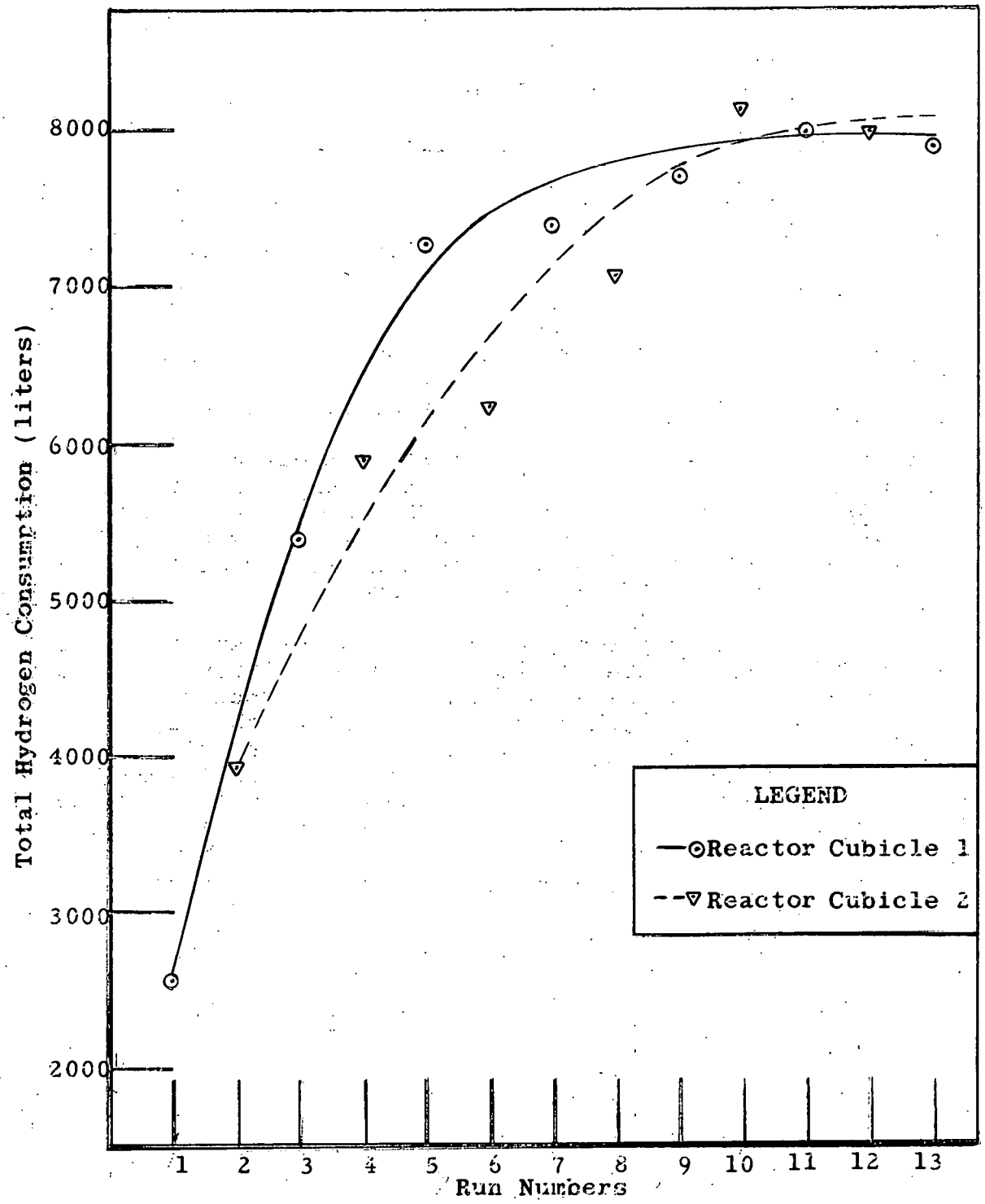

FIGURE 4: EFFECT OF REPEATED USE OF REACTOR VESSEL ON TOTAL HYDROGEN CONSUMPTION

Troubles that necessitate the complete shutdown of the unit are:

1. Breaking of the dip line.
2. Breaking of the transfer line.
3. Plugging of filter unit.
4. Leaks in transfer line, filter, or sampler.
5. Leaks in flange connections.
6. Breaking of gas line and receiver gauge line.
7. Leaks in welds of riser, especially on receiver.

When any of the troubles occur it is necessary to cool the reactor and receiver to a temperature where it will be possible to work on the equipment. The reactor requires 8 to 10 hours to cool to a temperature safe for working conditions and 1 to 2 hours for heating and melting of the material after the repair job is completed.

An increase in difficulties in the latest series of runs includes the plugging and breaking of the dip lines which is due to an increase in the amount of metallic impurities in the zirconium fluoride salts that plate out on the lines. The plugging of the ZrF_4 trap, transfer line, and filters is caused by the increase in the amount of carbon in the zirconium fluoride salts. High costs and low production of the pure hafnium free zirconium salts have made it necessary to use the commercial grade salts in all cases except for those in which the fluoride salts will be used with enriched uranium.

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I. DESCRIPTION OF EQUIPMENT

The equipment used in the preparation of the fluoride salt mixtures is divided into two categories: Group I consists of the equipment used in the weighing and blending of the salts, and is located in Cubicle I. Group II includes the reactors, receivers, furnaces, and related equipment, and are in Cubicles II and III.

Cubicle I

Scales

Duty: Used in the weighing of the NaF_4 and ZrF_4 salts.

No. required: one

Selection:

Manufacturer: Toledo Scale Company

Model: 82 Serial No. 8778

Accuracy: ± 100 grams

Type: PRINTWEIGHT

Capacity: 500 Kg

Weighing hopper built on the scale

Scales

Duty: Used in weighing the UF_4 salts

Number required: one

Selection:

Mfg: Toledo Scale Company

Model: 407S C Serial No. 915

Type: Gravitygram

Capacity: 14 kilograms

Accuracy: ± 1.0 gram

Blender

Duty: A device for mixing dry salts. Should be capable of producing a uniform mixture in a short mixing time.

Number required: one

Selection:

Mfg: Patterson Kelly Co., Inc.

Model: Twin shell dry blender

Serial No.: 99923

Capacity: 5 cu ft - 100 lbs/cu ft

Material of construction: 316 S.S.

Motor:

Mfg: Electra Motor Co.

Horsepower: 1 hp, 60 cycle, 3 phase

RPM: 1800/20

Transportation Hopper

Duty: A hopper capable of handling the dry mixed salts from the blender; also used in loading the reactor.

Number required: two

Selection:

Capacity: 300 lbs

Material of construction: 316 S.S.

Hoists

Duty: To transport the weighing hopper between the scale and blender, and between the blender and reactors. The hoist is to be used for moving and replacing the reactor and receiver.

Number required: two

Selection:

Mfg: Manning, Maxwell and Moor, Inc.

Model: Load Lifter Junior Hoist

Capacity: 1000 lbs

Volts, 440, cycle 60, phase 3

Cubicles II and III

Reactor

Duty: Vessel for holding the molten fluoride salts while they are being treated with HF and H_2. Vessel must be capable of withstanding a pressure of 30 psi, a complete vacuum at a temperature of 1500°F, and capable of withstanding the corrosive action of HF and H_2 gas.

Number required: two

Selection:

Reactor 48 inches high by 13-1/2 inches in diameter, constructed of 1/4" nickel plate with top and base of 1/2" nickel plate. Connections on top of reactor include a 3-inch diameter pipe used for loading the fluoride salts. A 1/2" Swagelok mounted on the pipe is used for connection of the ZrF_4 trap.

Thermocouple connection is made through a 1/4-inch nickel tubing. A 1/2-inch nickel tubing is used for the gauge line, and a 3/8-inch nickel tubing line is used for the dip leg. The dip leg is protected inside the reactor by being encased within a one-inch nickel pipe. (See Figure 5.)

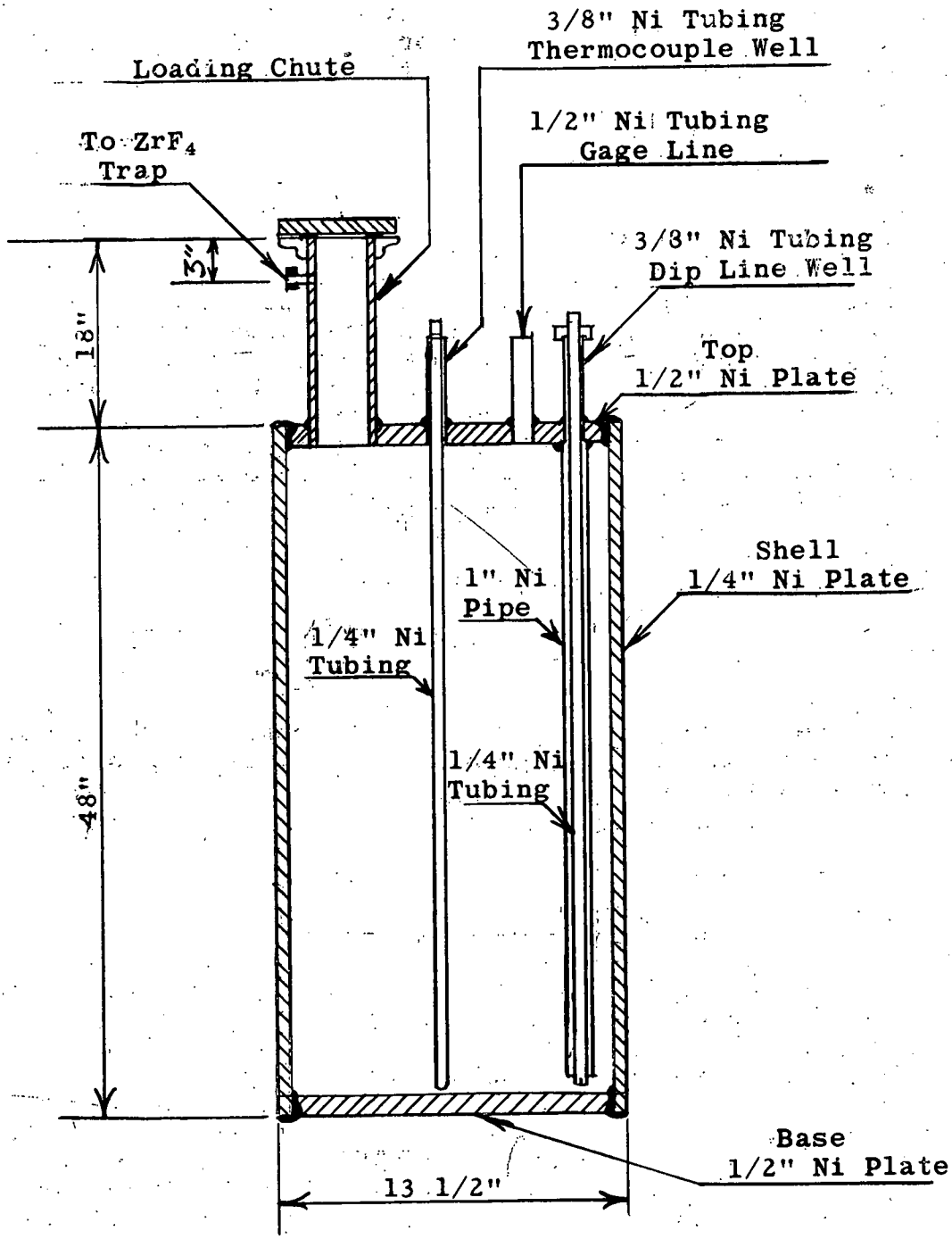

FIGURE 5: REACTOR VESSEL DETAIL

Zirconium Fluoride Trap

Duty: A means of trapping the zirconium fluoride dust and carbon produced in the reactor and carried out by the purge gases. The trap prevents the dust from plugging the lines.

Number required: two in use

Selection:

Trap 11 inches high constructed of 4-inch O.D. nickel tubing. The trap is packed with copper shot. A perforated nickel plate is placed two inches above the base for supporting the copper shot. Two 1/2-inch Swagelok connections are located on the trap; one is used for connecting to the reactor and the other is an outlet for the purge gases. A 1/4-inch copper line mounted in the base of the trap is used for draining liquids from the trap and obtaining samples of the purge gas. (See Figure 6.)

Receivers

Duty: To receive and hold the molten fluoride salts. Vessel should be capable of withstanding 30 psig pressure, a complete vacuum at a temperature of 1500°F, and withstand the corrosive action of HF and H_2 gas.

Number required: One per batch

Selection:

Receiver 23 inches high and 13 inches in diameter side constructed of 1/4-inch nickel plate with top and base of 1/2-inch nickel plate. Connections on top of receiver include a 3/8-inch nickel tubing for the dip leg, which is used in transferring the mixture from the receiver; a 3/8-inch nickel tubing as the transfer line connection; a 1/2-inch nickel pipe connection for the thermocouple; and a 3/8-inch nickel tubing line for the connection of a gauge. A 1/2-inch nickel line is used for the purge gas. A special welding code is used on all welds so that they will be able to withstand the conditions present in the receiver. (See Figure 7.)

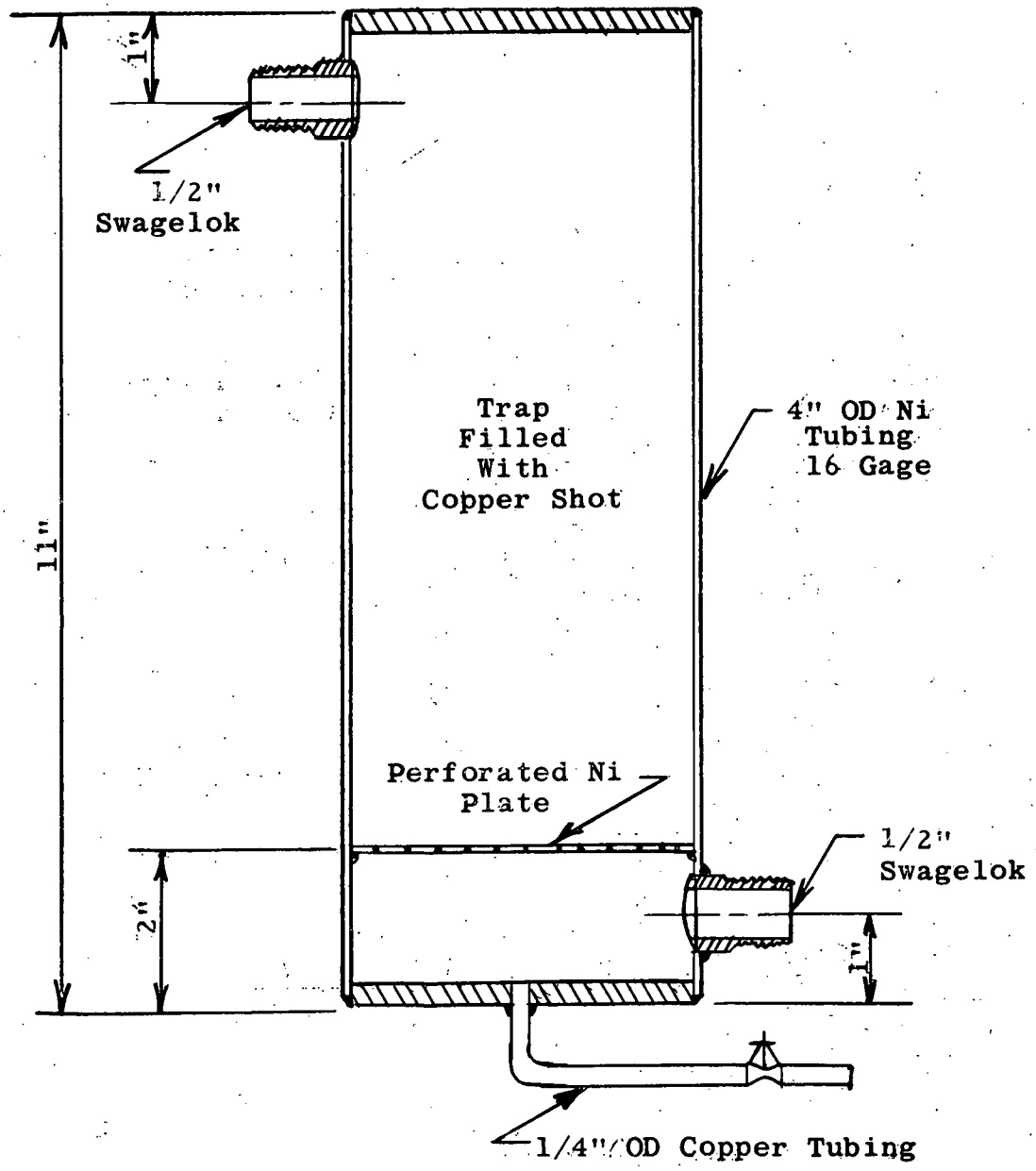

FIGURE 6: ZIRCONIUM FLUORIDE TRAP

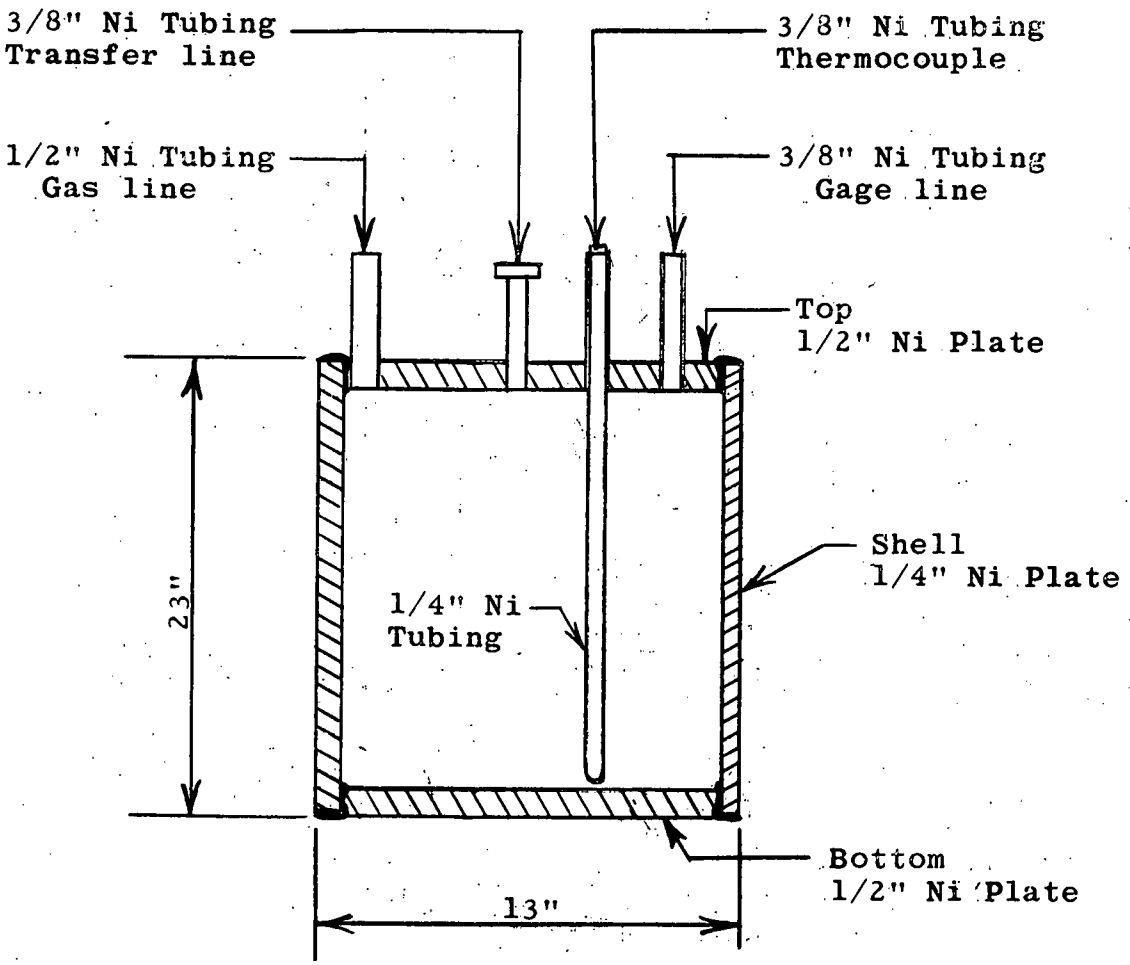

FIGURE 7: RECEIVER VESSEL DETAILS

Receiver Furnace

Duty: A furnace capable of heating the receiver and fluoride salts to 1300°F and must be capable of continuous service.

Number required: Two

Selection:

Mfg: Hevi-Duty

Model: HD 1236

Type: Hevi-duty

Voltage: 230 volts single phase

Capacity: 23 kilowats

Size: 23-inch O.D., 60 inches tall

The furnace is installed so that it may be lowered from the receiver by the use of pulleys and counterweights.

Reactor Furnace

Duty: A furnace capable of heating the receiver and fluoride salts to 1500°F and capable of continuous service.

Number required: Two

Selection:

Mfg: Trent, Inc.

Voltage: 440 volts, 3 phase

Capacity: 50 kilowatts

Size: 40-inch O.D., 78 inches tall

Filter

Duty: A unit through which the molten salts can be filtered to remove any salt crystals or metal chips that might have been present in the reactor.

Number required: One per receiver

Selection:

A 2-inch diameter filter unit; included in the unit is a 1/8-inch thick sintered Ni disc 1-5/6-inch diameter. The nickel disc is supported with a 1/8-inch thick perforated nickel plate. Filter porosity is 0.0015 inch. (See Figure 8 for detail drawing.)

Sampler

Duty: A unit to collect a representative sample of the molten fluoride salts.

Number required: One per batch

Selection:

Sampler is constructed of a 2-inch length of 2-inch nickel pipe. A 3/8-inch nickel tubing extends approximately 1 inch into the sampler and acts as an overflow weir. After the sampler is removed it is cut open with pipe cutters and the sample is removed. (See Figure 9 for details.) The sampler capacity is 200 grams.

Transfer Line

Duty: A line through which the molten salt is transferred from the reactor to the receiver. Line to be heated to 1300°F.

Number required: One per unit

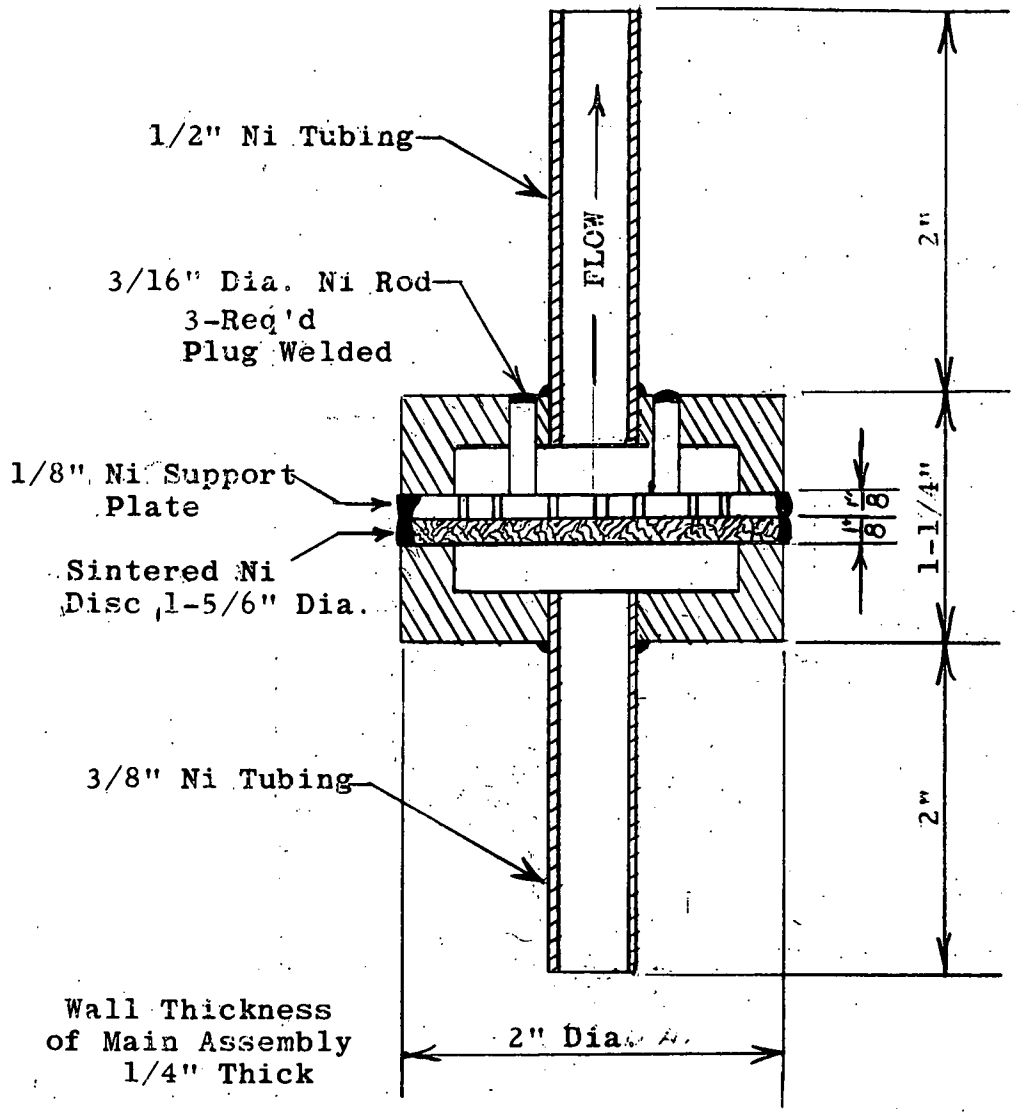

FIGURE 8: NICKEL FILTER DETAIL

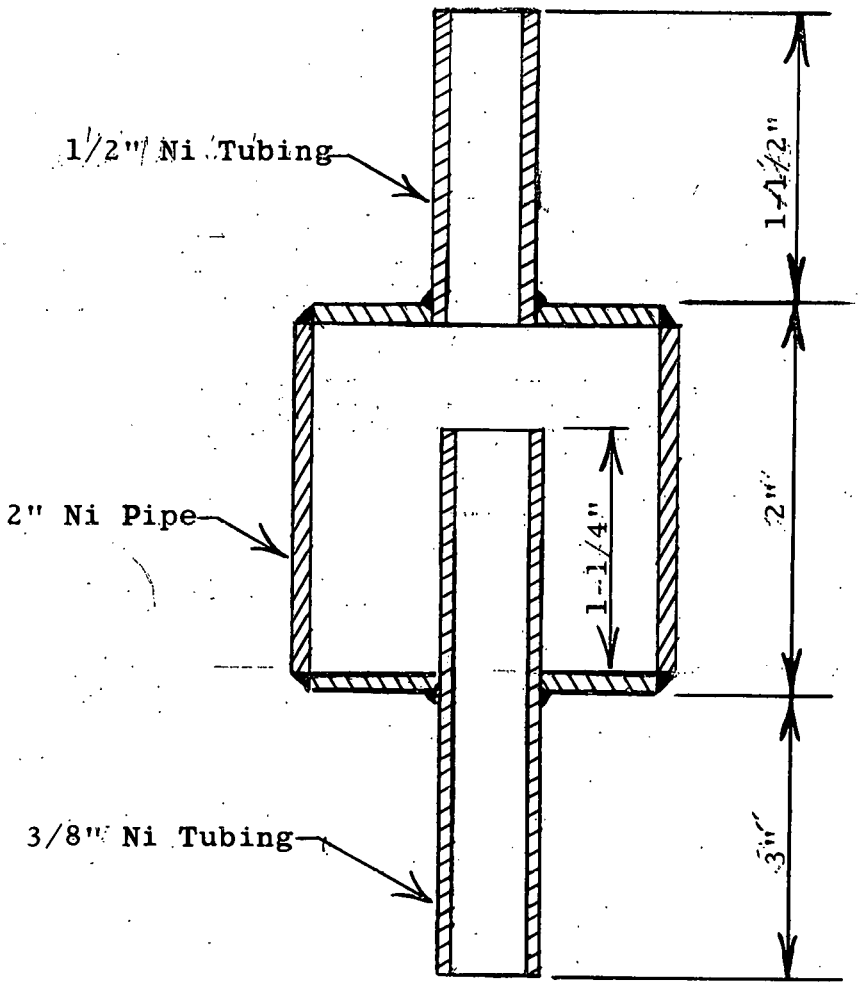

FIGURE 9: FLUORIDE SALT SAMPLER DETAIL

SECRET

Selection:

3/8-inch nickel tubing that is connected to the reactor. A filter and sampler is connected between the transfer line and receiver. The line is heated by two parallel calrod heaters. Six thermocouple connections are maintained on the line to insure that the line has reached the proper temperature before a transfer is attempted.

Panel Board

Duty: To provide a central location for all the controls of the reactor and receiver.

Number required: Two, one per unit. (See Figure 2.)

Selection:

The Panel Board is constructed of Plexiglas so that all connections and valves may be seen and hydrogen fluoride leaks detected more easily. All connections with the board are made with 1/2-inch copper tubing. All valves are 1/2-inch Fulton Sylphon brass bellows valves. To prevent the condensation of hydrogen fluoride gas in the lines of the control panel it is heated to 130°F by the use of strip heaters.

Pressure gauges: Ten required per panel board

Duragauge - bronze tube, nylon

Capacity:

Pressure: 30 psig

Vacuum: 30 inches

Control Instruments:

Furnace controls - two required per panel board

Mfg: Leeds-Northrup Company

Model: Micromax controller

Range: 0-2000°F

Thermocouple Recorder: One required per panel board

Mfg: Minneapolis-Honeywell Reg. Co.

Model: Brown Potentiometer - 12-point

Range: 0-2000°F

Voltage: 110 volts, 60 cycle, 0.75 amps

Vacuum Pump

Duty: A pump of sufficient capacity to evacuate the reactor and receiver to 20 inches of vacuum.

Number required: Two, one per unit

Selection:

Mfg: Beach-Russ Co.

Type: High capacity rotary vacuum pump

Model: 4W

Horsepower: 3 hp; volts: 220/440; cycles: 60

Soda-Lime Traps

Duty: A protection unit to prevent hydrogen fluoride gas from getting into the vacuum pumps or into the atmosphere.

Reaction:

$$NaOH + HF \longrightarrow NaF + H_2O$$

$$Ca(OH)_2 + HF \longrightarrow CaF_2 + 2H_2O$$

Number required: Two per unit

Selection:

A rounded-bottom vessel 48 inches high and 8 inches I.D., with flanged top, constructed of 316 S.S. plate. The hydrogen fluoride gas enters through the bottom of the vessel and exits through connection on top flange. The vessel is packed with five cartridges which contain soda lime. The openings in the cartridges are staggered to prevent channeling; between each cartridge is a perforated nickel plate and a copper screen. A thin layer of soda-lime is packed on each plate and the edges are sealed with soda lime; this prevents channeling of the hydrogen fluoride gas. The flanged top is sealed with a Teflon gasket.

Activated Alumina Drying Tube

Duty: To prevent the moisture formed in the soda lime traps from getting into the vacuum pump.

Number required: One per unit

Selection:

4-inch diameter glass pipe 48 inches long. Filled with activated alumina. The inlet side of the tube is packed with copper wool to distribute the gas. To prevent channeling, two sections of copper wool are placed in the tube. The exit end of the tube is packed with glass wool.

II. SAFETY PROCEDURE

The materials in use at the production facilities are extremely poisonous and highly corrosive. Care must be exercised at all times and especially whenever it is necessary to work with or around the chemicals and equipment.

General safety instructions to be used at the Production Facilities are:

1. Dust masks must be worn whenever it is necessary to weigh, blend, or transport any dry chemicals (i.e., NaF, ZrF_4, or UF_4).

2. Rubber gloves are to be worn when working with or around any line or equipment that may or has contained hydrogen fluoride.

3. Face shields and masks are to be used when it is necessary to break into the reactor or disconnecting any lines that may contain hydrogen fluoride, hydrogen gas or zirconium dust.

4. No smoking is to be allowed around the hydrogen cylinders or in the cubicles.

5. All standard procedures and precautions must be observed while working around or using hydrogen fluoride gas.

6. When lowering the receiver furnace the heat shield is to be used to prevent heat burns.

7. Tools and equipment are not to be placed on the hot furnaces.

III. OPERATING PROCEDURE

The standard procedure for the operation of equipment in the Fluoride Production Facilities in 9201-3 is given below:

A. Preliminary Operation

1. The receiver can must be weighed as it will be shipped; that is, with all blank connections on it. Record this weight as "Tare weight" in the record book. Keep all blank connections with the proper receiver cans.

2. After receiver has been connected to the apparatus check to see that all ZrF_4 traps and filters have been cleaned and the soda-lime traps checked since the last run.

3. Make sure all valves and lines in the control panel are clear, especially the emergency valve.

4. Check the hydrogen fluoride cylinder and line pressures; apply heat if necessary to raise the pressure to 10 pounds.

5. Set the helium and hydrogen line pressures at 10 pounds each.

B. Mixing Fluoride Salts

1. Carefully weigh all salts into the tared dolly. All weights must be recorded on cards and in record book.

2. Transfer salts to P-K blender. Care must be used to prevent dusting and loss of salts.

3. Allow to mix one (1) hour in blender.

4. Transfer salts from blender to portable dolly.

5. Move dolly into position above reactor.

C. Loading the Reactor Vessel

1. Make sure there is no pressure in the reactor.

2. Remove reactor loading tube cap and load the mixed fluoride salts into the reactor vessel.

3. After all of the mixed fluoride salts have been loaded into the reactor vessel, check the gas exit line from the loading tube to make sure it is not filled with fluoride salts. Blow helium through valves 2 and 3 to help clear this line.

4. Replace the loading tube cap and fasten securely.

5. Pressure test the system once more to make sure the apparatus is gas tight.

D. Helium Flushing

To remove the excess air or oxygen from the system after loading the fuel into the reactor, and to seal the system:

1. All valves closed. Turn calrods on to desired setting.

2. Open valve 3 and leave open. Open valve 7.

3. a. Open valve 4 and evacuate.

b. If transfer line is plugged, evacuate receiver through valve 5.

 399 028

4. a. Close valve 4, open valve C and (slowly) valve 1 to pressurize system with helium to 10 pounds.

   b. Under condition (b) above, close valves 4 and 5, open valve C and valves 1 and 2.

5. a. Close valve 1, open valve 4 slowly and evacuate.

   b. Close valves 1 and 2, and evacuate through valves 4 and 5.

6. Repeat steps 4 and 5 twice.

E. Melting the Mixture

Close valve C, open valves A and 7.

7. Close valve 4 and pressurize with hydrogen fluoride, by opening valve 1 slowly.

8. Close valve 1 and evacuate by opening valve 4 slowly.

9. Repeat steps 7 and 8 twice.

10. Close valve 4 and pressurize with hydrogen fluoride through valve 1 to zero pressure.

11. Close valve 1. Set reactor vessle micromax furnace control at 1500°F and receiver vessel micromax furnace control at 1300°F and allow furnaces to heat. Bleed off any excess hydrogen fluoride pressure developed through valve 4, keeping reactor vessel at 5 inches vacuum until the melt temperature (thermocouple No. 1) reads 1300°F.

Drain the ZrF_4 trap!

F. H_2 Treatment

Close valves A and 7, open valve B, set furnace control for reactor at 1475°F.

12. Close valve 1, open valve 4 and evacuate to zero reading (atmospheric pressure) on gauge G.

13. Close valve 4, open valve 1 slowly and pressurize system to 10 pounds on gauge G.

SECRET

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14. Repeat steps 12 and 13 for 1 hour allowing hydrogen pressure to remain in reactor for about 30 seconds each time before evacuating. Drain ZrF_4 trap at end of hydrogen treatment.

G. Hydrogen Fluoride Treatment

Close valve B, open valve A.

15. Flush with hydrogen fluoride as in steps 12 and 13 for 1-1/2 hours. At the completion of the hydrogen fluoride treatment, in order to clear the system of excess hydrogen fluoride, use the following procedure:

a. Shut off hydrogen fluoride cylinder.

b. With the main hydrogen fluoride valves on control panel open, evacuate system to at least 20 inches through valves 4, 3, and 1.

c. When gauge A reads 20 inches of vacuum, close valve A and main hydrogen fluoride valve.

d. Pressurize with helium by closing valve 4 and bleeding helium in slowly through valve 1 to zero pressure on gauge D.

e. Close valve 1 and evacuate to 20 inches again through valve 4.

f. Pressurize with helium again as in (d).

g. Proceed with hydrogen flushing.

H. Hydrogen Flushing

Close valve A, open valve B.

16. Close main vacuum valve between vacuum tank and soda lime traps.

17. Adjust the hydrogen flow through valves 1, 3, and 4, to about 3 to 5 liters per minute. The flow rate is never to exceed 5 liters per minute and should be kept around 4 liters per minute. Hydrogen flushing will continue for a minimum of 12 hours with frequent checks on flow rates and gas samples taken at least every hour, until the hydrogen fluoride content of the effluent gas reaches 1.0×10^{-4} mols HF per liter.

18. Sampling Procedure

Fill the sample bottle with 100 ml of 0.01 M H_3BO_3. Place the bubbler in the sample bottle. Set the gas flow through the wet test meter to 0.3 - 0.8 liters per minute. Allow exactly two liters of gas to bubble through the sampler. Record the total readings of the wet test meters. Remove the sample bottle and test the conductivity by the use of the Solu Bridge conductance meter. The value obtained on the meter is converted into mols of hydrogen fluoride per liter of sample by the use of a graph. Mols of hydrogen fluoride per liter is calculated from the value obtained from the graph. These values will be recorded in the log book along with information regarding the time sample was obtained, size of sample, temperature of reactor, gas flow rate through sampler, and the total amount of gas through the reactor.

I. Fuel Transfer

Calrod units - all points between 1100°F and 1300°F.

Filter - 1300°F

Receiver - 1300°F

Before preparing to transfer, the system should be pressure checked to determine if leaks have developed during operation. To do this, close valve 4, and pressurize to 10 pounds of helium through valve 1. Close valve 1 and allow the system to set for 5 minutes, noting any pressure drop in gauges D and E and the reactor and receiver gauges. If the pressure should drop more than one pound within the 5 minutes on any or all of these gauges, a leak is present which should be located and repaired before transferring the melt from the reactor to the receiver. If the leak cannot be repaired without shutting down the apparatus, do not attempt to transfer the melt unless the leak is known to be small, that the molten fluorides will not contact it, and no air will be drawn into the system through it.

For example:

A slight leak on the gauge line from the reactor still permits transfer of the melt as long as the reactor is never allowed to go under vacuum again until

that batch has been transferred, cooled, and removed from the system. On the other hand, a slight leak on the receiver gauge line must be repaired before transfer is possible since the receiver is under vacuum during the transfer and air will be drawn into the system.

19. Adjust system such that gauge G is reading zero or at a slight vacuum.

20. Close all valves except 3. Open main vacuum valve.

21. Have a helium pressure of 10 pounds on gauge C.

22. Open valve C.

23. Open valves 2 and 5 slowly and approximately at the same time until they are wide open.

24. Fuel is transferred when gas can flow freely through valves 2, 3, 5. Turn off all heating units. Lower receiver furnace from the receiver.

25. Allow gas to flow through continuously until disconnecting procedure is complete.

26. Shut off valves 4 and 5 and keep system under slight positive pressure until receiver and sampler have been disconnected.

J. Disconnecting the Receiver Can

After the inside temperature (thermocouple No. 8) of the loaded receiver can has dropped below 850°F, it is to be disconnected in the following manner (see Figure 10):

1. Keep a positive helium pressure on the system through valves 1 and 2 with valves 4 and 5 closed.

2. The first connection to be broken is at point "A" on Figure 10. Attach a copper line with a valve to this point and make connections to the helium supply by means of plastic tubing.

3. With helium coming in through the copper connection attached in step 2 above, the next disconnect point is "B", the gas line connection. Cap this point with the appropriate blank connection.

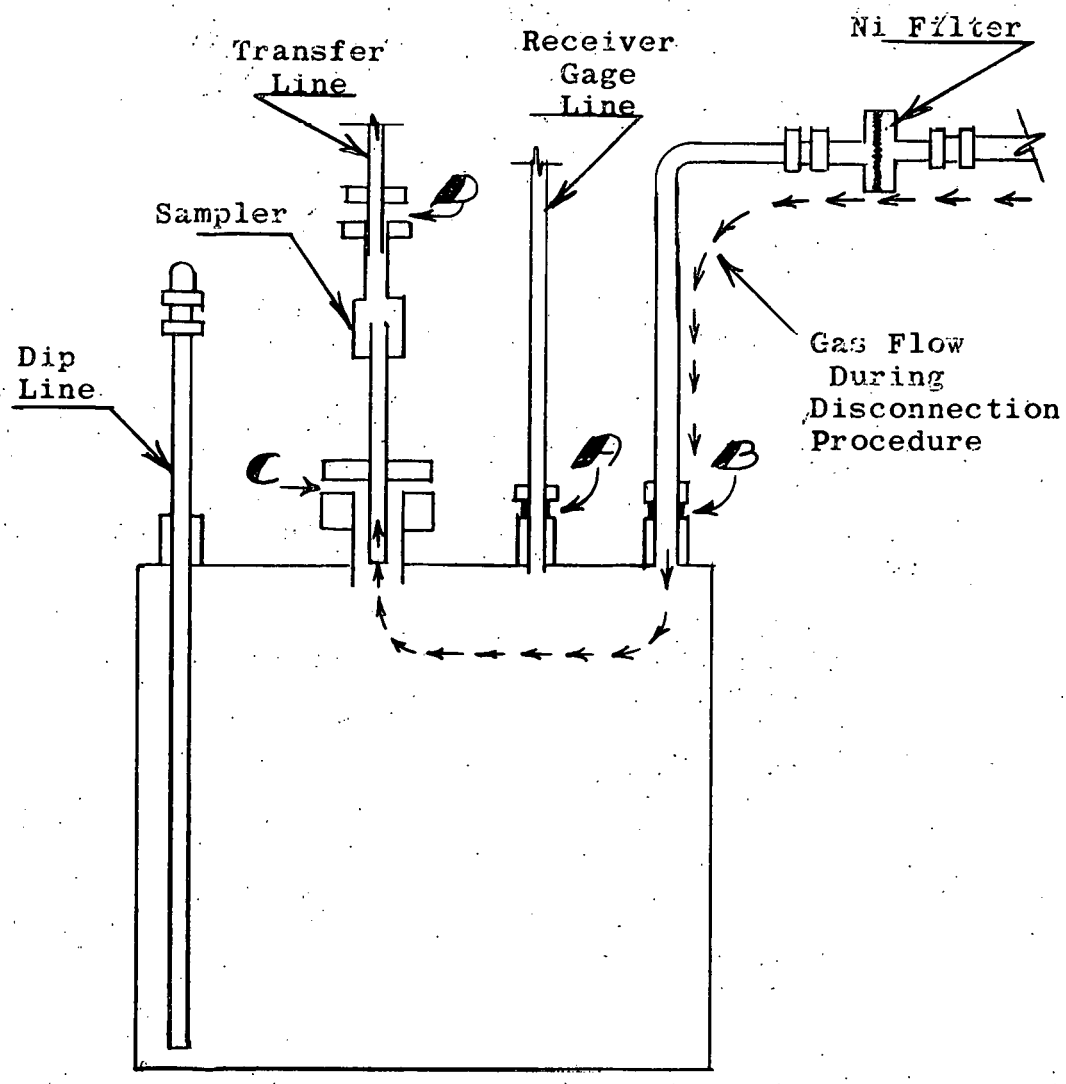

FIGURE 10: DISCONNECTING POINTS ON LOADED RECEIVER

4. Point "C" is the next disconnect point on the receiver. Cap with the appropriate blank flange. The receiver is now free of the apparatus.

5. Pressurize the receiver with about 10 pounds of helium through the copper valve and test for any leaks. Close the copper valve with 10 pounds pressure on the receiver and remove the receiver to storage helium header area. Valves 1 and 2 and A on the panel board are to be shut off.

6. The last disconnect point is "D" which removes the sampler. After the sampler has been removed, it should be cut open as soon as possible and the sample removed for chemical analysis.

20 grams	chemical analysis	glass vial
remainder	held for reference	glass bottle

7. After the sampler has been removed a new receiver and sampler should be attached as rapidly as possible for the next run.

K. Complete the check list. (See Table 2.)

IV. TROUBLE PROCEDURE

If;

A. The reactor vessel cannot be evacuated through valves 3 and 4:

1. Evacuate through emergency valve 8.

B. There is an hydrogen fluoride leak in the control panel manifold:

1. Close the hydrogen fluoride valve A and evacuate system to 5 inches vacuum.

2. Open helium valve C and fill system with helium.

3. Evacuate system again to 5 inches of vacuum.

4. Repeat steps 2 and 3 two more times.

Table 2. Check List

After each completed run, the following list will be checked for each cubicle:

Cubicle No._____

	Batch Number													
1. Remove & clean copper shot ZrF_4 trap														
2. Remove & clean sintered Ni. filter														
3. Check all lines for clear flow														
4. Complete all cards														
5. Check gas cylinders & replace empties														
6. Check that blanks are in proper place														
7. Are 4 completed samplers available														
8. Check dry ice supply														
9. Check sleeves on hoppers														
10. Check gasket on loading chute of reactor														
11. Check wet test meters														
12. Change H_2O in wet test meters														
13. Check all Tygon tubing for leaks & decomposition														
14. Check valves & oil level on vacuum pump														
15. Check H_2 trap for dry ice														
16. Close valves on ZrF_4 trap. All valves leading to W.T.M.														

5. Finally with a slight positive pressure of helium on the system, repair the leak.

C. There is an H_2 leak in the control panel manifold:

1. Follow the procedure given in "B" replacing the hydrogen with helium before making repairs.

D. There is an HF or an H_2 leak in the receiver can:

1. Follow the procedure given in "B" and repair leak or replace receiver can with a new receiver. If the furnaces are hot and the mixture melted, the following procedure is to be used for replacing the receiver or breaking the transfer line connection:

a. Lower the receiver furnace, after shutting it off, and place transite plate over furnace opening to prevent heat from interfering with the repair operations. Turn off calrod heating units on transfer line.

b. Use emergency valve 8 to bleed off any excess pressure in reactor vessel.

c. Using valve 2 (inlet gas line) and 8 (exit gas line) keep the reactor at atmospheric pressure at all times during the repair work.

d. After the leak has been repaired and all connections tight, normal operating procedure can be resumed.

E. There is a fuel leak in the receiver can:

1. Stop transfer immediately and reverse the gas flow. (Close valves 2 and 5 and open valves 1 and 4.)

2. Turn off receiver furnace and cautiously lower furnace away from receiver, after the receiver can and furnace have cooled to 1000°F or less.

3. In all cases of a fuel leak in the receiver always lower the furnace away from the receiver enough so that the receiver will not stick to the furnace liner when the melt solidifies.

4. Shut down the apparatus and when everything is cool make the necessary repairs to the furnace liner, the receiver can, and whatever else has come in contact with the molten salt.

SECRET

F. There is an HF or H_2 leak in the transfer line:

1. Follow the procedure given in "B". If the leak occurs early enough in the treatment, replace the transfer line and continue the run.

G. The calrod heating units on the transfer line burn out:

1. Replace the calrod units as soon as possible without interrupting the run, taking special care not to damage the transfer line.

H. Control panel valves plug or jam:

1. Flush system thoroughly with helium.

2. Apply positive helium pressure to system, making sure the pressure in the receiver is always higher than the pressure in the reactor.

3. Replace or repair the faulty valve.

I. The vacuum pump jams or stops:

1. Have vacuum pump repaired or replaced immediately.

2. Continue the operation as long as possible using the vacuum reservoir.

3. If the vacuum pump cannot be repaired in time to finish the run the apparatus must be shut down completely.

J. The ZrF_4 trap leaks:

1. Drain the trap and flush system thoroughly with helium as in the procedure under "B".

2. Remove the ZrF_4 trap and replace with a spare.

K. The connections on the reactor vessel leak hydrogen or hydrogen fluoride:

1. Flush the system with helium, as under procedure "B", and make repairs if possible.

2. If it is not possible to repair the leak during the run, shut down the apparatus and wait until system is cool.

L. There is a fuel leak in the reactor vessel connections or any kind of leak in the reactor vessel itself:

1. Shut down immediately and flush thoroughly with helium until the system is cooled below the melting point of the molten salt.

2. Repair the leak, if possible, or replace with new reactor after the system is cool enough to handle.

M. This procedure must be adhered to strictly.. No changes are to be made in operation without the express approval of the engineer in charge.

V. GENERAL OPERATING PROCEDURE FOR BATCHING DOWN OPERATIONS

Shipping Container

The container is to be weighed with all proper connections as it will be shipped to the requestor. (See Figure 11.)

Flushing Procedure

Attach the shipping can to the transfer line as shown in Figure 3, with the spark plug probe in place, flush the system with helium in the following manner:

1. Open valve 3 and leave open.

2. Start the vacuum pump.

3. Evacuate both the transfer can and receiver by opening valves 4 and 5.

4. Close valves 4 and 5 and open valves 1 and 2 and helium valve until gauges D, I, and E read approximately 10 lbs pressure.

5. Repeat steps 3 and 4 twice.

6. Close valve 1 and open valve 5 to bring receiver down to 10 inches of vacuum.

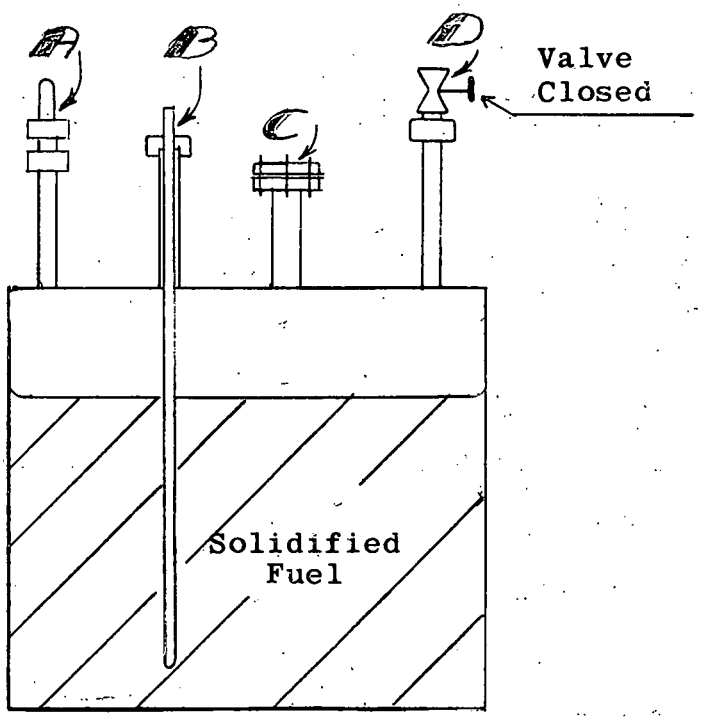

FIGURE 11: LOADED TRANSPORT CONTAINER
READY FOR SHIPMENT

Heating

7. Start the heat to the receiver furnace, keeping 10 lbs helium pressure on the shipping can and 10 inches of vacuum on the receiver. Do not heat the transfer line.

8. When the receiver temperature (Thermocouple No. 8) reaches 1000°F, start the heat to the transfer line maintaining the same pressure relation as in Step 7.

9. When the transfer line has reached a temperature between 1100°F and 1300°F, gas should flow through the transfer can to the receiver through valves 2 and 3 and out of valve 5, a distinct bubbling action should be apparent in the receiver.

10. When the receiver has reached a temperature of 1400°F, attach indicator light to the probe of the shipping can.

Transfer

11. To transfer, close valves 2 and 5 and open valves 1 and 4 making sure that the vacuum in the shipping can does not exceed 10 inches of vacuum.

12. When the melt reaches the probe in the transfer can and the light indicates, reverse the flow immediately in the following manner:

    a. Open valves 2 and 5 first.

    b. Close valves 1 and 4.

    c. Try not to cause a vigorous bubbling action in the receiver as to endanger the lines and connections from vibrations.

13. Turn off the heat on the transfer line.

14. Shut off the vacuum pump and after dissipating the vacuum, place the system on continuous flow through valves 2, 3, and 5 at a rate of approximately 2 liters per minute.

15. When the transfer line temperatures are 800°F or lower and the shipping can is cool enough to be moved,

close valve 2 and open the Cubicle helium valve enough to maintain 2 liters per minute.

16. With Cubicle helium valve open, the shipping can and sampler may be disconnected and a new can and sampler installed.

17. When installing a new shipping can, flush it thoroughly with helium before making the final connection to the transfer line.

18. After the new can has been installed, open valve 2 just enough to maintain the set rate of continuous flow. Start heating the transfer line.

19. Close the Cubicle helium valve and the small bellows valve connected to the wet test meter.