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SUBJECT: Volatility: Fluorinator Design FV-100, Zr-U Fuel Element Processing Phase

TO: R. P. Milford

FROM: J. B. Ruch

1.0 ABSTRACT

Volatility Pilot Plant Mark III Fluorinator will be a double-chamber type vessel, each chamber 2-1/2 ft. by 16 in. o.d. separated by a 5 in. pipe 15 in. long. ASME flanged and dished heads will be used for the chamber tops and conical sections with a 60° apex angle for the chamber bottoms. A new furnace designed to maintain the complete lower chamber (molten salt + freeboard) above melt temperature will eliminate past experiences of salt solidification on the wall, heads, and in or on the internal process lines. External pipe runs will be autoresistance heated to allow melting and drain back of salt plugs. The upper chamber serves as a gas de-entrainment and solids precipitation device to retain most of the entrained salt and condensable fluorides in the 100-400°C temperature range.

NOTICE

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DISCLAIMER

2.0 INTRODUCTION

Oxidation of UF_4 to UF_6 and subsequent volatilization accomplished by bubbling fluorine gas through a liquid mixture of molten fluoride salts of zirconium, lithium, sodium and fission products at 500-600°C requires a special reaction vessel. Design information for the Volatility Pilot Plant Mark III Fluorinator is furnished. Vessel replacement is required because of bottom damage caused while jack hammering a waste salt charge from the Mark II-B Fluorinator. Three previous pilot plant models revealed the following major needs: (1) a method for melting and draining unwanted solidified salt from those process lines extending into the molten salt, (2) to maintain the entire reaction chamber temperature above the salt melting point, avoiding salt solidification on the wall and in process lines above the salt level, (3) a solids settling chamber for precipitation of solids and entrainment from the gas stream at temperatures lower than those of the molten salt chamber, and (4) a satisfactory method of agitation for sampling purposes.

Preliminary studies by G. I. Cathers et al. indicated possible build up of UF_6 in a large gas chamber above the salt resulting in an equilibrating effect causing suppression of UF_6 evolution from the melt. The double chamber model avoids this condition by creating a slightly higher velocity through the reduced passage of the connecting section, thereby preventing back flow. Laboratory work indicated increased particle settling rates as the gas chamber approached isothermal conditions, believed to be the result of turbulence reduction brought about by approaching a streamlined gas flow pattern.

The Mark III double chamber model will consist of two 16 in. o.d. cylindrical sections connected by a 5 in. NPS pipe with an overall height of about 7 ft. (see Fig. 1). ASME heads will be used for the chamber tops. The chamber bottoms will be conical to facilitate funneling solids through the 5 in. neck into the lower reaction chamber and to facilitate liquid agitation in the lower chamber. The ASME Boiler Code, Section 8, Unfired Pressure Vessels, with a minmum corrosion allowance of 0.110 in. will apply. The bottom chamber is sized for a 55 liter charge plus about 50% freeboard. The upper chamber is about equal in volume. All process nozzles except the gas stream exit will enter the lower chamber top dished head in the heated zone. The product gas stream will exit through the nozzle via way of the 5 in. inspection nozzle located on top of the upper chamber. See Figures 1, 2 and 3 for general features.

3.0 GAS ENTRAINMENT STUDIES USING THE MARK II FLUORINATOR

3.1 METHODS

A barren molten salt charge was used (equi-molar $NaF-ZrF_4$) in the Mark II vessel to saturate nitrogen gas with ZrF_4 at process flow conditions of 22.5 slm. The normal gas exit through the CRP trap was sealed off and the entire stream forced through a glass wool filter cartridge attached to a corrosion specimen nozzle. Cartridge weight increase over a specific time was used for determination of gas stream entrainment rate. Studies were attempted under temperature conditions listed in Table 1.

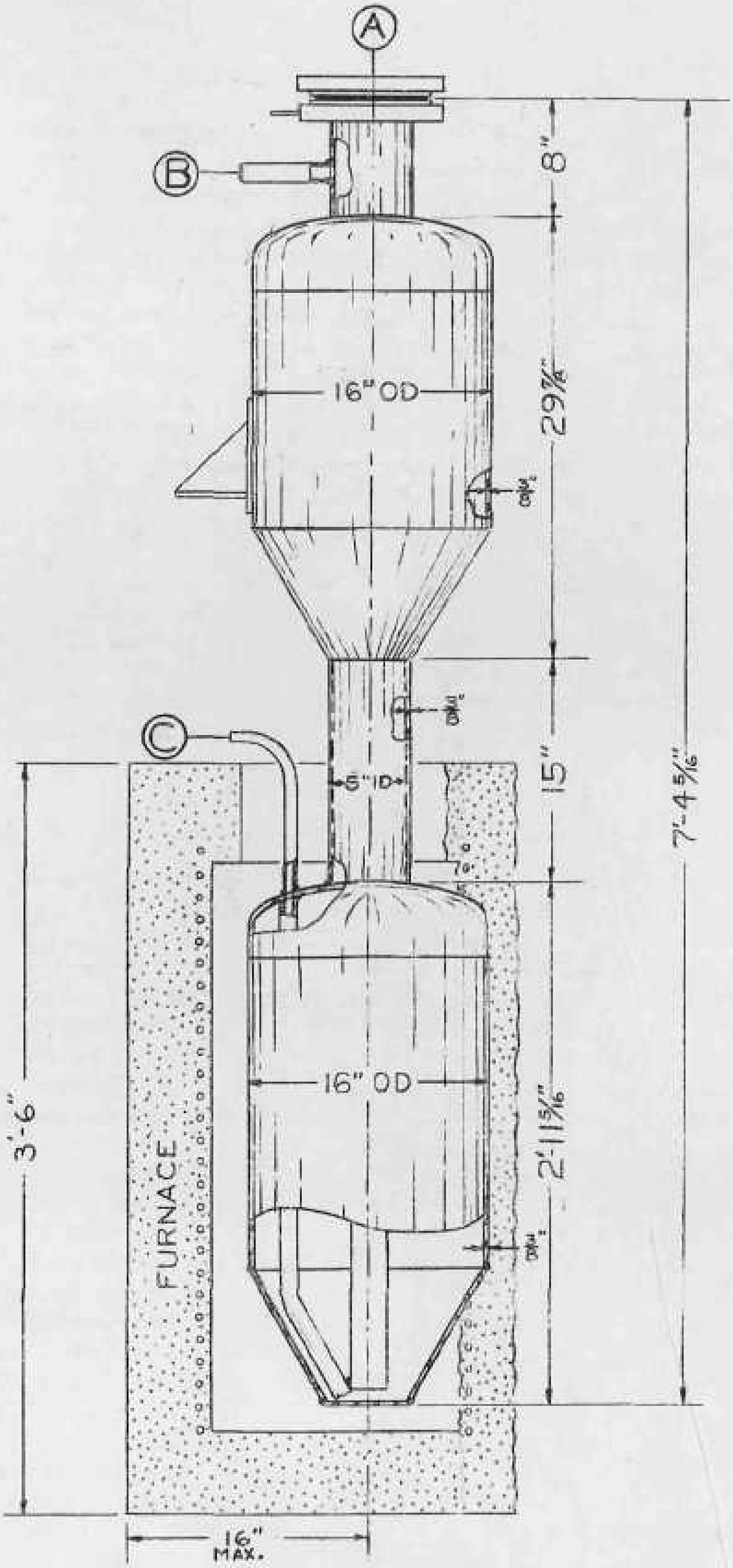
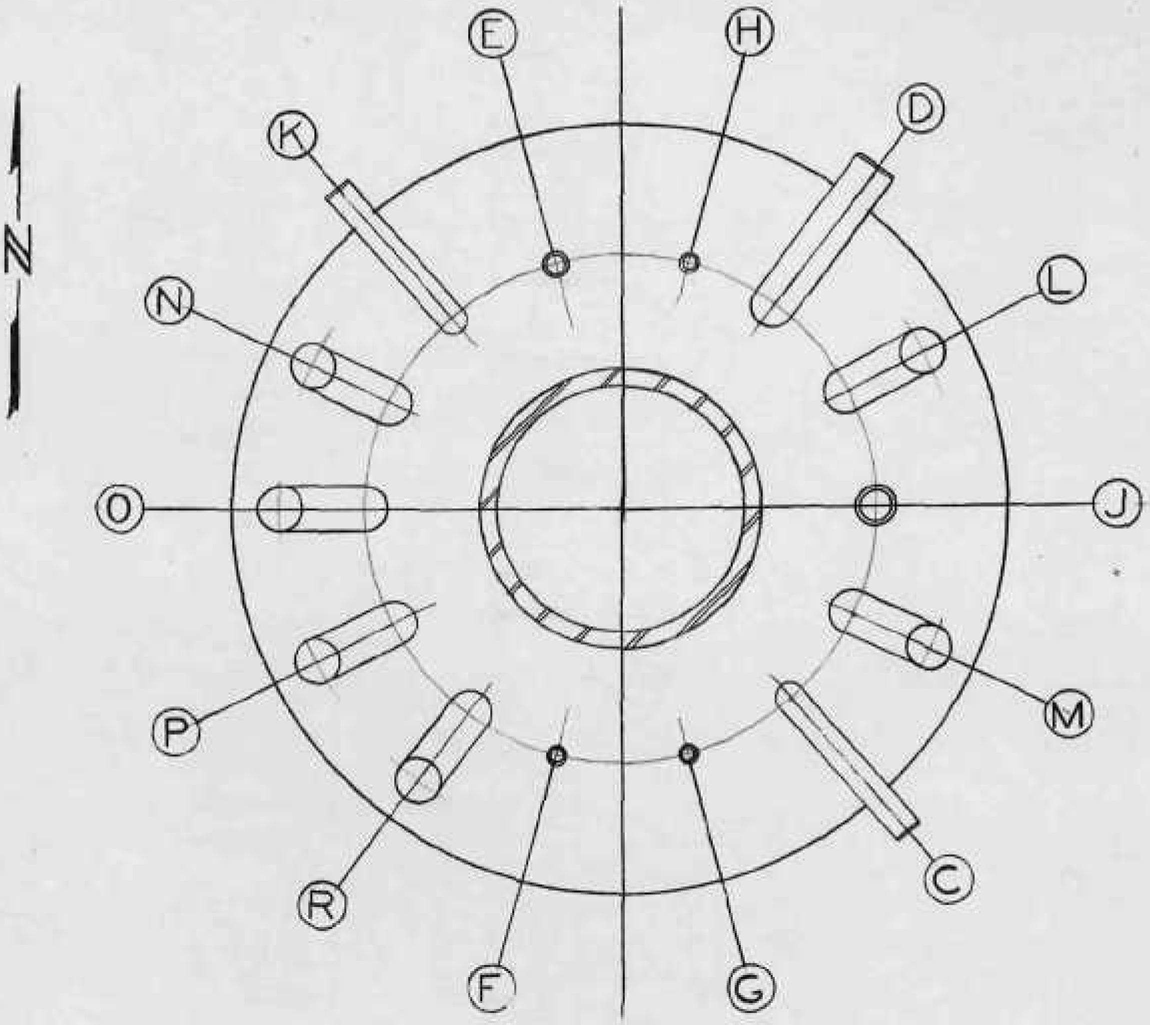


FIG. 1 FLUORINATOR FV-100 MARK III



Nozzle	Size	Service
A	5" NPS	Inspection
B	1"	UF_6 Outlet
C	3/8"	Salt Outlet
D	1/2"	F_2 Inlet
E	3/8"	Thermocouple Well
F	1/8"	LL HP and SG HP
G	1/8"	SG LP
H	1/8"	LL LP
J	3/4"	Sampler
K	3/8"	Salt Inlet
L	1/2"	Corrosion Study
M	1/2"	Corrosion Study
N	1/2"	Corrosion Study
O	1/2"	Corrosion Study
P	1/2"	Corrosion Study
R	1/2"	Corrosion Study
S	1/4"-T	Leak Detector

FIG 2 NOZZLE ORIENTATION MARK III FV-100

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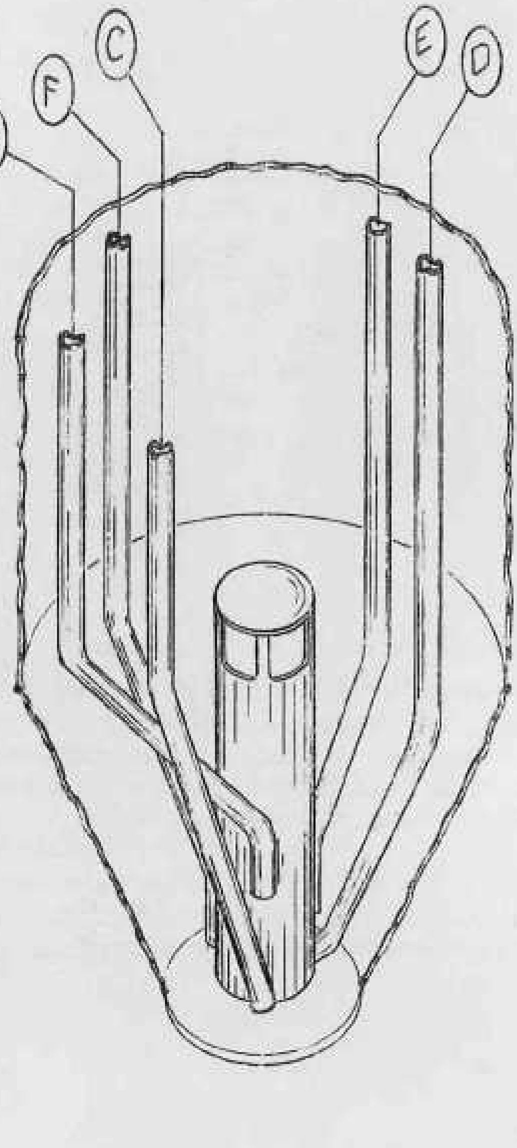

FIG. 3 DRAFT TUBE FV-100 MARK III

TABLE I

TEMPERATURE CONDITIONS FOR GAS PHASE ENTRAINMENT STUDIES

Condition Number	Molten Salt Temp. C°	FV-100 Mantle Heater(1)	Gas Phase Temp. C°	55 Gal Drum Temp.
1	600	Off	(140-250)	
2	650	Off	(190-400)	
3	600	On	(245-500)	
4	650	On	(260-530)	
5	600	On	600	
6	650	On	650	
7	600	On	600	100 (2)
8	650	On	600	100 (2)

(1) For Conditions 1 through 4 the fluorinator mantle was uninsulated except for a 4 mil sheet of aluminum foil wrapped around both the side and top flange. For all remaining conditions the mantle was heated with tubular heaters and insulated with 2 inches of diatomaceous silica-asbestos fiber insulation using the VPP standard method.

(2) For conditions 7 and 8 a 55-gallon drum was connected to the gas discharge line and the glass wool filter cartridge was moved to the far end of the drum. No heat or insulation was used on the drum.

3.2 DISCUSSION OF TEST RESULTS

Results of nine test runs using temperature conditions 1-4 above are shown in Table 2. Calculations using ZrF_4 vapor pressure data show that entrainment rates increase sharply with molten salt temperature. Experimental rates were higher for higher molten salt temperatures, but the ratio of actual solids discharge rate to the calculated ZrF_4 entrainment rate decreased rapidly with increased molten salt temperature, indicating a greater ZrF_4 vapor condensation rate at higher liberation rates or/and entrainment of another material such as the molten salt at a rate independent of temperature, and most likely a function of gas flow rate. Further proof of molten salt entrainment as indicated above was evidenced by the presence of considerable quantities of crystals of sodium-zirconium fluoride compounds determined by X-ray diffraction analysis of the collected solids from two runs. Approximately 70% of the material was sodium-zirconium fluoride crystals during periods of low ZrF_4 vapor generation (solids collection rate of 0.60 g/hr-Run 3) and only 10-30% during periods of high ZrF_4 vapor generation (solids collection rate of 2.3 g/h Run 4).

In a series of runs with an uninsulated mantle and a melt temperature of 600°C, the gas phase exit temperature was increased from 140°C to 245°C, this increased the solids discharge rate by about 10%. In another series of runs where the molten salt temperature was held at 650°C the gas phase exit temperature was increased from 200°C to 260°C and the solids discharge rate was increased by about 30%. This may indicate better solids retention conditions exist with lower melt and/or lower exit temperatures.

Runs using conditions from No. 5 on in Table 1 failed because of plugging in the gas-discharge line at the fluorinator top flange. Attempts to heat this line and flange above the ZrF_4 condensation temperature were unsuccessful because of equipment heating limitations, and since time consuming extensive modifications would have been required to correct this situation, these tests were discontinued in favor of testing in the Mark III model prior to radioactive runs. However, before plugging occured in the last series of runs, indications were that only about 10 to 20% of the solids entering the drum were discharged to the glass wool collector. Plugging of the exit line was not experienced during the cooler mantle temperature runs probably because ZrF_4 solids (or snow) were formed in the gas phase and carried through the cool line (200°C) as entrained particles rather than a vapor.

3.3 CONCLUSIONS

1. All fluorinator operations should be carried out at the lowest possible molten salt temperature.

2. The upper zone and preferably an upper chamber should operate at temperatures of 400°C or lower to minimize entrainment.

3. A series of similar runs should be carried out in the Mark III Fluorinator prior to any cold run testing. Experimental data of this nature would be extremely valuable during future process runs.

TABLE 2

ZrF_4 ENTRAINMENT RESULTS FROM PILOT PLANT STUDIES

Run No.	Molten Salt Temperature, °C	Mantle Heaters	Gas Phase Temperature Range, °C	Calculated Entrainment of ZrF_4 From Vapor Pressure Data (1) grams/hr	Experi-mental Rate, grams/hr
1	650	Off	(200-425)	4.12	1.1
2	670 ave.	Off	(190-400)	6.13	1.5
3	690 ave.	Off	(175-380)	11.25	2.3
4	600	Off	(140-300)	0.94	0.60
5	600	Off	(140-250)	0.94	0.66
6	650	On	(260-530)	4.12	1.44
7	600	On	(245-500)	0.94	0.60
8	600	On	(245-495)	0.94	0.72
9	600	On	(240-490)	0.94	0.70

(1) BMI - 1064 p 13

4.0 VESSEL REQUIREMENTS

4.1 PHYSICAL FEATURES

General size and shape requirement of the Mark III Fluorinator double chamber unit is shown in Fig. 1. Both chambers have a nominal 16 in. dia, are 2-1/2 ft. high and are connected by a 5-3/4 in. dia x 15 in. cylindrical section. ASME flanged and dished 3/8 in. standard heads will be used for the chamber tops. The chamber shell wall, conical sections and cylindrical connecting section will be fabricated from 3/8 in. nickel plate. The inspection nozzle located on top of the vessel will be 5 in. sch 40 pipe sealed with a blind flange. Flanges will be 5 in. 150 lb. ASA ring joint with an octagonal soft copper No. R-40 ring and VPP leak test device.

4.2 NOZZLES

4.2.1 Orientation

Nozzle orientation for the lower chamber top head is shown in Fig. 2. The inspection, UF_6 outlet, and leak detection nozzles, A, B and S in the Fig. 2 nozzle list, are located on the upper chamber top section shown in Fig. 2. All nozzles are sch 40 pipe except Nozzle S which is a standard tube size.

4.2.2 Internal Process Lines

All internal lines will be 1/2 in. NPS sch 80 "A" Nickel pipe. The waste salt outlet, fluorine inlet, and instrument lines will be rigidly supported by the draft tube. The high-pressure liquid level and specific gravity lines will have snubbers on their outlets similar to those of the previous design. An inverted funnel-shaped guide will be located beneath the sample nozzle.

A draft tube fabricated from 2 in. sch 40 pipe will be attached to the internal pipes as shown in Fig. 3. Its function will be to provide adequate mixing during sampling operations.

4.2.3 Corrosion Specimens

Corrosion specimen nozzles will be equipped with Swagelok butt weld connectors as previously used. Proper alignment is essential to permit free passage of the rod into the lower chamber. Six nozzles will be provided.

4.2.4 Autoresistance Heating

Process lines extending into the molten salt mixture will be autoresistance heated for a distance of about 10 ft. from the vessel. Lines involved are: the waste salt, fluorine inlet, liquid level high pressure, and specific gravity high pressure. The split circuit method will be used to heat the lines from the vessel head to a high point in the cell. Heated sections will require high temperature insulation for utilization of existing and new electrical transformers of reasonable size and capacity.

4.3 VESSEL SUPPORT

Vessel supports attached to the upper chamber will carry the entire vessel weight plus charge at process conditions. The bottom chamber will be suspended freely in the FV-500 Furnace.

4.4 MATERIALS OF CONSTRUCTION

The vessel shell and heads will be fabricated from 3/8 in. "L" Nickel plate. All external nozzles will be fabricated from "L" Nickel pipe except where autoresistance heating requires Inconel. All internals will be fabricated from "L" Nickel materials but "A" Nickel pipe will be substituted where "L" Nickel cannot be procured in reasonable time. Where advisable Inconel pipe and flanges will be substituted for "L" Nickel to avoid procurement delays. Vessel supports may be fabricated from Inconel or other satisfactory metals. Swagelok connectors for corrosion specimen nozzles should be Inconel.

4.5 CORROSION ALLOWANCE

A minimum corrosion allowance of 0.110 in. will be used for design purposes.

5.0 PROCESS REQUIREMENTS

5.1 PRESSURE

During normal fluorinator operations the vessel will be at atmospheric pressure, except 5 psi N_2 is required to start siphoning of the waste salt transfer. A plug in the gas stream, or waste salt line, could result in a pressure increase equal to that of the maximum regulated nitrogen or fluorine supply in use. In past pilot plant operations the maximum pressure of regulated supplies was 12 psi. The 20 psi vessel design pressure selected includes a reasonable safety factor.

5.2 TEMPERATURE

The normal operating range will be 450-600°C for both fluorination and waste salt transfer operations. The desired exit gas temperature range is 100-400°C. The lower chamber and intermediate connecting section between chambers will require a 750°C maximum operating temperature to melt off-specification salt mixtures and material splattered up on the walls during processing.

5.3 CORROSION

Past VPP operations produced a corrosion rate of 0.055 in. per month under the most severe conditions, hence the 0.110 in. allowance should allow continuous operation for a minimum of two months.

5.4 SAMPLING

Feed and waste samples for S. F. accountability and process control are necessary. In pilot plant experiments[1] waste samples were taken by three methods: the standard copper ladle (top sampling), flowing stream, and solid sections from the waste container. The average uranium concentration was about a factor of 10 higher for the flowing stream over the standard method and about a factor of 20 lower than the solid sampling method. This indication of inadequate mixing led to a series of draft tube experiments[2] that showed a conical bottom with center spaced draft tube should produce adequate agitation.

6.0 HEATING REQUIREMENTS

6.1 FLUORINATOR FURNACE

A pot type furnace capable of heating and maintaining the lower chamber shell at 750°C is required. An elevator device similar to the previous one will be required to lower the furnace for Vidigage measurements, and removal for maintainance purposes. The power requirement will be 35 kw. Specifications are given in Section 7. The furnace will contain a 3/16 in. Inconel liner.

6.2 INTER-CHAMBER HEATER - FV-500A

The Inter-chamber Heater will cover the intermediate cylindrical section between chambers and the conical bottom section of the upper chamber using a single control device. It will have the capacity to heat the inside vessel wall to a 750°C maximum temperature. A design power input of 2 kw per sq ft of surface area was adequate on previous units. Tubular heaters with alloy sheath for recommended maximum serivce of 1500°F will be used as the basic heating units. They will be wrapped with stainless steel shim stock and 2 in. of diatomaceous silica-asbestos fiber insulation where needed. Techniques applied in building previous heaters will be used.

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[1] C. L. Whitmarsh, "Reliability of the Salt Sampling Procedure in VPP," CF-59-1-131.

[2] J. B. Ruch and S. H. Stainker, "VPP - Fluorinator Design, Effect of a Conical Bottom on the Solids Circulation Rate," CF-59-5-15.

6.3 UPPER CHAMBER HEATER, FV-500B

The upper chamber heater covers the surface above the conical bottom. The product gas outlet nozzle will be heated by line heaters rather than this heater. A maximum service temperature of 500°C will be required for this zone. A minimum power input of 2-kw per sq ft. of surface area will be used. Tubular heating units will be used in the same manner as described in Section 6.2 above.

7.0 APPENDIX

7.1 DRAWING LIST

D-33393	FV-100 Mark III Fluorinator
D-33394	Fluorinator Details Sheet No. I
D-33395	Fluorinator Details Sheet No. II
D-35410	Fluorinator Details Sheet No. III
D-56592	Heaters, FV-500A and FV-500B

7.2 SPECIFICATIONS FOR FV-500 MARK II

OAK RIDGE NATIONAL LABORATORY
Operated by
Union Carbide Nuclear Company
Chemical Technology Division
Process Design Section

Issued: March 24, 1959

SPECIFICATIONS FOR ELECTRIC FURNACE, ITEM FV-500
VPP HYDROFLUORINATION ADDITION, BUILDING 3019

I. SCOPE

This specification covers the requirements for one electric furnace, for use in melting salt contained in a vertical, cylindrical vessel. The furnace is to be rated at 30 kw minimum. This specification includes the furnishing of the furnace complete with internal wiring, but with no electrical equipment beyond the incoming power terminal box unless a transformer is required for furnace operation. No cover is required.

The following drawing is a component part of this specification: D-33393 FV-100 Mark III Fluorinator.

II. TYPE OF SERVICE

The furnace is required to heat a vessel in which a radioactive salt is processed at a temperature of 1400°F maximum. The vessel to contain the radioactive salt will be supplied by the buyer. This vessel will be supported independently of the furnace.

III. SPECIFICATIONS

A. Mechanical

1. The portion of the vessel that the furnace is to heat is 16 in. o.d. x 30 in. high. The inside diameter of the furnace will have to be 19 in. diameter to receive this vessel with adequate clearance. The heated height of the furnace shall be 42 in. The outside diameter of the furnace shall be held to a minimum with a 32 in.-maximum o.d. if feasible.

2. The exterior surface of the furnace shall be covered with at least a No. 10 USS gauge (0.137 in.) sheet steel jacket to protect the insulation from damage. All exterior metal surfaces are to be finished with two coats of aluminum paint.

3. All metal parts which are exposed to the heat of the furnace shall be of a suitable stabilized austenitic stainless steel.

B. Thermal Insulation

The furnace shall have sufficient insulation to limit the outside surface temperature to 150°F maximum with an ambient air temperature of 70°F when the temperature inside the furnace is 1400°F.

C. Electrical Requirements

1. Available Power

The characteristics of available power are 230 volts, three phase, 60 cycles A. C.

2. Power Rating

The total power input to the furnace shall be 35 kw minimum. Three separate heating elements shall be delta connected into a single control zone. The delta connection is required to allow continued operation in the event that a single element fails.

3. Control

The operating temperature inside the furnace will be maintained by control equipment to be furnished by the Buyer. The Seller shall supply a suitable thermocouple protecting tube inserted through the wall of the furnace.

4. Heating Elements

The heating elements shall be sutiable for exposure to furnace atmosphere and continuous operation at 1600°F with maximum life expectancy; they shall be round cross section (rod-type element); and the watt density per square inch of exposed surface shall not exceed 13. Heating elements shall have a nominal chemical composition of 80% nickel and 20% chromium.

Elements shall be supported in a manner which allows even and free sliding of the element with sufficient provision for expansion and contraction throughout the entire length without shorting out or grounding any portion. Sufficient electrical insulation shall be provided to permit ground-free operation at 1600°F when connected to the required voltage. Heating elements shall be supported at sufficient intervals so that when thermal expansion and contraction occur, the elements do not contact any object other than element insulator-supports. All materials used in contact with the heating elements shall be of such composition that essentially no oxidation or high temperature corrosion will occur at normal operating temperature in air. The heating elements themselves shall remain essentially oxidation and corrosion-free at 1600°F in air.

No copper shall be used where the temperature exceeds 350°F. The terminals shall be so arranged that the buyer can make power supply connections without disturbing the heating element leads. If splices are to be made to the elements to bring out the leads, such splices shall be brazed or welded to the elements.. All leads which run through the insulation shall be of adequate size and current capaci.y to avoid excessive temperature rise at these points at full load.

IV. GENERAL CONSIDERATIONS

A. Information Required

1. The Seller shall submit with his quotation approximate outline and internal furnace dimensions. In addition, he shall submit complete data on the elements he proposes to use, including geometries of element arrangement, watt density, and element brand designation.

2. Prior to the start of construction the Seller shall submit for approval nine copies of detailed drawings covering dimensions, assembly, non-standard details, weights, materials, and wiring diagrams.

3. Within 15 days after written approval, the Seller shall furnish nine certified copies of each drawing. Only after the Buyer grants approval in writing shall the Seller proceed with fabrication.

4. The Seller shall furnish the Buyer eight complete sets of installation, operation, and maintenance manuals of instructions, including parts lists and a recommended list of spare parts, within 30 days after notification of the contract award.

5. The Buyer's purchase order number shall be shown in the title block of each drawing.

Specifications prepared by ______________________
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