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SUBJECT: A Metal to Graphite Joint for a Molten Salt System

TO: Listed Distribution

FROM: J. L. Crowley and W. B. McDonald

Abstract

A two region graphite moderated molten salt thermal breeder reactor with a graphite separator between fuel and blanket fluids will require a graphite-to-metal seal between the core and the blanket regions. One method of sealing is to form a frozen salt seal separating the fuel and blanket salts. This report describes a freeze seal between an Inconel header and a 5 inch diameter graphite (type S-4) crucible. Molten salt was circulated past the seal at 1150°F for 24 hours with a differential pressure of 18 psi across the seal. Heat was removed from the seal at a rate of approximately 4.5 KW by passing water through a coil brazed to the outside metal wall of the seal. No salt leakage was detected past the seal.

Introduction

This report describes a frozen salt, metal-to-graphite joint and gives the results of two tests. This type of joint is proposed as a method of preventing leakage between the breeder blanket salt and fuel bearing salt in a graphite moderated, two region molten salt thermal breeder reactor.

The tests were performed in a circulating molten salt system with a graphite crucible and a graphite bayonet tube to simulate a unit fuel tube construction[1] configuration of a molten salt thermal breeder reactor.

Two conditions of seal differential pressure (ΔP) were tested: (a) with back-up gas pressure to limit ΔP to within 0.5 psi, and (b) with an 18 psi ΔP across the seal.

Description of Test Apparatus

The test piece is shown in cross section in Fig. 1. The dimensions of the graphite crucible are 5 in. diameter and 12 in. deep with 3/4 in. wall. The crucible was machined from a molded block of type S-4 graphite[2] originally 3 feet in diameter and 1 foot thick.

The graphite crucible is supported in the flanged pot by a lava insulator. The upper flange contains the inlet and outlet lines, a fill line, external cooling coils, and seal annulus. A 1/16 in. gap is maintained around the outside diameter of the graphite by set screws. Just inside of the graphite crucible, opposite the seal area, is a 3 in. diameter metallic skirt which extends slightly below the level of the frozen seal area, as shown in Fig. 1. This skirt serves as a thermal barrier between the flowing stream of high temperature molten salt and the seal annulus. Cooling for the formation and maintenance of the seal is provided by copper tubing coils brazed to the outside wall.

A catch basin is mounted directly beneath the seal to retain any salt leakage for observation. The sheet metal basin is attached to the graphite crucible by welding to a metal hoop closely fitted in the circumferential groove around the crucible. A port is provided above this catch basin through which observations can be made without disassembly of the test. A borescope can be inserted for examination of the seal mouth and catch basin after the test has been cooled to room temperature.

The flanges are bolted and sealed with an asbestos gasket to maintain an inert atmosphere around the graphite during the filling and operation of the test.

As the molten salt is discharged from the pump into the test piece it is conducted through a 3/4 in. OD graphite tube extending the entire length of the chamber. The graphite tube is positioned with slip fit sockets at either end to simulate a unit fuel tube configuration[1] and otherwise has no direct bearing on the conduction of the test.

The salt flow is down through the center graphite tube and upward through the graphite crucible to the test piece outlet. The salt seal is formed initially during the filling when molten salt spills over the rim of the graphite crucible into the annulus next to the cooled wall. When the salt freezes in the annulus and forms a seal, the filling is continued until circulation is established and normal operating level is attained in the pump.

The auxiliary equipment is shown in Fig. 2. The test piece is shown at the right connected to the pump which is located at the upper left. The fill and drain tank, shown at the lower left, is connected to the test piece through a frozen plug valve. This photograph shows the loop prior to insulation and pump installation. The loop tubing and pump are fabricated of Inconel. The pump used is a centrifugal model LFB[3] (shown in Fig. 3).

All heating of the loop piping and pump bowl is accomplished with calrod heaters. The test piece is heated from the outside of the flanged pot with ceramic clam shell type heaters, as shown in Fig. 1.

Helium is supplied to the pump, drain tank, and to the flanged pot through separate control valves. The loop and test piece are filled by applying helium pressure at the drain tank. Draining is accomplished by the application of helium pressure to the pump surge tank and forcing the salt from the test piece into the drain tank.

Conduction of the Tests

The first test was to determine whether or not the frozen joint could be successfully established with no seal ΔP. The entire loop and test piece were preheated to above the melting point of the salt to be used. Salt composition 30 ($NaF-ZrF_4-UF_4$, 50-46-4 mole %) with a melting point of $968^{\circ}F$ was used because of less hazard involved in handling a non-beryllium bearing salt. Its melting temperature is close to that of beryllium bearing salts[4] contemplated for use in a molten salt reactor.

No special preparation, other than that given in the manufacture of type S-4 graphite,[2] was given the crucible before operation of the test. The graphite was installed as received from machining. A positive pressure helium blanket was maintained in the loop and around the outside of the graphite crucible while preheat was applied and during the filling operation. A helium atmosphere was maintained around the crucible and at the molten salt free surface in the pump during operation.

Approximately 0.1 gpm of water was circulated through the coil to cool the seal area during preheat previous to the first test. As the loop was filled, the cooling water was increased to 0.9 gpm. When the frozen plug valve was established in the loop fill line, the pump speed was increased to establish circulation of salt through the loop and test piece.

The pump speed was increased gradually up to 2000 rpm while the regulated helium pressure to the flanged pot was also increased to match the calculated pump head at that speed. The calculated internal salt pressure at the seal was 9.5 psig at this pump speed and the helium back-up pressure was maintained between 9.0 and 10.0 psig.

Conditions of the first test as shown in Table I were maintained for 45 hours before the loop was drained and allowed to cool for inspection. A borescope inspection was made through the inspection port to determine that there was no salt leakage past the seal to the catch basin. Also, the absence of HF odor, which is characteristic of salt leaks, in the helium vented from the flanged metal pot confirmed that there was no salt leakage below the catch basin.

The same start-up procedure was used for the second test until the loop was filled and salt circulation was established. The frozen salt formed in the seal annulus during the first test was maintained in the solid state by cooling during the filling operation for the second test. In effect the same frozen seal was used for both tests.

For the second test the pump speed was increased to 3000 rpm, which increased the calculated pressure at the seal to 13.2 feet of fluid or 19 psig. The helium pressure in the flanged pot was held between 1.0 to 1.5 psig giving a seal ΔP of from 17.5 to 18.0 psi.

Several hours after the start of the second test there were indications of salt leakage into the flanged pot. These indications were a blinking low level probe in the pump surge chamber and the odor of HF in the gas vented from the flanged pot. Operation was continued, however, at the conditions given in Table I, Test No. 2, for a period of 24 hours.

After the loop was drained and cooled the test piece was cut from the loop and the pot removed from around the graphite crucible for inspection. There was no salt in the catch basin under the seal nor was there any indication of salt leakage past the seal. As shown in Fig. 4, extrusion of salt occurred through the wall of the graphite crucible. The leakage took place through the graphite in the area below that covered by the internal skirt. An estimated 20 cu. in. of salt was extruded through the pores of the graphite over an internal surface area of approximately 70 sq.in. or a rate of about 0.3 in^3/ sq.in. of surface per day.

The test piece shell was cut just above the rim of the graphite crucible as shown in Fig. 5. The frozen salt shown on the inside surface of the graphite container indicates that cooling rate used in maintaining the seal was excessive. The 3/16 in. thick layer of salt extends 5 1/2 in. into the graphite container to occupy the entire annulus between the skirt and the inner surface of the graphite crucible. The 1/16 in. outer seal annulus and upper rim of graphite crucible may be seen where part of the salt has been chipped away.

Conclusions

It has been demonstrated that a frozen salt, metal-to-graphite joint is feasible. From the amount of frozen salt seen in Fig. 5 in the internal annulus, it is concluded that the cooling rate was excessive and could be considerably reduced for a seal of this configuration. Further tests should be conducted to determine limitations of seal ΔP and heat removal rate. Since the first two tests indicated that the limiting factor is the penetration of the graphite by the salt and not the seal under test, a less porous type should be used in future testing of joints.

Acknowledgements

The authors wish to acknowledge the assistance in the test and in the preparation of the report of A. S. Olson and W. H. Duckworth. A. Taboada of Metallurgy Division furnished the graphite for the test and W. R. Osborn of Reactor Projects Division handled fabrication of test components.

Appendix A

National Carbon Company
Division of Union Carbide Corporation
Niagara Works, Niagara Falls, N.Y.
April 6, 1960

Mr. A. S. Olson
Oak Ridge National Laboratory
Union Carbide Nuclear Company
P.O. Box Y
Oak Ridge, Tennessee

Dear Mr. Olson:

Mr. Janes has referred your letter to me regarding your request for physical properties of low permeability grades S-4 and RLM-24. "S-4" was a temporary designation for a grade which is now known as R-0025. The typical properties of these grades are as follows:

	R-0025		RLM-24	
	W. Gr.	A. Gr.	W. Gr.	A. Gr.
Density, gr/cc	1.90		1.85	
Sp. Resistance, ohm cm x 10^6	1200	1500	700	(1000)
Thermal Conductive BTU/ft^2/ft/^oF	65	56	95	(60)
Tensile Strength, psi	3000	2900	2200	---
Flexural Strength, psi	4000	3900	3400	---
Compress Strength, psi	8600	8000	9000	---
Mod. of Elast.	1.90	1.50	1.95	---
CTE, $10^{-6}/^oC$	2.4	2.9	1.5	(3.5)
Admittance, cm^2/sec N_2 x 10^{-3}	3	2	--	2

W. Gr. and A. Gr. = with and against the grain.
Figures in parenthesis are estimated since stock in form of tubes.

We hope that the above supplies you with the information you need. If not, please let us know.

Very truly yours,

/s/ R. L. Mansfield

Product and Process Development Laboratory

R.L. Mansfield
blj

TABLE I

Operating Conditions During Graphite Joint Tests 1 and 2

		Test #1	Test #2
1.	Avg. Loop Temperature, °F	1110	1140
2.	Salt Inlet to Test Piece Temperature, °F	1127	1155
3.	Salt Outlet From Test Piece Temperature, °F	1112	1140
4.	Graphite Crucible Temperature, Bottom, °F	1137	1150
5.	Metal Surface Temperature Above Rim of Graphite Crucible, °F	550	643
6.	Metal Surface Temperature Around Seal Annulus, °F	116	127
7.	Cooling Water ΔT, °F	21	28
8.	Cooling Water Rate, gpm	0.96	1.1
9.	Heat Removal Rate of Cooling Water, KW	2.95	4.5
10.	Pump Speed, RPM	2000	3000
11.	Approximate Salt Flow Rate, gpm	2	3.5
12.	Calculated Salt Pressure at Seal, psig	9.5	19.0
13.	Regulated Helium Pressure to Flanged Pot, psig	9.1	1.0
14.	Frozen Salt, Graphite to Metal Seal ΔP, psi	0.4	18.0
15.	Length of Operation, hours	45	24

References

1. H. G. MacPherson, Molten-Salt Breeder Reactors, ORNL CF 59-12-64 (January 12, 1960)

2. R. L. Mansfield, Personal Communication, Physical Properties of S-4 Graphite, April 6, 1960. (See appendix)

3. A. G. Grindell, E. F. Boudreau, and H. W. Savage, "Development of Centrifugal Pumps for Operation with Liquid Metals and Molten Salts at 1100-1500°F, Nuclear Science and Engineering, 7, 83-91, (Jan. 1960)

4. J. P. Blakely, Molten-Salt Compositions, ORNL CF-58-6-58 (June 12, 1958)

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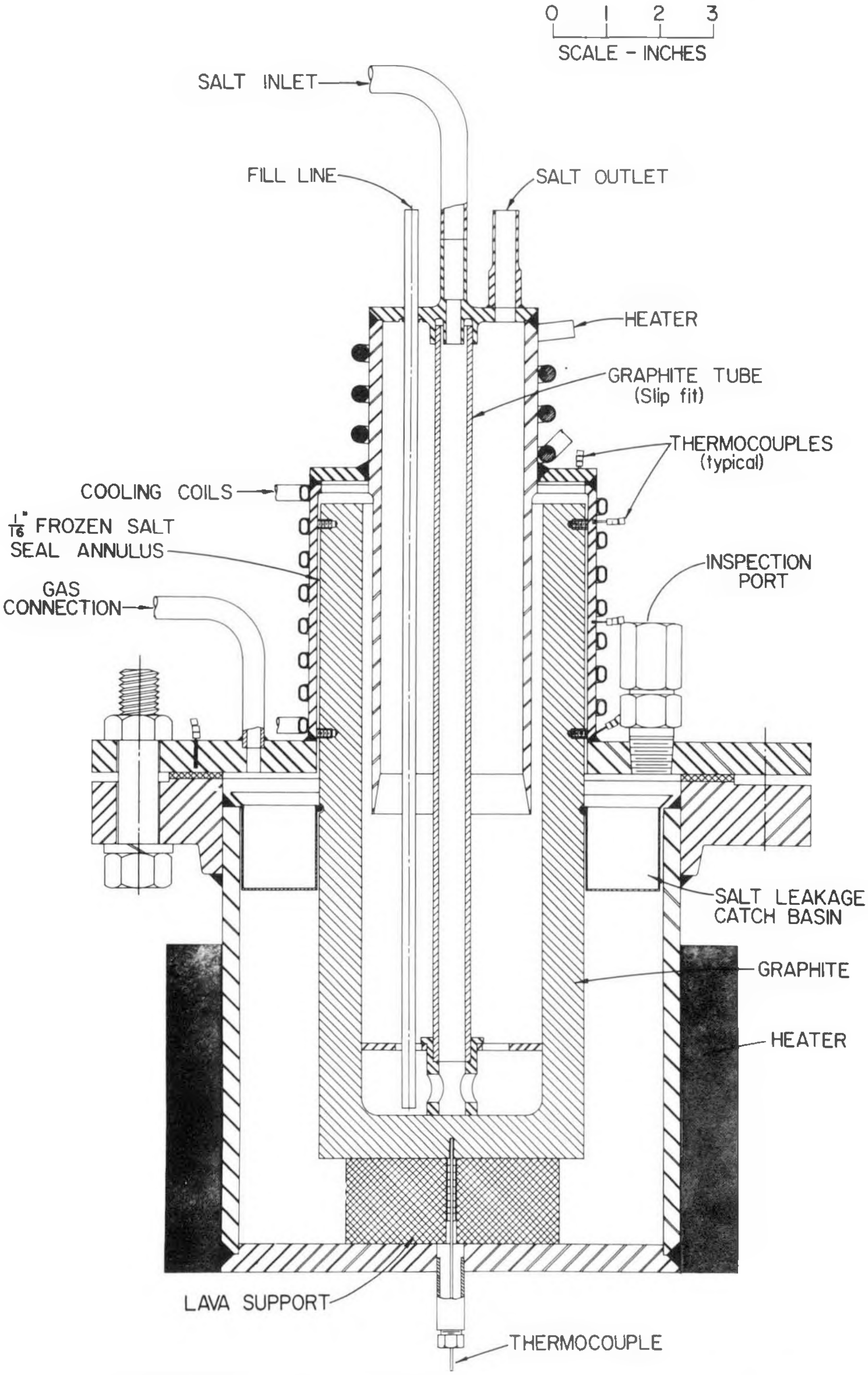

Figure 1. Frozen Salt Metal to Graphite Joint.

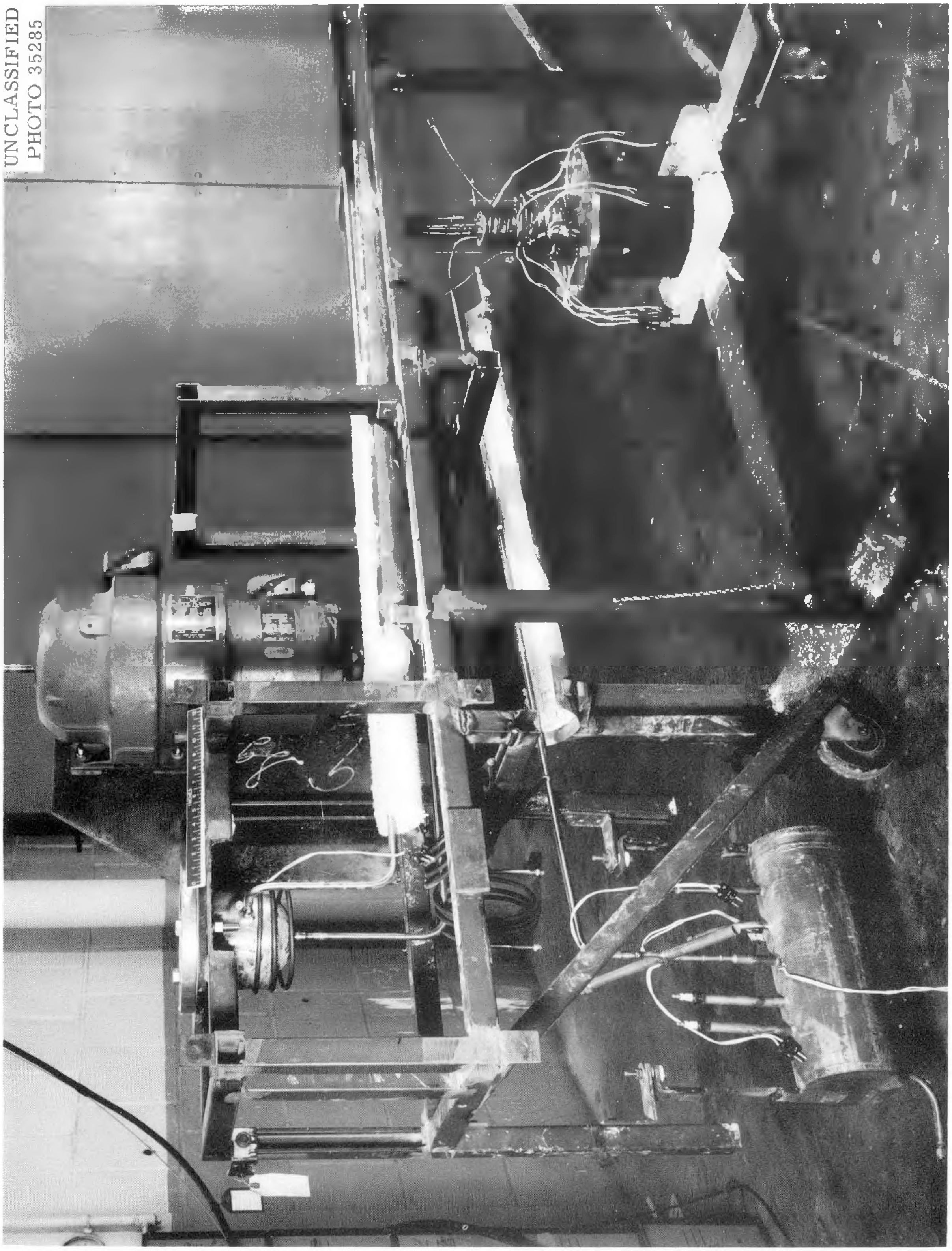

Figure 2. Facility for Testing Metal to Graphite Joint.

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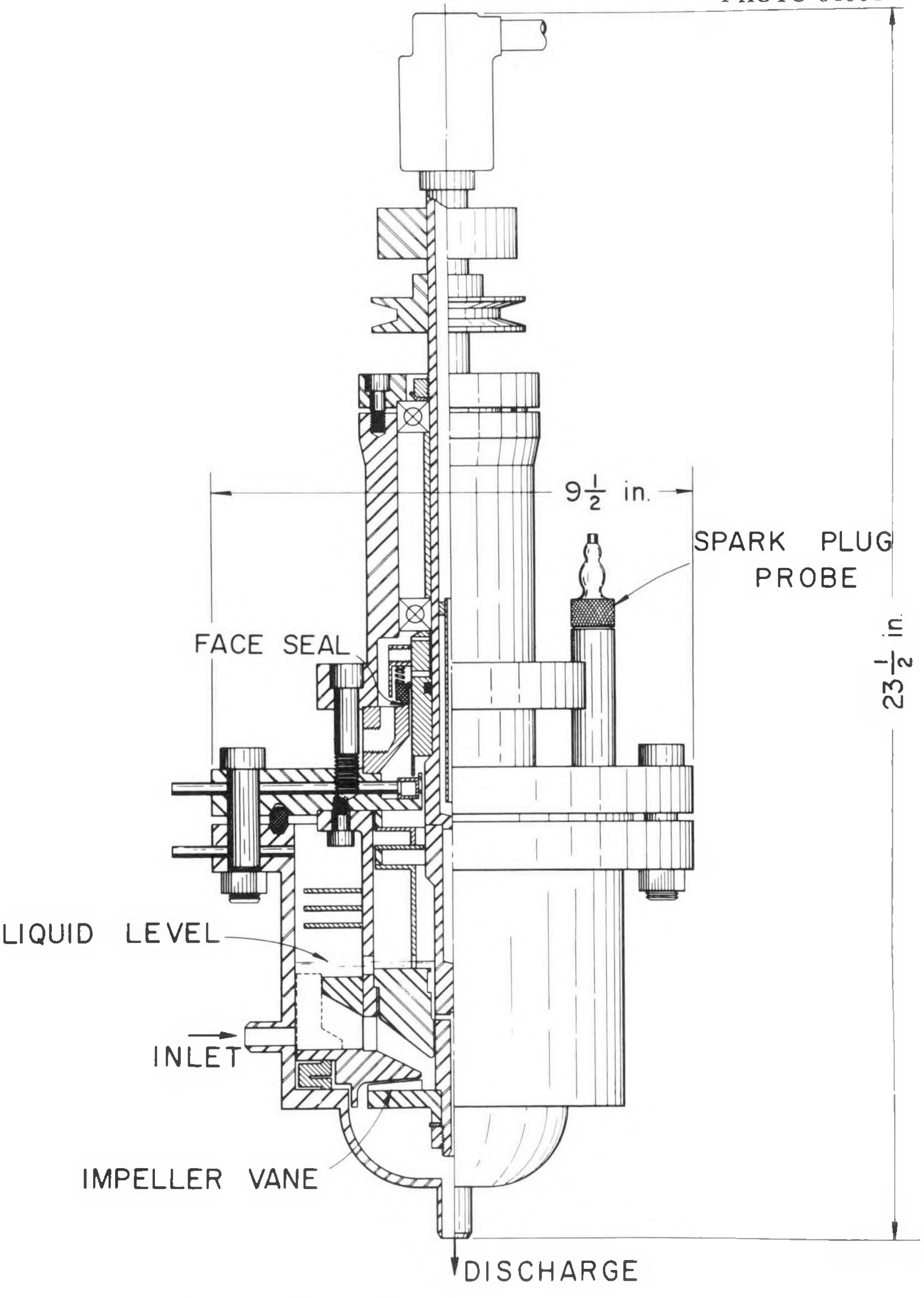

Figure 3. Centrifugal Model LFB Pump.

Figure 4. Extrusions of Salt Through Pores of Graphite Crucible.

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Figure 5. Annuli of Frozen Salt Internal and External to Graphite Crucible.

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