13

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SUBJECT: HRP In-Pile Corrosion Test Loops, Operation of In-Pile Loop L-2-19.

TO: E. G. Bohlmann

FROM: R. A. Lorenz

SUMMARY

Loop L-2-19 operated in HB-2 of the LITR for 1148.9 hours of circulation and 2705 Mwhr of LITR operation. The solution contained 0.17 m enriched UO_2SO_4, 0.02 m $CuSO_4$, 0.12 m excess H_2SO_4, and 0.20 m Li_2SO_4 in H_2O. The main stream was 280°C and the pressurizer 295°C.

There was no unusual difficulty during loop operation. A revised loop sample procedure gave better flushing of the sample lines. Four valves failed due to severe corrosion and fracturing of the 420 SS stems. I-131 was not detected in a sample of gas taken from the gas hold-up tank. The run was terminated when the circulating pump failed.

DISCLAIMER

HRP IN-PILE CORROSION TEST LOOPS, OPERATION OF IN-PILE LOOP L-2-19

TABLE OF CONTENTS

HRP IN-PILE CORROSION TEST LOOPS, OPERATION OF IN-PILE LOOP L-2-19

1. Introduction

Loop L-2-19 was the fifteenth completed in-pile loop experiment and the fifth in the HB-2 beam hole at the LITR. The loop was inserted on October 2, 1957 and removed from HB-2 on November 17, 1957.

Total Circulation time with enriched fuel	1148.9 hr
Total Circulation time with enriched fuel before irradiation commenced	93.4 hr
Total accumulated LITR energy	2705 Mwhr
Oxygen pressure in pressurizer	42 to 157 psi
Pressurizer temperature - CA (TC #9)	295°C
Main stream temperature - core outlet (TC #3)	280°C
Approximate concentration of original fuel	
UO_2SO_4 (enriched) in H_2O	0.17 m
$CuSO_4$	0.02 m
Acid (excess), H_2SO_4	0.12 m
Li_2SO_4	0.20 m

The loop temperature was lowered once during in-pile operation to allow replacement of a valve in the large equipment chamber (see sections 6.1 and 8.3).

2. Radiation Effects

2.1 Fission and Gamma Heat. The average total fission and gamma heat at 3 Mw reactor power, as determined by electrical power differences was 2558 ± 31 watts[1]. There was no significant drift or shift of the fission and gamma heat during the run. The loop core was made of type 347 stainless steel and contained a measured fuel solution volume of 239 cc at room temperature.

2.2 Radiolytic Gas. The average radiolytic gas pressure in the pressurizer at 3 Mw reactor power was 4.7 ± 0.5 psi using chromel-alumel thermocouple #9 as a pressurizer temperature reference. The average radiolytic gas was 2.9 psi using iron-constantan thermocouple #10 as a reference. Chromel-alumel thermocouple #9 is measured with a manually operated potentiometer generally considered to be more sensitive than the electronic null-balance recording potentiometer used with iron-constantan thermocouple #10. This helps explain the better precision of the chromel-alumel measurements, but does not explain the consistent difference between the C-A and I-C readings. It may be that one thermocouple was more affected by radiation heating than the other thermocouple. There was no significant shift of the radiolytic gas pressure during the run.

2.3 Radiation Intensities. Radiation intensities encountered during sampling and loop removal were higher than those encountered in previous HB-2 loops. A summary of radiation measurements is presented in Table II.

3. Mechanical Performance

3.1 Circulation Rates. The flow rate in the main stream was 5.3 gpm as determined by room temperature pressure drop measurements at Y-12. The flow rate through the pressurizer, as measured at Y-12, was 6.6 ± 0.9 cc/sec. The pressurizer flow rate, as measured by electrical power differences, remained constant at 6.4 ± 0.3 cc (287°C)/sec. during operation at the LITR.

3.2 Pump Performance. Run L-2-19 was terminated by failure of the ORNL 5-gpm circulating pump. From all outward appearances, the failure of this pump was very similar in nature to the pump failure encountered in run L-2-17[2]. During attempts to restart the pump and to rinse the loop with water, there were indications that some circulation was obtained.

Wheatstone Bridge measurements of the resistance of the start and run windings were taken both before and after pump failure. These data, which are tabulated below demonstrate that the abnormally high currents encountered were caused by turn to turn or winding to winding shorts in the run windings.

Resistance of Pump Stator Windings Before and After Pump Failure:

	H to I Run Windings	H to J Start Windings	J to I	(HI + HJ-JI) This is twice the resistance of lead H
Before In-Pile Operation	0.788	3.911	4.581	0.118
After In-Pile Operation	0.542	3.635	4.065	0.112

3.3 Sequence of Events and Response of Safety Interlocks During Pump Failure.

3.3.1 Pump Power. After 1148.85 hours of operation, the pump power suddenly increased from a normal of 1100 watts to 1550 watts giving simultaneous pump power high warning and reactor fast set-back and scram. The pump power continued to rise. An attempt was made to re-establish normal operation by switching the pump control to "start" position. The power increased to 1700 watts. Five minutes later the power increased to 1800 watts, the current went to full scale, and the voltage dropped to zero. The pump power then dropped to zero cutting off the pump power and loop and pressurizer heaters. Two minutes later the Emergency Drain was started.

3.3.2 Loop Temperature Recorder. After the reactor was scrammed by high pump power, the loop temperature as indicated by the loop temperature recorder dropped sharply from 276°C to 268°C. This temperature decrease was probably caused by loss of fission and gamma heat. After dropping to 268°C the temperature increased rapidly to 285°C. This rapid increase in temperature was caused by loss of circulation.

3.3.3 Loop Temperature Controller. The main stream temperature indicated by the loop temperature controller dropped rapidly from 278°C to 270°C when the heater was cut off during pump failure. While trying to re-start the pump, the temperature rose to 273°C, but fell rapidly off scale when the drain was started.

3.3.4 Pressurizer Heater Controller. Immediately after pump failure, the pressurizer heater controller decreased from 278.1 to 276°C. After the heater was cut off, the temperature dropped to 259.4°C. Each time an effort was made to start the pump, an increase in temperature was noticed.

3.3.5 Pressurizer Temperature. After the pump failed, the pressurizer temperature dropped immediately from 294.2°C to 293.0°C. A slight increase to 293.8°C occurred when an attempt to start the pump was made. After this, the temperature dropped rapidly.

3.3.6 Pressurizer Pressure. After pump failure, the pressure oscillated between 1260 and 1300 psi and then dropped gradually to 1135 psi. At this point, an effort was made to re-start the pump and the pressure immediately increased to 1575 psi. Pressure then dropped rapidly when the Emergency Drain was started.

3.3.7 Core Temperature. After pump failure, the core temperature immediately dropped from 288.3°C to 269°C due to loss of fission and gamma heat. A very slight increase was noticed when efforts were made to re-start the pump, but afterward the temperature dropped rapidly.

4. Instruments, Thermocouples, and Pressure Cells.

4.1 Thermocouple Performance. The C-A and I-C thermocouples in the pressurizer were calibrated using the vapor pressure of water to determine the actual temperature. Results were 1176.3 psia vapor pressure (295.93°C actual temperature), 12.023 mv C-A (295.35°C), and 293.76°C I-C. At a higher temperature the results were 1249.7 psia (300.20°C actual temperature), 12.219 mv C-A (300.28°C), and 298.61°C I-C. The I-C pressurizer temperature, as indicated on the null-balance electronic potentiometer, shifted during the first few hours of operation and then held steady for the remainder of the run, about 0.5°C lower than the C-A pressurizer temperature.

Temperature behavior which may be of interest follows:

Pressurizer Heater, Control, TC #8, was 0.5°C lower with reactor on.
Pressurizer Heater Temperature, TC #6, was 1°C lower with reactor on.
Core Nose Temperature, TC #5, was 14.4°C higher with reactor on.
Core Nose Temperature, TC #2, was 18.2°C higher with reactor on.
Core Inlet Temperature, TC #4, was 1.0°C higher with reactor on.
Core Outlet Temperature, TC #3, was 2.3°C higher with reactor on.
Temperature drop across the pressurizer (TC #6-TC #12) was 0.8°C less with reactor on.

4.2 Pressure Cell. A Baldwin pressure transducer made of 410 stainless steel was used to measure the pressure in the pressurizer. This pressure cell was calibrated from atmospheric pressure to 2000 psig within ± 1.4 psi.

At 145 hours of circulation the pressure measuring system gave a false indication of low pressure. This low pressure reading, which lasted for several minutes, was the first of a number of similar irregularities which occurred during the run. Both high pressure and low pressure "pips" were observed. These false pressure indications always returned to within 5 psi of the original calibration as determined by pressure comparisons with P-5 and P-6 made during fuel and water expansions. At the end of the run a dead-weight calibration showed that the pressure transducer would not hold a consistent calibration.

5. Oxygen and Solution Balances

5.1 Loop Volume. The total loop volume at room temperature as measured at HB-2 by oxygen additions was 1659.9 cc including pressure lines and the pressure cell. A summary of the volumes of the system is given in Table III.

5.2 Fuel Loading. The loop was originally filled with 1178.0 g (1092.4 cc) of enriched fuel and 14.1 cc of water in lines 1, 4, 5, and the pressure cell.

5.3 Oxygen Pressure Factor. The average quantity of oxygen in the loop, at a given partial pressure of oxygen, was 15.3 cc (STP)/psi. The oxygen factor for the original fill was 16.57. Oxygen factors for the second and third additions were 13.95 and 13.86, respectively.

Oxygen used with L-2-19 was high purity oxygen supplied by Southern Oxygen

Company. Cylinder number and vendor's analysis follows:

Oxygen Addition	Cylinder	Per Cent "Purity"
L-2-19-1 depleted	310261	99.64
L-2-19-1 enriched	"	"
L-2-19-2	59221	99.65
L-2-19-3	42585	99.62

5.4 Metering Tank Volume. The volume of the metering tank was measured to be 213.92 cc. The metering tank volume is measured at the same time as the loop volume and may be used as a rough guide to the accuracy of the loop volume measurement. Other measurements are L-2-10, 211.0 cc; L-2-15, 213.4 cc; L-2-14, 209.2 cc; L-2-17, 209.24 cc; and L-2-21, 209.5 cc.

5.5 Fission Product Iodine. A sample of gas was taken from the Gas Hold-up Tank 20 days after draining and gas flushing loop L-2-19. An 8.9 cc sample was taken during the venting and analyzed the same day by S. A. Reynolds using a gamma spectrometer. The only component detected was Xe-133. Its disintegration rate was approximately $6x10^8$ dpm. The upper-limit value for I-131 was $6x10^4$ dpm.

Assuming that all the fission product gas was distributed uniformly in the gas hold-up tank, the calculated sample disintegration rates are $6.3x10^8$ dpm for the Xe-133 and $9.1x10^8$ dpm for the I-131.

It appears from this one measurement that the I-131 may not be the controlling health hazard when gas is vented from the hold-up tank after several weeks of storage. No information is available to explain why the I-131 was more than a factor of 10^4 low.

6. Auxiliary Components

6.1 Valve and Piping Failures. (Superficial examinations reported by A. R. Olsen).

6.1.1 Valves 1, 4, and 5. These valves were received on the loop from Y-12. They leaked across the seat and were replaced. (A.E. 30V-4001-DJ valves).

6.1.2 Valve 16. During fuel expansion L-2-19-7A, V-16 was found to be plugged. After lowering the loop temperature, this valve was replaced. Section 8.3 gives the replacement procedure used.

Examination by Olsen: "Before dismantling the stem of this valve could be turned although some pressure was required. After disassembly the stem was found to be broken in two places. The small tip was broken off at the seating line and the tip was stuck in the seat. The second fracture was between the guide and packing washers. Both fracture areas were covered with corrosion products. The guide washer was frozen to the section of stem while the packing washer was covered with corrosion products on all surfaces. Several small pieces of the stem fell out of the valve body on disassembly. The tip was examined under the microscope and found to be badly corroded. The valve seat was partially covered with dried white crystals and some rust like scale."

Valve History: Ser. No. 563, A.E. 30 V-4001-DJ, processed 4-21-56, 420 SS stem heat No. 17632, 316 SS body, operated more than 225 times.

6.1.3 Valve 17. This valve was found plugged during a sample check-out before radiation began.

Examination by Olsen: "The valve stem turned easily before disassembly. After disassembly the extreme tip of the stem was found to be missing. This tip was not recovered. The fracture line appeared to match the seating ring on the stem. The fracture surface was covered with rust like material. The seating area of the valve appeared to be clean although some scale was seen down in the port. The stem proper was partially covered with a rust like scale up to the teflon packing, as was the guide washer. There were a number of flakes of teflon which had extruded past the packing washer."

Valve History: Ser. No. 564, identical to V-16, but operated more than 1000 times.

6.1.4 Valve 20. At the end of the run this valve was found to leak across the seat.

Examination by Olsen: "The valve stem turned easily before disassembly. After disassembly the valve stem was found to be intact but severely corroded, scaled and cracked. The valve seat was covered over approximately 1/4 of the area with a rust like scale. It is strongly recommended that this stem which appears to be on the verge of complete failure be examined metallographically."

Valve History: Ser. No. 557, identical to valves 16 and 17, but operated more than 225 times.

6.1.5 Valve 23. At the end of the run this valve was found to leak across the seat.

Examination by Olsen: "The valve stem turned easily before disassembly. After disassembly the valve stem was found to be cleanly fractured at the seating ring. The fracture was clean, apparently a fresh fracture. Some rust like scale was found on the stem up to the teflon packing. Machine marks were still visible on the stem. The valve seat was shiny and clean with some slight evidence of galling."

Valve History: Ser. No. 576, identical to valves 16, 17 and 20, but operated more than 80 times.

6.1.6 Valve 40. At the end of the run this valve was found to leak across the seat.

Examination by Olsen: "The stem turned freely in the valve before disassembly. After disassembly the stem and washers were all bright and clean. There was some evidence of moderate galling in the seating area on both the stem and the valve seat. As with all of the valves examined there were some flakes of teflon which had extruded past the packing washer."

Valve History: Ser. No. 771, A.E. 30V-4001-DJ, processed 2-14-57, 420 SS steam heat No. 3845, 316 SS body, operated more than 140 times.

6.1.7 Valves 38, 39, 127, 511, 512. These valves were found to leak across the seat at the end of run L-2-19. They are similar to the above valves, but were not examined. All were in gas-phase lines and were operated from 50 to 285 times.

6.1.8 Sample Connector #1. The sample system plugged in the vicinity of S.C. #1 several times during run L-2-19. The first was before irradiation at which time a rusty deposit was found where the line from V-36 joins the sample connector. This line was replaced.

The sample system plugged again during rinsing after sample L-2-19-5. The plug was opened by back-flushing. A similar plug was encountered on the next four samples. The sample connector and rinse capillary were replaced when the loop temperature was lowered for replacement of V-16.

6.2 Weigh Tank. The amount of dilute fuel removed from the soup weigh tank according to weight data was 9920 grams. The net amount of liquid in the weigh tank at the time of soup transfer was 9377 cc by book inventory.

6.3 Small Line Filters. To help prevent the spread of corrosion products and other solid particles, small filters were installed below valves 101 and 105 in the wash tank system. These filters are Autoclave Engineers' 30-F-4400 stainless steel filters using filter discs of sintered 304 SS with 65 and 35 micron pores.

7. Procedure Changes

7.1 Loop Sample. The loop sample procedure was changed to permit flushing of all sample lines with solution purges from the loop. This is done by draining the two purges immediately preceding the sample from the standard drain volume to the soup weigh tank through valves 27, 36, 20, 23, and 14. The draining is done at low pressure. This procedure has reduced dilution of loop samples from 15% to 4% based on the amount of tracer ion from the rinse water found in the sample. L-2-19 was the first loop to use potassium sulphate as the rinse water tracer.

8. Special Operations

8.1 Preparation.

8.1.1 Pre-Run Check-Out. Pre-run testing included measuring volumes of the capillary lines, measuring the loop and oxygen metering tank volumes by oxygen additions, steam calibration of the pressurizer thermocouples against the pressurizer pressure cell, and a pressurizer flow check. Pre-run testing of auxiliary equipment included the usual valve leak testing, instrument cleaning and calibration, and weigh tank calibration.

8.1.2 Depleted Uranium Run. A run of 67.9 hours was made using depleted uranium and natural lithium in a mock-up fuel of the same composition as the in-pile fuel. The load was 1178.0 grams of fuel plus 14.1 cc of water in lines 1, 4, 5, and the pressure cell. High purity oxygen from Southern cylinder #310261 was used. Since six solution samples were removed from the loop without replacement of solution, accurate oxygen consumption data were not obtained. A jacket calibration and pressurizer flow check were made.

8.1.3 Enriched Fuel Run. The loop was operated out of pile with enriched fuel for 59.35 hours.

8.2 Installation. Insertion of the loop was accomplished without difficulty.

8.3 Replacement of V-16 and S.C. #1.

0837	Started lowering loop and pressurizer temperatures to 160 and 180°C, respectively.
0915	Closed V-6 and opened V-211. Started removing lead shielding from front of L.E.C.
0932	Shielding removed. Maximum radiation through cover plates 43 mr/hr.
0937	Opened V-721 (hot drain to L.E.C.).
0942	Removed cover plates. Maximum radiation near V-16 was 230 mr/hr.
0959	Removed V-16. Valve was 20 mr/hr.
1006	V-16 replaced.
1127	V-16 leak checked.
1128	Removed cover plates in preparation for replacing sample connector. Maximum radiation inside L.E.C. 1 R/hr.
1445	Sample connector replaced and leak checked.
1449	Cover plates replaced.
1500	Closed V-211 and V-721. Opened V-6.
1607	Lead shielding replaced.
1630	Loop returned to normal operating conditions.

8.4 Removal from HB-2. No difficulties were encountered during removal of L-2-19. Approximately 24 hours were required for draining and rinsing the loop, disconnection in the small equipment chamber, removal of the loop package, separation of the loop and shielding plug, and insertion of shielding plugs into HB-2.

As with previous loops, the maximum radiation dosage received by the operators was less than laboratory tolerance (60 mr/day).

Table I

Typical Operating Data L-2-19

Hours Circulation on Enriched Fuel	°C Average with Reactor at Zero Power			°C Average with Reactor at 3 Mw		
	536-568	704-720	1040-1064	456-536	776-856	1072-1148
Press. Htr. Temp. Cont.	279.62	278.89	279.61	278.61	278.42	278.58
Press. Htr. Temp. Pt #7	288.03	287.42	287.86	287.24	287.09	287.54
Press. Temp. I.C.	294.03	294.00	293.81	294.22	294.08	294.11
Press. Temp. C.A.	294.67	294.61	294.57	294.68	294.67	294.64
Press. Jacket Temp. Pt. #6	291.11	291.61	291.03	291.17	291.41	291.28
Press. Outlet Temp. Pt. #4	285.26	285.00	284.86	285.37	285.25	285.35
Loop Temp. Core Outlet Pt. #2	274.08	274.00	274.00	276.50	276.47	276.56
Loop Temp. Cont., Thermowell	277.98	277.99	278.00	278.06	278.03	278.02
Loop Temp. Rec. after Heater	277.63	278.07	277.42	276.82	276.51	276.74
Loop Temp.Core Inlet Pt. #5	272.61	272.50	272.50	273.60	273.60	273.95
Core Temp. Rec.	273.09	273.50	273.73	287.66	287.81	288.21
Core Temp. Pt. #1	268.16	268.00	268.64	286.19	286.34	286.86
Pump Temperature	51.17	50.75	50.41	51.04	50.45	50.02
Pump Power	1096.65	1099.15	1102.87	1103.72	1099.00	1100.37

Pump Current - 0-95 Hours Circulation -15.8 amps   95-1148 Hours Circulation - 16.3 amps
Plug Coolant Flow ~0.95 GPM Process H_2O
Pump & liner Coolant flow 1.94 GPM demineralized H_2O
Loop Coolant - 2.07 SCFM Process Air plus 2.00 cc/min water
Equipment Chamber flow 0.48 SCFM filtered air
Equipment Chamber Vacuum 3.1 in. Hg.
Loop Container Flow (RD-5) 0.32 SCFM Process air
Continuous Air Bleed 0.2 SCFM Process air
Loop Container Pressure 50 psig

Table II

Radiation Intensities L-2-19

RD-5	Reactor at Zero Power Reactor at 3 Mw	0.9×10^4 cpm 2.2×10^4 cpm

	Radiation Detector Readings During Sample Purge; Reactor at 3 Mw	24 hours after sample and fuel expansion
RD-1	86 R/hr (after 30 sec.)	104 mr/hr
RD-2	116 R/hr (after 5 min.)	200 mr/hr
RD-3	380 R/hr (after 56 min.)	1.3 R/hr
RD-4	118 R/hr (after 28 min.)	330 mr/hr
RD-5	3.5×10^4 cpm	2.3×10^4 cpm

Radiation Leakage During Sample in Front of S.E.C. During Sample Purges:

Front of Paraffin Shield:	140 mrep/hr	Boron Cutie Pie
	90 mrep/hr	Plain Cutie Pie
	18 mrep/hr	Fast Neutrons
Below Paraffin Shield:	140 mrep/hr	Boron Cutie Pie
	75 mrep/hr	Plain Cutie Pie
	220 mrep/hr	Fast Neutrons

Large Equipment Chamber During Valve Replacement:

With cover plates on:	43 mr/hr maximum, at sample connector
Cover plates off:	230 mr/hr at V-16; 1 R/hr maximum
Valve 16:	20 mr/hr removed.

Table III

Summary of Loop Volumes - L-2-19

	Volume at 25°C cc	Operating Conditions °C	Volume cc
Pump	160	75	160.4
Main Stream	781.3	279	791.7
Pressurizer Inlet Line	18.7	287	19.1
Pressurizer Outlet Line	2.1	295	2.1
Pressurizer	675	295	684.5
Liquid		(295)	(432.5)
Vapor		(295)	(252.0)
Circulating Loop Total	1637.1		1657.8
Pressure Cell	7.5	25	7.5
Line 1	1.0	25	1.0
2	5.0	25	5.0
3	4.7	25	4.7
4	3.7	25	3.7
5	0.9	25	0.9
Lines & Pressure Cell	22.8		22.8
Total Loop Volume	1659.9		1680.6

(Core volume measured at 25°C = 239.0 cc)

Table IV

Electric Heater Power With Reactor At 3 Mw, Watts

Test No.	Hours Circulation	Loop Heater		Pressurizer Heater		Pressurizer Jacket Heater		Total	
1	204.2	275.4	(2528.1)	778.8	(48.0)	286.9	(-23.4)	1341.2	(2587.5)
2	370.0	354.5	(2473.1)	759.5	(44.5)	287.7	(-21.1)	1401.7	(2529.3)
3	443.2	366.1	(2516.1)	741.9	(79.1)	290.2	(-10.1)	1398.2	(2585.4)
4	535.7	348.3	(2516.9)	744.3	(77.3)	285.2	(-10.1)	1377.7	(2584.2)
5	706.7	319.6	(2547.0)	733.7	(75.6)	293.3	(-12.4)	1346.6	(2610.1)
6	782.9	450.6	(2426.7)	737.8	(63.3)	291.4	(- 8.9)	1479.7	(2481.2)
7	877.0	350.6	(2504.8)	722.8	(66.8)	289.9	(- 8.1)	1363.3	(2563.7)
8	1039.7	383.2	(2477.3)	717.9	(61.5)	287.7	(-11.0)	1388.8	(2529.8)

Numbers in parenthesis are power changes upon reactor shut-down, watts.

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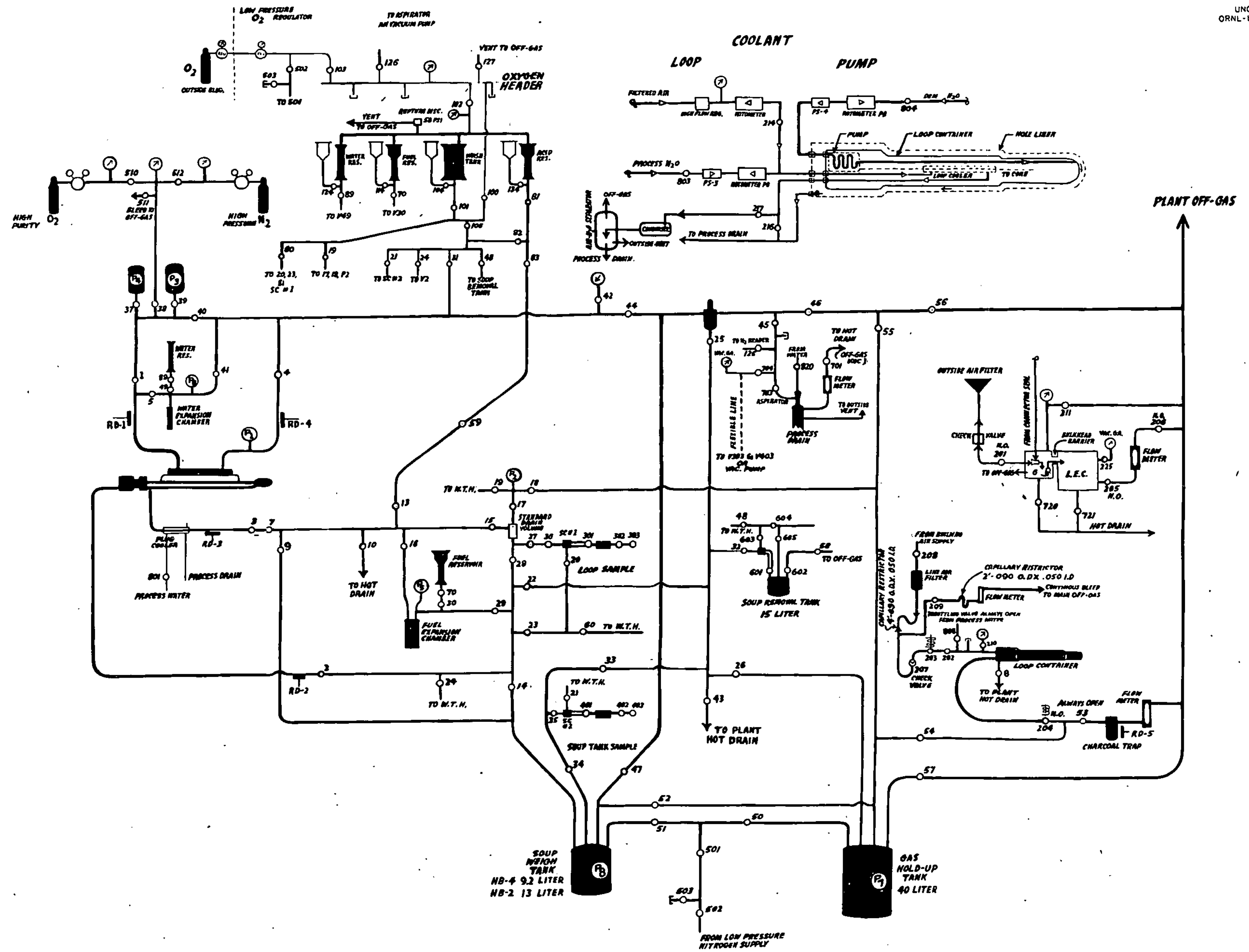

NOTE: ALL VALVES ARE NORMALLY CLOSED UNLESS OTHERWISE SPECIFIED

Figure 1

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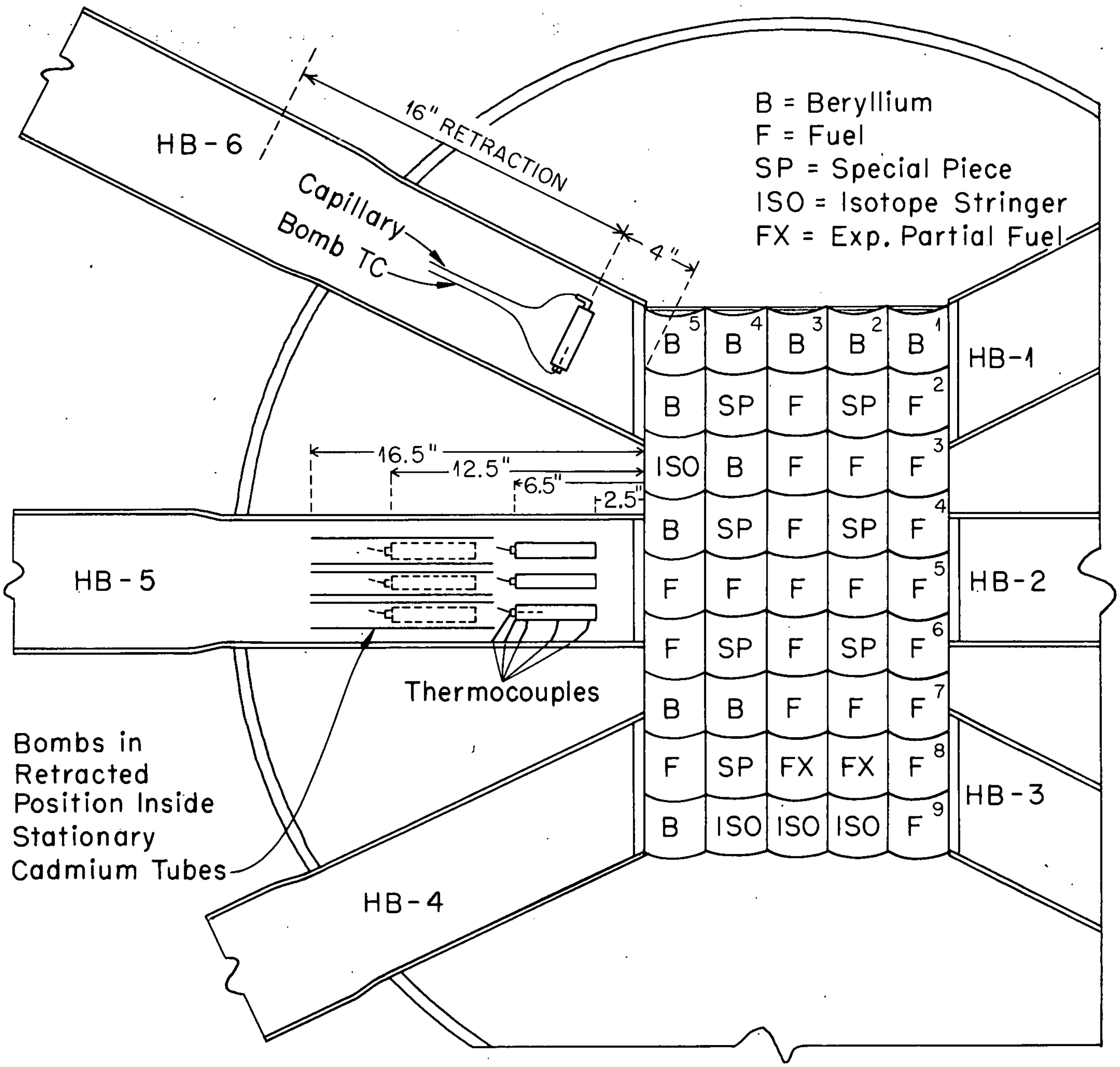

LITR ROCKING AUTOCLAVE CORROSION TEST FACILITIES

Figure 2

REFERENCES

1) All plus or minus values represent the 90% confidence limit of the mean based on the range of the data.

2) F. J. Walter, HRP In-Pile Corrosion Test Loops, Operation of In-Pile Loop L-2-17, August 30, 1957, CF Memo 57-8-112.

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75. E. M. Shank
76. M. D. Silverman
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79. R. W. Stoughton
80. A. Shor
81. J. A. Swartout
82. E. H. Taylor
83. D. G. Thomas
84. D. S. Toomb
85. W. C. Ulrich
86. W. E. Unger
87. R. Van Winkle
88. A. M. Weinberg
89. K. W. West
90. S. H. Wheeler
91. J. C. Wilson
92. C. E. Winters
93. F. C. Zapp
94-95. REED Library
96-97. Central Research Library
98-99. Document Reference Library
100-115. Laboratory Records
116. ORNL-RC

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117. F. C. Moesel, AEC, Washington
118-124. Westinghouse PAR Project (7)
125-139. TISE-AEC (15 for CF Memos)