



ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

QUARTERLY PROGRESS REPORT NO. 15

For Quarter Ending January 15, 1969

**prepared by
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**prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION**

**NASA Lewis Research Center
Contract NAS 3-6474
Robert L. Davies, Project Manager
Materials Section**

**NUCLEAR SYSTEMS PROGRAMS
SPACE SYSTEMS
GENERAL  ELECTRIC
CINCINNATI, OHIO 45215**

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NUCLEAR SYSTEMS PROGRAMS
MISSILE AND SPACE DIVISION
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Cincinnati, Ohio 45215

prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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FOREWORD

The work described herein is sponsored by the National Aeronautics and Space Administration under Contract NAS 3-6474. R. L. Davies of NASA - Lewis Research Center is the NASA Technical Manager.

The program is being administered for the General Electric Company by E. E. Hoffman, and R. W. Harrison is acting as the Program Manager. J. Holowach, the Project Engineer, is responsible for the loop design, facilities procurement, and test operations. Personnel making major contributions to the program during the current reporting period include:

Alkali Metal Purification and Handling - Dr. R. B. Hand,
L. E. Dotson, and H. Bradley.

ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

I. INTRODUCTION

This report covers the period from October 15, 1968 to January 15, 1969. The primary task of this program is to fabricate, operate for 10,000 hours and evaluate a T-111 Rankine System Corrosion Test Loop. Materials for evaluation include the containment alloy, T-111 (Ta-8W-2Hf) and the turbine candidate materials Mo-TZC and Cb-132M which are located in the turbine simulator of the two-phase potassium circuit of the system. The loop design will be similar to the Cb-1Zr Rankine System Corrosion Test Loop; a two-phase, forced convection, potassium corrosion test loop which has been tested under Contract NAS 3-2547.⁽¹⁾ Lithium is being heated by direct resistance in a primary loop. Heat rejection for condensation in the secondary potassium loop is being accomplished by radiation in a high vacuum environment to the water cooled chamber. The compatibility of the selected materials will be evaluated at conditions representative of space electric power system operating conditions, namely:

- a. Boiling temperature, 2050°F
- b. Superheat temperature, 2150°F
- c. Condensing temperature, 1400°F
- d. Subcooling temperature, 1000°F
- e. Mass flow rate, 40 lb/hr
- f. Boiler exit vapor velocity, 50 ft/sec

⁽¹⁾ Hoffman, E. E. and Holowach, J., Cb-1Zr Rankine System Corrosion Test Loop, Potassium Corrosion Test Loop Development Topical Report No. 67, R66SD3016, General Electric Company, Cincinnati, Ohio, May 1, 1968.

- g. Average heat flux in plug (0-18 inches), 240,000 Btu/hr ft²
- h. Average heat flux in boiler (0-250 inches), 23,000 Btu/hr ft²

In addition to the primary program task cited above the program also includes capsule testing to evaluate advanced tantalum alloys of the ASTAR 811 type (Ta-8W-1Re-1Hf) in both potassium and lithium.

Also included in the program is the fabrication, 5000-hour operation and evaluation of a 2600°F, high flow velocity, pumped lithium loop designed to evaluate the compatibility of the ASTAR 811 type alloys, T-111, T-222, and the tungsten alloy, W-25Re-30Mo at conditions simulating an out-of-pile thermionic reactor system.

II. SUMMARY

Reinstrumentation and reinsulation of the T-111 Corrosion Loop was completed, and the vacuum chamber was sealed and evacuated.

Purification of the alkali metals was completed. Both loop circuits were cleaned by alkali metal flushing and filled with charges for test operation.

The alkali metals are being circulated and the loop is being brought to temperature.

III. PROGRAM STATUS

A. T-111 RANKINE SYSTEM CORROSION TEST LOOP

1. Reinstrumentation of the Boiler

Reinstrumentation and reinsulation of the boiler commenced following leak checking of the loop after postweld annealing of the installation welds.⁽¹⁾ Over thirty W-3Re/W-25Re thermocouples required replacement. The thermocouple installation has been completed, and insulation with Cb-1Zr dimpled foil is in progress.

A heater was installed on the bottom of the metering valve as shown in Figure 1. The heater shown before assembly in Figure 2 was originally designed for the vapor nucleator installed in the Cb-1Zr Rankine System Corrosion Test Loop, NASA Contract NAS 3-2547.⁽²⁾

2. Test Facility Operations

Reinstrumentation and reinsulation were completed, and the chamber was closed on November 15, 1968. The chamber was evacuated with the turbo-molecular pump, and mass spectrometer leak checking was performed. No leaks were found, and ion pumping was initiated. The bakeout heaters were then turned on, and on November 21, 1968, a pressure of 4×10^{-7} torr was recorded with the chamber at 500°F. At that time a final helium leak

(1) Advanced Refractory Alloy Corrosion Loop Program, Quarterly Progress Report No. 14 for Period Ending October 15, 1968, NASA Contract NAS 3-6474.

(2) Hoffman, E.E., Holowach, J., "Cb-1Zr Rankine System Corrosion Test Loop," Topical Report No. 7 NASA Contract NAS 3-2547, General Electric Company Report R67SD3016.

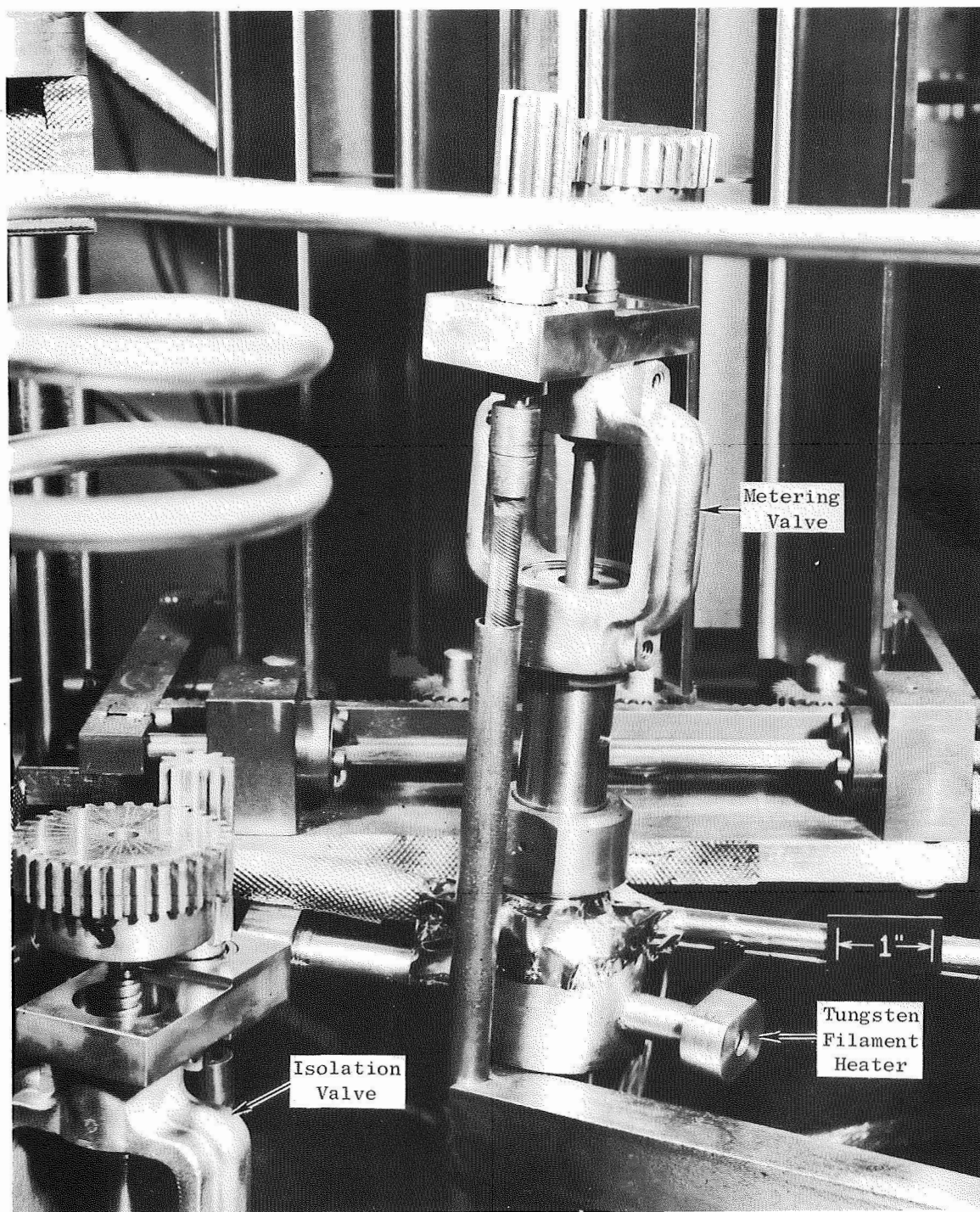


Figure 1. Tungsten Filament Heater Installed on Metering Valve of T-111 Rankine System Corrosion Test Loop. (P68-11-15A)

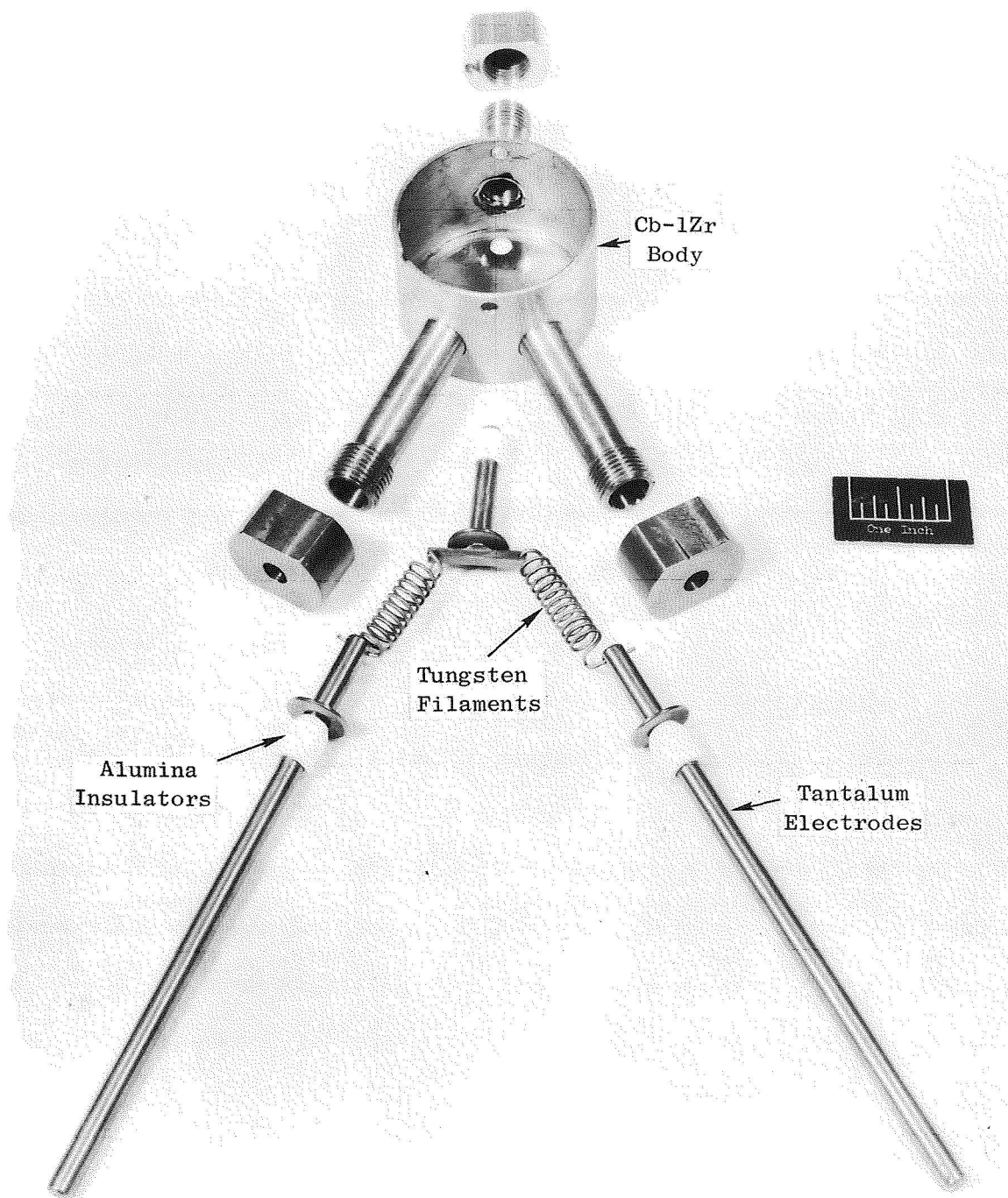


Figure 2. Tungsten Filament Heater Before Assembly. (C65012245)

check was performed between the potassium and lithium circuits with the loop at 375°F, and no leaks were found. Bakeout will continue until alkali metal flushing of the loop circuits is completed.

3. Alkali Metal Purification

Additional analytical results were obtained on the lithium received from Foote Mineral Company, Exton, Pennsylvania. A sample was previously taken during the transfer of 28 pounds of lithium to the hot trap.⁽³⁾ The analysis of this sample (No. 2039) is compared with that for the hot trapped lithium (No. 2067) in Table I. The lithium was hot trapped for 200 hours at 1500°F.

Before distillation was initiated, the vacuum manifold on the lithium purification system was disconnected from the receiver and cleaned. As described previously,⁽³⁾ lithium had been inadvertently blown back into the vacuum manifold during a sample operation. The manifold was reinstalled and subsequently twenty pounds of hot trapped lithium was distilled at 1230°F. The sample (No. 2081) taken from the receiver was analyzed and had a high nitrogen concentration as shown in Table I. Examination of the lithium purification facility indicated the Granville-Phillips high vacuum valves, previously cleaned of lithium, had leaks in the bellows. It is believed that these leaks were primarily responsible for the nitrogen contamination of the lithium.

The valves were replaced with new valves and the system leak checked. The contaminated lithium was returned to the hot trap and hot trapping

(3) Advanced Refractory Alloy Corrosion Loop Program, Quarterly Progress Report No. 14 for Period Ending October 15, 1968, NASA Contract NAS 3-6474.

TABLE I

ANALYSIS OF LITHIUM

Sample No.	Concentration, ppm		
	As Received ^(a)	Hot Trapped ^(b)	Distilled ^(c)
Element	2039	2067	2081
O	95		30
C	75	69	49
N	278	21,23,33	295
Ag	< 5	< 5	< 5
Al	< 5	< 5	5
B	< 50	< 50	< 25
Ba	< 50	< 50	< 75
Be	< 5	< 5	< 5
Ca	< 5	5	25
Cb	< 25	< 25	< 25
Co	< 5	< 5	< 5
Cr	< 5	< 5	< 5
Cu	5	50	5
Fe	< 5	< 5	5
Mg	5	< 5	5
Mn	< 5	< 5	< 5
Mo	< 5	< 5	< 5
Na	< 75	< 50	< 75
Ni	< 5	< 5	< 5
Pb	< 50	< 50	< 50
Si	5	5	5
Sn	< 25	< 25	< 25
Sr	25	25	25
Ti	< 25	< 25	< 25
V	< 25	< 25	< 25
Zr	< 25	< 25	< 25

(a) From shipping container received from Foote Mineral Co.,
Exton, Pa.

(b) 28 pounds hot trapped for 200 hours at 1500°F

(c) 20 pounds distilled at 1230°F subsequently found to be
contaminated by leaks in the vacuum manifold valves
during distillation.

initiated. The contaminated lithium from the still receiver was hot trapped 305 hours at 1500-1550°F and sampled by flushing two pounds of lithium through the sample tube. Subsequent analysis indicated 15 ppm nitrogen. Distillation was initiated, and samples were taken after five and twelve pounds of lithium distillate was obtained in the still receiver. The respective nitrogen analyses, 380 ppm and 146 ppm, indicated that the high nitrogen concentration in the lithium in the still was being diluted by the distillate but not sufficiently to reduce the nitrogen level to an acceptable value. The lithium in the still receiver was therefore returned to the hot trap for further purification. The lithium was hot trapped for 200 hours at 1500-1550°F and sampled. A nitrogen concentration of 13 ppm was obtained, and distilling was again resumed. Twenty-two pounds of lithium was distilled. The analysis of a sample of this material is presented in Table II. The use of the purified lithium for flushing and filling the loop was approved by the NASA Program Manager, and preparations were made to connect the lithium purification system to the loop transfer system.

4. Lithium-Potassium Solubility Study

The mutual solubilities of lithium and potassium are being determined over the 600°F to 1200°F temperature range in the apparatus described previously.⁽⁴⁾ The data obtained to date are presented in Table III. These data were obtained by equilibrating equal volumes of lithium and potassium at constant temperature for at least 16 hours. The data shown

⁽⁴⁾ Advanced Refractory Alloy Corrosion Loop Program, Quarterly Progress Report No. 14 for Period Ending October 15, 1968, NASA Contract NAS 3-6474.

TABLE II

ANALYSIS OF THE DISTILLED LITHIUM TO BE USED IN THE
T-111 RANKINE SYSTEM CORROSION TEST LOOP^(a)

<u>Element</u>	<u>Concentration, ppm</u>
N	27,33
O	31
C	46
Ag	< 5
Al	5
B	< 50
Ba	< 75
Be	< 5
Bi	< 25
Ca	25
Cb	< 25
Co	< 5
Cr	< 5
Cu	< 5
Fe	< 5
Mg	5
Mn	< 5
Mo	< 5
Na	< 10
Ni	< 5
Pb	< 50
Si	< 5
Sn	< 25
Sr	25
Ti	< 25
V	< 25
Zr	< 25

(a) Analysis obtained on a sample removed from the still receiver following hot trapping and vacuum distilling.

TABLE III

MUTUAL SOLUBILITIES OF POTASSIUM AND LITHIUM

<u>Temperature</u> <u>°F</u>	<u>Wt. % K^(a)</u> <u>in Li</u>	<u>Wt. % Li^(a)</u> <u>in K</u>	<u>Run No.</u>
610	0.10	0.23	4
610	(b)	0.23	5
800	0.19	0.36	1
800	(b)	0.37	2
1000	0.70	1.12	3
1230	2.60	-	6

(a) Determined by spectrophotometric analysis

(b) Sample lost in preparation

for 800°F represent times of 16 hours and 24 hours with no change in solubility indicated. No data was obtained for the solubility of lithium in potassium at 1200°F because the valves used did not function properly after prolonged exposure to this temperature. The temperatures were measured with calibrated chromel/alumel thermocouples accurate to $\pm 10^\circ\text{F}$.

Details on the apparatus and evaluation of the data will be reported at a later date.

5. Alkali Metal Flushing of the Loop

As previously reported⁽⁵⁾ examination of the alkali metals drained from the loop indicated lithium in the potassium and potassium in the lithium as a result of the boiler leak. Particulate matter was also found in the potassium. The particles and contaminated alkali metals will be removed from the loop by repeatedly flushing the circuits with pure alkali metals.

The apparatus required for filling and flushing the potassium loop circuit is shown in Figure 3. The transfer system was modified for flushing operations by inserting a dump line between valves FF, KK, and the disposal tank. This permits the transfer of flush charges of potassium directly to the disposal tank without contaminating the fill system upstream from valve FF. In addition, the dump line is connected to a small potassium still where flush potassium charges can be diverted for subsequent distillation and analysis of the residue for lithium concentration and particulate matter.

⁽⁵⁾ Advanced Refractory Alloy Corrosion Loop Program, Quarterly Progress Report No. 12 for Period Ending April 15, 1968, NASA Contract NAS 3-6474, NASA-CR-72452.

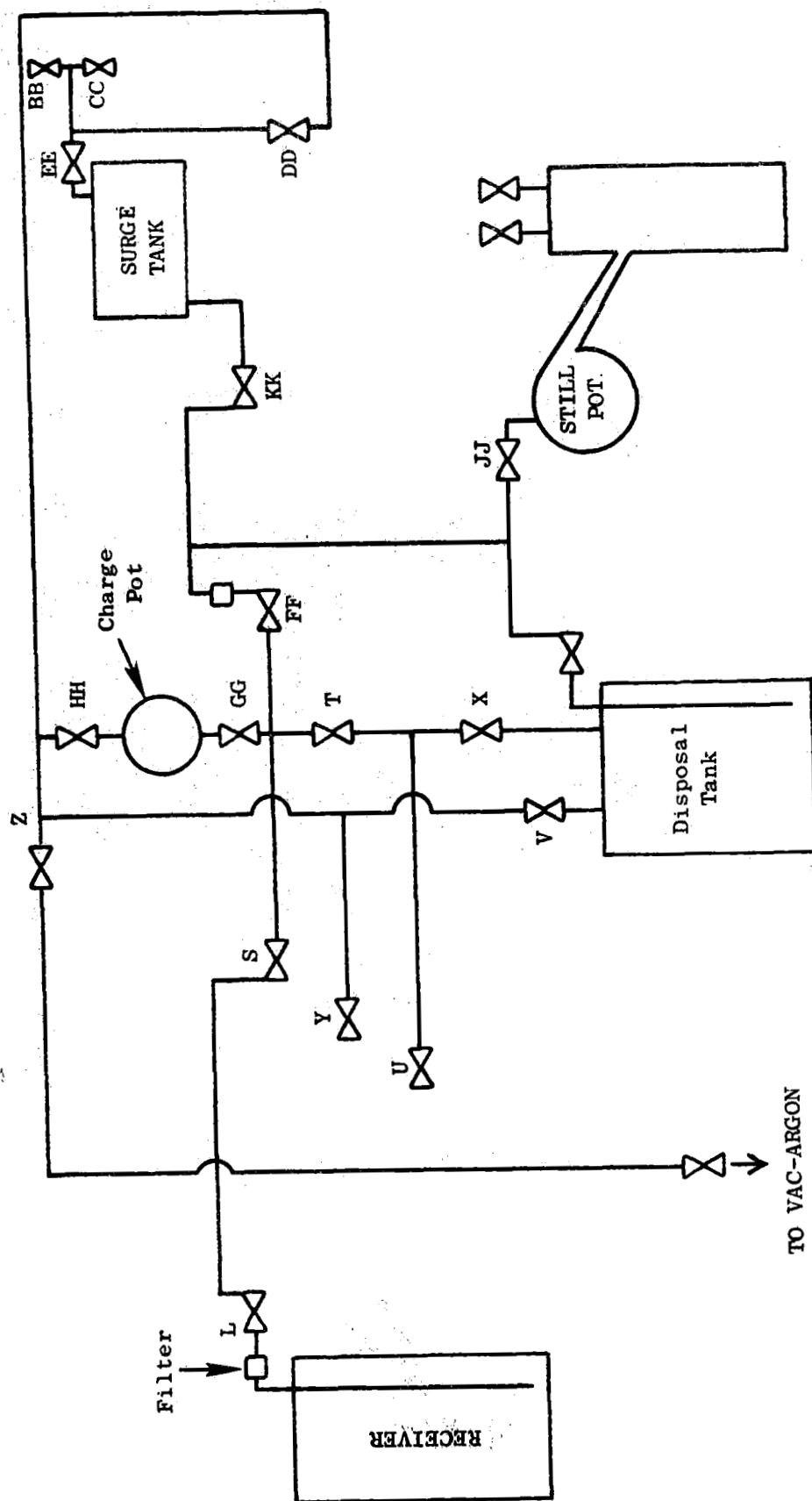


Figure 3. Potassium Transfer System for Filling and Flushing the T-111 Corrosion Test Loop.

The lithium transfer system was modified similarly; however, after the lithium circuit is flushed, a sampler is attached directly to valve KK on the lithium side of the transfer system. The sample taken is then analyzed to determine the potassium concentration in the lithium.

The entire transfer system was baked out at temperatures up to 500°F, and on December 12, 1968, the pressure rise rate was measured to be less than 0.2 micron-liters per minute.

The potassium side of the transfer system was filled, and the potassium was dumped into the disposal tank to flush the system. The system was refilled with potassium and a sample obtained. The analysis of this sample, shown in Table IV, was acceptable and the use of this potassium was approved by the NASA Program Manager. The potassium surge tank was filled with an 1800 cc charge on December 13, 1968, and flushing operations on the potassium loop circuit were initiated.

The secondary circuit was flushed with four 1800 cc charges of potassium while the final purification of lithium was in progress. The loop temperatures and pressures obtained during these flushing operations are presented in Table V.

The first charge was used to establish circulation and, after the flow was reversed to agitate particles,⁽⁶⁾ was quickly dumped into the disposal tank. Flow indications during circulation indicated some plugging of the metering valve with particles, and the valve was actuated to its full open position. The observed pressure drop across the valve, shown in Table V, confirms particulate matter plugging in the valve. Improved flow indications were obtained during the circulations of the second and third potassium charges with some observed decrease in the pressure drop across the valve. Both the second and third charges were circulated four times before removal from the surge tank. The second charge was dumped into the disposal tank. The third charge was dumped into the still for subsequent analysis. The potassium was distilled off

(6) Advanced Refractory Alloy Corrosion Loop Program, Quarterly Progress Report No. 12 for Period Ending April 15, 1968, NASA Contract NAS 3-6474, NASA-CR-72452.

TABLE IV

ANALYSIS OF THE POTASSIUM USED IN FLUSHING
THE T-111 CORROSION TEST LOOP^(a)

<u>Element</u>	<u>Concentration, ppm</u>
O	7,14
C	51
Ag	< 2
Al	< 2
B	< 30
Ba	< 10
Be	< 2
Ca	< 2
Cb	< 10
Co	< 2
Cr	< 2
Cu	< 2
Fe	< 2
Mg	< 2
Mn	< 2
Mo	< 2
Na	< 20
Ni	< 2
Pb	< 20
Si	< 2
Sn	< 20
Sr	< 2
Ti	< 10
V	< 20
Zr	< 10

(a) Analysis obtained on a sample removed from the transfer system before transferring the potassium into the loop surge tank.

TABLE V

TEMPERATURES AND PRESSURES RECORDED DURING FLUSHING OF THE T-111 CORROSION LOOP SECONDARY CIRCUIT
WITH POTASSIUM BEFORE FILLING THE PRIMARY CIRCUIT WITH LITHIUM^(a)

Circulation (b)	First Charge		Second Charge				Third Charge			
	1 (c)		1	2	3	4	1	2	3	4
Maximum Chamber Pressure, torr	4.7×10^{-6}		1×10^{-6}	2×10^{-6}	7×10^{-7}	4×10^{-7}	4×10^{-7}	1.5×10^{-7}	2×10^{-7}	1.5×10^{-7}
Loop Temperatures, °F (c)										
Boiler	785		787	905	938	886	910	886	900	880
Potassium Preheater Inlet	1025		689	759	762	743	759	748	702	550
Potassium Preheater Exit	785		897	970	1025	961	975	954	971	960
Pump Discharge	970		839	900	919	907	911	905	920	905
Metering Valve	1040		895	958	912	900	910	900	914	900
Subcooler	875		674	733	698	713	691	670	693	670
Pressures, psia										
Metering Valve Inlet	82 (d)	4 (c) 70 (d)	111	104	100	100	101	103	101	101
Metering Valve Exit	53	13 (c) 38	54	53	53	51	54	54	54	54
ΔP (valve full open)	29	9 (c) 42	57	50	47	49	47	49	46	46
Surge Tank Temperature, °F										
Before	320									
After Dumping	410		77	72	62	76	73	86	84	91
ΔT	90									

(a) Heat supplied by the potassium preheater.

(b) The second and third charges were circulated and dumped into the surge tank four times. After the fourth circulation the second charge was dumped through the surge tank into the disposal tank and the third charge was dumped into the still for subsequent analysis.

(c) Reverse flow

(d) Forward flow. The flow direction of the potassium was changed to agitate particulate matter.

and the 39 mg of residue analyzed for lithium. A concentration of approximately 30 ppm lithium in the potassium was determined. A very small amount of particulate matter was found in the still. The still was cleaned, welded together and reinstalled in the transfer system as shown previously in Figure 3.

On December 26, 1968 bakeout of the lithium transfer system and purification system was completed and a pressure rise rate of less than 0.4 micron liters/minute was obtained. The lithium transfer system was subsequently flushed by filling and draining the lithium into the disposal tank. The transfer system was refilled and the lithium sampled. The analysis, presented in Table VI, was acceptable to the NASA Program Manager and the loop surge tank was filled with lithium.

The first lithium charge was discarded into the disposal tank, and the surge tank was refilled with a 1600 cc charge of lithium. The lithium was circulated in the primary circuit and used to heat a fourth charge of potassium to higher temperature than previously possible with only potassium in the loop. The potassium charge was circulated and dumped into the surge tank three times. The temperatures and pressures recorded just before the final dump into the small potassium still, are presented in Table VII.

The potassium was distilled off and the 28 mg residue analyzed for lithium. A concentration of approximately 20 ppm lithium in the potassium was determined. No particulate matter was found in the still. The data indicated sufficient flushing of the potassium circuit had been performed.

TABLE VI

ANALYSIS OF THE LITHIUM USED IN FLUSHING THE
T-111 CORROSION TEST LOOP^(a)

<u>Element</u>	<u>Concentration, ppm</u>
N	34
O	20
C	46
Ag	< 5
Al	25
B	< 50
Ba	< 50
Be	< 5
Bi	< 25
Ca	25
Cb	< 25
Co	< 5
Cr	< 5
Cu	5
Fe	< 5
Mg	5
Mn	< 5
Mo	5
Na	< 50
Ni	5
Pb	< 50
Si	25
Sn	< 25
Sr	25
Ti	< 25
V	< 25
Zr	< 25

(a) Analysis obtained on a sample removed from the still receiver following hot trapping and vacuum distilling.

TABLE VII

TEMPERATURES AND PRESSURES RECORDED DURING FLUSHING
OF THE T-111 CORROSION LOOP SECONDARY CIRCUIT WITH
POTASSIUM USING LITHIUM IN THE PRIMARY CIRCUIT TO SUPPLY THE HEAT^(a)

<u>Maximum Chamber Pressure, torr</u>	5×10^{-8}
<u>Loop Temperatures, °F</u>	
Primary Heater	1420
Boiler	1393
Potassium Preheater, Inlet	660
Potassium Preheater, Exit	525
Pump Discharge	775
Metering Valve	770
Subcooler	675
1st Stage Turbine Simulator	1384
Stages 2-10	1260
<u>Pressures, PSIA</u>	
Metering Valve, Inlet	36
Metering Valve, Exit	26
ΔP (valve full open)	10
<u>Surge Tank Temperature, °F</u>	
Before	476
After Dumping	528
ΔT	52

(a) Data for the third circulation. The charge was dumped into the surge tank and returned to the loop twice before final dumping into the still for subsequent analysis.

The lithium charge in the primary circuit was dumped into the disposal tank and the loop filled with a third charge of lithium (1600 cc). The lithium sampler was installed at valve KK and, after circulating the lithium in the primary, a sample was taken. Analytical results indicated a concentration of 90 ppm potassium in the lithium. Since the solubility limit for potassium in lithium at the sampling temperature (580°F) was 1000 ppm and the potassium concentration in the distilled lithium was 55 ppm, further flushing of the lithium primary circuit was believed unnecessary.

6. Filling the Loop With Alkali Metals for Operation

The secondary loop was filled with 2300 cc of potassium and the primary loop was filled with 2200 cc of lithium. Samples were withdrawn from each loop for final qualification analyses. The analytical results, shown in Table VIII, indicated acceptable purities which were subsequently approved by the NASA Program Manager. Circulation of the alkali metals was initiated and start-up of the loop initiated.*

B. ADVANCED TANTALUM ALLOY CAPSULE TESTS

Testing of two ASTAR 811C and one ASTAR 811CN lithium thermal convection capsules continues. As of January 15, 1969, 2070 hours of testing had been completed. The chamber pressure at that time was 1.1×10^{-8} torr.

C. 2600°F LITHIUM LOOP

Final assembly of the 2600°F Lithium Loop will be accomplished upon completion of the lithium heater subassembly. This subassembly

* On 1-25-69 operation of the T-111 Rankine System Corrosion Test Loop was initiated and as of 2-7-69 over 300 hours of trouble-free operation were attained.

TABLE VIII

ANALYSIS OF THE ALKALI METALS USED IN OPERATION OF
THE T-111 CORROSION TEST LOOP^(a)

Element	Concentration, ppm	
	Lithium	Potassium
N	35, 39, 46, 49	-
O	49	3, 6
C	31	32
Ag	< 5	< 2
Al	5	10
B	< 50	< 30
Ba	< 50	< 20
Be	< 5	< 2
Ca	5	10
Cb	< 25	< 10
Co	< 5	< 2
Cr	< 5	< 2
Cu	5	2
Fe	< 5	2
Mg	5	2
Mn	< 5	< 2
Mo	< 5	< 2
Na	< 50	< 20
Ni	5	< 2
Pb	< 50	< 20
Si	5	2
Sn	< 25	< 10
Sr	5	< 2
Ti	< 25	< 10
V	< 25	< 10
Zr	< 25	< 10
Li	-	31
K	106	-

(a) Analysis obtained on samples removed from the loop prior to initiation of loop start-up.

cannot be made until the tensile and corrosion test specimens of the ASTAR alloys are heat treated at conditions to be specified by the NASA Program Manager.

IV. FUTURE PLANS

- A. Initiate test operation of the T-111 Rankine System Corrosion Test Loop.
- B. Continue testing of the advanced tantalum alloy capsules.

PUBLISHED REPORTS

<u>Quarterly Progress</u>	<u>For Quarter Ending</u>
Report No. 1 (NASA-CR-54477)	July 15, 1965
Report No. 2 (NASA-CR-54845)	October 15, 1965
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Report No. 4 (NASA-CR-72029)	April 15, 1966
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Report No. 7 (NASA-CR-72230)	January 15, 1967
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