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ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

Quarterly Progress Report No. 11

For Quarter Ending January 15, 1968

By

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and

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prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

CONTRACT NAS 3-6474

SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC
CINCINNATI, OHIO 45215

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ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

QUARTERLY PROGRESS REPORT 11

Covering the Period
October 15, 1967 to January 15, 1968

Edited by

R. W. Harrison

Project Metallurgist

Approved by

E. E. Hoffman

Manager, Corrosion Technology

Prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Lewis Research Center

Under Contract NAS 3-6474

February 2, 1968

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FOREWORD

The work described herein is sponsored by the National Aeronautics and Space Administration under Contract NAS 3-6474. For this program, Mr. R. L. Davies is the NASA Project Manager.

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

Refractory Alloy Procurement - R. G. Frank and L. B. Engel, Jr.

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

Welding and Joining - W. R. Young and S. R. Thompson.

Partial Pressure Gas Analyzer Calibration - Dr. T. F. Lyon.

ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

I. INTRODUCTION

This report covers the period from October 15, 1967 to January 15, 1968. 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 developed under Contract NAS 3-2547. Lithium will be heated by direct resistance in a primary loop. Heat rejection for condensation in the secondary potassium loop will be 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

g. Average heat flux in plug (0-18 inches), 240,000 BTU/hr ft^2

h. Average heat flux in boiler (0-250 inches), 23,000 BTU/hr ft^2

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-1Hf-1Re) 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.

II. SUMMARY

The fabrication of the T-111 Rankine System Corrosion Test Loop was completed. The loop has been moved to the test site and is presently being instrumented.

All necessary alkali metal for filling the T-111 Corrosion Test Loop has been prepared.

The partial pressure gas analyzer for the 4-foot diameter vacuum chamber has been calibrated.

The EM pump for the 2600°F Lithium Loop has been fabricated.

Three advanced tantalum alloy thermal convection capsules have been filled with lithium and two advanced tantalum alloy reflux capsules have been filled with potassium.

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III. PROGRAM STATUS

A. T-111 RANKINE SYSTEM CORROSION TEST LOOP

1. Loop Fabrication

The fabrication status of T-111 Corrosion Test Loop components is as follows:

As reported previously[(1)], the welding of all T-111 Corrosion Test Loop Components has been completed. During this report period, the last major subassembly was completed. The four major subassemblies were then joined to complete the loop fabrication.

The potassium surge tank subassembly, Figure 1, illustrates the intermediate fixturing required to maintain component orientation. During this fabrication the pressure transducer tee welds and the weld between the surge tank and EM pump duct were made and postweld annealed locally. Each component had been postweld annealed previously.

The lithium heater subassembly which consists of the lithium heater, EM pump duct, and surge tank, Figure 2, was completed. The three welds required to join these components were annealed locally at 2400°F for 1 hour in accordance with Specification SPPS 03-0037-00-A, "Postweld Vacuum Annealing of Cb-1Zr and T-111 (Ta-8W-2Hf) Alloys".

The completed lithium heater, condenser, boiler, and potassium surge tank subassemblies were positioned in the stainless steel support structure attached to the vacuum chamber spool section for reference alignment. The tubing which joins the boiler and potassium surge tank subassemblies was match marked for alignment and the two subassemblies were removed from the spool section for welding in the eight-foot diameter extension to the welding chamber. This assembly step was required because this particular weld could not be reached with the entire loop

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(1) Advanced Refractory Alloy Corrosion Loop Program, Quarterly Progress Report No. 10, for Period ending October 15, 1967, NASA Contract NAS 3-6474.

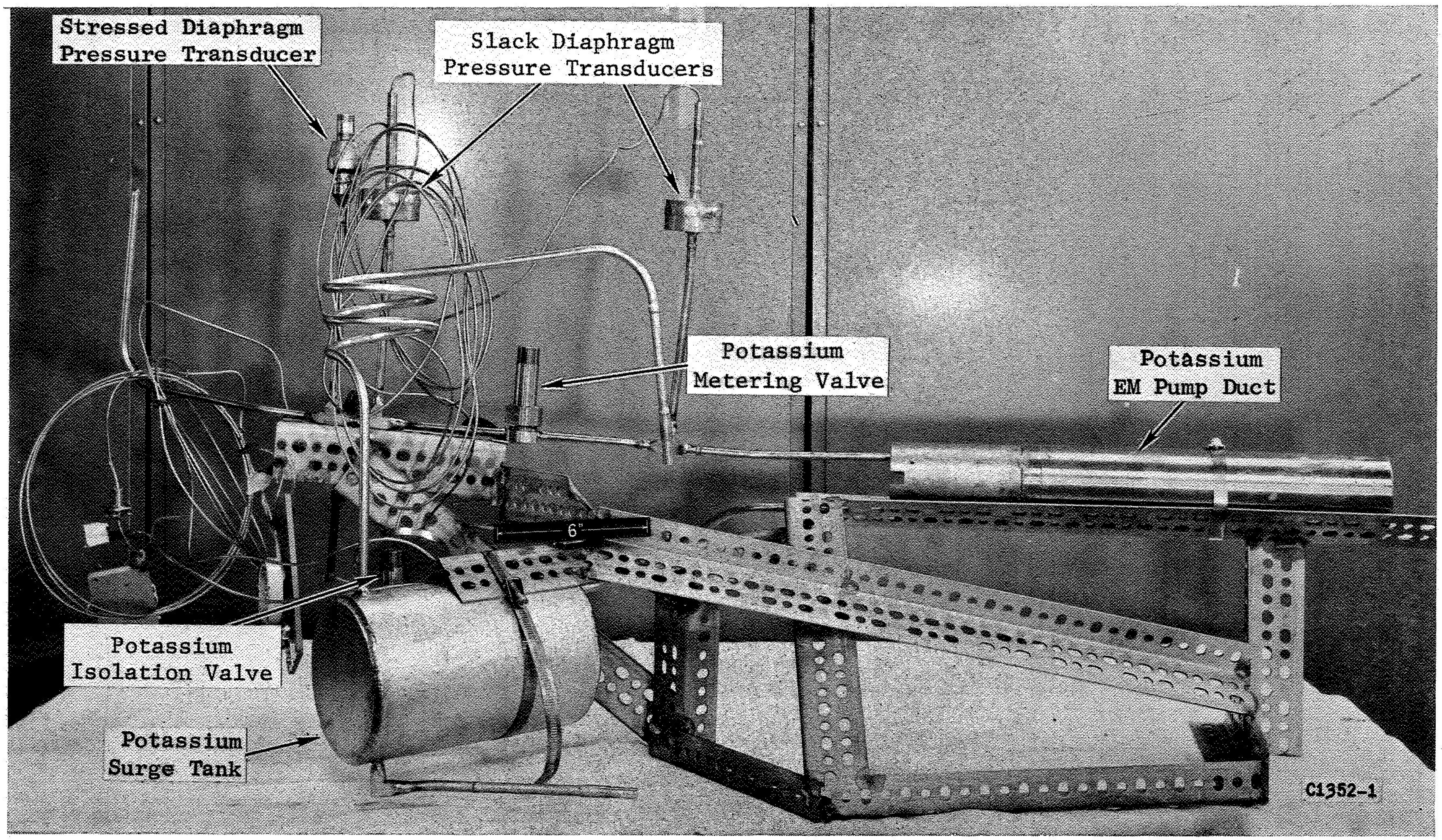

Figure 1. Potassium Surge Tank and EM Pump Subassembly of the T-111 Corrosion Test Loop Mounted on the Welding Fixture. (Orig. C67110669)

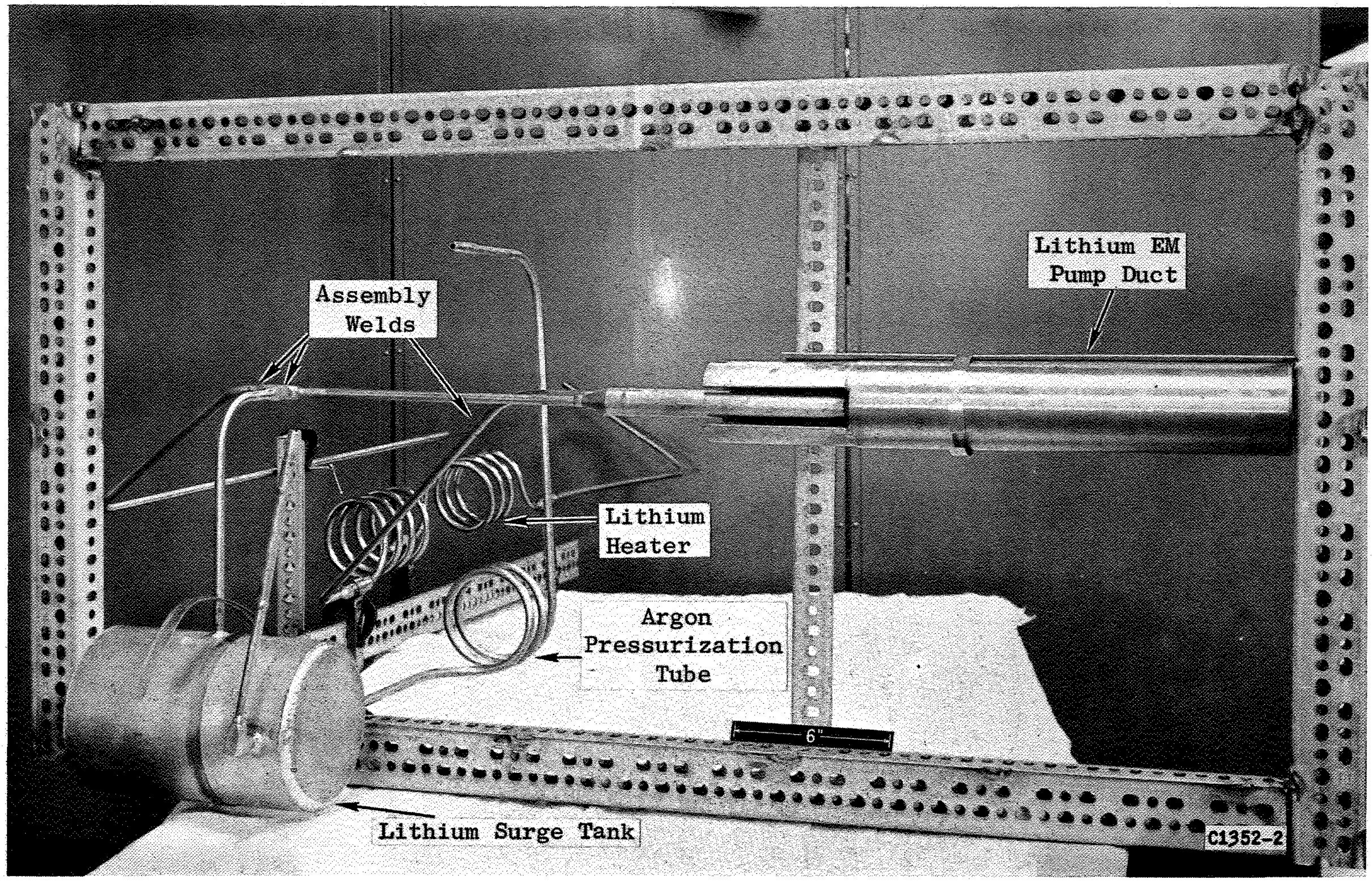

Figure 2. Lithium Surge Tank and EM Pump Subassembly of the T-111 Corrosion Test Loop Mounted on the Welding Fixture. (Orig. C67110668)

positioned in the welding chamber. After radiographic inspection of this weld, this unit was repositioned in the permanent support structure.

The lithium heater and condenser subassemblies were also positioned in the support structure. The final assembly weld fixture was attached to the loop, Figure 3, holding the subassemblies and three slack diaphragm pressure transducers (not shown) in alignment. The permanent support structure was disassembled and the loop, now supported by the welding fixture, was removed from the vacuum chamber spool section and placed in the welding chamber as shown in Figure 4.

The seven welds required to join the subassemblies and attach the three pressure transducers were inspected radiographically and subsequently annealed in the welding chamber at 2400°F for 1 hour in accordance with Specification SPPS 03-0037-00-A.

The loop was removed from the welding chamber and the electrode area of the lithium heater and the potassium preheater were grit blasted to increase emittance per Specification SPPS 03-0011-00-A, "Grit Blasting of Columbium and Columbium Alloy Products". The loop was then positioned in the vacuum chamber spool section and the permanent support structure was affixed. After the loop was supported properly the final welding fixture was removed. An overall view of the loop and spool piece is shown in Figure 5. A close-up photograph of the components in the lower portion of the loop is given in Figure 6.

The stainless steel tube attachments to the drain and gas pressurization lines were welded to the loop connections and vacuum chamber feedthroughs. A final mass spectrometer helium leak test was then performed on the entire loop with no leak indication. The vacuum connections for the NaK-filled tubes from the slack diaphragm pressure transducer and the EM pump duct stainless steel outer cans were welded to the appropriate vacuum spool section feedthroughs. The completed loop was then removed to the test site.

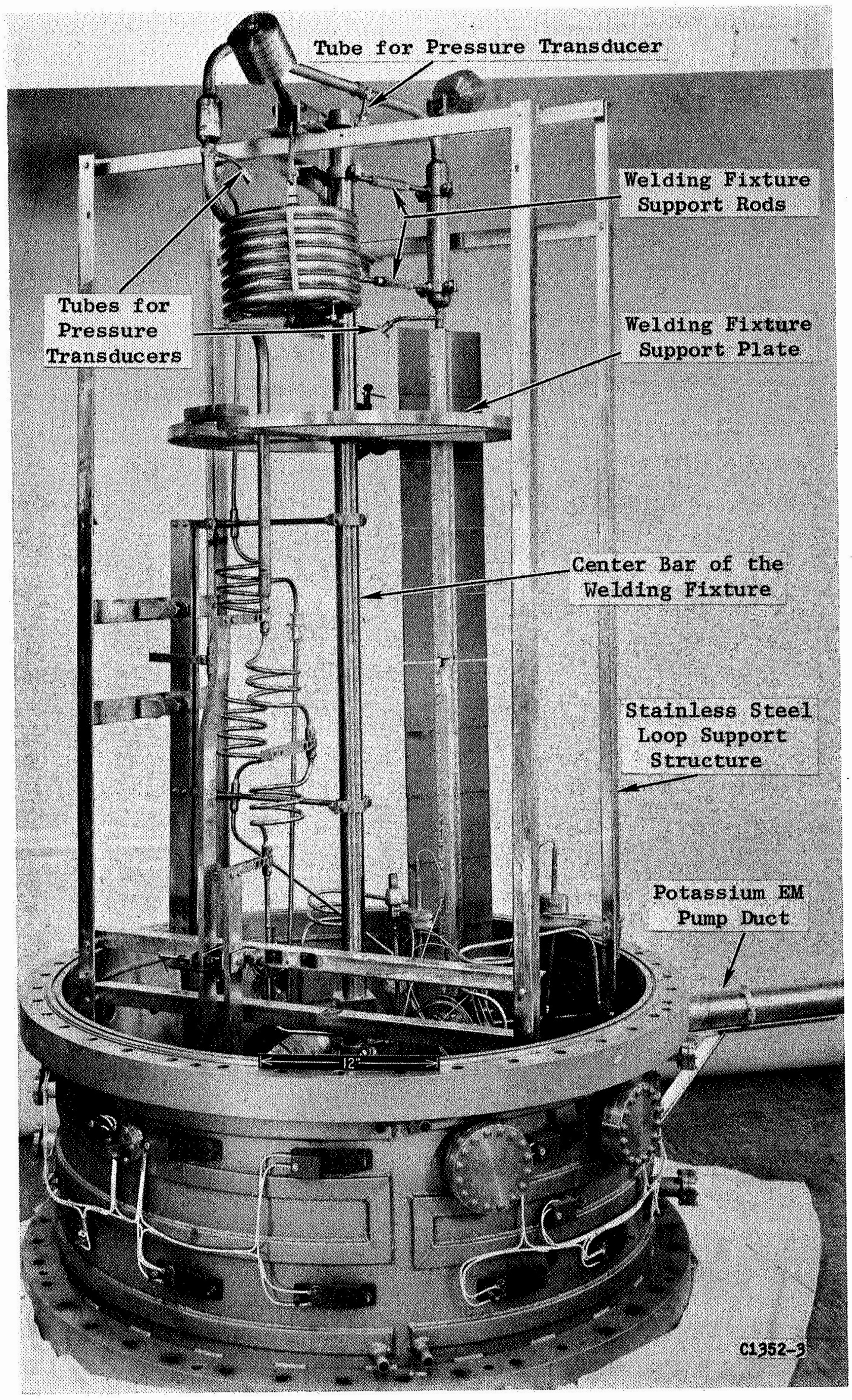

Figure 3. T-111 Corrosion Test Loop and Chamber Spool Piece During the Transfer of the Loop from the Support Structure to the Welding Fixture. (Orig. C67112232)

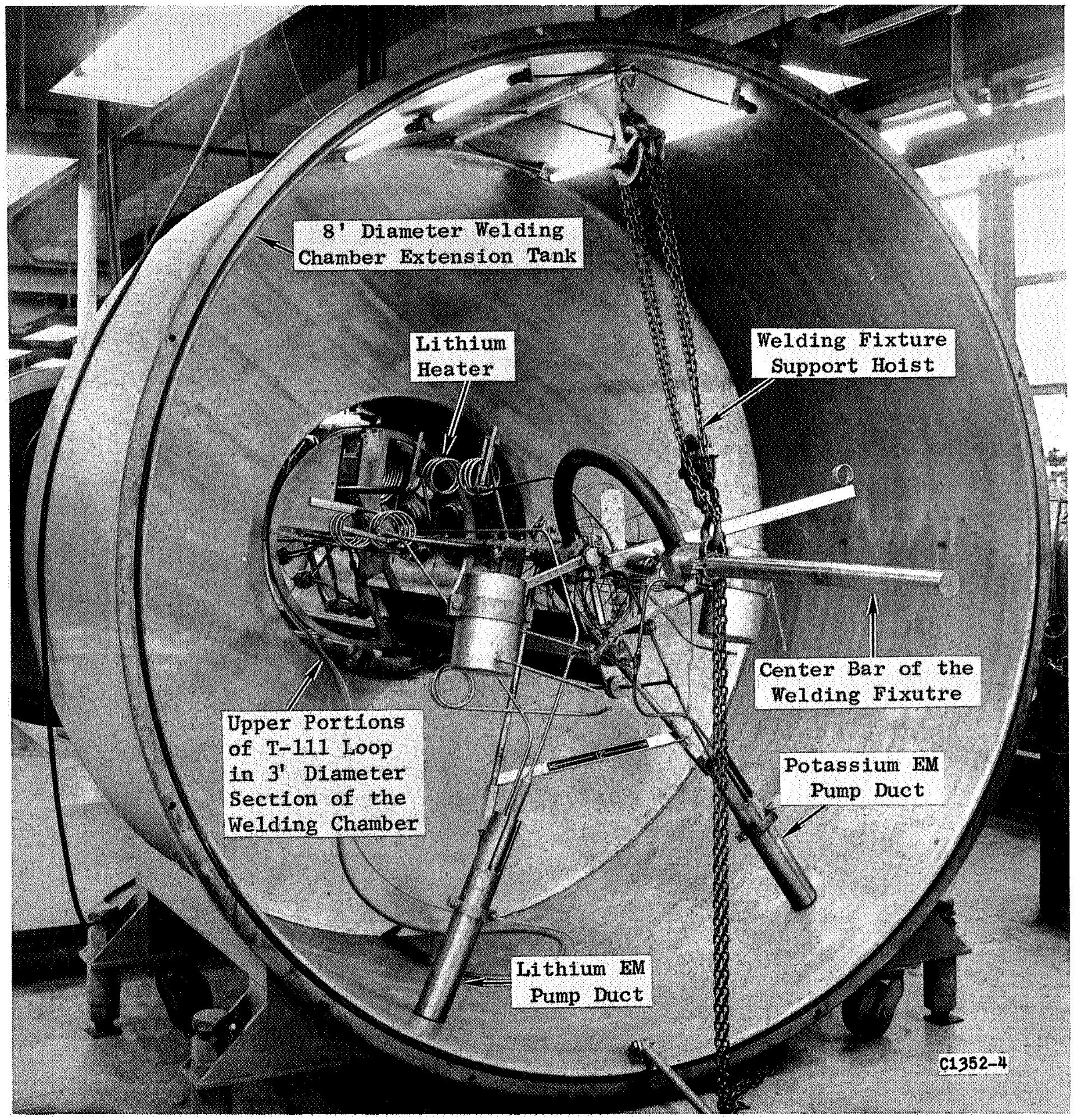

Figure 4. T-111 Corrosion Test Loop Mounted on the Welding Fixture Prior to Final Welding and Weld Heat Treatment Operations. (Orig. C67120714)

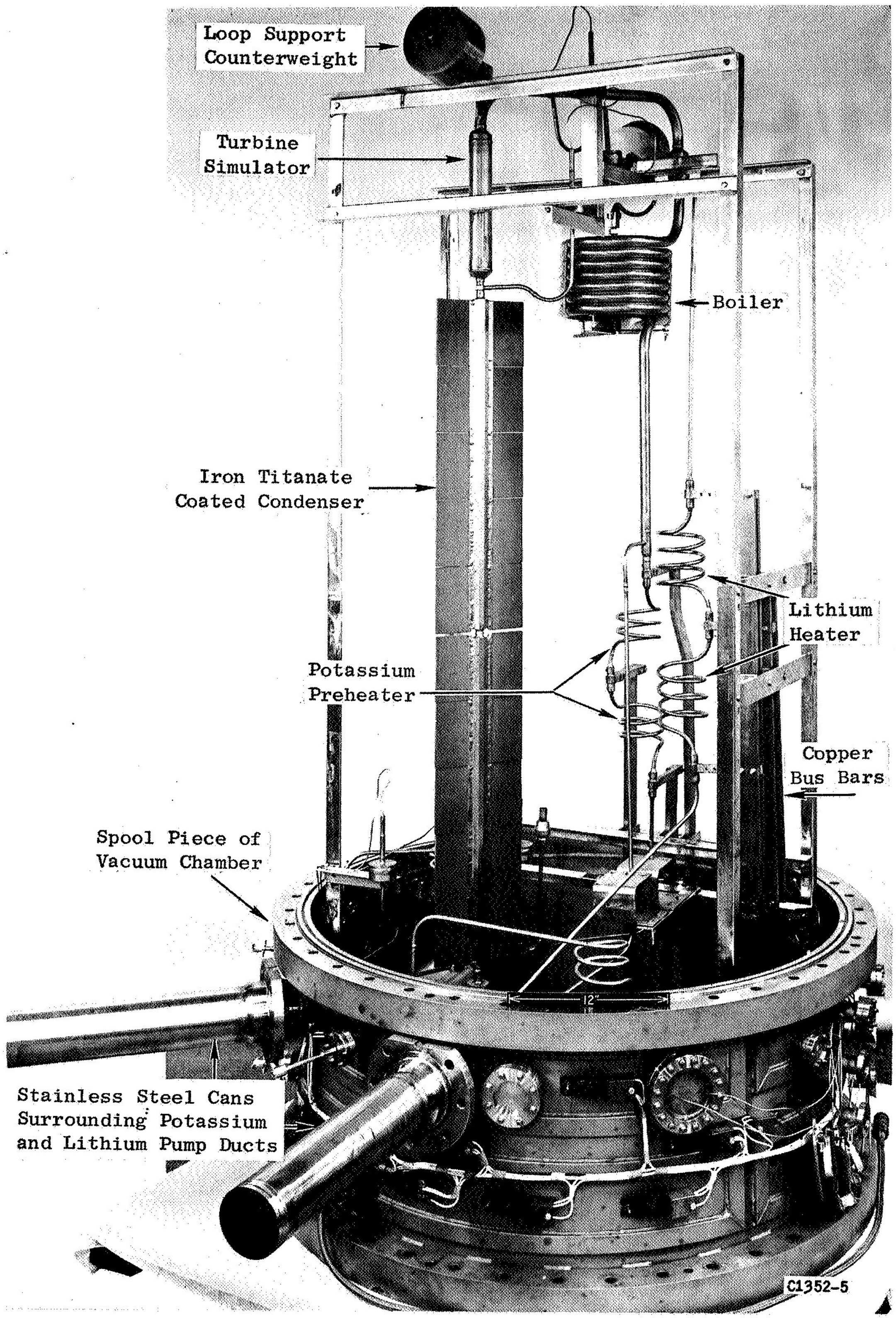

Figure 5. T-111 Corrosion Test Loop and the Vacuum Chamber Spool Piece Following Completion of Loop Fabrication. (Orig. C67122143)

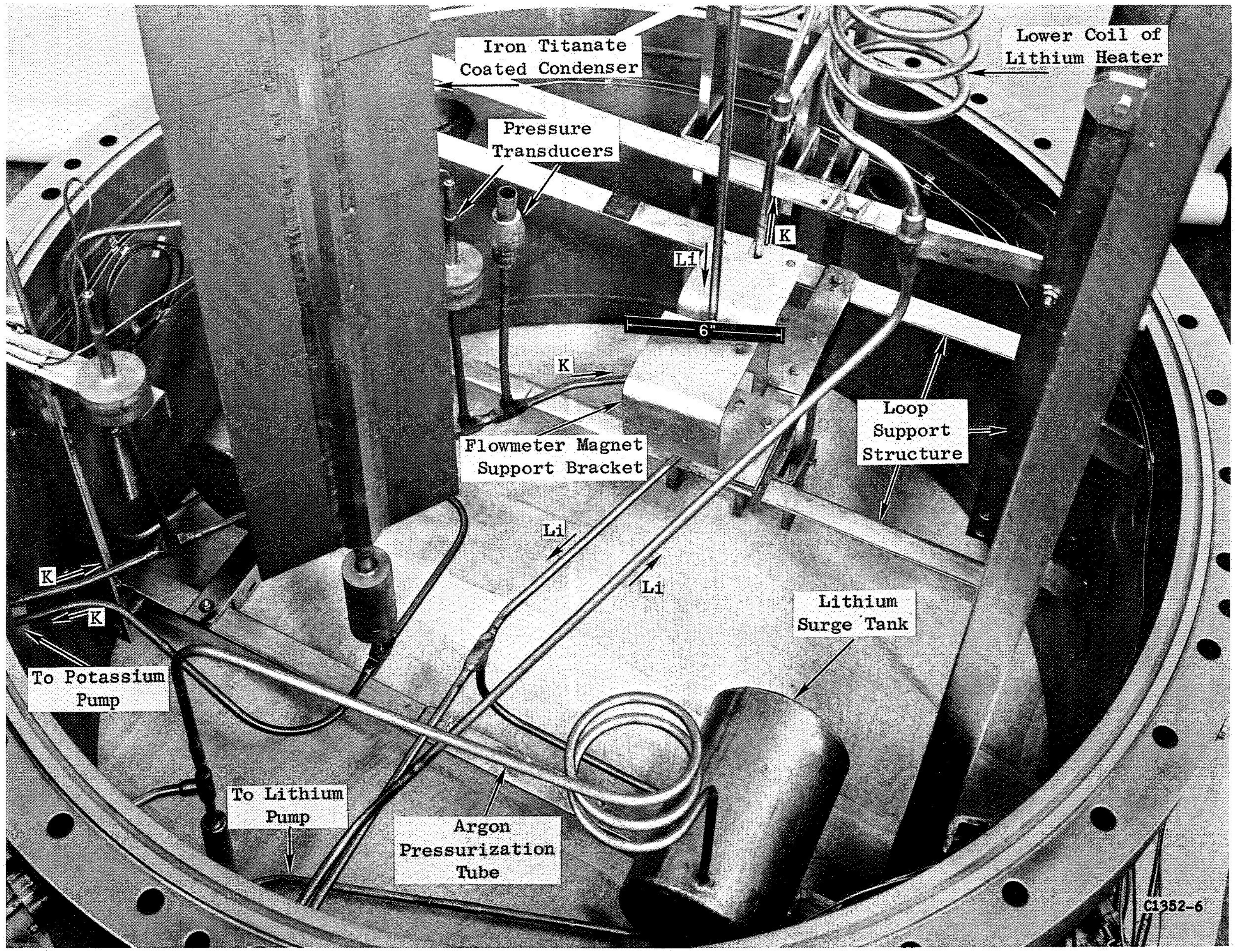

Figure 6. Lower Portion of the T-111 Corrosion Test Loop Following Completion of Fabrication. (Orig. C67122147)

2. <u>Alkali Metal Purification</u>

Twelve pounds of hot trapped lithium were distilled at 1235°F, thereby making a total of 15 pounds of distilled lithium available for filling the T-111 Corrosion Test Loop. Analysis for nitrogen indicated a concentration of less than 5 ppm. Analysis for oxygen, by fast neutron activation at Gulf General Atomic, indicated a concentration of 273 ±56 ppm. Oxygen analysis using the vacuum distillation method will be performed by R. Gahn at NASA-Lewis Research Center at a later date. The anomalously high values for oxygen which have been obtained by neutron activation are believed in error. This conclusion is based on similarly high neutron activation oxygen results obtained for potassium of known low oxygen concentration.

Five pounds of hot trapped potassium were distilled at 550°F, making 18 pounds of potassium available for the T-111 Corrosion Test Loop. Analysis of the distillate indicated an oxygen concentration of 5 ppm. A sample of the distilled potassium will also be analyzed for oxygen by fast neutron activation at Gulf General Atomic as an additional check on the techniques and procedures employed.

The dual transfer system for filling the T-111 Corrosion Test Loop System was prepared for installation and will be attached to the loop and purification systems when the loop has been installed in the vacuum chamber.

3. <u>Installation and Instrumentation of the T-111 Corrosion Test Loop</u>

Following the completion of loop fabrication and mounting of the loop on the test chamber spool piece, the loop was transferred from the fabrication area to the test operation area by pneumatic-wheeled truck traveling at a maximum speed of 5 miles per hour. During the transfer, the entire loop was covered with a polyethylene bag and all gas pressurization and liquid metal fill lines were sealed with gas-tight OD compression fittings*.

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* Imperial Brass Mfg. Co. Model No. 240-F

The test loop was positioned near the vacuum system, and an air shelter was assembled over the loop. The air shelter, which was previously used for instrumentation of the Cb-1Zr Rankine System Corrosion Test Loop[2], is a reinforced polyethylene hemisphere approximately 15 feet in diameter by 15 feet high and is supported by 0.2 psig air pressure supplied by a centrifugal blower. The loop is at ground level and is readily accessible for instrumentation. All personnel are required to wear clean, white dacron gloves, shoe covers and coveralls while working in the air shelter.

The temporary support brackets used in the fabrication and transfer of the loop were removed and permanent support brackets and instrumentation channels for routing thermocouple and pressure sensor lead wires were installed. The copper bus bars for both the potassium preheater and lithium heater were also installed at this time, since they also serve as structural support members to maintain the helical heater coils in position.

Thermal insulation consisting of multiple layers of Cb-1Zr foil is simultaneously being applied to the loop as the thermocouples are being installed. The procedure used is similar to that developed in instrumenting the Cb-1Zr Rankine System Corrosion Test Loop[3]. All thermocouples are made from single lots of the 0.005-inch W-3Re/W-25Re wire which was also used in the Cb-1Zr Corrosion Test Loop. Beryllium oxide (99.5%) insulators are used at the hot junction of the thermocouple where contact with the T-111 surface is in excess of 1800°F. Aluminum oxide (99.5%) insulators are used where the temperature is less than 1800°F and for all thermocouple lead wires inside the vacuum chamber. All insulators were baked out at a pressure of 1×10^{-5} torr for 1 hour at 2000°F before installation.

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(2) Potassium Corrosion Test Loop Development, Quarterly Progress Report No. 8 for Period Ending July 15, 1965, NASA Contract NAS 3-2547, NASA-CR-54843, p. 10.

(3) ibid., p. 17.

4. Calibration of the Partial Pressure Gas Analyzer

The partial pressure gas analyzer for the Corrosion Test Loop vacuum chamber has been calibrated for hydrogen, helium, nitrogen, and argon. The analyzer tube (GE Model 22PT 120) has recently been received from the manufacturer where it was cleaned and adjustments made to the ion source. The tube was mounted on the chamber, and the empty chamber was evacuated without the spool piece. After the chamber had been baked out, and prior to calibration of the analyzer, a base pressure of 3.9×10^{-9} torr was obtained.

The calibration of the partial pressure gas analyzer was made by admitting a pure gas to the chamber through a variable leak valve (Granville-Phillips Series 203). When the pressure had stabilized, a reading of the system ion gauge was made, and a mass spectrum was obtained. This process was repeated at various pressures for each of the four calibrating gases. From these data, calibration factors for each of the gases were calculated. The calibration factor (torr per amp) is here defined as the ion gauge reading per unit ion current from the analyzer for a particular pure gas. Appropriate corrections were applied when appreciable partial pressures of gases other than the calibrating gas were present.

The calibration factors thus obtained are shown in Table I. Calibration factors for gases other than those for which a direct calibration was made were obtained by interpolation or extrapolation. Also given in Table I are ionization gauge sensitivities relative to nitrogen for each of the gases tabulated.

Quantitative data may thus be obtained from the mass spectra as follows: to obtain indicated ionization gauge partial pressure, the ion current at a particular mass number is multiplied by the appropriate calibration factor; to obtain true partial pressure, the indicated ionization gauge partial pressure is divided by the relative ionization gauge sensitivity.

TABLE I. PARTIAL PRESSURE GAS ANALYZER CALIBRATION FACTORS

Parent Specie	m/e	Calibration Factor (torr/amp)	Relative Ionization Gauge Sensitivity
H_2	2	1.02	0.42
He	4	5.9	0.19
CH_4	16	(2.5)	1.07
H_2O	18	(2.7)	0.89
Ne	20	(2.9)	0.33
CO or N_2	28	3.9	1.00
O_2	32	(4.6)	0.85
Ar	40	6.3	1.56
CO_2	44	(7.2)	1.37

NOTE: Values in parentheses obtained by interpolation or extrapolation.

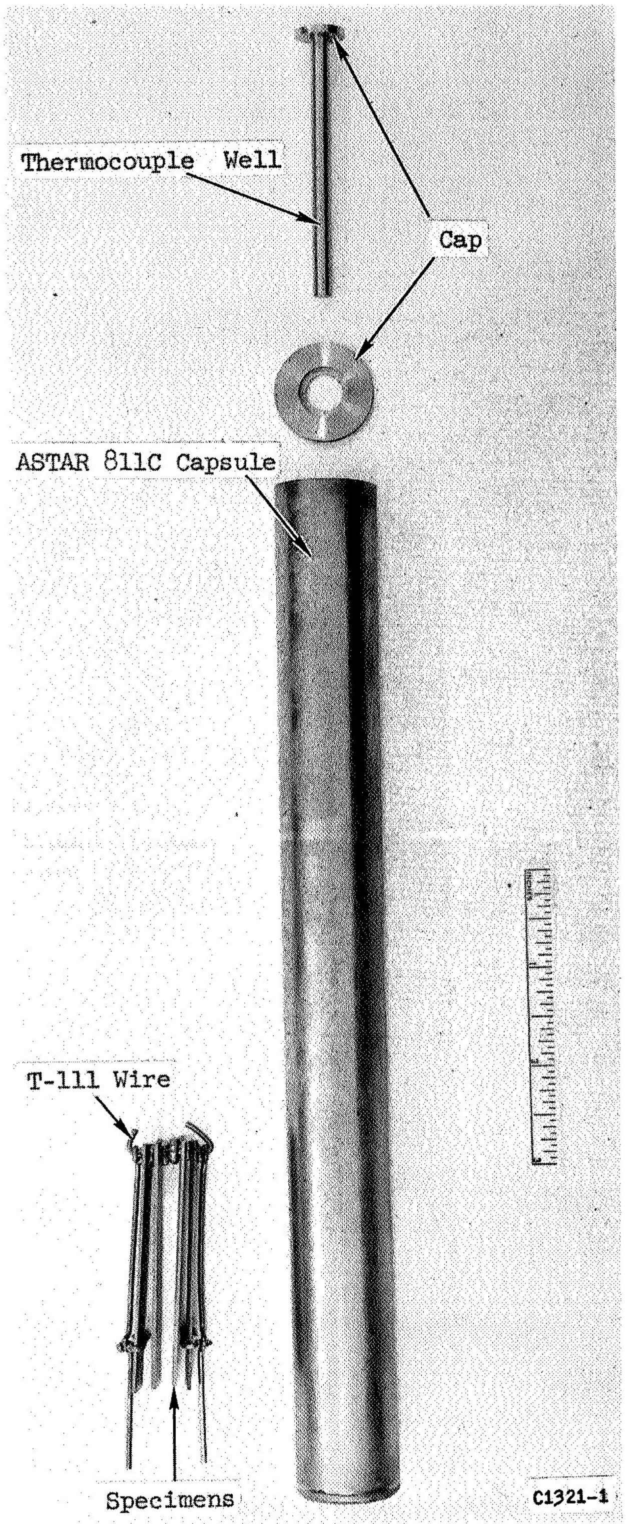
5. Test Plan for the T-111 Rankine System Corrosion Test Loop

The Test Plan for the T-111 Rankine System Corrosion Test Loop was completed and submitted to the NASA Program Manager for review and approval. The Test Plan was approved on January 12, 1968 and will be issued during the next reporting period.

B. ADVANCED TANTALUM ALLOY CAPSULE TESTS

The advanced tantalum alloy capsules are shown, prior to final assembly, in Figures 7-9. The two ASTAR 811C potassium reflux capsules contain oxygen contaminated specimens of ASTAR 811C and T-111 in the as-welded and welded and annealed conditions as shown in Figure 7. The two ASTAR 811C lithium thermal convection capsules contain similar specimens to the potassium reflux capsules. Specimens of ASTAR 811C in the as-received, welded, and annealed conditions are also included in these capsules, as shown in Figure 8. The ASTAR 811CN lithium thermal convection capsule shown in Figure 9, contains six (6) ASTAR 811CN stress rupture specimens.

After assembly of the specimens into the capsules, the larger top cap was welded to the capsule body. The smaller cap with the thermocouple well was welded in place after the capsules were filled with alkali metal. The assembled capsules were placed in the 30 KV electron beam welding chamber, shown in Figure 10, for filling with alkali metal. The potassium reflux capsules were filled with potassium procured from Mine Safety Appliances Research Corporation. This high purity grade potassium was hot trapped and distilled before shipment to General Electric. The potassium was further purified at General Electric by vacuum distillation at approximately 600°F at a pressure in the 10^{-5} torr range into a titanium lined hot trap. The purification apparatus is shown in Figure 11. The hot trap was used in this case as a clean storage and transfer container. In the filling operation potassium was transfered under argon pressure from the hot trap into the annular dispensing reservoir, shown in Figure 12, to a precalculated height corresponding to the volume required to fill each capsule. The liquid



SPECIMENS

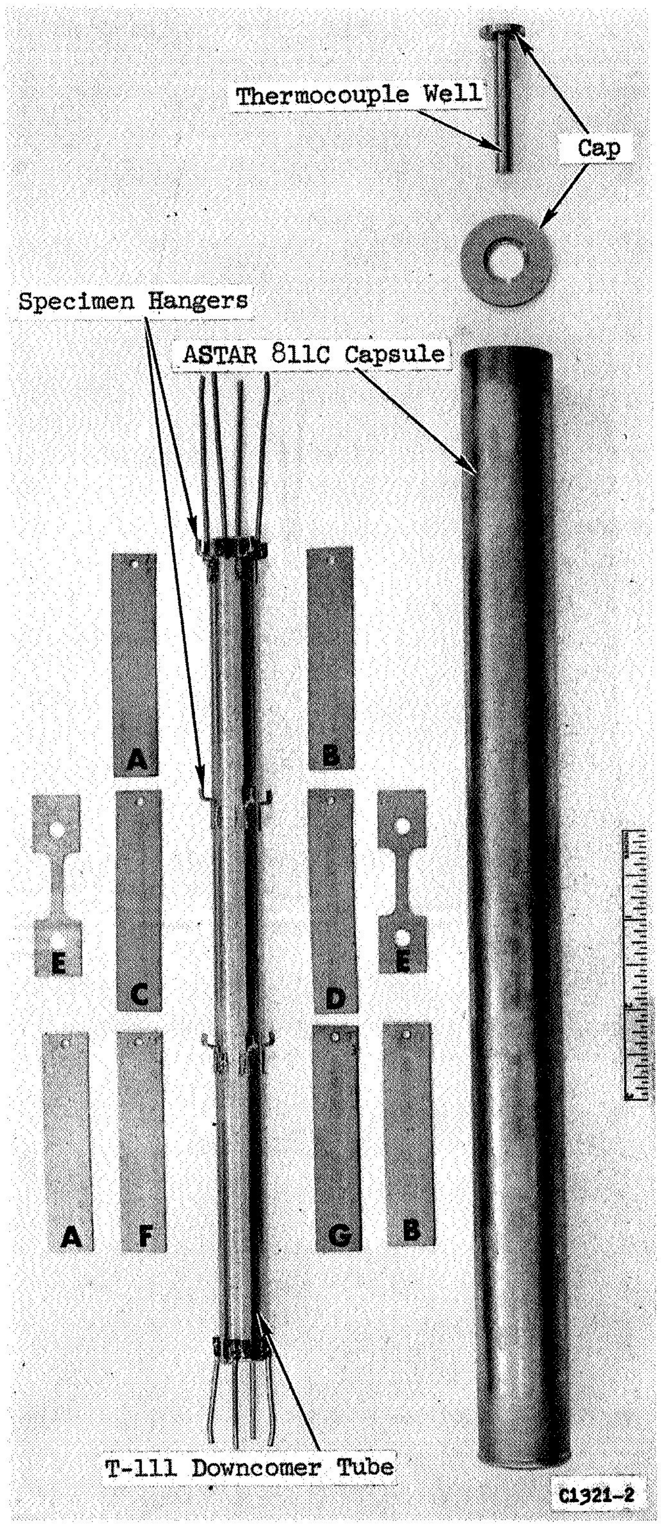
Oxygen Contaminated ASTAR 811C Welded

Oxygen Contaminated ASTAR 811C Welded and Postweld Annealed

Oxygen Contaminated T-111 Welded

Oxygen Contaminated T-111 Welded and Postweld Annealed

Figure 7. Advanced Tantalum Alloy ASTAR 811C - Potassium Reflux Capsule. (Orig. C67111011)



SPECIMENS

A. Oxygen Contaminated ASTAR 811C Welded

B. Oxygen Contaminated ASTAR 811C Welded and Postweld Annealed

C. ASTAR 811C Welded

D. ASTAR 811C Welded and Postweld Annealed

E. ASTAR 811C Stress Rupture Specimens

F. Oxygen Contaminated T-111 Welded

G. Oxygen Contaminated T-111 Welded and Postweld Annealed

Figure 8. Advanced Tantalum Alloy ASTAR 811C - Lithium Thermal Convection Capsule. (Orig. C67111017)

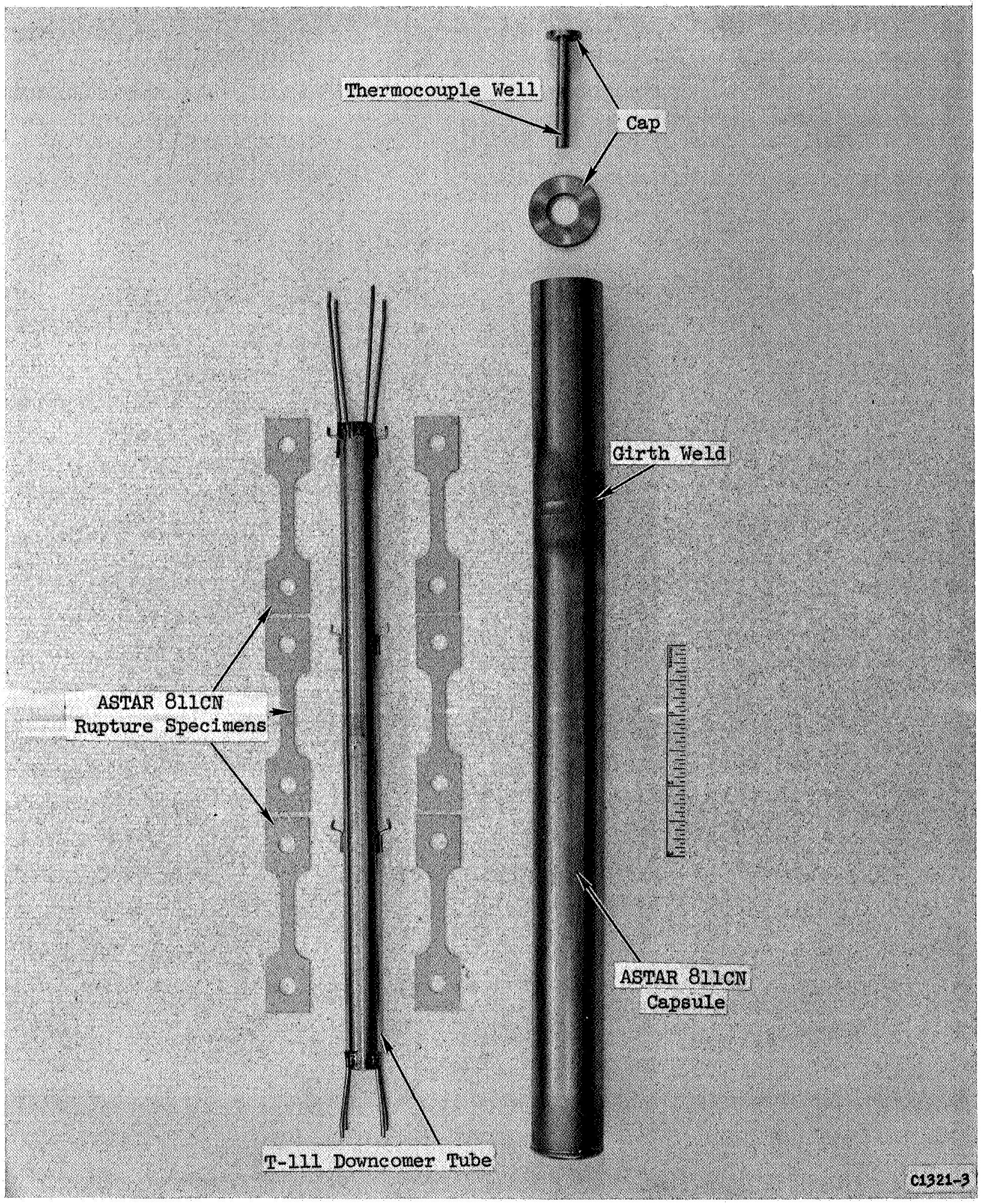


Figure 9. Advanced Tantalum Alloy ASTAR 811CN - Lithium Thermal Convection Capsule. (Orig. C67111018)

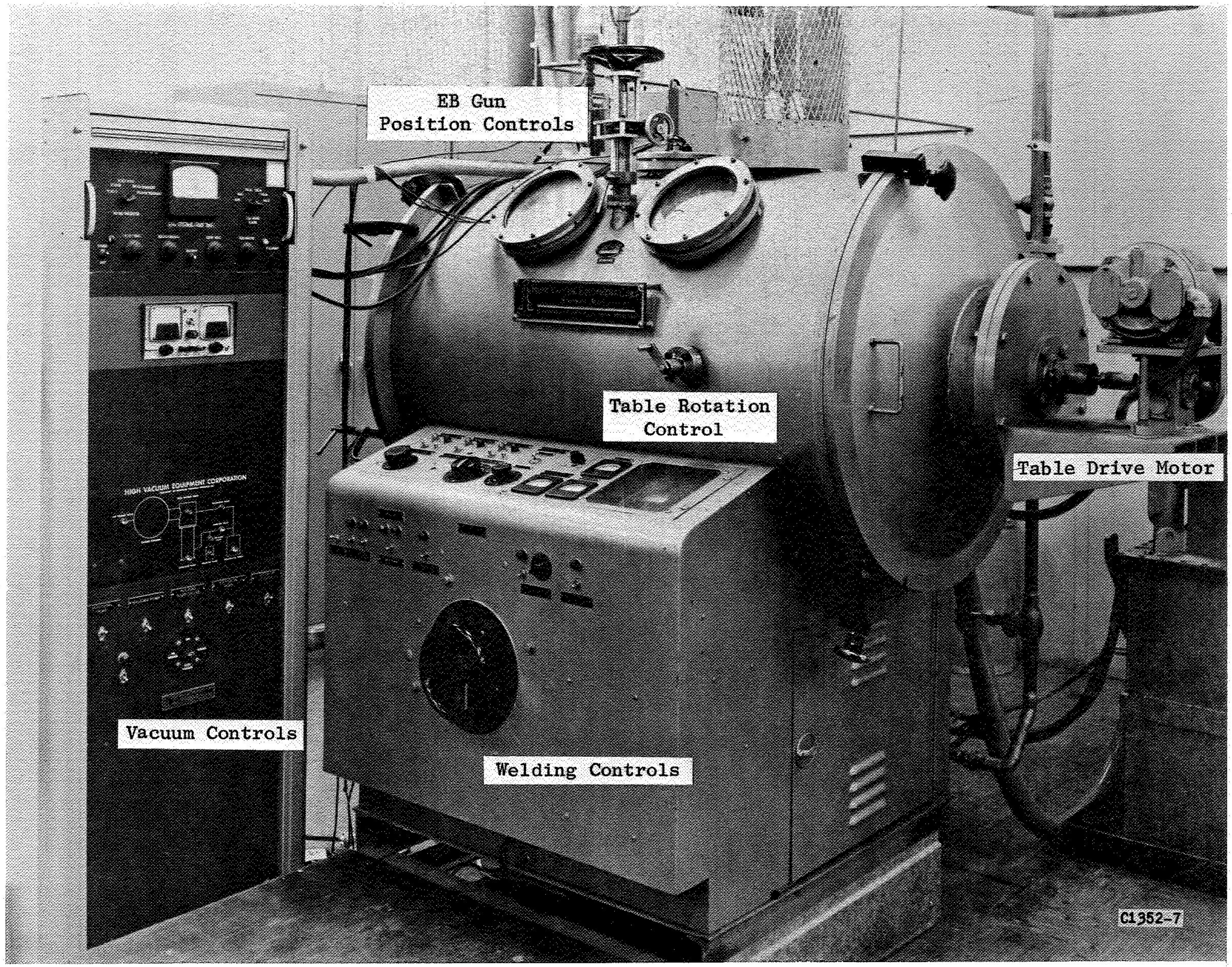


Figure 10. 30KV Electron Beam Welding Chamber and Controls Used to Fill Corrosion Capsules with Alkali Metal. (C67040153)

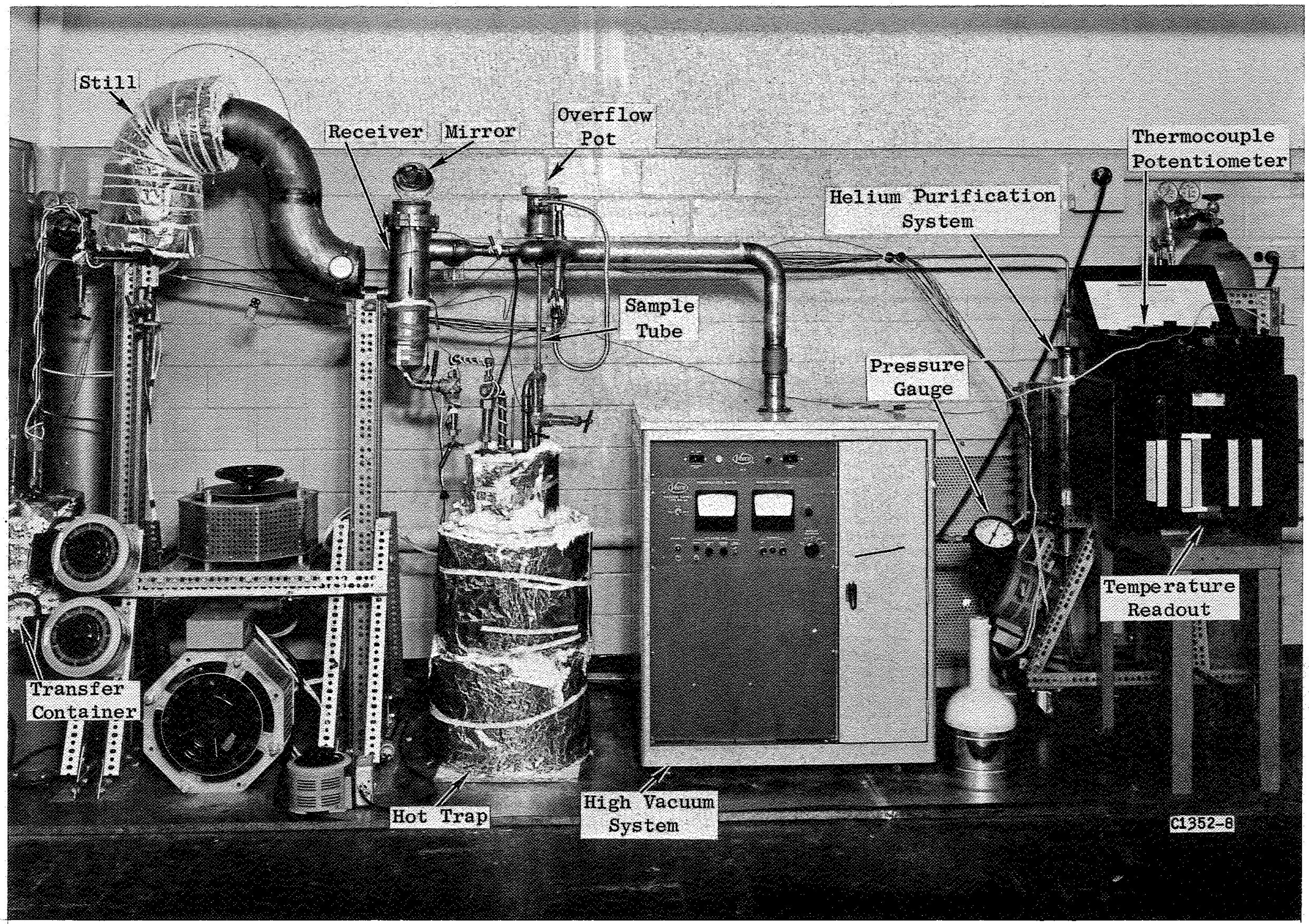


Figure 11. Potassium Purification System.

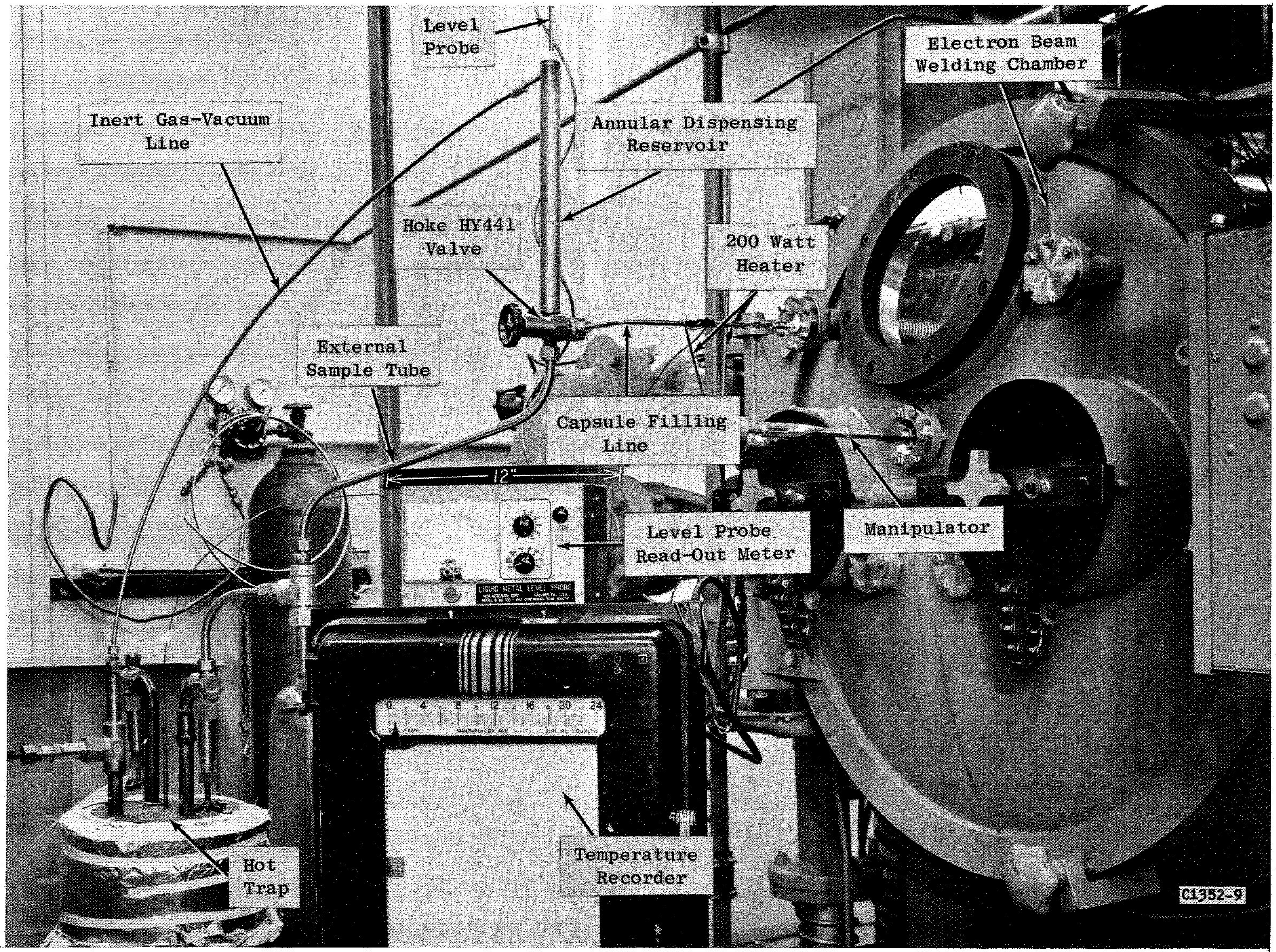


Figure 12. External Apparatus for Filling Capsules with Potassium in the EB Welding Chamber. (Orig. C66051941)

metal level was determined by means of a MSA Research Corporation Level Probe. The measured volume was then transfered under argon pressure through the bellows-sealed filling line, shown in Figure 13, into the capsule. Each capsule was positioned under the fill line by means of a manipulator and a flexible cable which was attached on one end to a gear train on the turntable and on the other end to a crank outside the chamber, Figures 10 and 13. A waste container was placed on the rotating table to provide a means of initial flushing out the fill line with potassium. When filled, each capsule was rotated from under the fill line and capsule lids were put in place with the manipulator. The manipulator is sealed to the chamber door by means of sliding O-ring seals. The capsule was positioned under the EB welding gun by moving the table holding the capsule utilizing the flexible cable drive. The gear on each capsule was adjusted to mesh with the welding drive gear such that the capsules rotated around their axes during welding. This gear was controlled by a variable speed motor to rotate at the optimum welding speed. Copper chill blocks were fitted on each capsule to reduce the heat conduction along the capsule during welding thereby minimizing the possible vaporization of the potassium which might cause unsound welds.

Analytical samples of the potassium were obtained by filling a stainless steel tube in the same manner as the capsules were filled. The resulting oxygen analyses by the mercury amalgamation method indicated the oxygen concentration to be 9 ppm. Metallic impurities were determined by spectrographic analysis and are presented in Table II.

The thermal convection capsules were filled with distilled lithium obtained from the transfer container shown in Figure 14. The lithium was procured from Lithium Corporation of America, Bessemer City, North Carolina, and further purified at General Electric by filtering through a 5 micron filter, hot trapping 126 hours at 1500°F in a titanium lined, zirconium getter hot trap, and distilling at 1235°F. The distillation

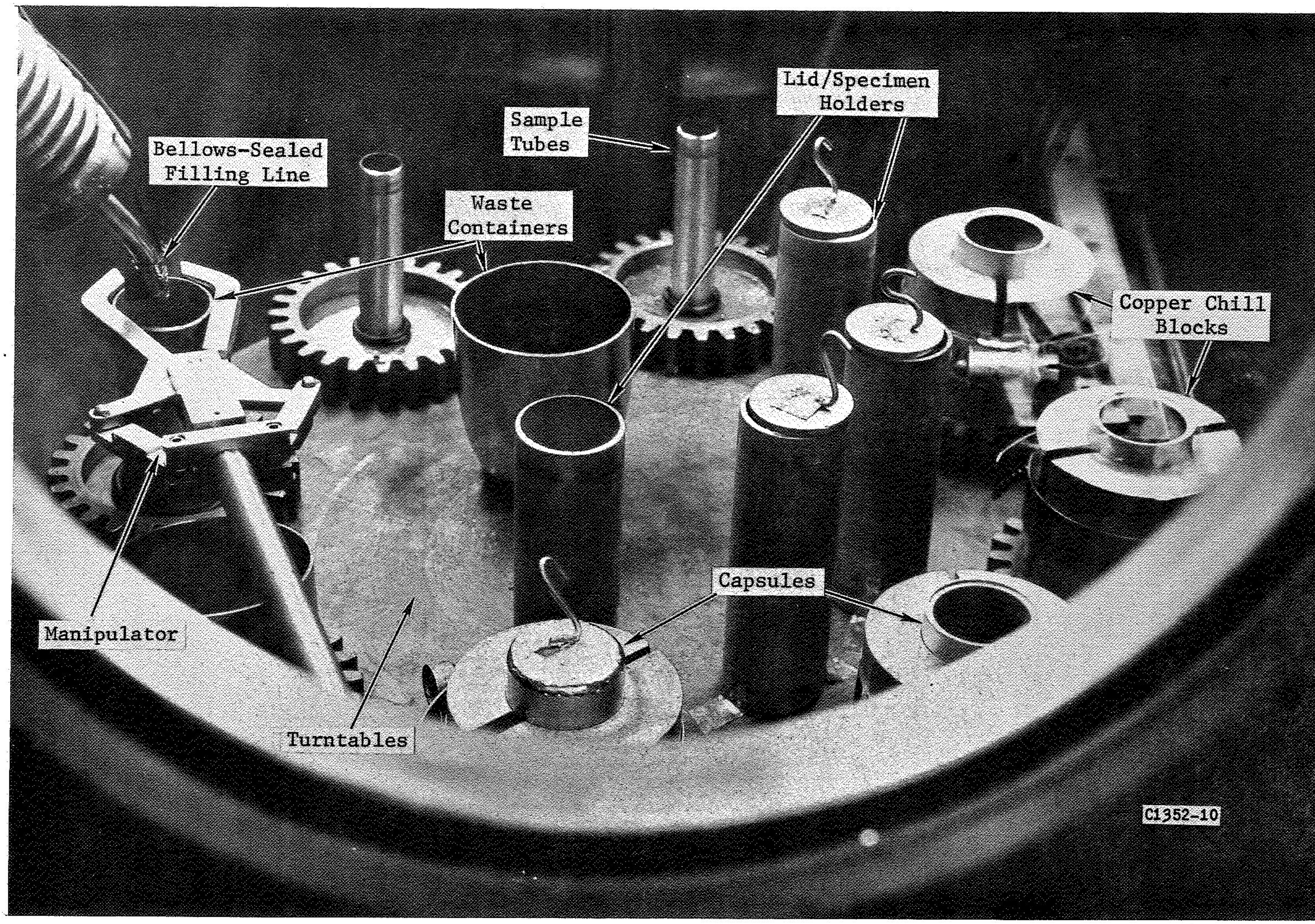


Figure 13. Internal Apparatus for Filling Capsules with Potassium in the EB Welding Chamber. (Orig. C66053119)

TABLE II. SPECTROGRAPHIC ANALYSIS OF ALKALI METALS USED IN ADVANCED TANTALUM ALLOY CAPSULE TESTS

Element	Concentration, ppm Potassium	Lithium
Ag	< 2	< 5
Al	< 2	< 5
B	< 10	< 50
Ba	< 10	< 50
Be	< 10	< 5
Ca	< 2	< 5
Cb	< 10	< 25
Co	< 2	< 5
Cr	< 2	< 5
Cu	< 2	< 5
Fe	< 2	< 5
Mg	< 2	< 5
Mn	< 2	< 5
Mo	< 2	< 5
Na	< 2	< 25
Ni	< 2	< 5
Pb	< 2	< 25
Si	< 2	5
Sn	< 2	< 25
Sr	< 2	< 5
Ti	< 2	< 25
V	< 10	< 25
Zr	< 2	< 25

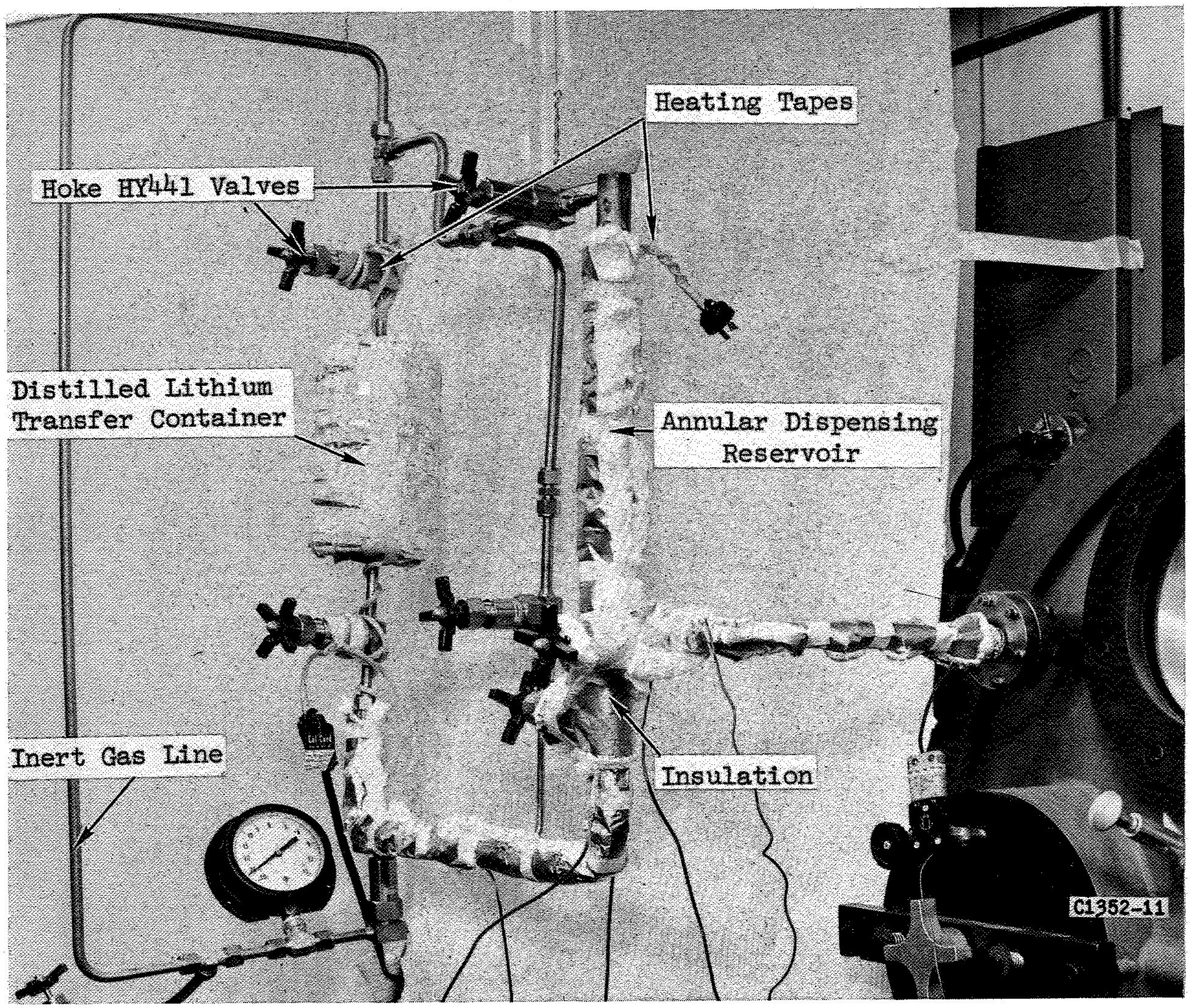


Figure 14. Lithium Capsule Filling System Mounted on the Door of the EB Welding Chamber. (Orig. C67110666)

procedure was described in an earlier report[(4)]. The chemical analysis of the distilled lithium is presented in Table II. The lithium filling operation was performed similarly to that previously described for filling capsules with potassium with two differences in the procedure. First, the thermal convection capsules were preheated to 400°F by means of tubular heating elements coiled around each capsule. Preheating was employed to keep the lithium molten in the capsule during filling thus limiting the possibility of freezing causing a bridge in the small annular space between the capsule wall and the specimens contained therein. Second, the thermal convection capsules were sealed by TIG welding in helium to obtain a room temperature helium pressure of 1 atmosphere inside each capsule. At the capsule temperatures of 2300°F the helium pressure above the lithium will be approximately 195 psi. This helium overpressure will reduce the possibility of lithium boiling instabilities interferring with thermal convection flow. Analytical analysis of the lithium sample taken during filling indicated a nitrogen concentration of 5 ppm. The thermal convection capsules were helium mass spectrometer leak checked after sealing to detect any outleakage of helium. Minor helium leakage was detected in the ASTAR 811C capsules. The capsules were subsequently re-evacuated and the lids resealed by TIG welding under 1 atmosphere of helium. Mass spectrometric leak checking indicated no leaks.

C. 2600°F LITHIUM LOOP FABRICATION

During this report period, orders were placed for machining of loop test section components. Delivery is scheduled for the end of January.

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(4) Advanced Refractory Alloy Corrosion Loop Program, Quarterly Progress Report No. 9 for Period Ending July 15, 1967, NASA Contract NAS 3-6474, NASA-CR-72336, p. 15.

The fabrication of the loop EM pump duct proceeded with the receipt of all machined components as shown in Figure 15. The weld between the helical duct and the pump exit line, 3/8-inch OD x 0.065-inch wall, was then made. The interference fit between the helical duct and wrapper was performed without incident. The helical duct was immersed in liquid nitrogen to provide a diametral clearance between it and the outer wrapper which remained at room temperature. The closure welds on the end cap and connector were then made. Final machining of the outside diameter, completed the EM pump duct as shown in Figure 15. The torsion tube (not shown) which prevents pump duct rotation will be welded in place prior to postweld annealing of this component.

The Cb-1Zr surge tank for this loop is also nearly completed. The gas pressurization, drain, and fill tubes have been welded in place. Welding of the end caps to the tank body will complete this component.

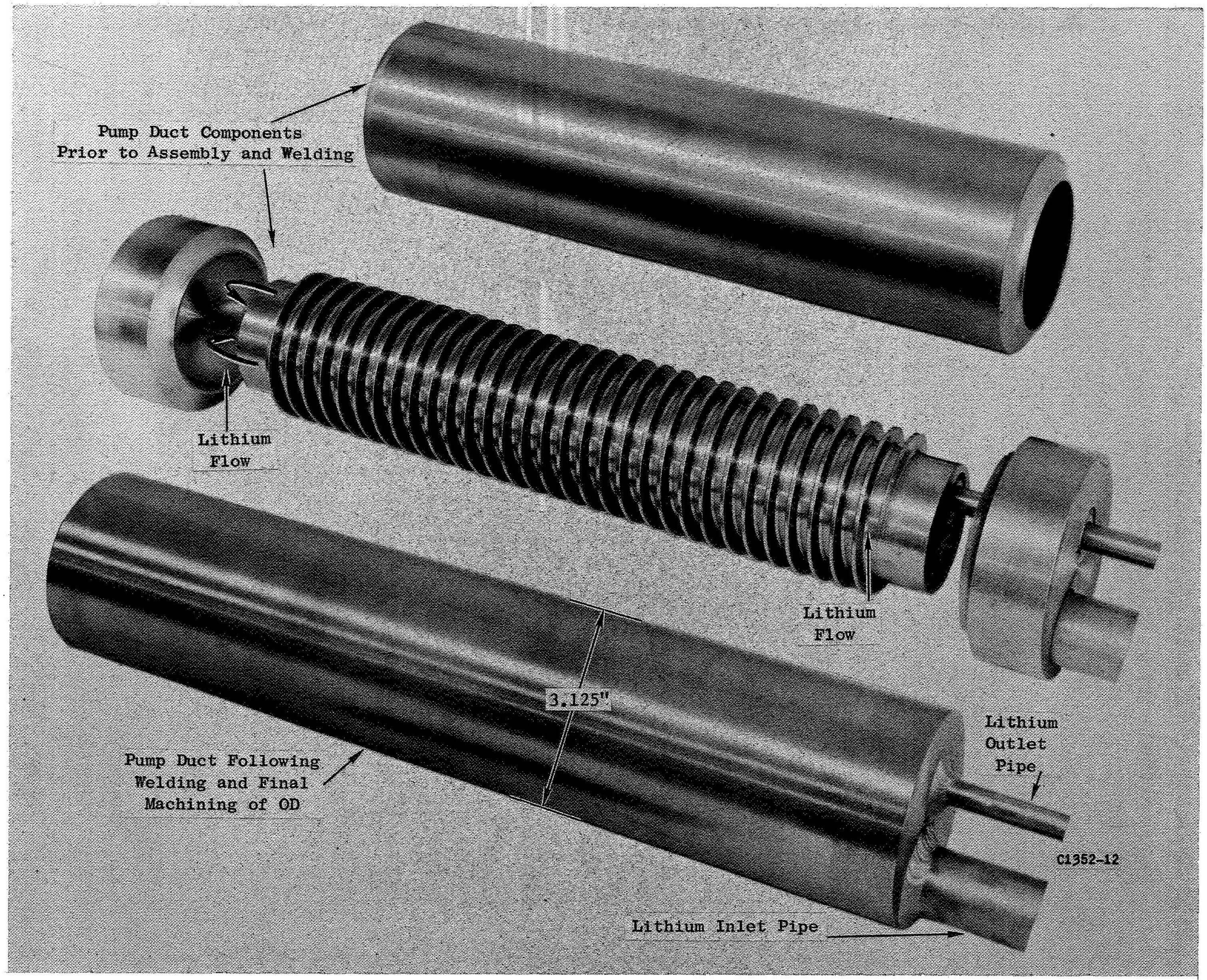

Figure 15. Components of the T-111 EM Pump Duct Before and Following Final Welding and Machining. (Orig. C68010311, C68011906)

IV. FUTURE PLANS

A. Instrumentation of the T-111 Corrosion Test Loop will be completed.

B. Lithium and potassium will be transferred from the purification systems to the two circuits of the T-111 Corrosion Test Loop.

C. Pre-test calibration of the T-111 Corrosion Test Loop instrumentation will be performed.

D. Fabrication of the 2600°F Lithium Loop will be completed.

PUBLISHED REPORTS

Quarterly Progress	For Quarter Ending
Report No. 1 (NASA-CR-54477)	July 15, 1965
Report No. 2 (NASA-CR-54845)	October 15, 1965
Report No. 3 (NASA-CR-54911)	January 15, 1966
Report No. 4 (NASA-CR-72029)	April 15, 1966
Report No. 5 (NASA-CR-72057)	July 15, 1966
Report No. 6 (NASA-CR-72177)	October 15, 1966
Report No. 7 (NASA-CR-72230)	January 15, 1967
Report No. 8 (NASA-CR-72335)	April 15, 1967
Report No. 9 (NASA-CR-72336)	July 15, 1967
Report No. 10 (NASA-CR-72352)	October 15, 1967

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