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# ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

## QUARTERLY PROGRESS REPORT NO. 23
For Quarter Ending January 15, 1971

Prepared by
J. P. Smith



prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

NASA Lewis Research Center
Contract NAS 3-6474
R. L. Davies and P. L. Stone, Project Managers
Materials and Structures Division

NUCLEAR SYSTEMS PROGRAMS
SPACE DIVISION
GENERAL ELECTRIC
CINCINNATI, OHIO 45215

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October 15, 1970, to January 15, 1971

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CONTRACT NAS 3-6474

NASA-Lewis Research Center
Cleveland, Ohio
R. L. Davies and P. L. Stone, Project Managers
Materials and Structures Division

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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 and P. L. Stone of NASA - Lewis Research Center are the NASA Technical Managers.

The Program Manager for the General Electric Company is E. E. Hoffman. Personnel making major contributions to the program during the current reporting period include:

T-111 Corrosion Loop - R. W. Harrison, J. Smith, A. Losekamp;
1900°F Lithium Loop - J. Smith, T. Irwin;
Lithium Thermal Convection Loop - G. Brandenburg, A. Losekamp, T. Irwin, P. Blanz.

ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

# I. INTRODUCTION

This report covers the period from October 15, 1970, to January 15, 1971 of a four-part program, as described below.

## A. T-111 Rankine System Corrosion Test Loop

The initial task of this program was to fabricate, operate for 10,000 hours, and evaluate a T-111 Rankine System Corrosion Test Loop. Materials for evaluation included the containment alloy, T-111 (Ta-8W-2Hf), and the turbine candidate materials, Mo-TZC and Cb-132M, which were located in the turbine simulator of the two-phase potassium circuit of the system. The loop design is similar to the Cb-1Zr Rankine System Corrosion Test Loop; a two-phase, forced convection, potassium corrosion test loop which was tested under Contract NAS 3-2547.[1] Lithium was heated by direct resistance in a primary loop. Heat rejection for condensation in the secondary potassium loop was accomplished by radiation in a high-vacuum environment to the water-cooled chamber. The compatibility of the selected materials was 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 $ft^2$.

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(1) Hoffman, E. E. and Holowach, J., Cb-1Zr Rankine System Corrosion Test Loop, Potassium Corrosion Test Loop Development Topical Report No. 7, NASA-CR-1509, June 1970.

This loop completed 10,000 hours of testing in March 1970; posttest evaluation is complete, and a final topical report is being written.

B. 1900°F Lithium Loop

Also included in the program is the fabrication, 7500-hour operation, and evaluation of a 1900°F, high-flow velocity (1 gpm), pumped lithium loop designed to evaluate the compatibility of T-111 clad uranium nitride fueled specimens, ASTAR 811-type alloys, T-111, Mo-TZM, and W-Re-Mo Alloy 256* at conditions simulating a space power reactor system. This loop initially completed 2500 hours of testing before undergoing a scheduled shutdown. The loop has been placed back on test with two new fuel pins to be tested for 5000 hours at the same conditions. One of the new fuel pins contains an intentional clad defect.

C. Advanced Tantalum Alloy Capsule Test

The program also included capsule testing to evaluate advanced tantalum alloys of the ASTAR 811 type (Ta-8W-1Re-1Hf) in both potassium and lithium. Refluxing potassium capsule tests at 2200°F and lithium thermal convection capsule tests at 2400°F have completed 5000 hours of testing, and a final report is being written.

D. Lithium Thermal Convection Loop

A fourth task in this program involves design, fabrication, and operation of a natural circulation lithium loop at 2500°-2700°F. The loop will be fabricated from T-111 alloy and will contain chemistry, metallography, and creep/tensile specimens of T-111 and advanced tantalum alloys of the ASTAR type.

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* W-25 a/o Re-30 a/o Mo (W-29 w/o Re-18 w/o Mo)

## II. SUMMARY

Posttest evaluation of the 10,000-hour T-111 Rankine System Corrosion Test Loop is complete. Microprobe examination of the high-oxygen area of the potassium swirler wire insert indicates the presence of a complex potassium compound. Tensile tests of specimens from the potassium vapor carryover line have confirmed the reduced ductility indicated by preliminary bend tests.

The 1900°F Lithium Loop continues to operate smoothly and trouble free. As of January 15, 1971, the loop has logged an additional 4025 hours since the replacement of fuel test specimens; the total accumulated test time is 6538 hours.

Except for final installation of the loop on the vacuum test chamber, assembly of the Lithium Thermal Convection Loop is complete. All refractory metal assembly, welding, and annealing are complete. Installation of thermocouples and Cb-1Zr dimpled foil thermal insulation is also complete.

## III. PROGRAM STATUS

### A. T-111 Rankine System Corrosion Test Loop

The planned posttest evaluation of the 10,000-hour test has been completed. Most of the results of the evaluation were reported previously;[2,3] however, the final test results are reported below.

#### 1. Microprobe Evaluation

Microprobe analysis has been performed on the two remaining specimens necessary to complete posttest evaluation of the loop. One area examined was the grain boundary corrosion in the swirler wire insert about six inches above the lithium exit from the plug section of the boiler.[4] The results show the grain boundary phase contains small amounts of potassium and carbon; no other elements were detected. Since the particles did not fluoresce in the electron beam, it can be concluded that they are not pure oxides; however, it does not preclude the possibility of oxygen being present in quantities below the detection limit of the microprobe, or possibly the presence of complex oxides. The precipitate particles were too small and the signals too weak in order to further identify their exact composition using the microprobe.

As reported earlier,[5] the boiler tube wall adjacent to the swirler wire section discussed in the preceding paragraph exhibited similar localized attack. The metallographic specimen of the tube wall from this region was reground, repolished, and rough polished dry to attempt to determine if any free potassium was present in the corroded area. Water was applied to the surface of the freshly polished surface while observing the specimen under the microscope. No evidence of gas evolution was detected which

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(2) Advanced Refractory Alloy Corrosion Loop Program Quarterly Progress Report No. 21, Period Ending July 15, 1970, NASA-CR-72782 (GESP-546), August 12, 1970.

(3) Advanced Refractory Alloy Corrosion Loop Program Quarterly Progress Report No. 22, Period Ending October 15, 1970, NASA-CR-72818 (GESP-562).

(4) Ibid, Figure 11.

(5) Ibid, Figure 6.

indicates that there is no free potassium metal in the corroded area. Additional polishing of the boiler tube specimen and examination of it in the unetched condition revealed a nonmetallic, semicontinuous phase in the grain boundaries of the corroded region. Based on these microprobe and metallographic evaluations, this phase is probably a complex potassium compound. Solubility experiments of oxygen-doped tantalum in potassium have revealed the presence of potassium tantalate ($K_3TaO_4$).[6]

Microprobe examination was also performed on the turbine simulator second-stage blade (Mo-TZC). A uniform layer approximately 0.0003 inch in thickness was noted on the leading edge of the blade specimen during metallographic examination.[7] The microprobe results showed that the coating actually consists of two separate layers. The outer layer is essentially pure hafnium. The second layer, located between the outer layer and the Mo-TZC substrate contains hafnium, but in much lower quantities than the outer layer. Also, the inner layer contains Ti and Zr in about the same quantities as the Mo-TZC substrate. These results indicate that hafnium is deposited from the potassium vapor onto the surface of the blade and then diffuses into the blade. Similar transfer of hafnium from the hot to the cold portions of the lithium circuit were noted previously[2,3] in connection with the hafnium nitride coating on the 3/8-inch tube of the plug section of the boiler.

2. Tensile Tests on Specimens from T-111 Tubing

Tensile tests were performed on specimens cut from the 1-inch-OD x 0.1-inch-wall T-111 pipe from the top of the plug section and from the potassium vapor crossover line. These two areas were selected because they represent the longest straight sections from the loop which were exposed to the highest ($\sim$ 2220°F) and lowest ($\sim$ 1880°F) operating temperatures. Also, preliminary screening tests by flattening ring sectors at room temperature from several locations in the loop[8] showed the crossover line to be extremely brittle; whereas, the boiler material exhibited

(6) Stecura, Stephan, "Apparent Solubilities of Commerically Pure and Oxygen-Doped Tantalum and Niobium in Liquid Potassium," NASA TN D-5875, July 1970.

(7) Advanced Refractory Alloy Corrosion Loop Program Quarterly Progress Report No. 22, Period Ending October 15, 1970, NASA Contract NAS 3-6474, NASA-CR-72818 (GESP-562), Figure 25.

(8) Ibid, Figure 21.

only slightly reduced ductility compared to the pretest pipe. As shown in Figure 1, the specimens except for the curved cross section were similar to conventional flat tensile specimens; however, they were longitudinal sections from the 1-inch-OD pipe. Although the results from this type of specimen may not be useful for engineering design purposes, they are valid for comparison of relative properties or property changes which was the intention of this study. The results of the 80°F and 2000°F tensile tests are summarized in Table I. At both 80°F and 2000°F, the strength of all the specimens is essentially the same except for the unexposed pretest material which may be slightly stronger. The only significant deviation in properties is the low ductility at 80°F of the vapor crossover line compared to either the boiler or unexposed pretest material. The rather sharp contrast in the 80°F ductility of the crossover line material compared to the other specimens is clearly seen in Figure 2. This reduced room temperature ductility confirms the validity of preliminary results on the ring sector tests from this loop and the High-Temperature Alkali Metal Valve Loop.[9]

The results of these evaluations on the 10,000-hour T-111 Loop and the HTVL indicate a possible aging reaction for T-111 at temperatures near 1900°F. Whether the embrittlement, or perhaps notch sensitivity, is purely thermally induced or associated with alkali metal exposure has not been resolved. The necessary tests to fully evaluate this problem are beyond the scope of the existing contract.

B. <u>1900°F Lithium Loop</u>

1. <u>Test Operation</u>

The loop continues to operate very stably and as of Janauary 15, 1971, has logged an additional 4025 hours since the replacement of the fuel test specimens for a total accumulated test time of 6538 hours. No shutdowns or interruptions were encountered during this report period.

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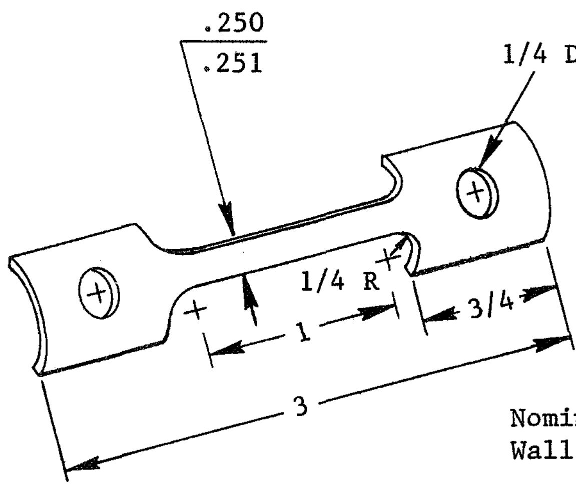
(9) Harrison, R. W. and Holowach, J., Refractory Metal Valves for 1900°F Service in Alkali Metal Systems, NASA Contract NAS 3-8514, GESP-508, p. 161, April 15, 1970.




Nominal Arc Length - 3/4
Wall Thickness - 0.10

Dimensions in Inches

Figure 1. Specimen Design for Posttest Tensile Evaluation of 1-Inch OD Tubing.

TABLE I

TENSILE TEST RESULTS ON SPECIMENS OF T-111 PIPE(1)
EXPOSED FOR 10,000 HOURS IN THE T-111 RANKINE SYSTEM CORROSION TEST LOOP

| | 80°F Tests | | | | |
|---|---|---|---|---|---|
| Specimen | Exposure Temperature °F | U.T.S. (ksi) | .02% Y.S. (ksi) | .2% Y.S. (ksi) | Elong. (%) |
| Unexposed | - | 92.6 | 69.4 | 76.5 | 34.0 |
| Boiler | 2200 | 90.4 | 61.9 | 70.5 | 32.0 |
| Boiler | 2200 | 89.6 | 61.3 | 71.6 | 33.0 |
| K Vapor Line | 1880 | 86.5 | 63.1 | 70.1 | 10.0 |
| K Vapor Line | 1880 | 88.6 | 61.1 | 68.9 | 13.0 |

| | 2000°F Tests(2) | | | | |
|---|---|---|---|---|---|
| Specimen | Exposure Temperature °F | U.T.S. (ksi) | .02% Y.S. (ksi) | .2% Y.S. (ksi) | Elong. (%) |
| Unexposed | - | 57.0 | 24.8 | 29.9 | 25.0 |
| Boiler | 2200 | 51.6 | 23.1 | 27.4 | 28.0 |
| Boiler | 2200 | 51.9 | 25.1 | 28.3 | 28.0 |
| K Vapor Line | 1880 | 52.3 | 28.9 | 30.9 | 26.0 |
| K Vapor Line | 1880 | 52.8 | 25.1 | 28.4 | 28.0 |

(1) 1 inch OD x 0.1 inch wall.

(2) Tests conducted in a vacuum of 1.0 - 2.8 x $10^{-6}$ torr.

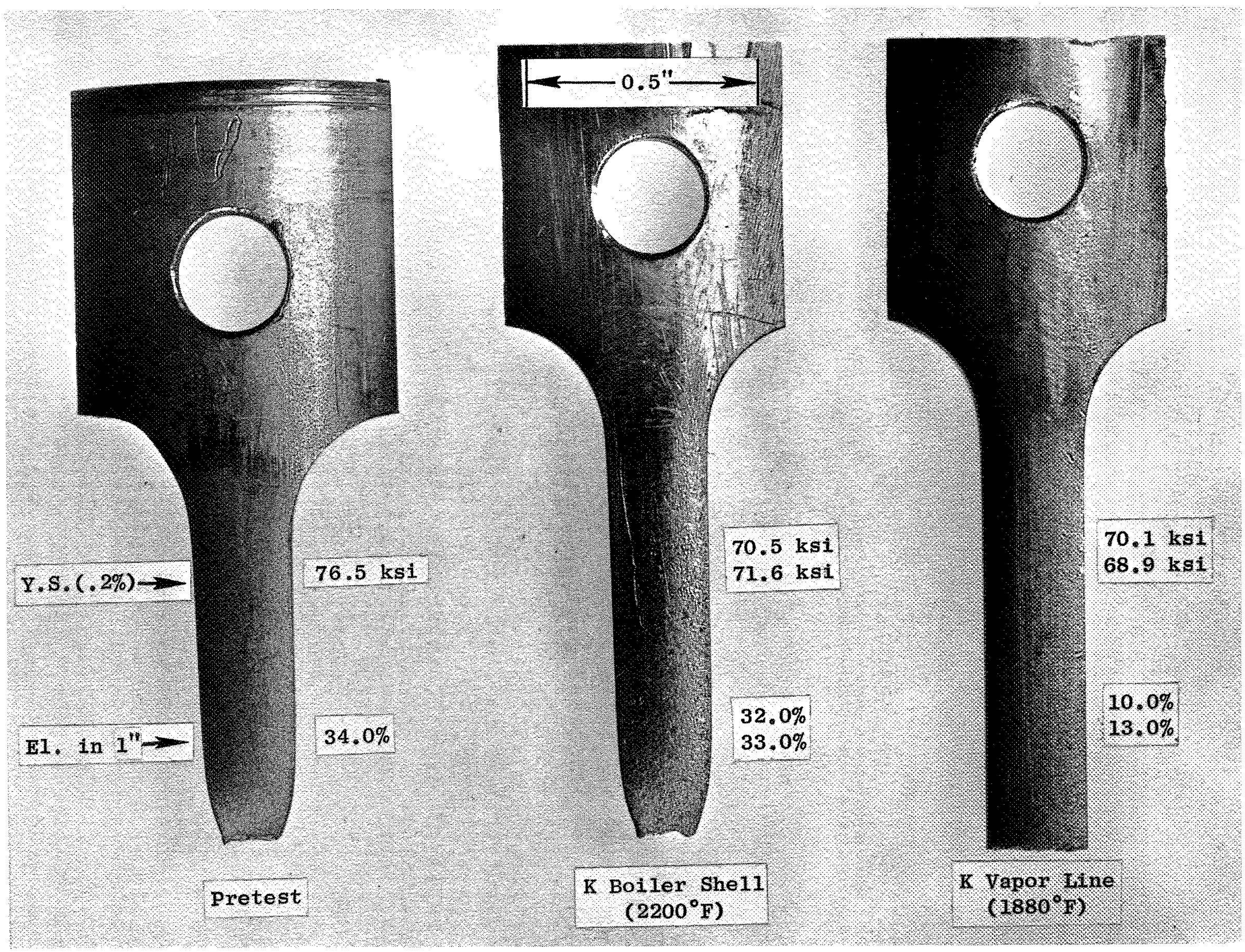



Figure 2. Fractured Tensile Test Specimens Cut From 1-Inch T-111 Pipe of the 10,000-Hour T-111 Corrosion Test Loop. Tensile Tests Performed at 80°F. (P70-10-16C)

Typical operating temperatures are shown in Figure 3. The test chamber environment continues to improve; the ion gage has indicated a chamber pressure in the $10^{-10}$ torr range since mid-November. Daily mass scans are still obtained; gaseous species with mass numbers 2, 18, 28, 40, and 44 are the only ones that remain in any measurable quantity.

## 2. Results of Evaluation of T-111 Clad UN Fuel Element Specimens from 2500-Hour Test*

The fuel test specimens consisted of 95 percent dense uranium mononitride (UN) cylinders clad with T-111 (0.750-inch-OD x 0.040-inch-wall). The T-111 was lined with 0.005-inch-thick tungsten foil to prevent contact and possible reactions of the UN with the T-111 clad. Visual examination of the two fuel element specimens (LT-1 and LT-3), removed from the loop after 2500 hours, revealed no discoloration and no evidence of corrosion. No dimensional changes were observed. Only minor weight changes were noted as was reported previously.[10] Except for a slight reduction in the oxygen content of the T-111, very little change in chemistry was observed as a result of the lithium exposure. Metallographic examination of the T-111 showed no evidence of lithium corrosion and no contamination of the T-111 by the UN. Although the T-111 appeared to be unaffected by the lithium exposure, the cladding from both LT-1 and LT-3 failed in a brittle, intergranular manner when subjected to a ring flattening test. Similar rings from unexposed samples were very ductile and could be flattened completely without any cracking. The cause for the embrittlement had not yet been determined, but it has been found that the ductility of the brittle T-111 can be restored by annealing at 2400°F for one hour in vacuum. Additional tests are planned in an effort to determine the reason for the observed T-111 embrittlement.

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* Except for dimensional and weight measurements, the evaluation of the fuel test specimens is being performed at the NASA-Lewis Research Center by G. K. Watson. The above discussion is a summary of the results to be issued in a NASA-Lewis Research Center report.

(10) Advanced Refractory Alloy Corrosion Loop Program, Quarterly Progress Report No. 21, Period Ending July 15, 1970, NASA CR-72782 (GESP-546), Aug. 12, 1970, p. 43.

Date January 15, 1971 Test Time 6538 Hrs.
Chamber Pressure 6.5 x $10^{-10}$ Torr
Heater 37.9 A 292 V
Flow 2.22 mv 1 GPM

TEMPERATURES IN °F

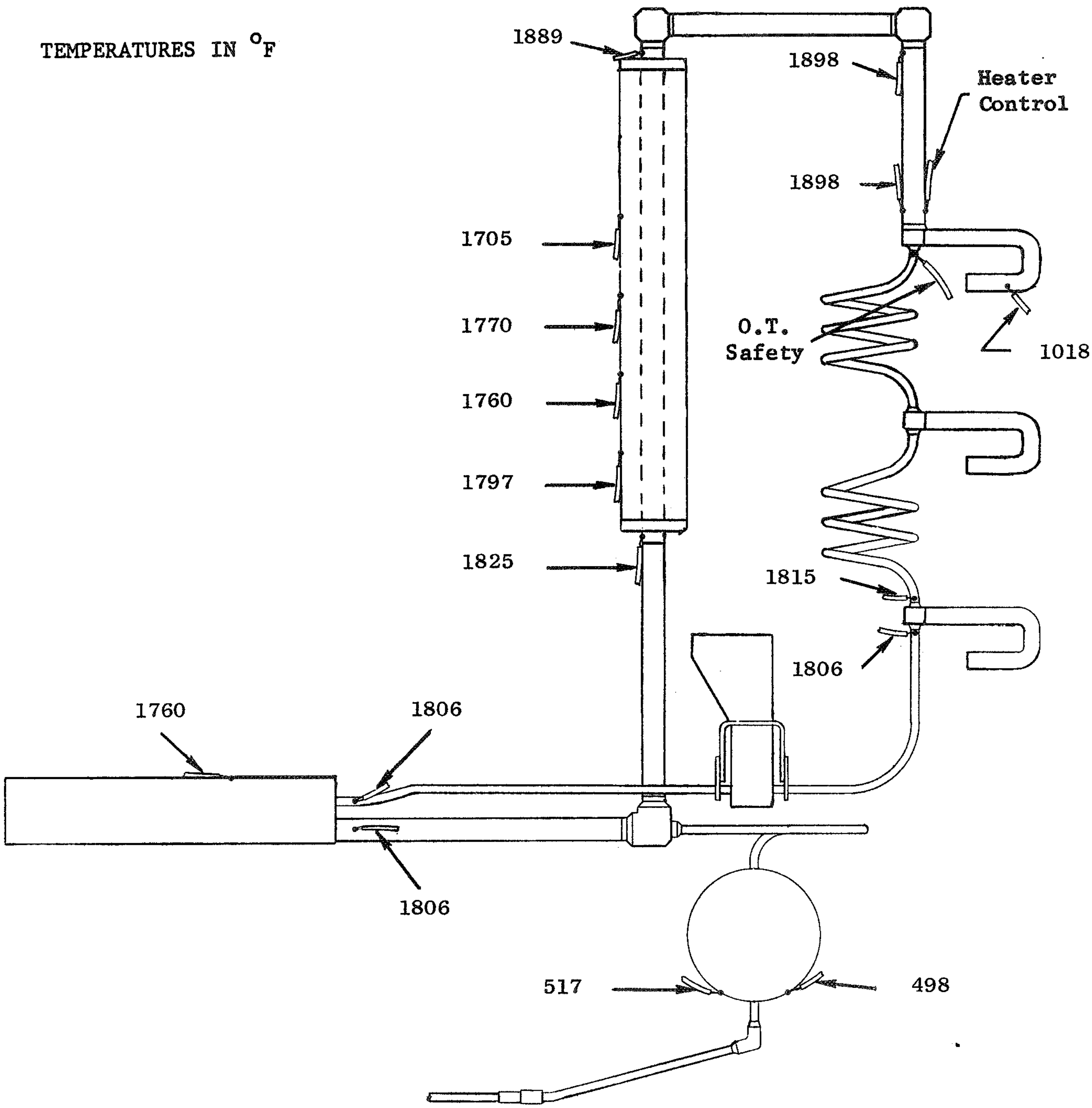


Figure 3. 1900°F Lithium Loop Operating Temperatures - 6538 hours.

## C. Lithium Thermal Convection Loop

Fabrication of all subcomponents required for the Lithium Thermal Convection Loop has been completed. Final assembly of all subcomponents, instrumentation, and thermal insulation of the loop was also completed. The relative position and orientation of these subassemblies in the loop are illustrated in the isometric drawing, Figure 4. All welds were made according to GE-NSP Specification P8AYA13, "Welding of Columbium, Tantalum, and Their Alloys by the Inert-Gas Tungsten Arc Process." All refractory metal alloy welds were radiographed according to GE-NSP Specification P3AYA14, "Radiographic Inspection," and helium mass spectrometer leak checked to GE-NSP Specification P3CYA16, "Leak Testing Using a Helium Mass Spectrometer Leak Detector." No weld defects were detected. All welds were given a postweld anneal in vacuum at 2400°F for one hour according to GE-NSP Specification P10DYA10, "Postweld Vacuum Annealing of Cb-1Zr and T-111 (Ta-8W-2Hf) Alloys."

The distribution of the T-111, ASTAR 811C, and ASTAR 1211C specimens in the loop is shown schematically in Figure 5. Original plans were to include only T-111 and ASTAR 811C; however, it was decided to also include the more advanced alloy, ASTAR 1211C. The ASTAR 1211C sheet was received at GE-NSP in the as-rolled condition and subsequently annealed for one hour at 3000°F in vacuum at a pressure of $< 1 \times 10^{-5}$ torr. Chemistry and metallographic specimens of the as-rolled and annealed material are presently being evaluated. Prior to assembly of the subcomponents, all specimens were cleaned and weighed to obtain data suitable for weight change measurements at the completion of the test. This data along with comparison of pretest and posttest chemical analyses, metallographic evaluation, and tensile strength will allow for a complete mapping of any mass transfer or other metallurgical changes and their effect on mechanical properties.

### 1. Split Tantalum Resistance Heating Element

Heating of the hot-leg vertical test section of the loop will be accomplished with a split tantalum resistance heating element. The details

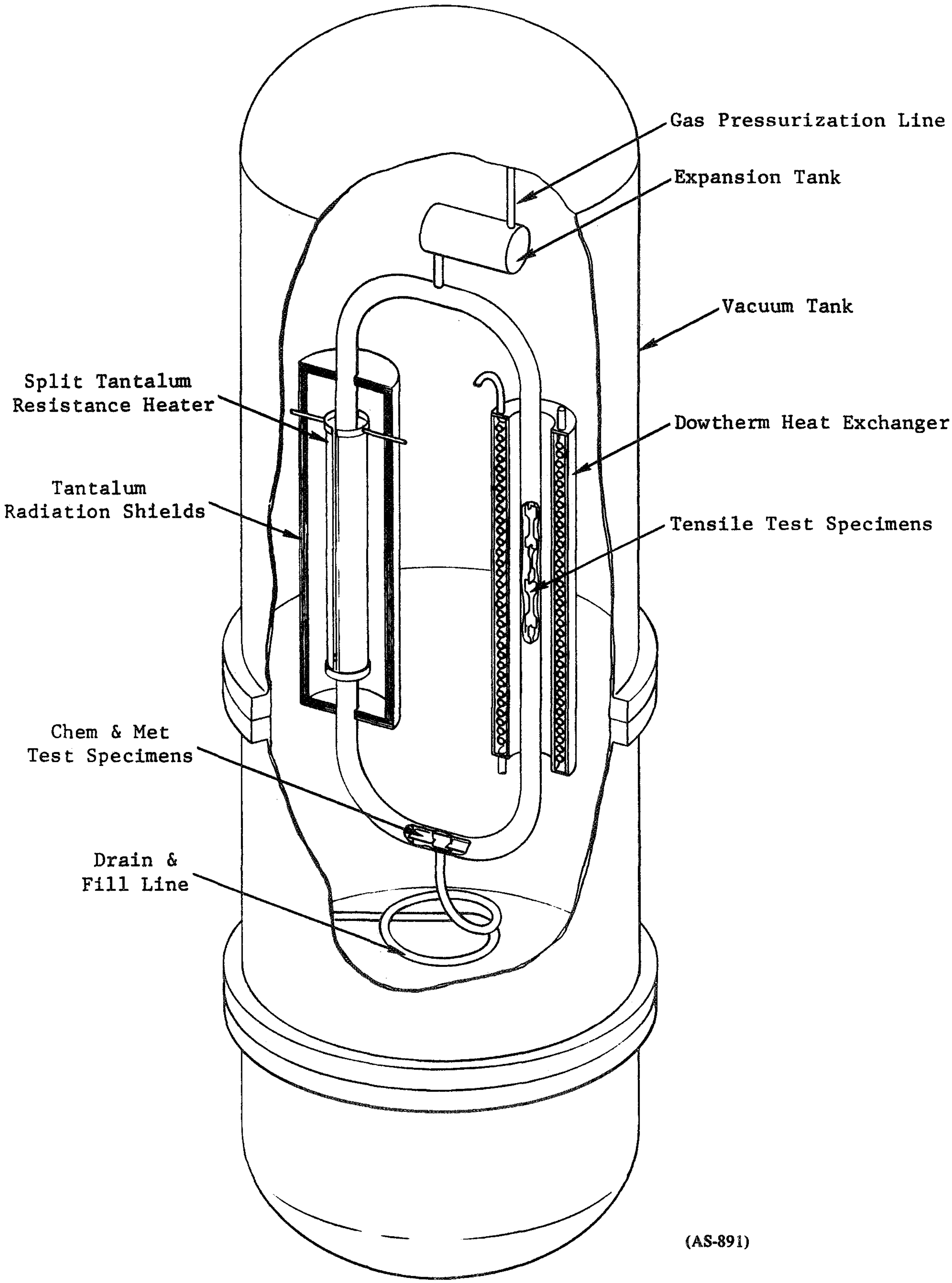


Figure 4. Isometric Drawing of Lithium Thermal Convection Test Loop.

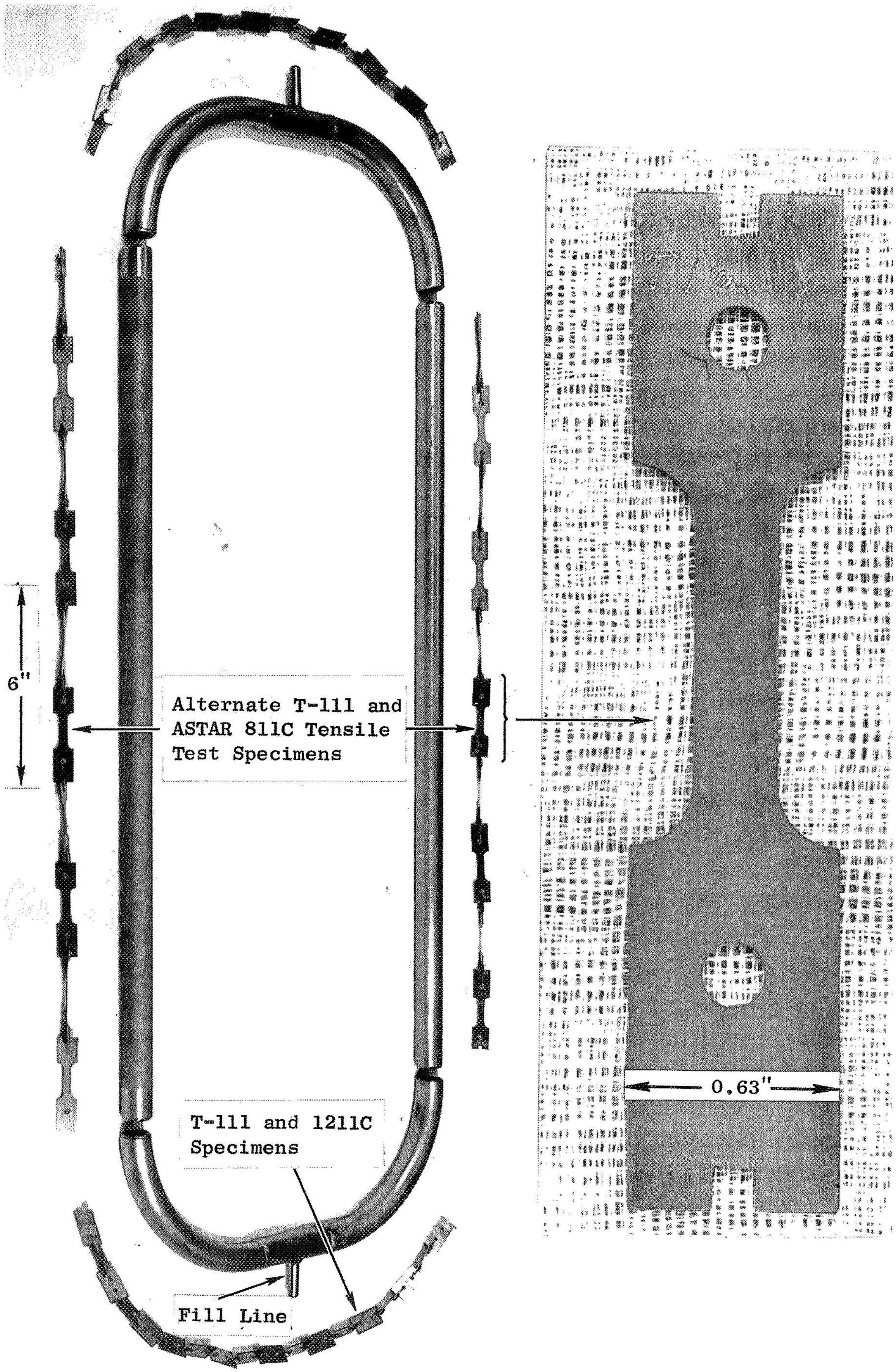


Figure 5. T-111 - Lithium Thermal Convection Loop Prior to Insertion of Tantalum Alloy Test Specimens. (P70-10-7H and P70-10-7C)

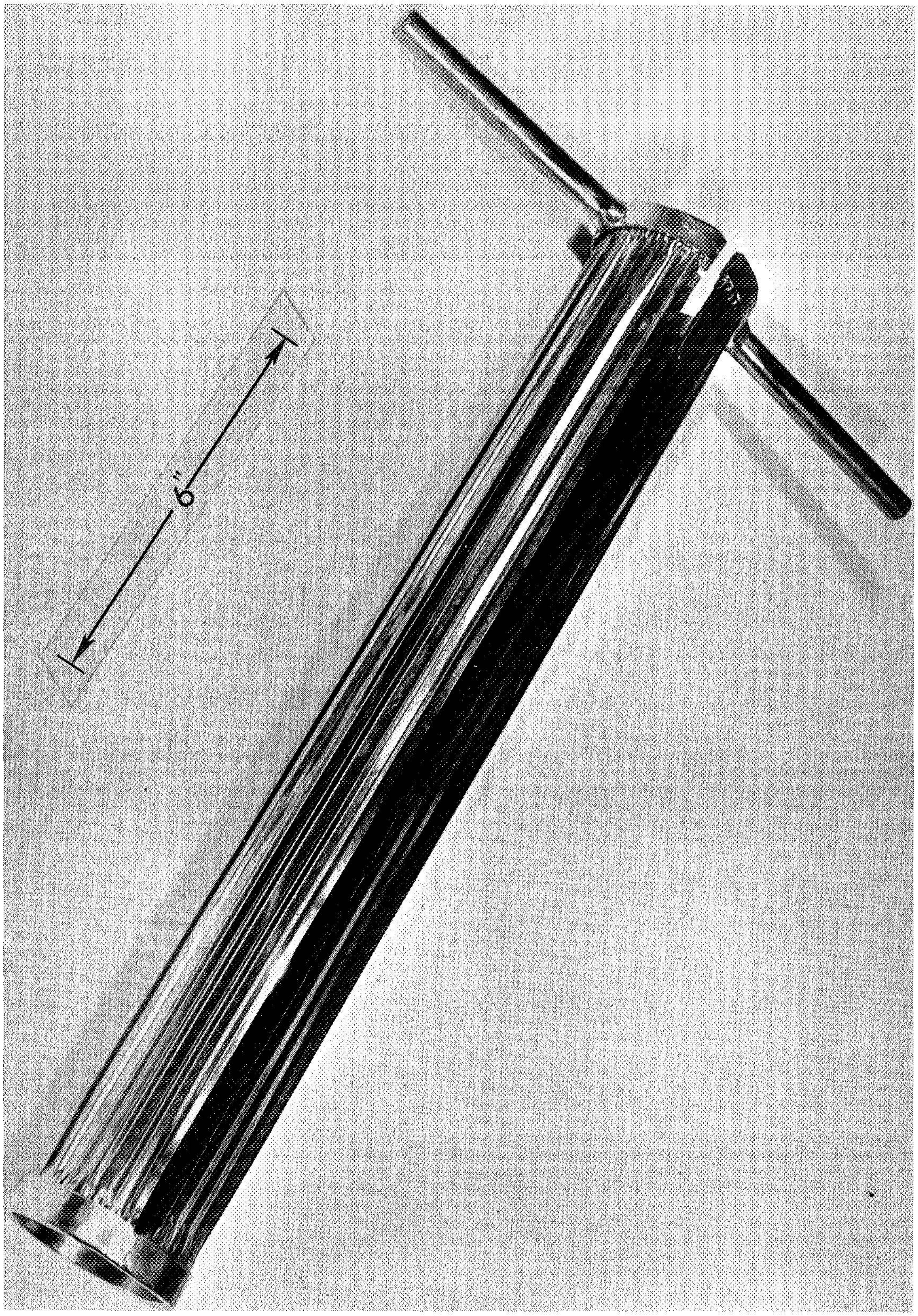


Figure 6. Split Tantalum Heater for Lithium Thermal Convection Loop. (P70-11-7A)

of the heater design have been previously described.[3] The fabricated heater showing the corrugated tantalum foil heater body is shown in Figure 6.

2. Dowtherm-Filled Water-Cooled Heat Sink

Heat shall be rejected in the cold-leg vertical test section by radiation to a Dowtherm-filled water-cooled heat sink surrounding the T-111 piping. The details of the heat sink have been previously described.[3]

The fabricated heat sink is shown in Figure 7. Since it is filled with Dowtherm at room temperature, an expansion tank connected to the heat sink with a 1/4-inch stainless steel tube will be installed on the outside of the test chamber to accommodate 25-percent volume expansion during 450°F chamber bakeout and loop filling operations.

3. Final Assembly

Following fabrication of these various subassemblies, final assembly was accomplished as described below.

a) The lithium expansion tank was welded to the top curved section of the loop;

b) The lithium fill line was welded to the lower curved section of the loop;

c) Specimens were inserted into the four loop subcomponents;

d) The two vertical, straight test sections were welded to the lower curved section;

e) The loop was positioned and attached to the stainless steel support structure on the spool piece;

f) The split tantalum heater, heat shields, and Dowtherm-filled heat sink were installed;

g) The top curved test section was welded to the vertical test sections utilizing the motor-driven rotary fixture shown in Figure 8.

Following the completion of each welding operation, the welds were annealed, radiographed, and leak tested according to the specifications discussed at the beginning of Section C of this report. After verifying the integrity of all welds, the loop and spool piece were transferred to

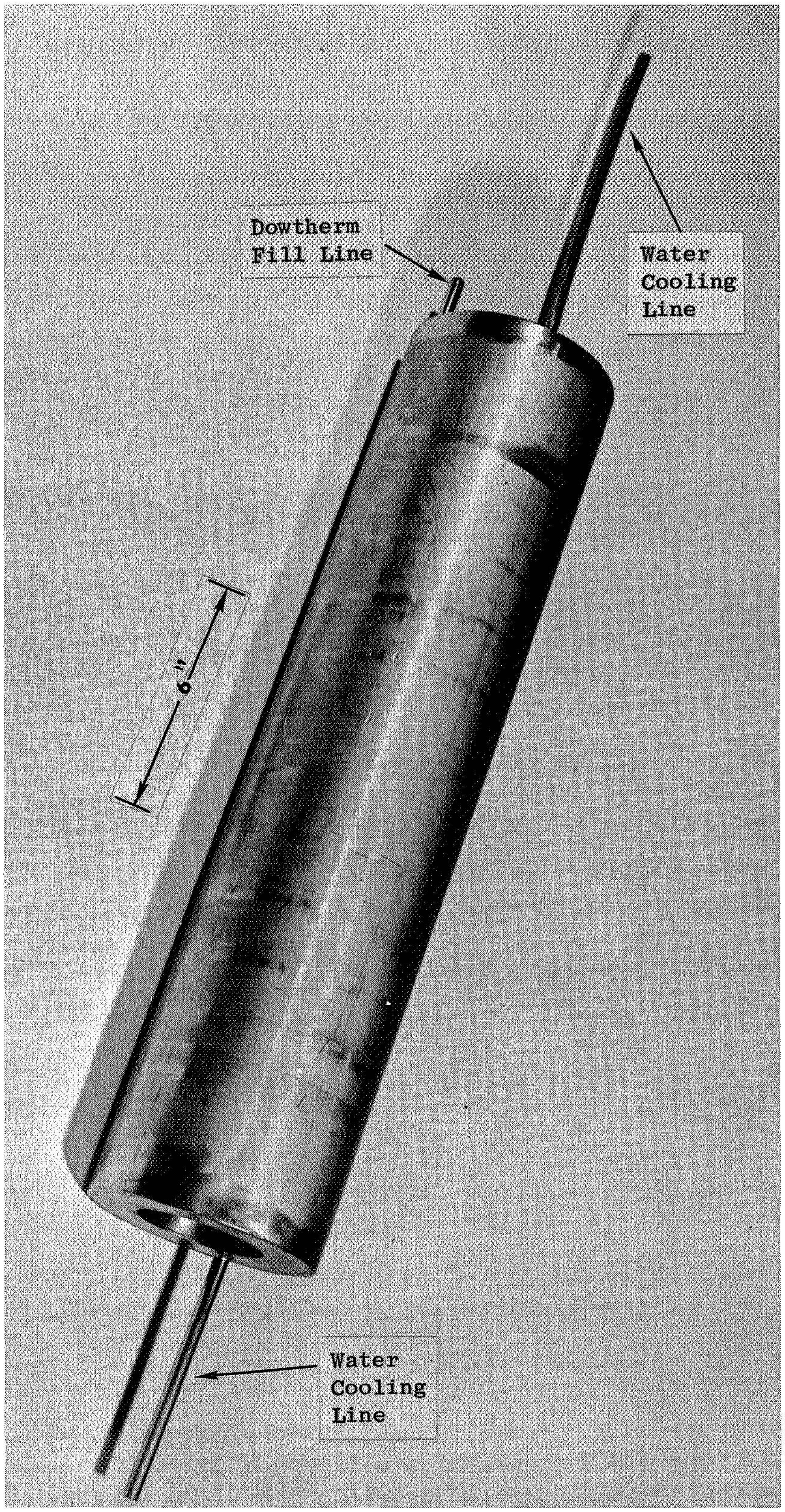


Figure 7. Dowtherm-filled, Water-cooled Heat Sink. (P70-11-7B)

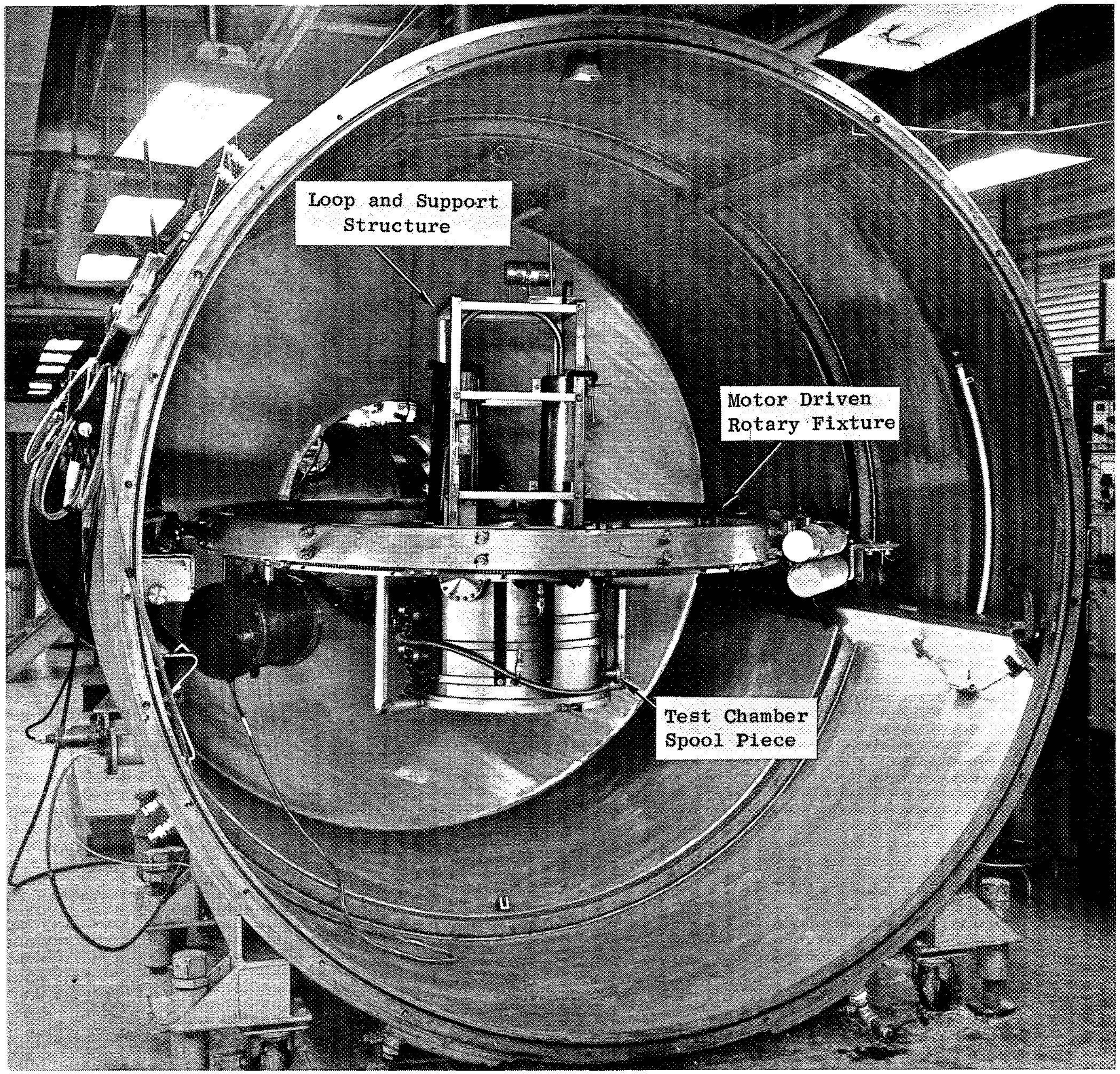


Figure 8. Lithium Thermal Convection Loop Mounted on Motor Driven Rotary Fixture in 8-Foot-Diameter Weld Chamber. (P70-12-10C)

the portable laminar flow clean room facility used previously during the fabrication of the 1900°F Loop.[11]

4. Thermal Insulation and Instrumentation

Thermal insulation consisting of multiple layers of Cb-1Zr foil was applied to the upper and lower curved sections of the loop as the thermocouples were installed. The foil used was 0.002 inch thick x 0.5 inch wide which had been dimpled by passing the foil between a hardened steel, coarse-knurled roller working against a hard plastic sheet. The effective thickness of the foil after dimpling was between 0.009 to 0.012 inch. The insulation was attached by spot welding the foil to the loop and to itself as succeeding layers were applied. A minimum number of spot welds were used to reduce conduction heat losses through the foil. A molybdenum spot welder electrode was used to avoid contamination of the foil surfaces with copper and an argon cover gas was used to protect all welded areas from oxidation.

Twenty-four split junction W-3Re/W-25Re thermocouples were installed on the loop. All thermocouples were insulated with high-density alumina. The high-purity (99.5 percent) alumina two-hole insulators were baked out at a pressure of $1 \times 10^{-5}$ torr for four hours at 1000°C (1832°F). To avoid contamination, several layers of tantalum foil were placed between the insulator and the loop to eliminate direct contact between the alumina and the T-111.

The assembled, insulated, and instrumented loop ready for installation on the test chamber is shown in Figure 9.

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[11] Advanced Refractory Alloy Corrosion Loop Program, Quarterly Progress Report No. 19, Period Ending January 15, 1970, Contract NAS 3-6474, NASA-CR-72662 (GESP-410), February 41, 1970, p. 13.

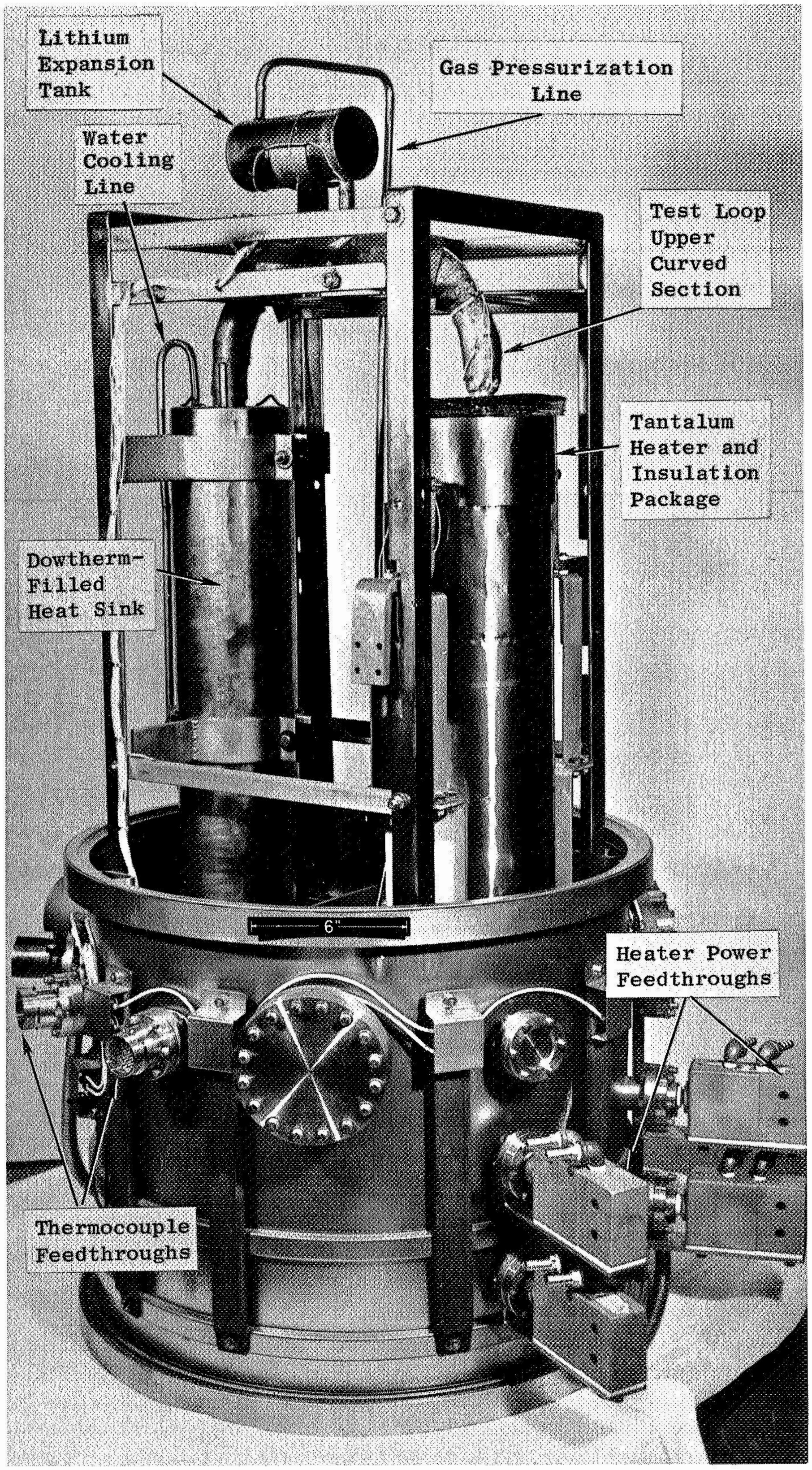


Figure 9. Lithium Thermal Convection Loop Ready for Installation on the Test Facility. (P71-1-15C)

## IV. FUTURE PLANS

Complete final topical report on the T-111 Rankine System Corrosion Test Loop.

Complete 5000-hour test on the defect fuel element specimen in the 1900°F Lithium Loop and begin preparations for posttest evaluation.

Initiate testing of Lithium Thermal Convection Loop.

## PREVIOUSLY PUBLISHED PROGRESS REPORTS FOR THIS CONTRACT

| 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 |
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| Report No. 20 (NASA-CR-72739) | April 15, 1970 |
| Report No. 21 (NASA-CR-72782) | July 15, 1970 |
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